Supporting Information

Coupling of α-Bromoamides and Unactivated Alkenes to Form γ-Lactams through EDA and Photoredox Catalysis

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Materials and Methods:

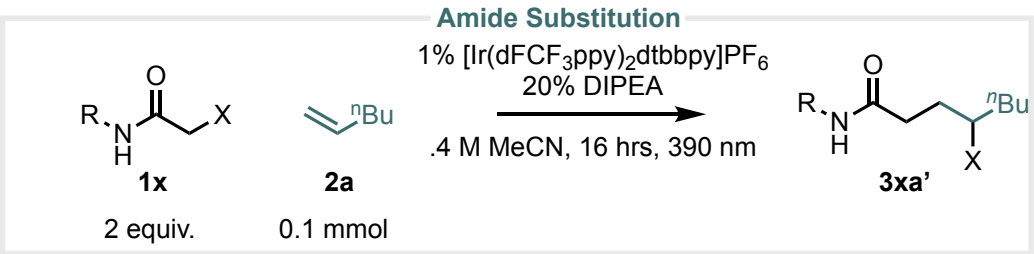
Unless otherwise noted, all reactions were performed in oven-dried glassware and carried out under an atmosphere of Argon with magnetic stirring. All photochemical reactions were run in 8 mL reaction tubes fitted with Teflon caps under irradiation from a PR-160 Kessil 390 nm 52W LED lamp or a PR-160 Kessil 440 nm 45W LED lamp with Teflon stir-bars under vigorous magnetic stirring in borosilicate glass vials between 3 and 6 cm from the irradiation source. All photochemical reactions were set-up in an Argon glovebox, though can also be performed with suitable Schlenk-line techniques. Chromatographic purification was accomplished by flash chromatography on SiliCycle® Silica Flash® 40-63 μm, 60 Å, silica or Teledyne ISCO CombiFlash®Rf+ LumenTM instrument. As most of the compounds listed do not exhibit a strong UV trace, ELSD was integral to the separation of product. Thin layer chromatography was performed on SiliCycle® 250 μm 60 Å plates. Visualization was accomplished with 254 nm UV light, potassium permanganate 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 electrospray ionization (ESI) probe.

Cyclic voltammetry was conducted on a BioLogic SP-200 potentiostat utilizing a glassy carbon working electrode, a platinum wire counter electrode, and a SCE reference electrode.

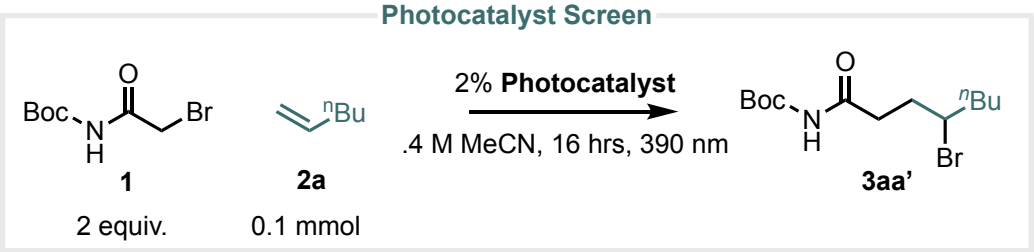
Unless otherwise mentioned, all starting materials were obtained from commercial sources including Millipore-Sigma, TCI, and Alfa-Aesar.

Table ESI1. Amide Substituents



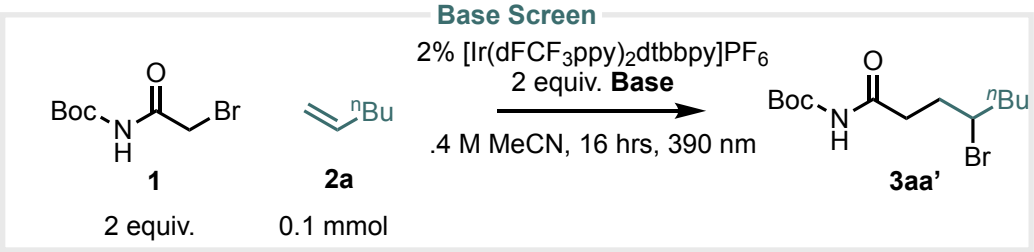
Entry	R =, X =	Yield (%, NMR)
1	H, Br	0
2	Ph, Br	0
3	Boc, Cl	0
4	$COCF_3$, Br	92
5	Boc, Br	94

Table ESI2. Photocatalyst Screen



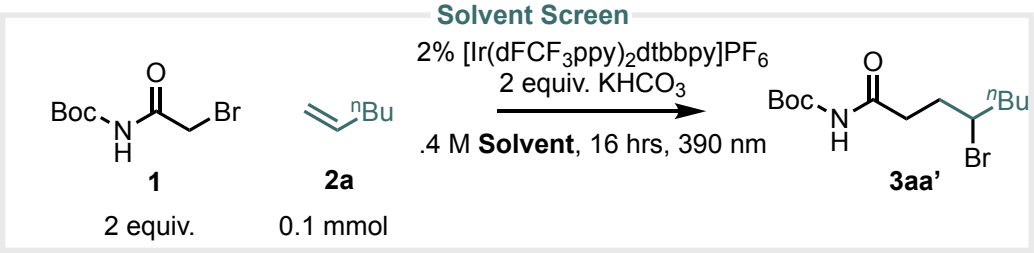
Entry	Photocatalyst	Yield 3a' (%, NMR)
1	**$[Ir(dFCF_3ppy)_2dtbbpy]PF_6$**	**32**
2	$[Ir(ppy)_2dtbbpy]PF_6$	16
3	$Ir(dFppy)_3$	23
4	$Ir(ppy)_3$	21

Table ESI3. Base Screen



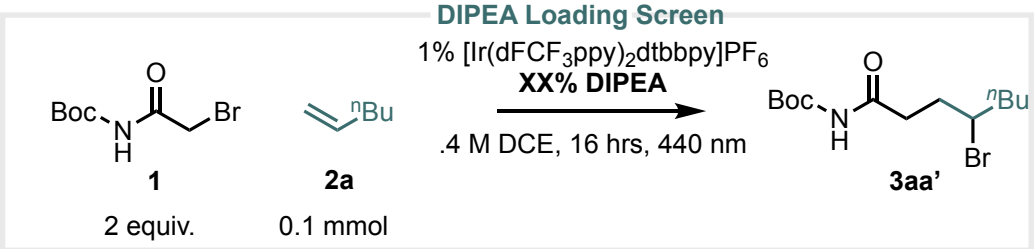
Entry	Base	Yield 3a' (%, NMR)
1	none	32
2	KOAc	14
3	KH_2PO_4	55
4	**$KHCO_3$**	**88**
5	K_2HPO_4	75
6	K_2CO_3	0
7	K_3PO_4	0

Table ESI4. Solvent Screen



Entry	Solvent	Yield 3a' (%, NMR)
1	MeCN	88
2	Acetone	45
3	EtOAc	82
4	**DCE**	**91**
5	$CHCl_3$	66

Table ESI5. DIPEA Loading Screen



Entry	DIPEA Loading (%)	Yield 3a' (%, NMR)
1	0	35
2	10	65
3	**20**	**78**
4	50	75
5	100	55

Table ESI6. Amine Base Screen

Yields determined by ^{1}H NMR of crude reaction mixtures.

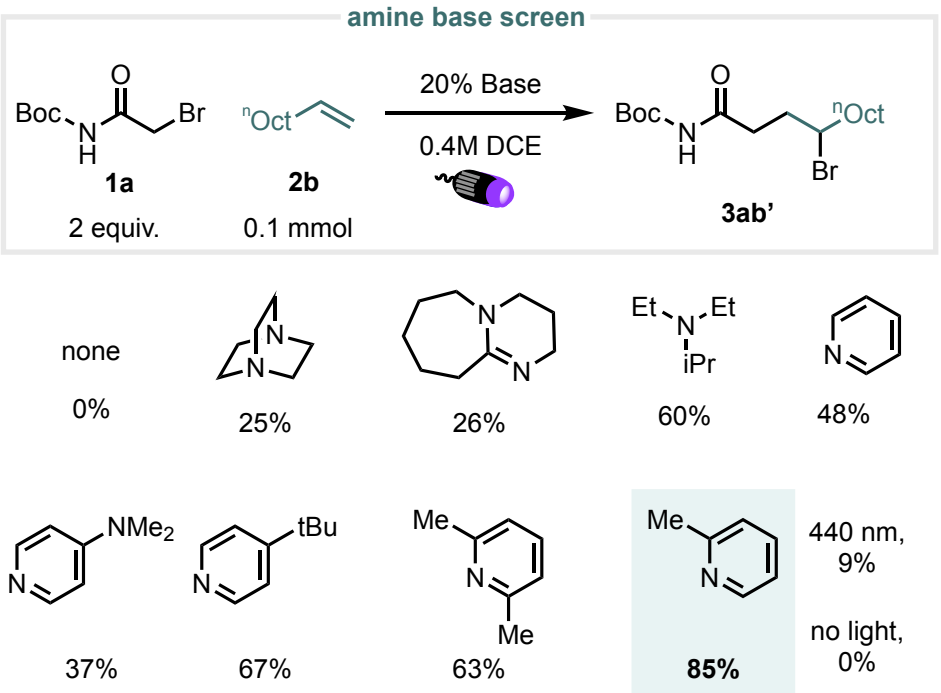
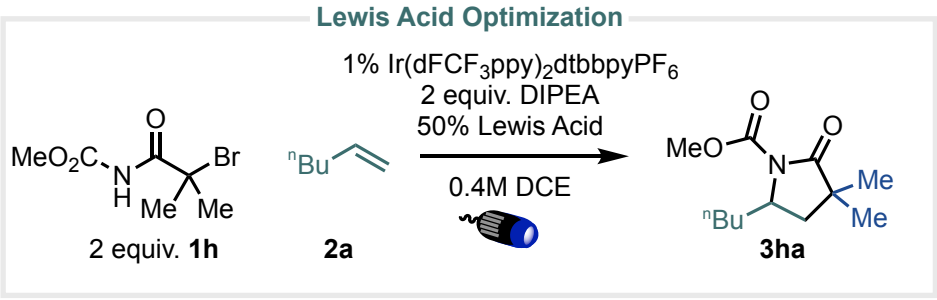
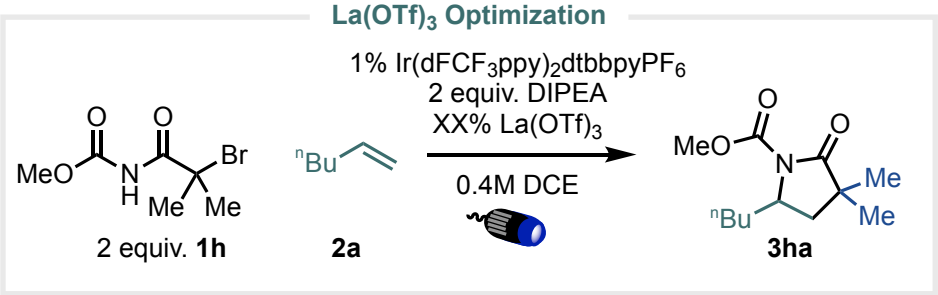


Table ESI7. Lewis Acid Screen



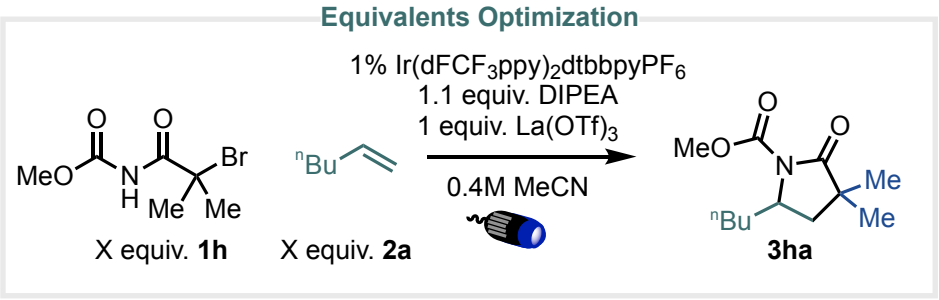
Entry	Lewis Acid	Yield 3ah (%)
1	$La(OTf)_3$	35
2	$Gd(OTf)_3$	30
3	$Sc(OTf)_3$	8
4	$Yb(OTf)_3$	17
5	$Eu(OTf)_3$	20
6	$Mg(OTf)_2$	16

Table ESI8. $La(OTf)_3$ Optimization



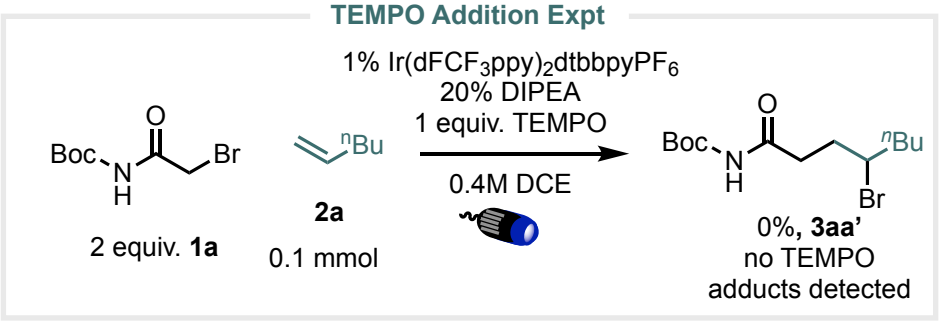
Entry	% $La(OTf)_3$	Yield 3ah (%)
1	0	0
2	10	8
3	25	15
4	50	28
5	75	31
6	100	38

Table ESI9. Equivalents Optimization



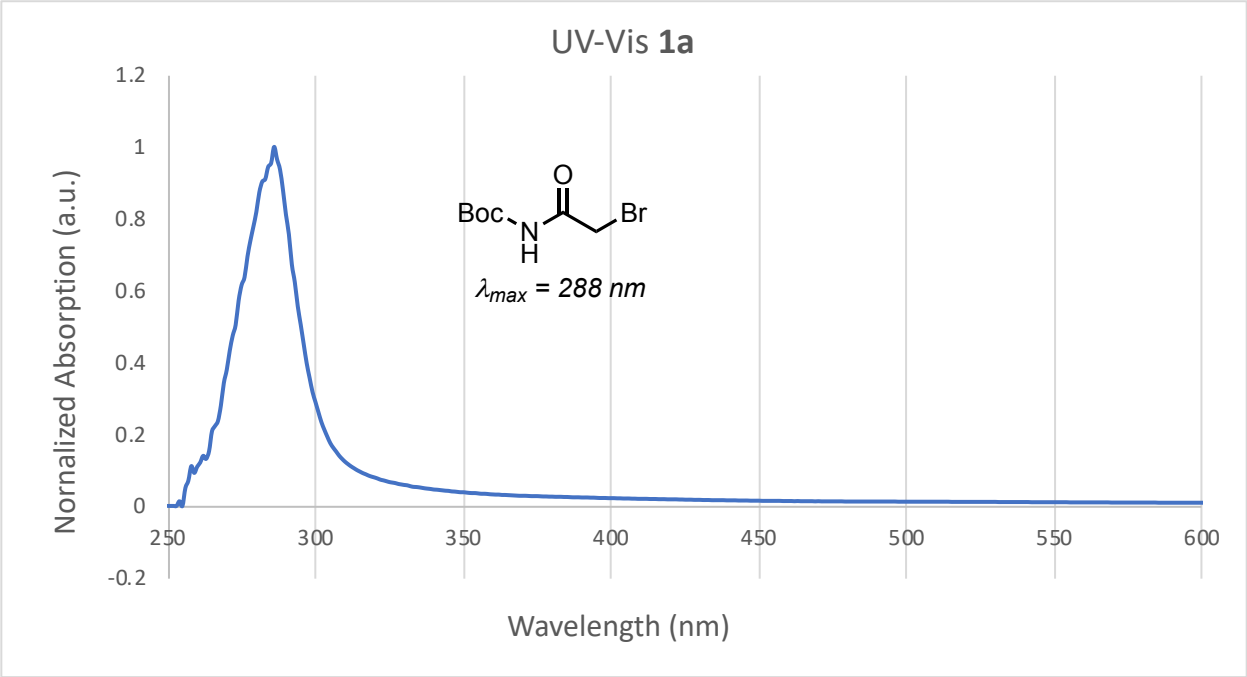
Entry	X equiv. 1h	X equiv. 2a	Yield 3ha (%)
1	2	1	26
2	1	2	40
3	1	3	66

Table ESI10. TEMPO Addition Experiment



UV-Vis and Cyclic Voltammetry Characterization

Figure S1. UV-Vis Absorption Spectrum of **1a**



General Procedure for cyclic voltammetry.

To an oven dried electrochemical cell was added 1 mmol tetrabutylammonium tetrafluoroborate followed by 10 mL dry MeCN. This solution was sparged with Ar gas for at least 10 minutes. A CV measurement of the solvent was taken to ensure the absence of oxygen or impurities. Then 1 mM substrate was added and the solution was sparged again with Ar gas to mix and reform the inert atmosphere.

Figure S2. Cyclic voltammetry of 1 mM **1h** in MeCN + 0.1 M *n*-NBu_4BF_4 at 0.2 V/s ($E_{1/2}$ = -1.47 V vs. SCE) and cyclic voltammetry of 1 mM **1h** + 1mM $La(OTf)_3$ in MeCN + 0.1 M *n*-NBu_4BF_4 at 0.2 V/s ($E_{1/2}$ = -1.32 V vs. SCE)

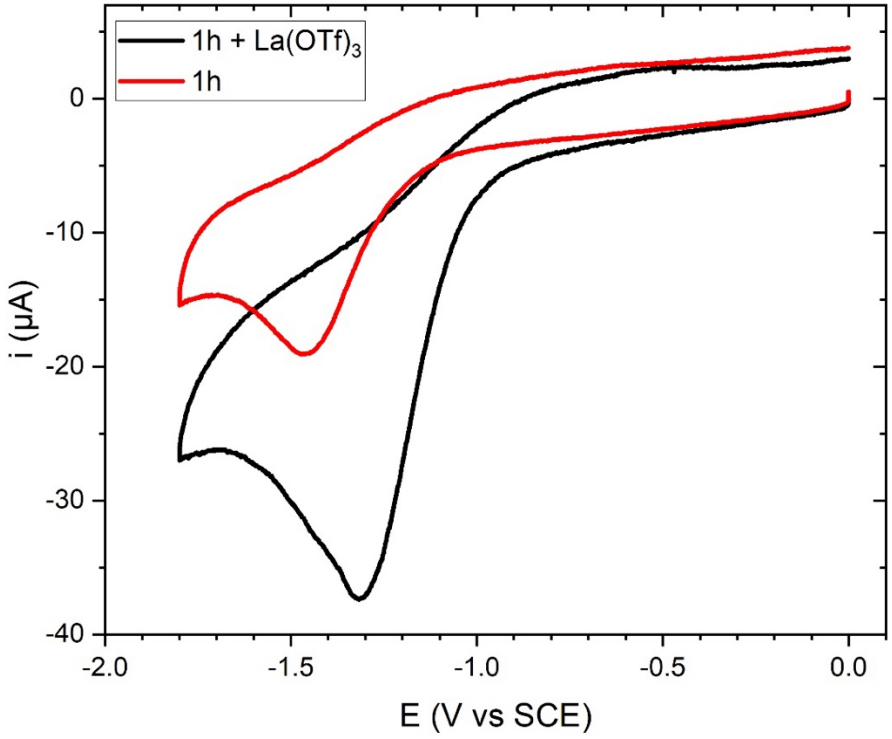


Figure S3. UV-Vis of 2-Me-pyridine-1a Mixtures in DCE

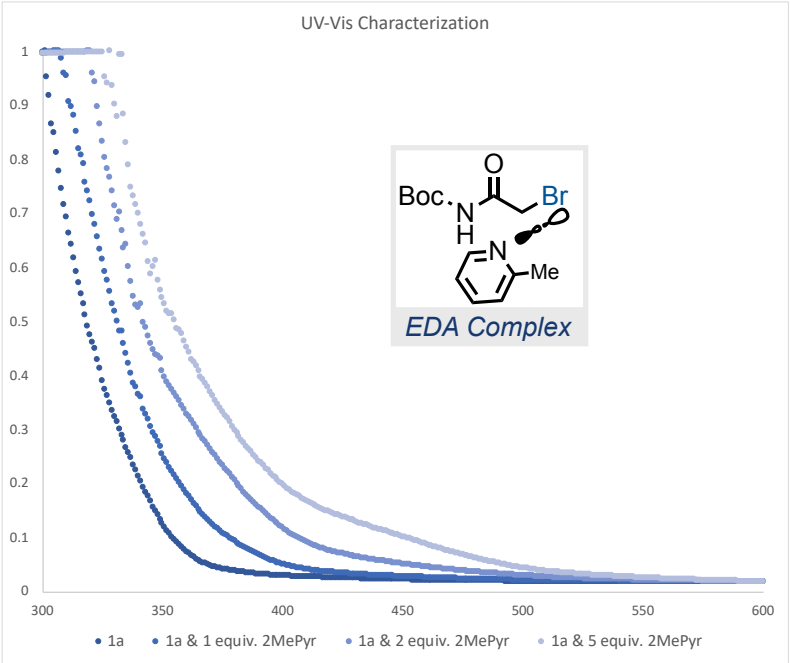
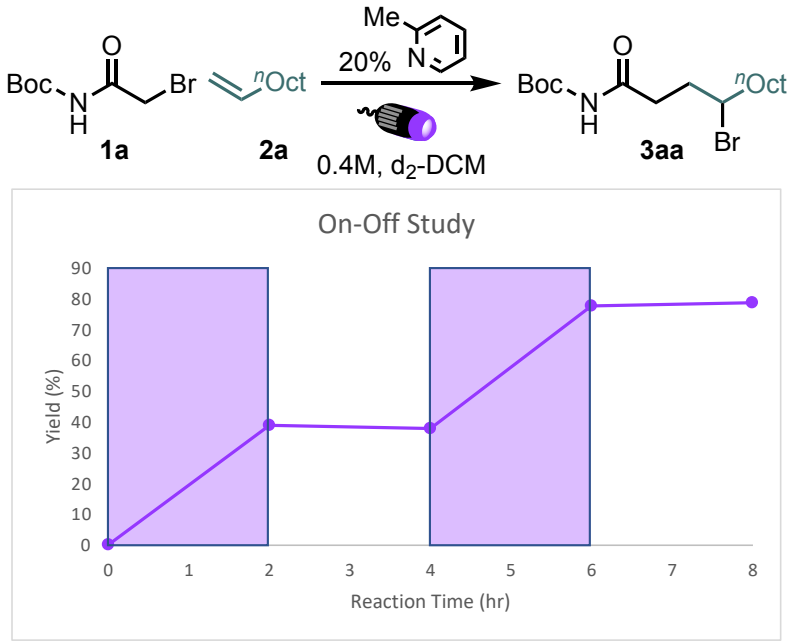


Figure S4. On-Off Study for EDA Reaction



Quantum yield calculation

Quantum yield was calculated according to reported literature.[1]

The measurement of the combined photon flux of two Kessil PR160s at maximum intensity (45W maximum, 440 nm), 11 cm away from each light source, was determined by standard ferrioxalate actinometry following already reported procedures.

Photon flux = 1.15 x 10^{-9} E/s

A cuvette was filled with standard reaction solution and placed 11 cm away from each light source. Under these conditions, the fraction of light absorbed by the photocatalyst was f = 0.9998. The sample was irradiated for 30 min (1800 s). Product was detected by ^{1}H NMR in 10% yield. The quantum yield was calculated as 14.

$$\Phi = \frac{\text{mol of } \mathbf{Product}}{\text{photon flux} \times \text{t} \times \text{f}} = \frac{0.1 \times 0.3 \times 10^{-3}\ \text{mol}}{\left(1.15 \times 10^{-9}\frac{\text{E}}{\text{s}}\right) \times 1800\ \text{s} \times 0.9998} \approx 14$$

Figure S5. Color Change of EDA Complex

Left vial: 0.1 M **1a** + 20% 2-Me-Pyridine

Right vial: 0.1 M **1a**

Figure S6. Photo of Photoreactor

Figure S7. Kessil Lamp Emission Spectra

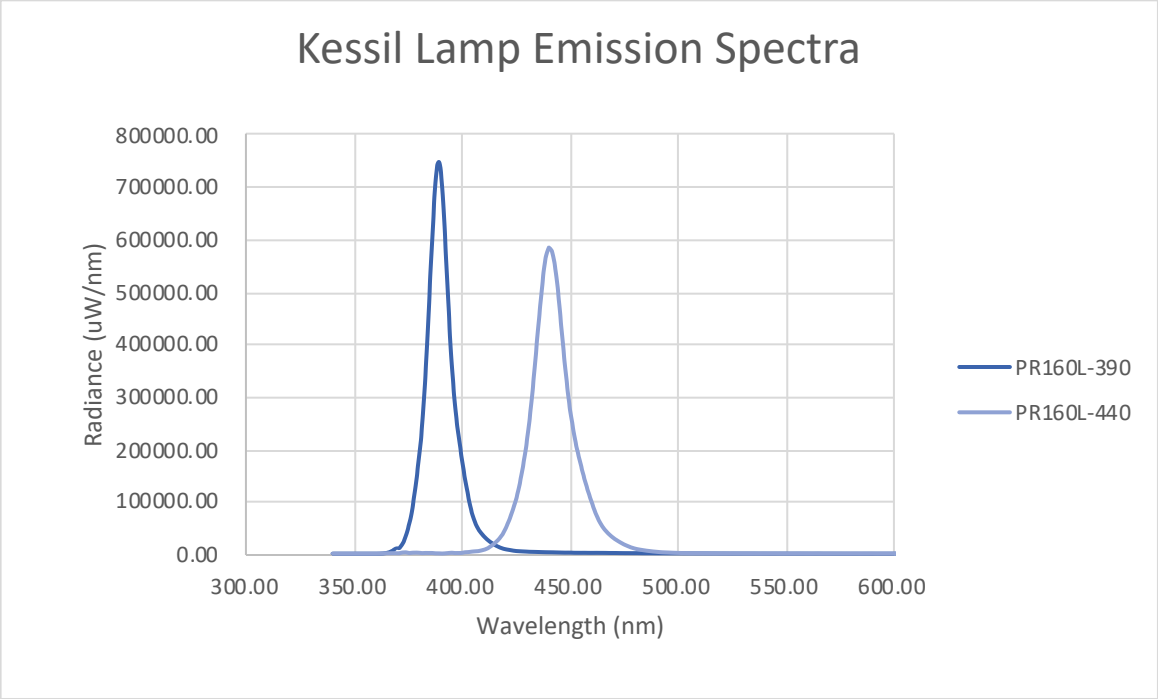


Figure S8. Enhancing the Electrophilicity of Intermediate Radicals in Substates **1t** and **1u**

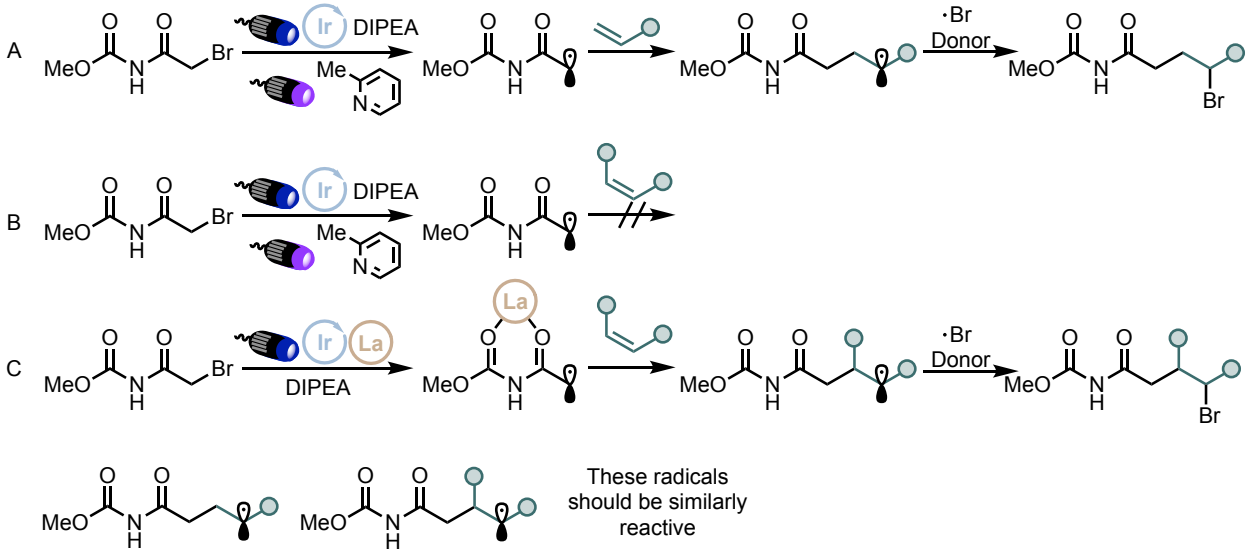
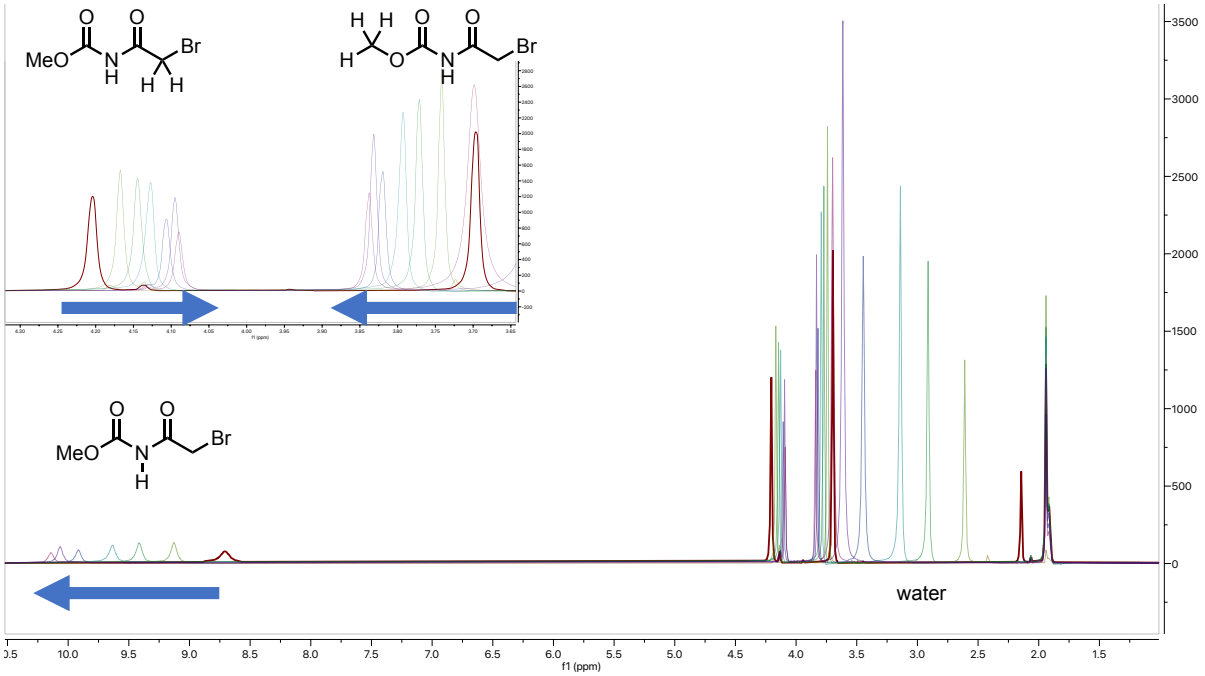


Figure S9. NMR Shifts of **1h** in Presence of $La(OTf)_3$



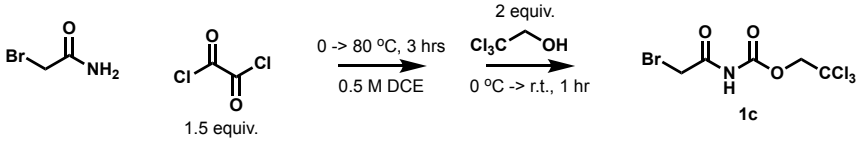
0.1 M 1h + X% $La(OTf)_3$ in d_3-MeCN, X = 0%, 10%, 20%, 40%, 60%, 80%, 100%

Water is introduced via the hygroscopic $La(OTf)_3$ addition

Starting Material Synthesis and characterization Data:

α-Br-imides **1a-b**[2] and **1e**[3] and **1g**[4] were synthesized according to known literature procedures.
α-Br-imides **1c-d**,**f-g**,[2] **1h-k,**[5,6] and **1l**[6,7] were synthesized via adapted literature procedures.
Olefins **2k**,[8] **2n,**[9] **2o,**[10] **2p,**[11] and **2r**[12] were synthesized according to a known literature procedure.
Olefins **2q** and **2s** were synthesized via an adapted literature procedure.[11]
All remaining olefins were purchased from Aldrich or Fisher and used as received.

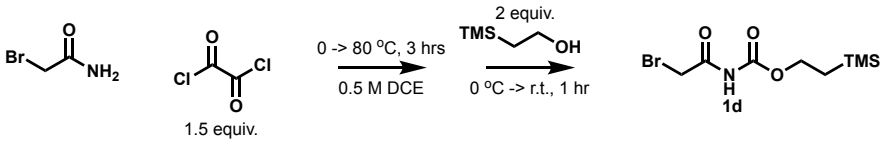
Synthesis of imide **1c**.



To a dry 100 mL flask was added 2-bromoacetamide (20 mmol, 2.56 g) and affixed a reflux condenser. The flask was evacuated and refilled with N_2 three times after which 40 mL dry and degassed DCE was added. The solution was cooled to 0 °C with an ice bath and placed under slight positive pressure of N_2 with an outlet needle due to gas evolution during the reaction. 30 mmol (2.54 g, 1.72 mL) of oxalyl chloride was added dropwise via syringe. The mixture was stirred at 0 °C, warmed to room temperature, and then refluxed at 80 °C for 3 hours. The solution was again brought to 0 °C with an ice bath and 40 mmol (5.98 g, 3.86 mL) of 2,2,2-trichloroethan-1-ol was added. The reaction was stirred for 15 minutes at 0 °C and allowed to warm to room temperature and stir for 1 hour. The solution was then poured into 100 mL 0 °C solution of $NaHCO_3$ and extracted with DCM x1 and EtOAc x2. The organic layers were combined and dried over Na_2SO_4, concentrated by rotary evaporation, and subjected to flash silica gel column chromatography (eluent 0-20% EtOAc in Hexanes) to furnish 1.5 g (4.6 mmol, 23 %) of desired imide **1c.**

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (s, 1H), 4.82 (s, 2H), 4.31 (s, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 166.67, 149.88, 94.25, 75.24, 28.42.
HRMS (ESI+) [$C_5H_5BrCl_3NNaO_3$+] m/z calculated 335.8391, found 335.8395

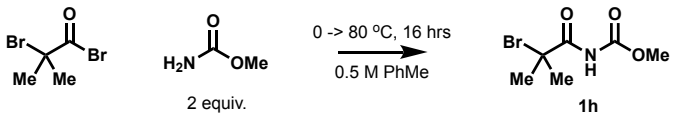
Synthesis of imide **1d.**



To a dry 100 mL flask was added 2-bromoacetamide (20 mmol, 2.56 g) and affixed a reflux condenser. The flask was evacuated and refilled with N_2 three times after which 40 mL dry and degassed DCE was added. The solution was cooled to 0 °C with an ice bath and placed under slight positive pressure of N_2 with an outlet needle due to gas evolution during the reaction. 30 mmol (2.54 g, 1.72 mL) of oxalyl chloride was added dropwise via syringe. The mixture was stirred at 0 °C, warmed to room temperature, and then refluxed at 80 °C for 3 hours. The solution was again brought to 0 °C with an ice bath and 40 mmol (4.73 g, 5.73 mL) of 2-(trimethylsilyl)ethan-1-ol was added. The reaction was stirred for 15 minutes at 0 °C and allowed to warm to room temperature and stir for 1 hour. The solution was then poured into 100 mL 0 °C solution of $NaHCO_3$ and extracted with DCM x1 and EtOAc x2. The organic layers were combined and dried over Na_2SO_4, concentrated by rotary evaporation, and subjected to flash silica gel column chromatography (eluent 0-20% EtOAc in Hexanes) to furnish 3.1 g (11 mmol, 55%) of desired imide **1d.**

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (s, 1H), 4.33 (s, 2H), 4.32 – 4.24 (m, 4H), 1.16 – 0.90 (m, 2H), 0.06 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 167.04, 151.49, 65.77, 28.81, 17.68, -1.36.
HRMS (ESI+) [$C_8H_{16}BrNNaO_3$+] m/z calculated 305.9955, found 305.9989

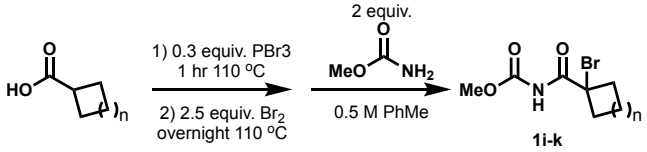
Synthesis of imide **1h**.



To a dry 100 mL flask was added methyl carbamate (40 mmol, 3.0 g) and affixed a reflux condenser. The flask was evacuated and refilled with N_2 three times after which 40 mL dry and degassed toluene was added. To the resulting solution was slowly added 2-bromo-2-methylpropanoyl bromide (20 mmol, 4.6 g, 2.5 mL) at 0 °C. The solution was heated at 80 °C overnight under a stream of N_2. The solution was then poured into 100 mL 0 °C solution of $NaHCO_3$ and extracted with DCM x1 and EtOAc x2. The organic layers were combined and dried over Na_2SO_4, concentrated by rotary evaporation, and subjected to flash silica gel column chromatography (eluent 0-10% EtOAc in Hexanes) to furnish 3.0 g (14 mmol, 68%) of desired imide **1h** (amorphous solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.50 (s, 1H), 3.82 (s, 3H), 1.95 (s, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.33, 151.21, 60.63, 53.41, 31.77.
HRMS (ESI+) [$C_6H_{10}BrNNaO_3$+] m/z calculated 247.9716, found 247.9767

Synthesis of imides **1i-k**.



To a dry 100 mL flask fitted with a reflux condenser and an addition funnel was added cyclohexanecarboxylic acid (23.5 mmol, 3.0 g). The flask was sparged with N_2 for 20 minutes and then PBr_3 (7.1 mmol, 0.670 mL) was added via syringe. The solution was heated to 110 °C for 1 hour. Then Br_2 (58.6 mmol, 3.0 mL) was added dropwise. The reaction was stirred overnight and then allowed to cool to room temperature. The reaction was then sparged with N_2 into a saturated solution of sodium thiosulfate. To the resulting crude a-Br-acid bromide was added methyl carbamate (40 mmol, 3.0 g) followed by 40 mL dry degassed toluene. The resulting mixture was heated to 80 °C overnight under a stream of N_2. The solution was then poured into 100 mL 0 °C solution of $NaHCO_3$ and extracted with DCM x1 and EtOAc x2. The organic layers were combined and dried over Na_2SO_4, concentrated by rotary evaporation, and subjected to flash silica gel column chromatography (eluent 0-10% EtOAc in Hexanes) to furnish 1.6 g (6 mmol, 26%) of desired imide **1i** (amorphous solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.37 (s, 1H), 3.82 (s, 3H), 2.38 – 1.88 (m, 4H), 1.70 (dtt, J = 21.0, 13.2, 4.1 Hz, 5H), 1.41 – 1.20 (m, 1H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 168.99, 151.41, 69.34, 53.39, 37.80, 24.78, 22.85.
HRMS (ESI+) [$C_9H_{14}BrNNaO_3$+] m/z calculated 286.0049, found 286.0078

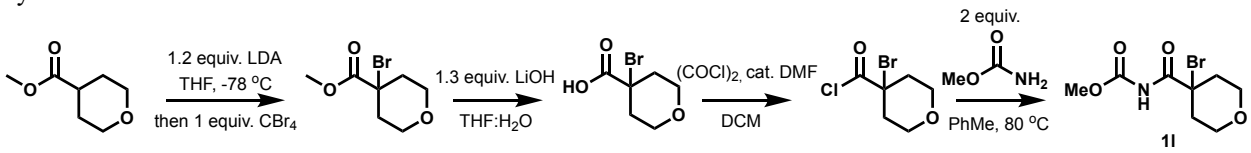
The above procedure provided **1j** in 23% yield from cyclopentanecarboxylic acid (amorphous solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (s, 1H), 3.84 (s, 3H), 2.45 (dddd, J = 15.3, 8.8, 5.5, 1.5 Hz, 2H), 2.36 – 2.17 (m, 2H), 2.08 – 1.81 (m, 4H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 168.52, 151.18, 71.34, 53.43, 41.99, 23.81.
HRMS (ESI+) [$C_8H_{12}BrNNaO_3$+] m/z calculated 271.9893, found 271.9924

The above procedure provided **1k** in 44% yield from cyclobutanecarboxylic acid (amorphous solid).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (s, 1H), 1.96 (s, 6H), 1.52 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.62, 149.24, 83.32, 61.14, 31.93, 28.14.
LRMS (EI) [$C_7H_{10}BrNNaO_3$+] m/z calculated 257.9736, found 257.0

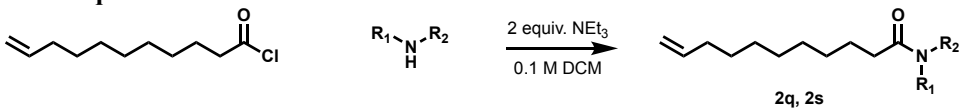
Synthesis of imide **1l**.



From commercially available methyl tetrahydro-2H-pyran-4-carboxylate was synthesized methyl 4-bromotetrahydro-2H-pyran-4-carboxylate via a known literature procedure in an 87% isolated yield.[6] The isolated material was then added to a 250 mL flask and dissolved in 150 mL THF. LiOH monohydrate 950 mg (1.3 equiv.) was dissolved in 75 mL of deionized water and added dropwise to the organic solution and allowed to stir at room temperature until disappearance of the starting material was seen by TLC. The reaction was quenched via the addition of 1M HCl, diluted with EtOAc and the organic layer separated. The aqueous layer was extracted twice more with EtOAc, dried with sodium sulfate, and concentrated. Analysis by ^{1}H NMR showed complete conversion to the acid. The crude acid was dissolved in dry DCM (35 mL, 0.5 M) with two drops of DMF. The solution was cooled to 0 °C and oxalyl chloride (4.42 mL, 2 equiv.) was added dropwise. After complete conversion of starting material by TLC the solution was concentrated under reduced pressure. The flask was fitted with a reflux condenser and dry degassed toluene was added under a N_2 atmosphere. Methyl carbamate (2.61 g, 2 equiv.) was then added quickly and the solution heated at 80 °C overnight under a stream of N_2. The solution was then poured into 100 mL 0 °C solution of $NaHCO_3$ and extracted with DCM x1 and EtOAc x2. The organic layers were combined and dried over Na_2SO_4, concentrated by rotary evaporation, and subjected to flash silica gel column chromatography (eluent 0-40% EtOAc in Hexanes) to furnish 600 mg (5.2 mmol, 26%) of desired imide **1l** (amorphous solid).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.28 (s, 1H), 3.92 (dd, *J* = 9.9, 6.0 Hz, 2H), 3.87 – 3.77 (m, 5H), 2.35 (ddd, *J* = 14.6, 10.1, 4.4 Hz, 2H), 2.03 (dd, *J* = 14.6, 3.0 Hz, 2H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 167.76, 151.25, 64.09, 53.58, 37.11.
HRMS (ESI+) [$C_8H_{12}BrNNaO_4$+] m/z calculated 287.9842, found 287.9870

Synthesis of olefins **2q** and **2s**

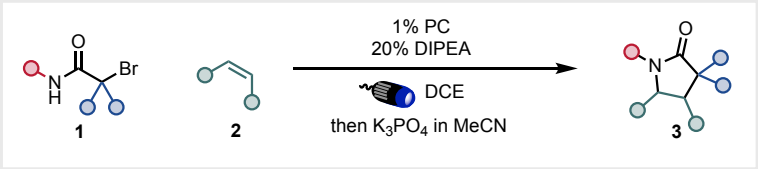


To a solution of amine (1 equiv., 10 mmol) and triethylamine (2 equiv., 20 mmol) in DCM (0.1 M) was added undec-10-enoyl chloride (1.2 equiv. 12 mmol) dropwise at 0 °C. The solution was allowed to warm to room temperature and stir overnight (16 hours). The reaction was quenched with saturated aqueous sodium bicarbonate and extracted with DCM twice. The organic phases were combined and dried over sodium sulfate and concentrated under reduced pressure. The crude material was then purified via silica column chromatography.

2q – *N*-cyclohexylundec-10-enamide
Yield 86% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.80 (ddt, *J* = 16.8, 10.0, 6.6 Hz, 1H), 5.32 – 5.19 (m, 1H), 5.16 – 4.75 (m, 1H), 4.02 – 3.55 (m, 1H), 2.19 – 2.07 (m, 1H), 2.07 – 1.96 (m, 2H), 1.90 (dd, *J* = 12.7, 4.0 Hz, 2H), 1.65 (ddt, *J* = 32.4, 10.7, 3.5 Hz, 5H), 1.44 – 1.22 (m, 13H), 1.13 (dddd, *J* = 23.2, 15.5, 9.4, 3.5 Hz, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 172.37, 139.37, 114.32, 48.19, 37.30, 33.96, 33.47, 29.50, 29.48, 29.45, 29.42, 29.26, 29.24, 29.07, 26.06, 25.74, 25.06.
HRMS (ESI+) [$C_{17}H_{32}NO$+] m/z calculated 266.2478, found 266.2549

2s – *tert*-butyl 4-(undec-10-enoyl)piperazine-1-carboxylate
Yield 92% (amorphous solid)
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.79 (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.10 – 4.84 (m, 1H), 3.58 (t, *J* = 5.2 Hz, 2H), 3.40 (d, *J* = 15.7 Hz, 5H), 2.38 – 2.22 (m, 2H), 2.11 – 1.93 (m, 2H), 1.68 – 1.54 (m, 2H), 1.46 (s, 7H), 1.40 – 1.13 (m, 10H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 172.08, 154.76, 139.32, 114.30, 80.44, 45.59, 41.49, 33.93, 33.53, 29.60, 29.52, 29.48, 29.22, 29.05, 28.53, 25.46.
HRMS (ESI+) [$C_{20}H_{36}N_2NaO_3$+] m/z calculated 375.2618, found 375.2646

Standard Reaction Conditions:



A) To an oven-dried 8 mL test tube vial, was added 1% [Ir(dFCF$_3$ppy)$_2$dtbbpy]PF$_6$ and α-bromoimide (0.6 mmol, 2 equiv.) electrophile. 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 degassed 1,2-dichloroethane was added (750 uL, 0.4 M) followed by the olefin nucleophile (0.3 mmol, 1 equiv.) and diisopropylethylamine (0.06 mmol, .2 equiv.) and sealed tightly. The vial was then placed ~3 inches from a 440 nm Kessil lamp to be irradiated and stirred for 16 hours at room temperature. The vial was then removed from the light box and K_3PO_4 (5 equiv. 1.5 mmol) was added along with 2.25 mL MeCN and sufficient H_2O to render the K_3PO_4 soluble. After stirring at room temperature for 3 hours, the vial was rinsed with EtOAc into a flask and then concentrated onto silica gel in vacuo. Products were purified using flash column chromatography using EtOAc:Hexanes as an eluent.

B) Identical to standard reaction conditions A but using 50% loading of N-isopropyl-N,2-dimethylpropan-2-amine instead of diisopropylethylamine.

C) To an oven-dried 8 mL test tube vial, was added 2% [Ir(dFCF$_3$ppy)$_2$dtbbpy]PF$_6$, 0.3 mmol (1 equiv.), of α-bromoimide, 0.3 mmol (1 equiv.) La(Otf)$_3$ and 750 uL of MeCN. This solution was allowed to stir at room temperature for at least 30 minutes. Then 3 equiv. (0.9 mmol) of the olefin nucleophile and 0.33 mmol diisopropylethylamine are added and the vial was placed ~3 inches from a 440 nm Kessil lamp to be irradiated and stirred for 36 hours at room temperature. In some cases, noted specifically, the intramolecular cyclization does not occur and so the vial was removed from the light box and K_3PO_4 (5 equiv. 1.5 mmol) was added along with 2.25 mL MeCN and sufficient H_2O to render the K_3PO_4 soluble. After stirring at room temperature for 3 hours or directly from the photoreactor the vial was rinsed with EtOAc and saturated aqueous ammonium chloride into a separatory funnel and extracted 3x with EtOAc. This was then dried with sodium sulfate and and concentrated onto silica gel in vacuo. Products were purified using flash column chromatography using EtOAc:Hexanes as an eluent.

D) To an oven-dried 3 mL vial, was added α-bromoimide (0.9 mmol, 3 equiv.) electrophile. 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 degassed 1,2-dichloroethane was added (750 µL, 0.4 M) followed by the olefin nucleophile (0.3 mmol, 1 equiv.) and 2-methylpyridine (0.06 mmol, .2 equiv.) and sealed tightly. The vial was then placed ~2 inches from a 390 nm Kessil lamp to be irradiated and stirred for 16 hours at room temperature. The vial was then removed from the light box and K_3PO_4 (5 equiv. 1.5 mmol) was added along with 2.25 mL MeCN and H_2O dropwise until the K_3PO_4 dissolved. After stirring at room temperature for 3 hours, the vial was rinsed with EtOAc into a flask and then concentrated onto silica gel in vacuo. Products were purified using flash column chromatography using EtOAc:Hexanes as an eluent.

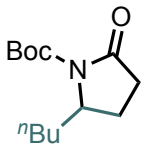
E) To an oven-dried 3 mL vial, was added α-bromoimide (3.0 mmol, 3 equiv.) electrophile. The vial was charged with a stir bar and transferred to a glovebox, where the solids were backfilled with an inert atmosphere. In the glovebox anhydrous degassed 1,2-dichloroethane was added (2.25 mL, 0.4 M) followed by the olefin nucleophile (1.0 mmol, 1 equiv.) and 2-methylpyridine (0.2 mmol, .2 equiv.) and sealed tightly. The vial was then placed ~2 inches from a 390 nm Kessil lamp to be irradiated and stirred for 16 hours at room temperature. The vial was then removed from the light box and the contents were transferred to a 20 mL scintillation vial rinsing the vial with 7.75 mL MeCN. Then K_3PO_4 (5 equiv. 1.5 mmol) was added followed by H_2O dropwise until the K_3PO_4 dissolved. After stirring at room temperature for 3 hours, the vial was rinsed with EtOAc into a flask and then concentrated onto silica gel in vacuo. Products were purified using flash column chromatography using EtOAc:Hexanes as an eluent.

Helpful Tips:

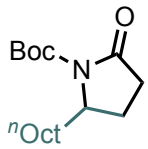
Sufficiently wet MeCN also works well for the cyclization as we found through control studies. Excess H_2O does not appear deleterious but does start to enable phase separation if too much is added. Sufficient mixing was found to be essential in these cases.

Depending on the structure of the α-bromoimide, complexation with the Lewis acid often results in an insoluble complex. The mixture typically becomes homogeneous after a few minutes of stirring under irradiation.

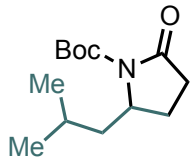
Characterization Data of Products:



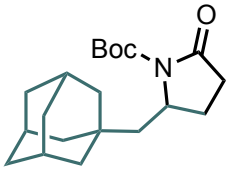
3aa – tert-butyl 2-butyl-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2a**.
Yield 77% (viscous oil)
Prepared using standard reaction conditions D from **1a** and **2a**.
Yield 64%
^{1}H NMR (400 MHz, $CDCl_3$) 4.09 (dddd, *J* = 9.5, 8.3, 3.4, 1.9 Hz, 1H), 2.57 (ddd, *J* = 17.7, 11.0, 9.2 Hz, 1H), 2.41 (ddd, *J* = 17.7, 9.5, 2.7 Hz, 1H), 2.16 – 2.01 (m, 1H), 1.83 – 1.71 (m, 2H), 1.52 (s, 9H), 1.52 – 1.42 (m, 1H), 1.42 – 1.23 (m, 4H), 0.91 (t, *J* = 7.0 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-d) δ 174.62, 150.21, 82.83, 58.25, 33.55, 31.60, 28.24, 27.96, 22.71, 22.67, 14.20.
HRMS (ESI+) [$C_{13}H_{23}NnaO_3$+] m/z calculated 264.1570, found 264.1606



3ab – tert-butyl 2-hexyl-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2b**.
Yield 69% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.09 (dddd, J = 9.3, 8.2, 3.4, 1.9 Hz, 1H), 2.57 (ddd, J = 17.7, 11.0, 9.1 Hz, 1H), 2.47 – 2.35 (m, 1H), 2.16 – 2.01 (m, 1H), 1.76 (dddd, J = 12.8, 9.2, 2.7, 2.0 Hz, 2H), 1.53 (s, 9H), 1.34 – 1.24 (m, 12H), 0.92 – 0.84 (m, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.62, 150.21, 82.83, 58.28, 33.90, 32.00, 31.61, 29.70, 29.65, 29.36, 28.24, 25.83, 22.82, 22.69, 14.26.
HRMS (ESI+) [$C_{15}H_{27}NnaO_3$+] m/z calculated 320.2196, found 320.2223

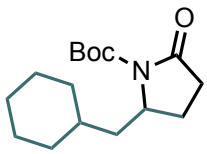


3ac – tert-butyl 2-isobutyl-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2c**.
Yield 50% (viscous oil)
^{1}H NMR (500 MHz, CDCl3) δ 4.28 – 4.06 (m, 1H), 2.58 (ddd, J = 17.6, 11.4, 9.0 Hz, 1H), 2.41 (ddd, J = 17.7, 9.4, 2.3 Hz, 1H), 2.15 – 2.02 (m, 1H), 1.76 (ddt, J = 12.9, 9.0, 2.0 Hz, 1H), 1.67 – 1.57 (m, 2H), 1.53 (s, 9H), 1.40 (ddd, J = 12.4, 10.6, 3.4 Hz, 1H), 0.98 – 0.84 (m, 6H).
^{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.
HRMS (ESI+) [$C_{13}H_{23}NnaO_3$+] m/z calculated 264.1570, found 264.1606

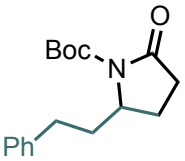


3ad – tert-butyl 2-(((3r,5r,7r)-adamantan-1-yl)methyl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A **1a** and **2d**.

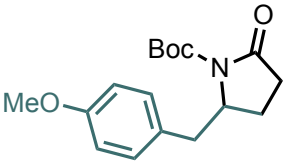
Yield 59% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.11 (dddd, J = 11.1, 8.1, 3.2, 1.6 Hz, 1H), 2.59 (ddd, J = 17.6, 11.5, 9.0 Hz, 1H), 2.47 – 2.35 (m, 1H), 2.15 – 2.00 (m, 2H), 1.98 – 1.76 (m, 6H), 1.53 (s, 10H), 1.37 (ddd, J = 13.4, 10.7, 4.1 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 174.37, 60.33, 37.01, 33.24, 32.74, 32.19, 27.40, 26.46, 25.57, 14.44.
HRMS (ESI+) [$C_{20}H_{31}NnaO_3$+] m/z calculated 356.2196, found 356.2227



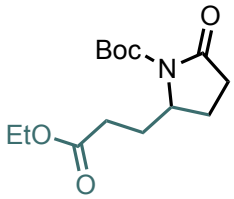
3ae – tert-butyl 2-(cyclohexylmethyl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2e**.
Yield 62% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.27 – 4.07 (m, 1H), 2.58 (ddd, J = 17.7, 11.4, 9.0 Hz, 1H), 2.40 (ddd, J = 17.7, 9.3, 2.3 Hz, 1H), 2.08 (ddddd, J = 12.6, 11.4, 9.3, 8.1, 1.1 Hz, 1H), 1.85 – 1.57 (m, 7H), 1.53 (s, 9H), 1.41 – 1.09 (m, 5H), 1.07 – 0.87 (m, 2H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.68, 150.05, 82.85, 56.33, 41.36, 35.06, 34.60, 32.69, 31.36, 28.28, 26.59, 26.49, 26.28, 23.02.
HRMS (ESI+) [$C_{16}H_{27}NnaO_3$+] m/z calculated 304.1883, found 304.1916



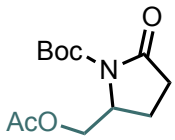
3af -tert-butyl 2-oxo-5-phenethylpyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2f**.
Yield 84% (viscous oil)
Prepared using standard reaction conditions D from **1a** and **2f**.
Yield 77%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 2H), 7.23 – 7.15 (m, 3H), 4.15 (dddd, *J* = 9.7, 8.3, 3.4, 2.0 Hz, 1H), 2.79 – 2.52 (m, 3H), 2.44 (ddd, *J* = 17.7, 9.5, 2.8 Hz, 1H), 2.22 – 2.05 (m, 2H), 1.93 – 1.75 (m, 2H), 1.50 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.47, 150.09, 141.03, 128.75, 128.45, 126.36, 83.00, 57.82, 35.50, 32.26, 31.51, 28.21, 22.57.
HRMS (ESI+) [$C_{17}H_{23}NnaO_3$+] m/z calculated 312.1570, found 312.1610



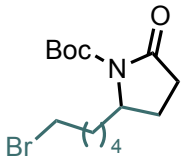
3ag – *tert*-butyl 2-(4-methoxybenzyl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A **1a** and **2g**.
Yield 50% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 – 7.05 (m, 2H), 6.89 – 6.81 (m, 2H), 4.32 (tdd, *J* = 8.6, 3.4, 1.5 Hz, 1H), 3.79 (s, 3H), 3.09 – 2.95 (m, 1H), 2.71 (dd, *J* = 13.5, 8.8 Hz, 1H), 2.49 – 2.16 (m, 2H), 1.96 (dtd, *J* = 13.0, 10.4, 8.4 Hz, 1H), 1.80 (dddd, *J* = 13.0, 7.7, 3.6, 1.5 Hz, 1H), 1.58 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.61, 158.77, 150.15, 130.55, 129.17, 114.36, 83.11, 59.27, 55.44, 38.79, 31.33, 28.32, 21.78.
HRMS (ESI+) [$C_{17}H_{23}NnaO_4$+] m/z calculated 328.1519, found 328.1557



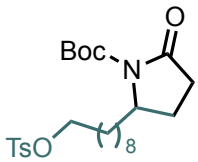
3ah – *tert*-butyl 2-(3-ethoxy-3-oxopropyl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2h**.
Yield 72% (viscous oil)
Prepared using standard reaction conditions D from **1a** and **2h**.
Yield 76%
Prepared using standard reaction conditions E from **1a** and **2h**.
Yield 74%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.27 – 4.02 (m, 3H), 2.59 (ddd, *J* = 17.8, 11.0, 9.1 Hz, 1H), 2.43 (ddd, *J* = 17.8, 9.5, 2.8 Hz, 1H), 2.34 (ddd, *J* = 8.5, 7.0, 2.0 Hz, 2H), 2.20 – 2.03 (m, 2H), 1.93 – 1.80 (m, 1H), 1.75 (dddd, *J* = 13.0, 9.1, 2.8, 2.0 Hz, 1H), 1.53 (s, 9H), 1.26 (t, *J* = 7.1 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.14, 172.75, 150.12, 83.25, 60.85, 57.38, 31.48, 30.79, 29.22, 28.21, 22.72, 14.37.
HRMS (ESI+) [$C_{14}H_{23}NnaO_5$+] m/z calculated 308.1468, found 308.1488



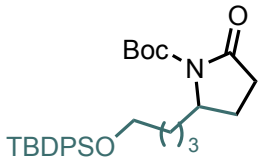
3ai - *tert*-butyl 2-(acetoxymethyl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2i**.
Yield 54% (viscous oil)
Prepared using standard reaction conditions D from **1a** and **2i**.
Yield 52%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.43 – 4.33 (m, 2H), 4.25 – 4.11 (m, 1H), 2.65 (ddd, *J* = 17.7, 11.1, 9.6 Hz, 1H), 2.45 (ddd, *J* = 17.7, 9.9, 2.3 Hz, 1H), 2.26 – 2.13 (m, 1H), 2.07 (s, 3H), 1.94 (ddt, *J* = 13.3, 9.6, 1.9 Hz, 1H), 1.54 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.25, 170.79, 149.85, 83.64, 65.07, 56.31, 31.93, 28.21, 21.19, 21.03.
HRMS (ESI+) [$C_{12}H_{19}NNaO_5$+] m/z calculated 280.1155, found 280.1195



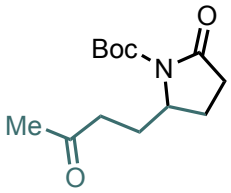
3aj - *tert*-butyl 2-(5-bromopentyl)-5-oxopyrrolidine-1-carboxylate
Prepared using reaction conditions A from **1a** and **2j**.
Yield 68% (viscous oil)
Prepared using reaction conditions D from **1a** and **2j**.
Yield 60%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.15 – 4.05 (m, 1H), 3.41 (t, *J* = 6.7 Hz, 2H), 2.57 (ddd, *J* = 17.7, 11.0, 9.1 Hz, 1H), 2.42 (ddd, *J* = 17.7, 9.5, 2.8 Hz, 1H), 2.10 (tt, *J* = 10.9, 9.1 Hz, 1H), 1.95 – 1.70 (m, 4H), 1.53 (s, 10H), 1.47 – 1.23 (m, 4H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.39, 150.23, 82.99, 58.02, 33.73, 33.70, 32.75, 31.58, 28.26, 28.16, 25.02, 22.69.
HRMS (ESI+) [$C_{14}H_{24}BrNNaO_5$+] m/z calculated 356.0832, found 356.0859



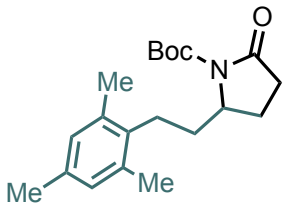
3ak - *tert*-butyl 2-oxo-5-(9-(tosyloxy)nonyl)pyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2k**.
Yield 86% (viscous oil)
Prepared using standard reaction conditions D from **1a** and **2k**.
Yield 47%
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 – 7.75 (m, 2H), 7.34 (d, *J* = 8.1 Hz, 2H), 4.08 (dddd, *J* = 9.5, 8.2, 3.4, 1.9 Hz, 1H), 4.01 (t, *J* = 6.5 Hz, 2H), 2.62 – 2.49 (m, 1H), 2.48 – 2.33 (m, 4H), 2.09 (tt, *J* = 12.1, 9.1 Hz, 1H), 1.75 (dddd, *J* = 14.7, 10.4, 6.3, 4.0 Hz, 2H), 1.67 – 1.57 (m, 2H), 1.52 (s, 9H), 1.38 – 1.15 (m, 13H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 174.62, 150.20, 144.81, 133.40, 129.97, 128.05, 82.84, 70.83, 58.23, 33.87, 31.61, 29.58, 29.44, 29.06, 29.00, 28.23, 25.80, 25.50, 22.67, 21.82.
HRMS (ESI+) [$C_{25}H_{39}NNaO_6S$+] m/z calculated 504.2390, found 504.2431



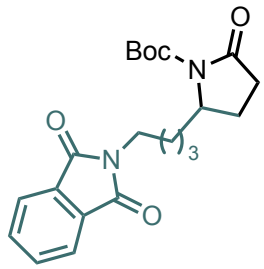
3al - *tert*-butyl 2-(5-((*tert*-butyldiphenylsilyl)oxy)pentyl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2l**.
Yield 78% (viscous oil)
Prepared using standard reaction conditions D from **1a** and **2l**.
Yield 75%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.62 (m, 4H), 7.43 – 7.33 (m, 6H), 4.07 (ddq, *J* = 9.4, 5.3, 1.9 Hz, 1H), 3.67 (t, *J* = 6.3 Hz, 2H), 2.55 (ddd, *J* = 17.7, 11.0, 9.1 Hz, 1H), 2.41 (ddd, *J* = 17.7, 9.5, 2.7 Hz, 1H), 2.08 (tt, *J* = 12.4, 9.3 Hz, 1H), 1.74 (ddt, *J* = 11.0, 6.4, 2.3 Hz, 2H), 1.63 – 1.35 (m, 15H), 1.05 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.51, 150.17, 135.72, 134.12, 129.77, 127.80, 82.84, 63.72, 58.19, 33.59, 32.51, 31.56, 28.23, 27.04, 22.61, 22.15, 19.39.
HRMS (ESI+) [$C_{29}H_{41}NNaO_4Si$+] m/z calculated 518.2697, found 518.2708



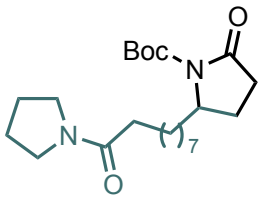
3am - *tert*-butyl 2-(4-((*tert*-butyldiphenylsilyl)oxy)butyl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2m**.
Yield 71% (viscous oil)
Prepared using standard reaction conditions D from **1a** and **2m**.
Yield 62%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.15 (tdd, *J* = 8.3, 4.5, 2.0 Hz, 1H), 2.70 – 2.25 (m, 4H), 2.17 (s, 3H), 2.11 – 1.95 (m, 2H), 1.83 (dtd, *J* = 13.7, 8.3, 6.6 Hz, 1H), 1.75 – 1.67 (m, 1H), 1.54 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 207.32, 174.09, 150.38, 83.26, 57.30, 39.76, 31.55, 30.14, 28.24, 27.93, 23.02.
HRMS (ESI+) [$C_{13}H_{21}NNaO_4$+] m/z calculated 278.1363, found 278.1386



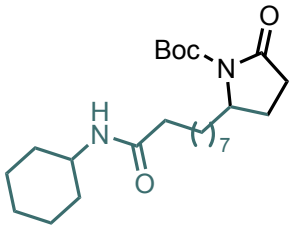
3an - *tert*-butyl 2-oxo-5-(2,4,6-trimethylphenethyl)pyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2n**.
Yield 88% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (s, 2H), 4.28 – 4.18 (m, 1H), 2.71 – 2.42 (m, 4H), 2.26 (d, *J* = 16.7 Hz, 10H), 1.98 – 1.84 (m, 2H), 1.69 (dddd, *J* = 13.7, 11.4, 9.0, 5.9 Hz, 1H), 1.51 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.38, 150.13, 135.83, 135.62, 134.77, 129.26, 83.02, 58.26, 33.18, 31.69, 28.22, 25.36, 22.75, 20.95, 19.85.
HRMS (ESI+) [$C_{20}H_{29}NNaO_3$+] m/z calculated 354.2040, found 354.2063



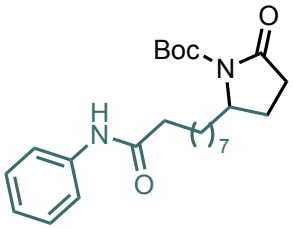
3ao - *tert*-butyl 2-(5-(1,3-dioxoisoindolin-2-yl)pentyl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2o**.
Yield 60% (amorphous solid)
Prepared using standard reaction conditions D from **1a** and **2o**.
Yield 79%
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.76 – 7.67 (m, 2H), 4.08 (dddd, *J* = 9.7, 8.2, 3.4, 1.9 Hz, 1H), 3.70 (t, *J* = 7.1 Hz, 2H), 2.64 – 2.47 (m, 1H), 2.40 (ddd, *J* = 17.8, 9.4, 2.6 Hz, 1H), 2.08 (tt, *J* = 12.6, 9.3 Hz, 1H), 1.90 – 1.62 (m, 5H), 1.51 (m, 11H), 1.45 – 1.28 (m, 2H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.35, 168.59, 150.21, 134.15, 132.27, 123.41, 83.00, 57.99, 37.87, 33.40, 31.55, 28.63, 28.23, 23.23, 22.67.
HRMS (ESI+) [$C_{21}H_{26}N_2NaO_5$+] m/z calculated 409.1734, found 409.1752



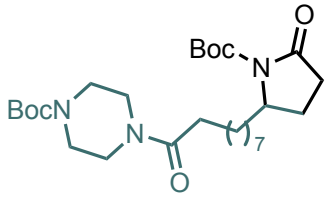
3ap - *tert*-butyl 2-oxo-5-(9-oxo-9-(pyrrolidin-1-yl)nonyl)pyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2p**.
Yield 83% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.08 (dddd, *J* = 9.4, 8.3, 3.4, 1.9 Hz, 1H), 3.43 (s, 4H), 2.56 (ddd, *J* = 17.7, 11.0, 9.2 Hz, 1H), 2.40 (ddd, *J* = 17.7, 9.5, 2.7 Hz, 1H), 2.30 – 2.19 (m, 2H), 2.08 (tt, *J* = 12.1, 9.2 Hz, 1H), 1.94 – 1.86 (m, 5H), 1.81 – 1.70 (m, 2H), 1.64 (h, *J* = 7.8 Hz, 2H), 1.52 (s, 9H), 1.30 (s, 10H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.61, 172.10, 150.20, 82.82, 58.25, 46.78, 45.92, 34.89, 33.90, 31.60, 29.65, 29.62, 29.60, 29.53, 28.24, 25.82, 25.10, 22.68.
HRMS (ESI+) [$C_{22}H_{38}N_2NaO_4$+] m/z calculated 417.2724, found 417.2740



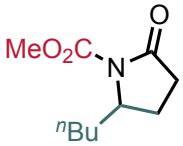
3aq - *tert*-butyl 2-(9-(cyclohexylamino)-9-oxononyl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2q**.
Yield 86% (viscous oil)
Prepared using standard reaction conditions D from **1a** and **2q**.
Yield 71%
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.42 (d, *J* = 7.3 Hz, 1H), 4.08 (dddd, *J* = 9.4, 8.2, 3.4, 1.9 Hz, 1H), 3.76 (dddd, *J* = 14.7, 10.6, 8.0, 3.9 Hz, 1H), 2.56 (ddd, *J* = 17.8, 11.0, 9.2 Hz, 1H), 2.40 (ddd, *J* = 17.7, 9.5, 2.7 Hz, 1H), 2.23 – 1.99 (m, 3H), 1.89 (dt, *J* = 11.9, 3.9 Hz, 2H), 1.81 – 1.66 (m, 4H), 1.65 – 1.57 (m, 3H), 1.52 (s, 9H), 1.40 – 1.24 (m, 13H), 1.17 – 1.04 (m, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.60, 172.39, 150.20, 82.84, 58.23, 48.31, 37.11, 33.88, 33.40, 31.61, 29.58, 29.54, 29.38, 29.34, 28.23, 26.01, 25.74, 25.71, 25.04, 22.67.
HRMS (ESI+) [$C_{24}H_{42}N_2NaO_4$+] m/z calculated 445.3037, found 445.3127



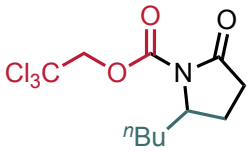
3ar - *tert*-butyl 2-oxo-5-(9-oxo-9-(phenylamino)nonyl)pyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2r**.
Yield 65% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.9 Hz, 2H), 7.37 – 7.24 (m, 1H), 7.09 (t, *J* = 7.4 Hz, 1H), 4.14 – 4.04 (m, 1H), 2.57 (ddd, *J* = 17.7, 11.0, 9.2 Hz, 1H), 2.46 – 2.30 (m, 3H), 2.17 – 1.99 (m, 1H), 1.81 – 1.66 (m, 4H), 1.52 (s, 9H), 1.32 (s, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.63, 171.46, 150.23, 138.18, 129.17, 124.33, 119.89, 82.88, 58.23, 37.93, 33.87, 31.64, 29.52, 29.46, 29.33, 29.29, 28.26, 28.25, 25.67, 22.69.
HRMS (ESI+) [$C_{24}H_{36}N_2NaO_4$+] m/z calculated 439.2567, found 439.2663



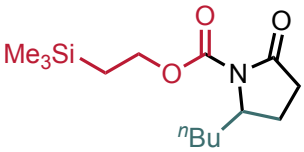
3as - *tert*-butyl 4-(9-(1-(*tert*-butoxycarbonyl)-5-oxopyrrolidin-2-yl)nonanoyl)piperazine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2s**.
Yield 82% (amorphous solid)
Prepared using standard reaction conditions D from **1a** and **2s**.
Yield 63%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.12 – 4.02 (m, 1H), 3.58 (s, 2H), 3.43 (s, 6H), 2.56 (ddd, *J* = 17.7, 11.0, 9.1 Hz, 1H), 2.40 (ddd, *J* = 17.8, 9.5, 2.7 Hz, 1H), 2.31 (t, *J* = 7.6 Hz, 2H), 2.20 – 1.97 (m, 1H), 1.82 – 1.69 (m, 2H), 1.61 (d, *J* = 7.4 Hz, 2H), 1.52 (s, 9H), 1.46 (s, 9H), 1.30 (s, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.39, 171.92, 154.60, 150.13, 82.66, 80.30, 58.06, 45.42, 43.65, 41.33, 33.72, 33.34, 31.53, 29.44, 29.42, 29.34, 28.37, 28.34, 28.07, 25.63, 25.24, 22.51.
HRMS (ESI+) [$C_{27}H_{47}N_3NaO_6$+] m/z calculated 532.3357, found 532.3441



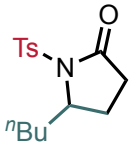
3ba - methyl 2-butyl-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1b** and **2a**.
Yield 72% (viscous oil)
Prepared using standard reaction conditions D from **1b** and **2a**.
Yield 79%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.18 (dddd, *J* = 9.5, 8.3, 3.4, 1.9 Hz, 1H), 3.86 (s, 3H), 2.62 (ddd, *J* = 17.9, 11.0, 9.2 Hz, 1H), 2.45 (ddd, *J* = 17.9, 9.4, 2.7 Hz, 1H), 2.13 (ddd, *J* = 20.2, 12.0, 9.0 Hz, 1H), 1.87 – 1.76 (m, 2H), 1.56 – 1.46 (m, 2H), 1.39 – 1.23 (m, 6H), 0.92 (t, *J* = 7.0 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.19, 152.50, 58.42, 53.62, 33.24, 31.61, 27.78, 22.75, 22.68, 14.14.
HRMS (ESI+) [$C_{10}H_{17}NNaO_3$+] m/z calculated 222.1101, found 222.1163



3ca - 2,2,2-trichloroethyl 2-butyl-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1c** and **2a**.
Yield 55% (viscous oil)
Prepared using standard reaction conditions D from **1c** and **2a**.
Yield 73%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.94 (d, *J* = 11.9 Hz, 1H), 4.78 (d, *J* = 11.9 Hz, 1H), 4.25 (dddd, *J* = 9.8, 8.3, 3.2, 1.7 Hz, 1H), 2.64 (ddd, *J* = 18.0, 11.3, 9.1 Hz, 1H), 2.49 (ddd, *J* = 18.0, 9.5, 2.5 Hz, 1H), 2.25 – 2.10 (m, 1H), 1.96 – 1.83 (m, 2H), 1.58 (ddtd, *J* = 11.3, 9.6, 5.3, 2.3 Hz, 1H), 1.44 – 1.18 (m, 4H), 0.91 (t, *J* = 6.9 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.36, 149.68, 94.71, 75.21, 58.56, 33.48, 31.18, 27.91, 22.80, 22.71, 14.20.
HRMS (ESI+) [$C_{11}H_{16}Cl_3NNaO_3$+] m/z calculated 338.0088, found 338.0118

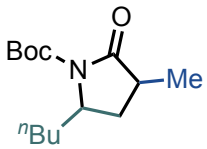


3da - (trimethylsilyl)methyl 2-butyl-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1d** and **2a**.
Yield 90% (viscous oil)
Prepared using standard reaction conditions D from **1d** and **2a**.
Yield 70%
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.40 – 4.24 (m, 2H), 4.16 (dddd, *J* = 9.4, 8.2, 3.4, 2.0 Hz, 1H), 2.60 (ddd, *J* = 17.8, 11.0, 9.2 Hz, 1H), 2.43 (ddd, *J* = 17.8, 9.5, 2.7 Hz, 1H), 2.18 – 2.03 (m, 1H), 1.87 – 1.74 (m, 2H), 1.57 – 1.42 (m, 1H), 1.40 – 1.24 (m, 4H), 1.17 – 1.05 (m, 2H), 0.91 (t, *J* = 7.0 Hz, 3H), 0.05 (s, 9H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.19, 152.02, 65.26, 58.28, 33.32, 31.64, 27.81, 22.73, 22.69, 17.82, 14.15, -1.40.
HRMS (ESI+) [$C_{13}H_{25}NNaO_3Si$+] m/z calculated 308.1652, found 308.1703

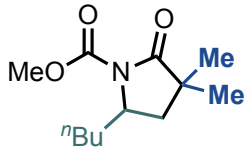


3ea - 5-butyl-1-tosylpyrrolidin-2-one
Prepared using standard reaction conditions C from **1e** and **2a** followed by addition of 5 equiv. K_3PO_4 (1.5 mmol) and stirring for 3 hours.

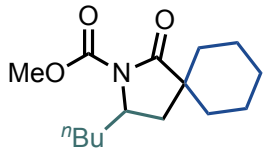
Yield 45% (amorphous solid)
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.32 (m, 2H), 4.39 (dddd, *J* = 9.3, 8.4, 3.1, 1.7 Hz, 1H), 2.52 (ddd, *J* = 17.7, 11.2, 9.2 Hz, 1H), 2.43 (s, 3H), 2.34 (ddd, *J* = 17.7, 9.6, 2.3 Hz, 1H), 2.24 – 2.10 (m, 1H), 1.96 (dddd, *J* = 13.7, 11.0, 5.3, 3.1 Hz, 1H), 1.85 (ddt, *J* = 13.0, 9.2, 2.0 Hz, 1H), 1.70 – 1.59 (m, 1H), 1.43 – 1.15 (m, 4H), 0.90 (t, *J* = 7.1 Hz, 3H).
^{13}C NMR (126 MHz, $CDCl_3$) δ 173.69, 145.05, 136.26, 129.60, 128.48, 60.58, 34.44, 30.97, 27.37, 23.72, 22.62, 21.81, 14.10.
HRMS (ESI+) [$C_{15}H_{21}NNaO_3S$+] m/z calculated 318.1134, found 318.1170



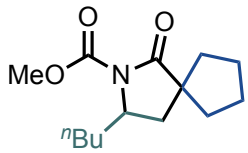
3fa - *tert*-butyl 5-butyl-3-methyl-2-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions B from **1f** and **2a**.
Yield 56% 1:1 *dr* (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.00 (td, *J* = 8.9, 3.3 Hz, 1H), 3.92 (dtd, *J* = 10.1, 7.4, 2.9 Hz, 1H), 2.74 (q, *J* = 7.4 Hz, 1H), 2.69 – 2.57 (m, 1H), 2.55 – 2.45 (m, 1H), 2.36 (ddd, *J* = 12.8, 9.7, 7.6 Hz, 1H), 2.10 – 1.93 (m, 2H), 1.53 (d, *J* = 1.9 Hz, 17H), 1.40 – 1.16 (m, 11H), 0.91 (t, *J* = 7.0 Hz, 6H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 177.34, 176.76, 150.67, 150.38, 82.87, 82.70, 56.34, 56.06, 37.68, 36.63, 34.89, 33.16, 31.84, 28.44, 28.24, 28.18, 27.00, 22.74, 22.71, 16.73, 15.56, 14.22, 14.20.
HRMS (ESI+) [$C_{14}H_{25}NNaO_3$+] m/z calculated 278.1727, found 278.1751



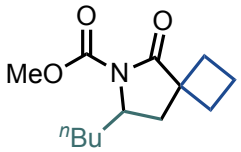
3ha - methyl 5-butyl-3,3-dimethyl-2-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions C from **1h** and **2a**.
Yield 59% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.99 (dddd, *J* = 9.4, 8.1, 6.0, 3.1 Hz, 1H), 3.86 (s, 3H), 2.12 – 1.99 (m, 2H), 1.63 (dd, *J* = 13.1, 6.0 Hz, 1H), 1.49 – 1.19 (m, 8H), 1.18 (s, 3H), 0.91 (t, *J* = 7.1 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 179.57, 153.01, 54.98, 53.61, 41.64, 38.52, 34.35, 27.32, 26.50, 26.13, 22.67, 14.16.
HRMS (ESI+) [$C_{12}H_{21}NNaO_3$+] m/z calculated 250.1414, found 250.1526



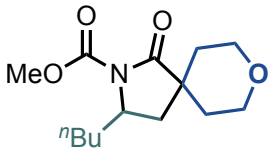
3ia - methyl 3-butyl-1-oxo-2-azaspiro[4.5]decane-2-carboxylate
Prepared using standard reaction conditions C from **1i** and **2a**.
Yield 80% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 3.99 (dddd, *J* = 9.7, 8.7, 5.6, 3.1 Hz, 1H), 3.86 (s, 3H), 2.10 (dd, *J* = 13.3, 8.5 Hz, 1H), 2.02 (dddd, *J* = 12.5, 9.4, 6.0, 3.1 Hz, 1H), 1.83 – 1.55 (m, 5H), 1.54 – 1.20 (m, 8H), 0.91 (t, *J* = 7.0 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 179.27, 153.02, 55.25, 53.59, 46.14, 34.74, 34.40, 34.32, 34.26, 27.38, 25.43, 22.68, 22.13, 22.01, 14.17.
HRMS (ESI+) [$C_{15}H_{25}NNaO_3$+] m/z calculated 290.1727, found 290.1839



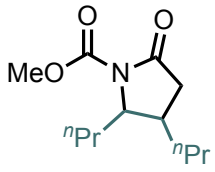
3ja - methyl 3-butyl-1-oxo-2-azaspiro[4.4]nonane-2-carboxylate
Prepared using standard reaction conditions C from **1j** and **2a**.
Yield 46% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.10 – 3.93 (m, 1H), 3.86 (s, 3H), 2.16 – 1.93 (m, 4H), 1.83 (td, *J* = 8.1, 4.3 Hz, 2H), 1.78 – 1.51 (m, 6H), 1.47 – 1.22 (m, 6H), 0.91 (t, *J* = 7.0 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 179.92, 152.90, 55.83, 53.59, 51.83, 39.11, 39.00, 38.14, 34.05, 27.70, 25.96, 25.55, 22.68, 14.17.
HRMS (ESI+) [$C_{14}H_{23}NNaO_3$+] m/z calculated 276.1570, found 276.1608



3ka - methyl 7-butyl-5-oxo-6-azaspiro[3.4]octane-6-carboxylate
Prepared using standard reaction conditions C from **1k** and **2a** followed by addition of 5 equiv. K_3PO_4 (1.5 mmol) and stirring for 3 hours.
Yield 63% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.09 – 3.93 (m, 1H), 3.85 (s, 3H), 2.58 – 2.40 (m, 2H), 2.24 – 1.79 (m, 7H), 1.44 – 1.20 (m, 5H), 0.99 – 0.81 (m, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 178.22, 152.76, 56.15, 53.55, 46.67, 36.99, 33.92, 33.57, 30.45, 27.98, 22.62, 16.53, 14.12.
HRMS (ESI+) [$C_{13}H_{21}NNaO_3$+] m/z calculated 262.1414, found 262.1535

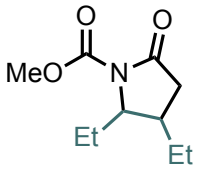


3la - methyl 3-butyl-1-oxo-8-oxa-2-azaspiro[4.5]decane-2-carboxylate
Prepared using standard reaction conditions C from **3l** and **2a**.
Yield 68% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.09 – 3.92 (m, 3H), 3.87 (s, 3H), 3.55 (ddt, *J* = 12.4, 9.5, 2.3 Hz, 2H), 2.19 (dd, *J* = 13.3, 8.4 Hz, 1H), 2.07 (dt, *J* = 14.1, 3.9 Hz, 2H), 1.95 (ddd, *J* = 13.8, 9.9, 4.1 Hz, 1H), 1.75 (dd, *J* = 13.3, 5.5 Hz, 1H), 1.53 – 1.17 (m, 7H), 0.92 (t, *J* = 6.9 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ (101 MHz, $CDCl_3$) δ 177.62, 152.84, 63.97, 63.86, 55.16, 53.74, 43.29, 35.04, 34.74, 34.57, 34.35, 27.37, 22.65, 14.15.
HRMS (ESI+) [$C_{14}H_{23}NNaO_4$+] m/z calculated 292.1519, found 292.1562



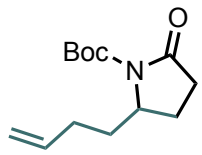
2:1 *dr*

3bt - methyl 5-oxo-2,3-dipropylpyrrolidine-1-carboxylate
Prepared using standard reaction conditions C from **1b** and **2t** followed by addition of 5 equiv. K_3PO_4 (1.5 mmol) and stirring for 3 hours.
Yield 48% 2:1 *dr (viscous oil)*
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.26 (q, *J* = 6.4 Hz, 1H), 3.86 (s, 1H), 3.85 (s, 3H), 2.86 (dd, *J* = 18.0, 8.5 Hz, 0H), 2.78 (dd, *J* = 17.9, 8.5 Hz, 1H), 2.55 – 2.36 (m, 2H), 2.35 – 2.13 (m, 2H), 1.84 – 1.61 (m, 1H), 1.53 – 1.25 (m, 13H), 1.02 – 0.81 (m, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 173.85, 152.71, 63.79, 62.91, 60.58, 53.63, 53.59, 38.37, 38.02, 38.00, 37.44, 37.34, 37.23, 35.79, 35.76, 34.86, 34.54, 32.03, 31.73, 21.24, 20.21, 20.15, 19.68, 18.99, 14.51, 14.31, 14.16, 14.10.
HRMS (ESI+) [$C_{12}H_{21}NNaO_3$+] m/z calculated 250.1414, found 250.1475

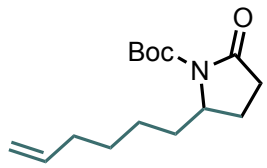


2:1 *dr*

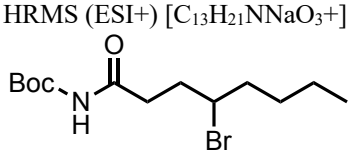
3bu - methyl 2,3-diethyl-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions conditions C from **1b** and **2t** followed by addition of 5 equiv. K_3PO_4 (1.5 mmol) and stirring for 3 hours.
Yield 55% 2:1 *dr (viscous oil)*
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.24 (dt, *J* = 7.2, 5.8 Hz, 1H), 3.86 (s, 4H), 3.85 (s, 3H), 3.81 (ddd, *J* = 9.1, 3.4, 1.3 Hz, 1H), 2.77 (dd, *J* = 18.0, 8.6 Hz, 1H), 2.55 – 2.44 (m, 1H), 2.40 – 2.11 (m, 3H), 1.97 – 1.87 (m, 1H), 1.88 – 1.71 (m, 2H), 1.65 – 1.32 (m, 8H), 1.09 – 0.82 (m, 13H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 173.87, 173.81, 152.71, 152.65, 64.82, 61.56, 53.64, 53.60, 39.09, 37.95, 37.81, 36.16, 28.06, 26.41, 22.53, 22.45, 12.57, 11.49, 10.84, 9.82.
HRMS (ESI+) [$C_{10}H_{17}NNaO_3$+] m/z calculated 222.1101, found 222.1133



3aw - *tert*-butyl 2-(but-3-en-1-yl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2w**.
Yield 67% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.81 (ddt, *J* = 17.0, 10.1, 6.6 Hz, 1H), 5.11 – 4.97 (m, 2H), 4.13 (dddd, *J* = 9.9, 8.3, 3.2, 2.0 Hz, 1H), 2.58 (ddd, *J* = 17.7, 11.0, 9.1 Hz, 1H), 2.43 (ddd, *J* = 17.7, 9.5, 2.7 Hz, 1H), 2.22 – 1.99 (m, 2H), 1.90 (dddt, *J* = 12.9, 9.8, 7.1, 3.1 Hz, 1H), 1.78 (ddt, *J* = 12.8, 9.1, 2.3 Hz, 1H), 1.53 (s, 11H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.48, 150.15, 137.40, 115.66, 82.99, 57.68, 32.91, 31.52, 30.12, 28.25, 22.50.
HRMS (ESI+) [$C_{14}H_{25}NNaO_3$+] m/z calculated 278.1727, found 278.1751



3ay - *tert*-butyl 2-(hex-5-en-1-yl)-5-oxopyrrolidine-1-carboxylate
Prepared using standard reaction conditions A from **1a** and **2y**.
Yield 40% (viscous oil)
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.79 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.10 – 4.84 (m, 2H), 4.15 – 4.05 (m, 1H), 2.57 (ddd, *J* = 17.7, 11.0, 9.1 Hz, 1H), 2.41 (ddd, *J* = 17.7, 9.5, 2.7 Hz, 1H), 2.17 – 2.02 (m, 3H), 1.77 (tdd, *J* = 12.2, 7.0, 3.1 Hz, 2H), 1.61 – 1.21 (m, 15H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 174.55, 150.22, 138.67, 114.89, 82.89, 58.19, 33.78, 33.74, 31.60, 28.83, 28.26, 25.25, 22.71.
HRMS (ESI+) [$C_{13}H_{21}NNaO_3$+] m/z calculated 262.1414, found 262.1443



3aa' - *tert*-butyl (4-bromooctanoyl)carbamate
Prepared using standard reaction conditions A from **1a** and **2a** without the addition of base and MeCN.
Yield 92% (viscous oil)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (s, 1H), 4.18 – 3.95 (m, 1H), 3.13 – 2.75 (m, 2H), 2.23 (dddd, *J* = 15.1, 8.2, 7.0, 3.5 Hz, 1H), 2.05 (dddd, *J* = 15.1, 9.6, 7.9, 5.8 Hz, 1H), 1.93 – 1.76 (m, 2H), 1.49 (s, 13H), 0.91 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 173.91, 150.54, 82.83, 57.66, 39.25, 34.51, 33.43, 29.88, 28.19, 22.29, 14.12.
LRMS (EI) [$C_{13}H_{23}NO_3$] m/z [M-HBr] calculated 241.2, found 241.1

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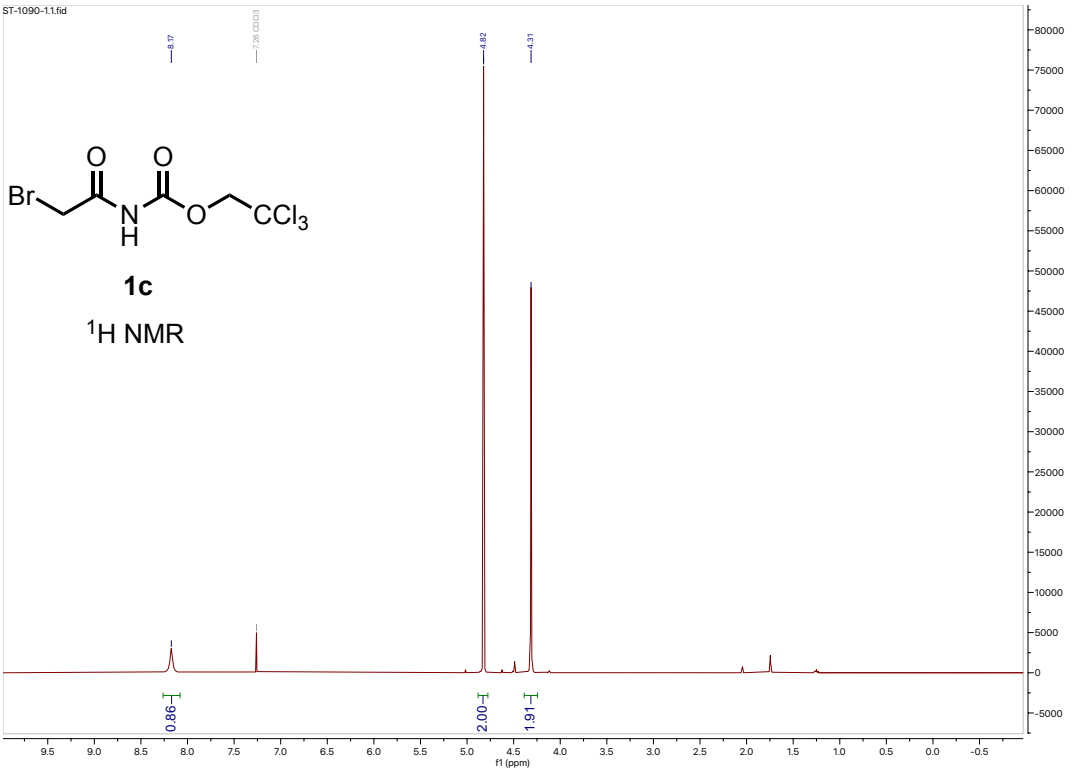
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CCl3
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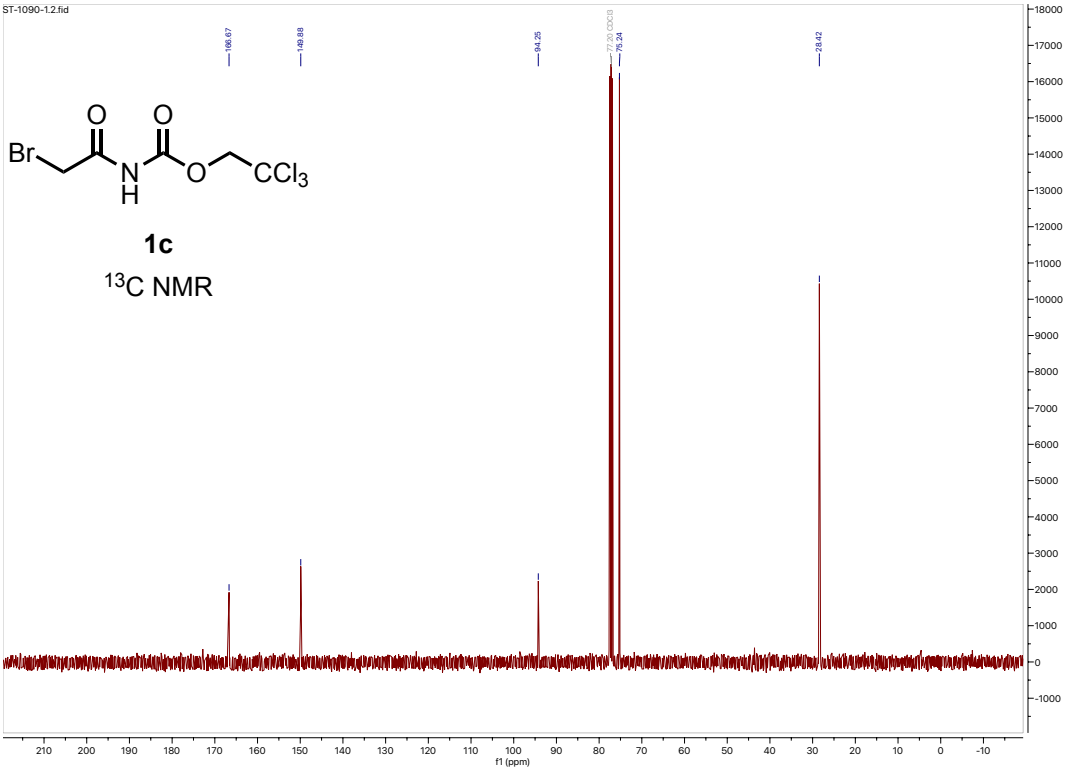
1H NMR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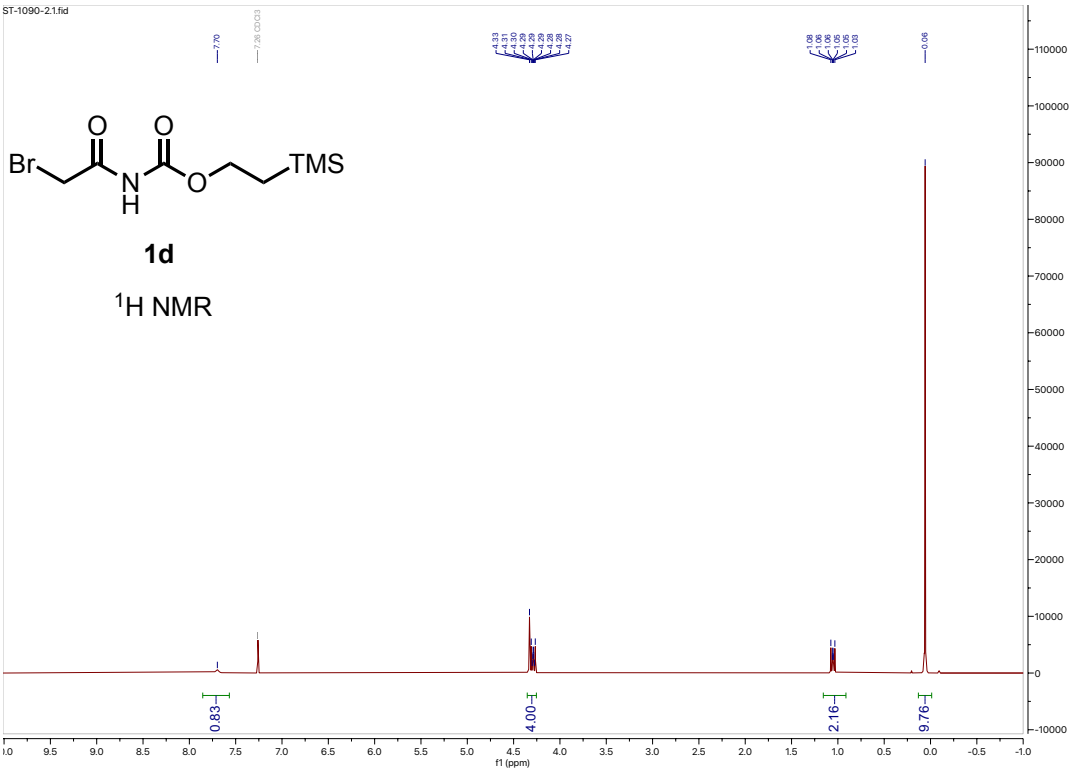
13C NMR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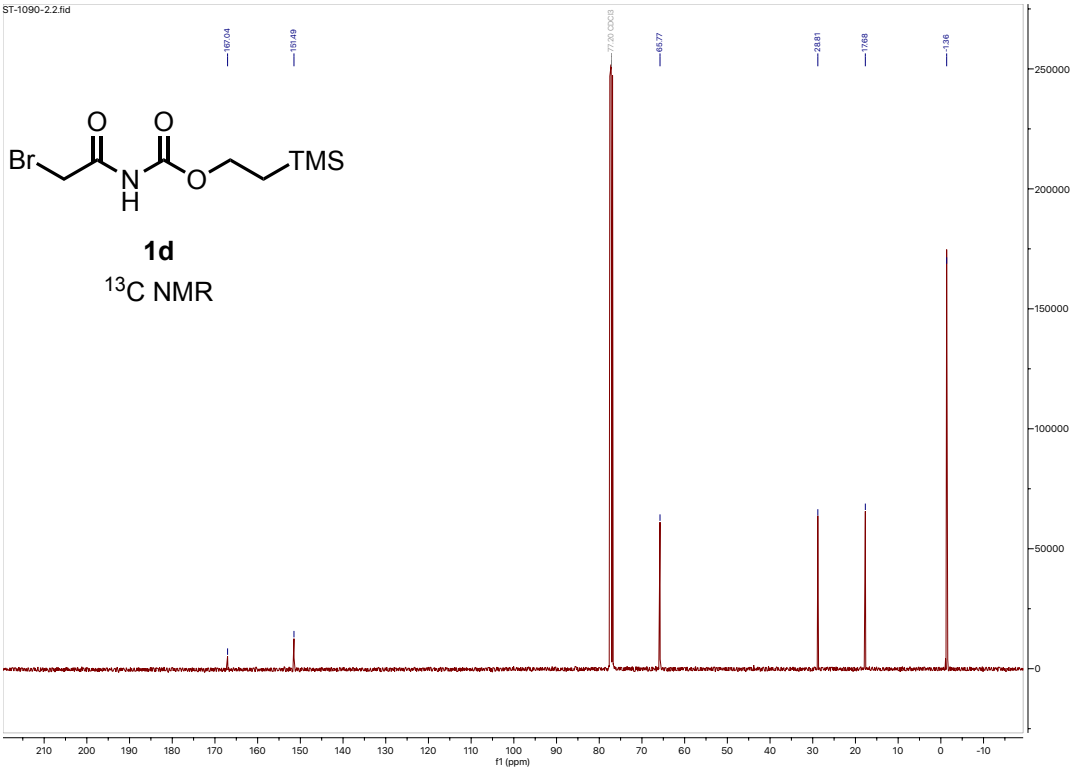
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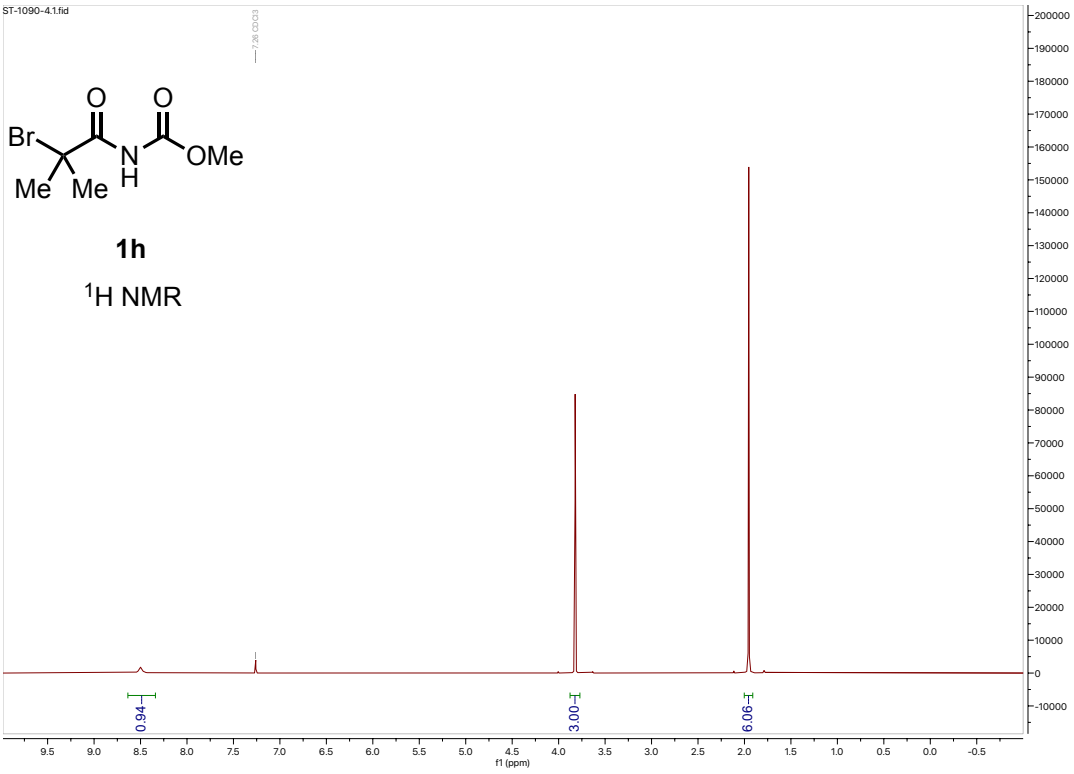
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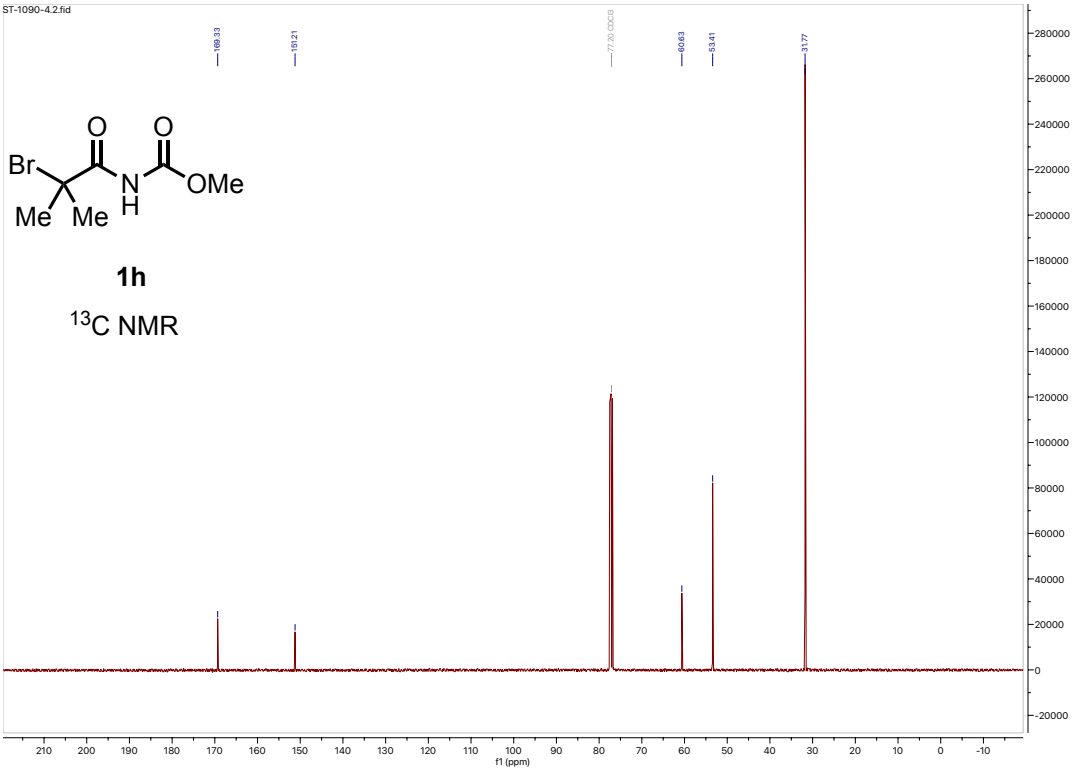
f1 (ppm)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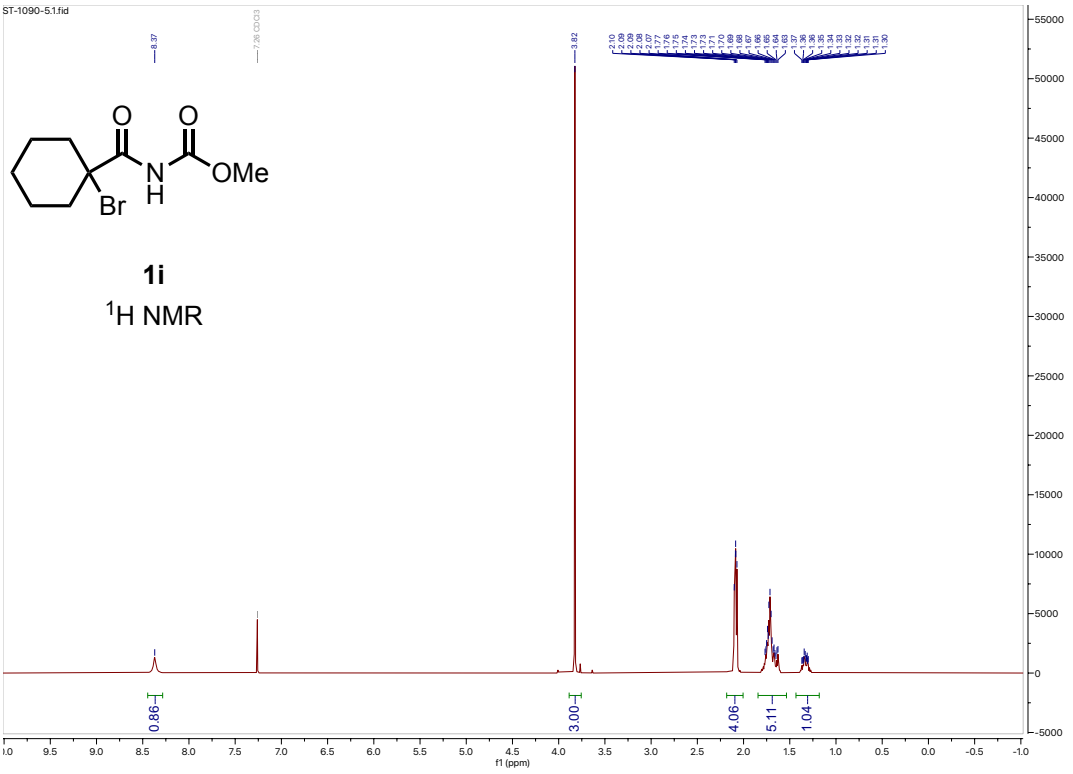
f1 (ppm)



1i

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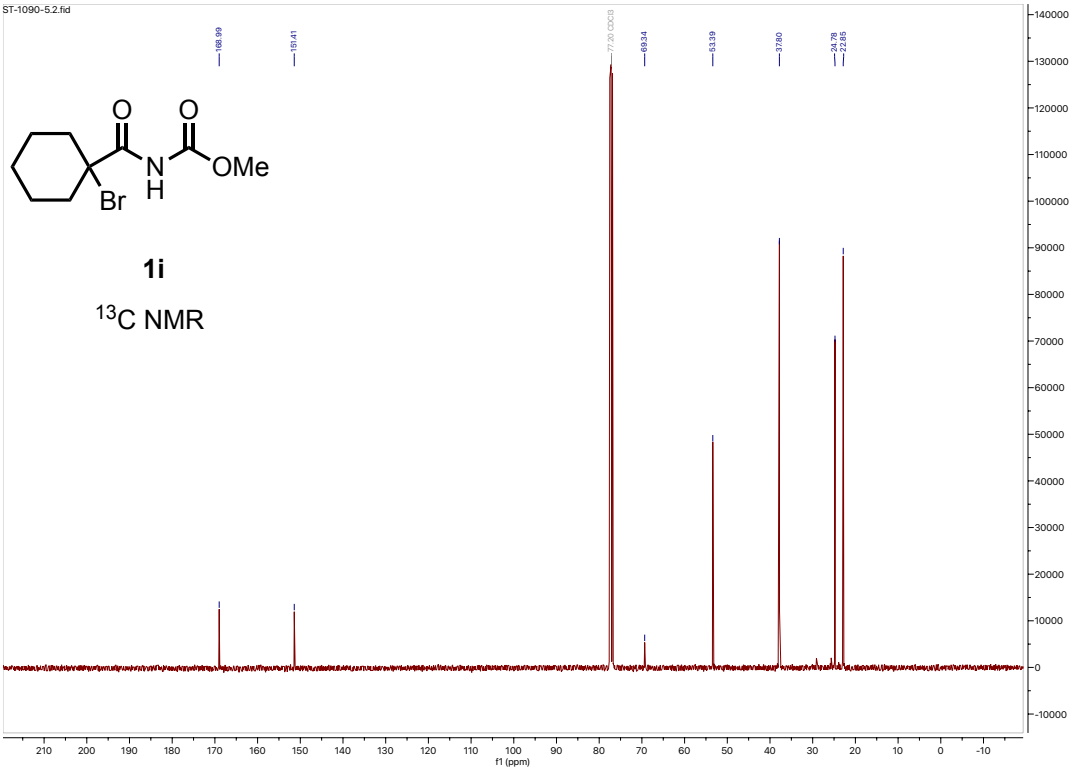
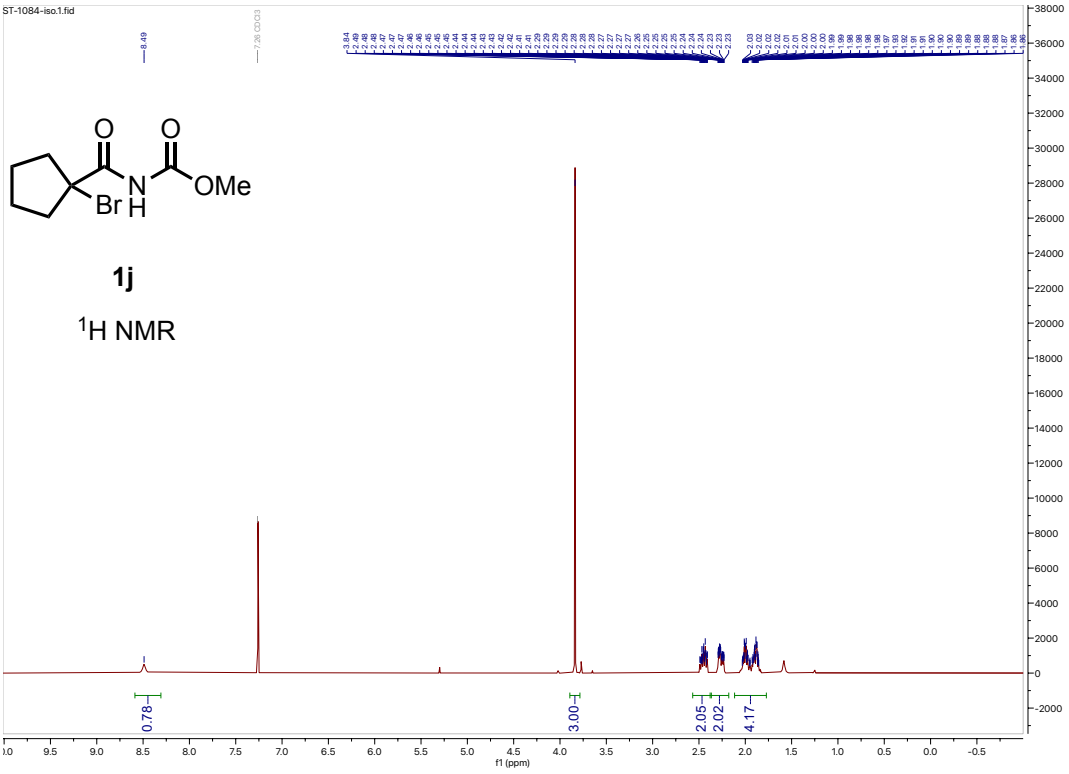


1i

^{13}C NMR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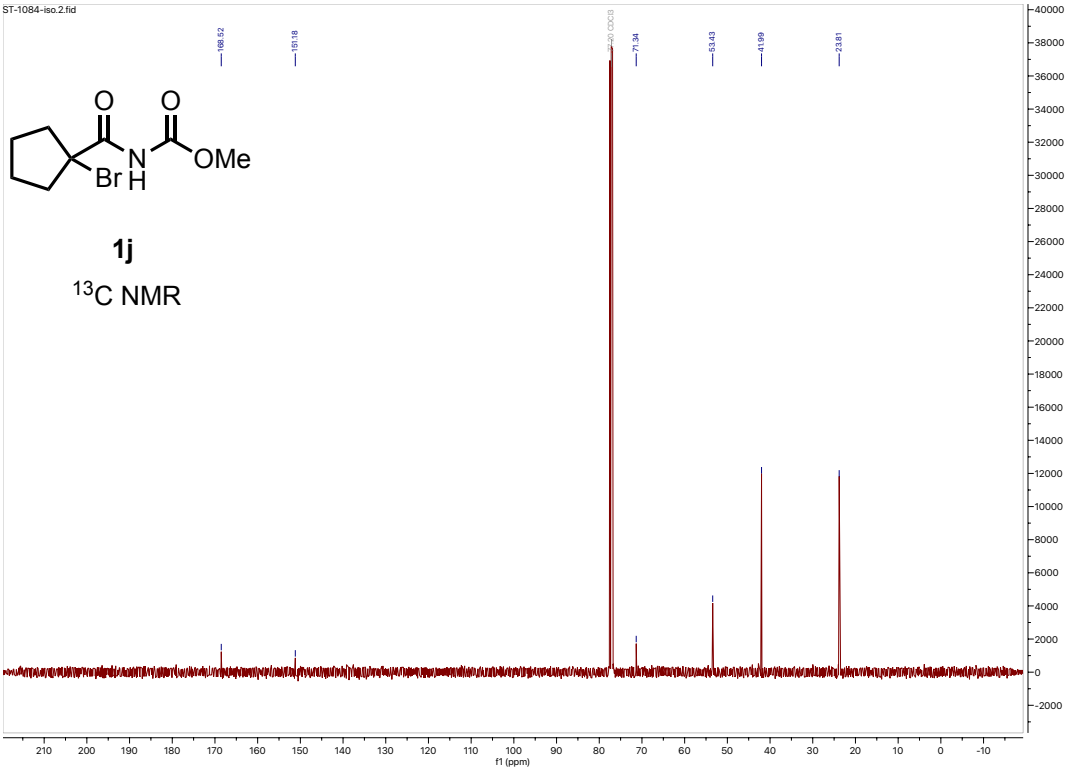
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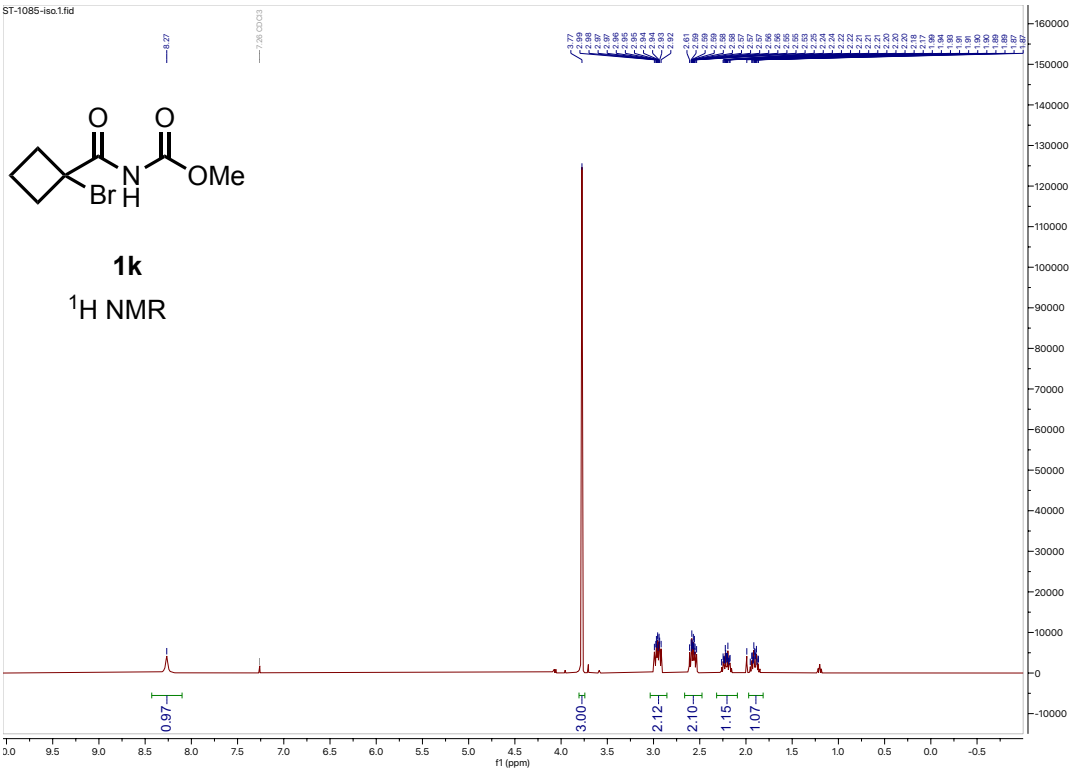
f1 (ppm)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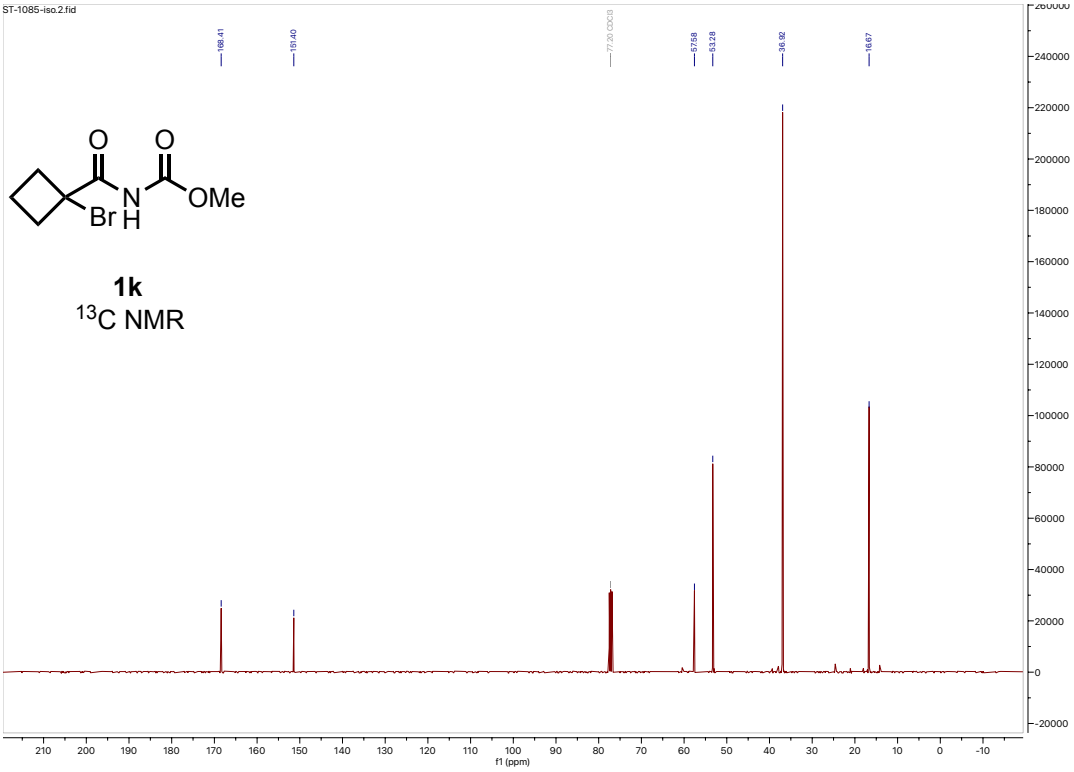
f1 (ppm)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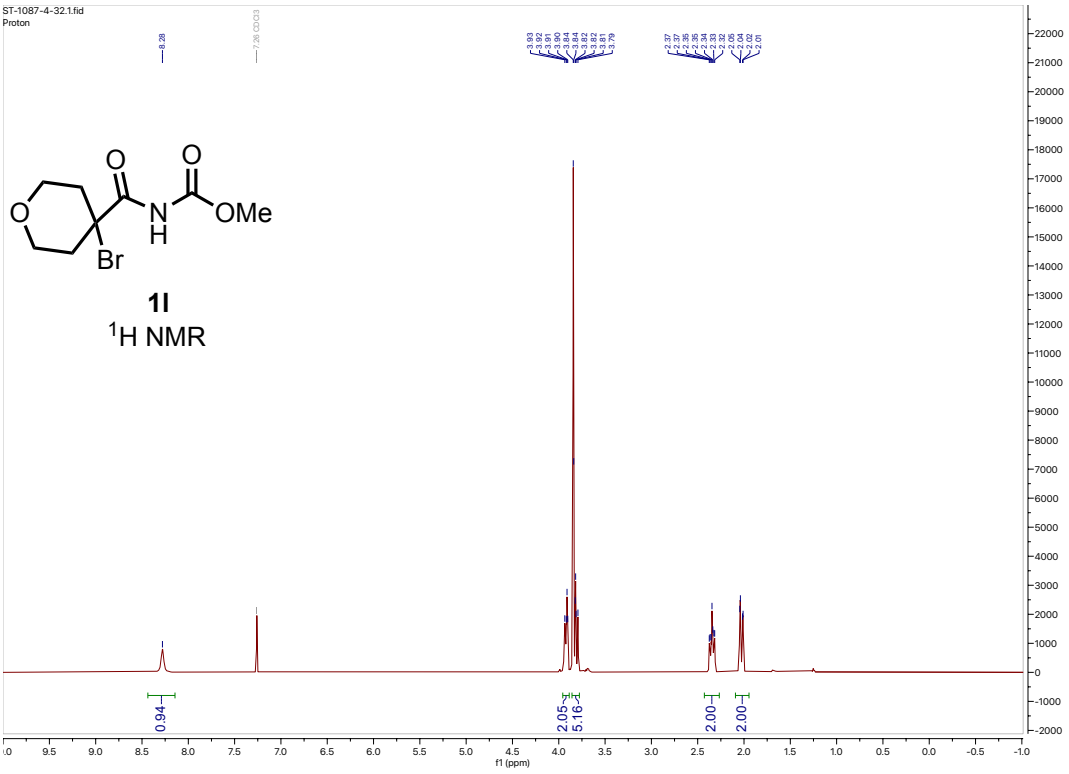

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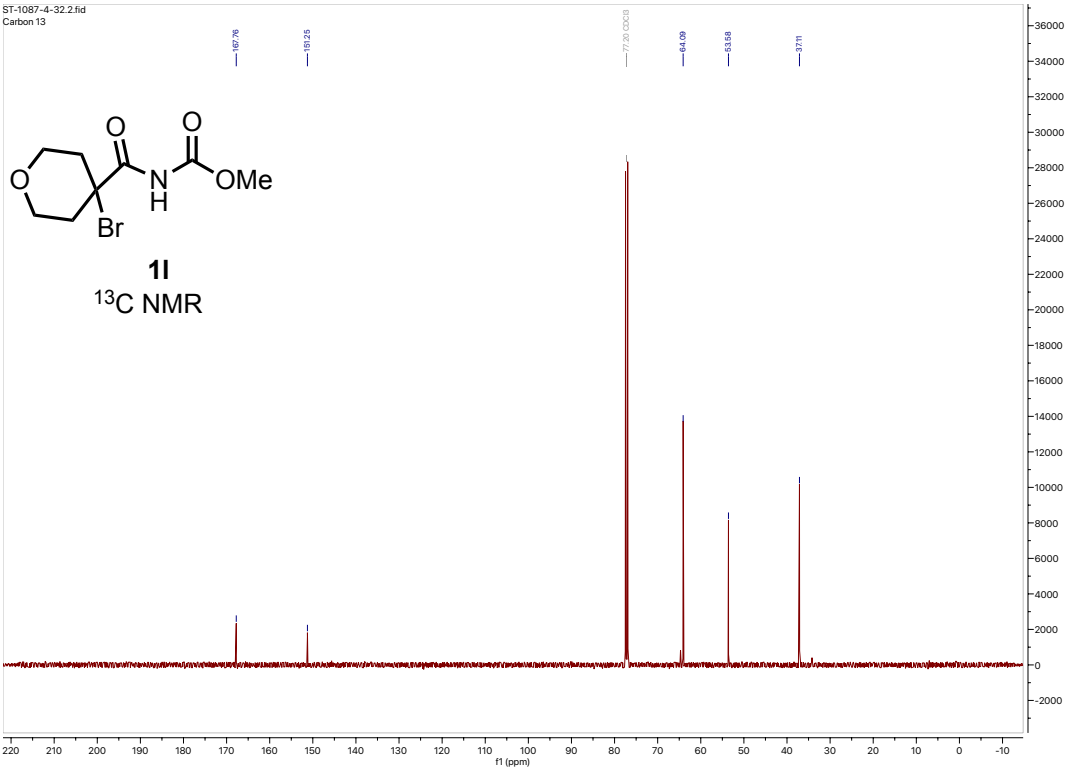
^{1}H NMR



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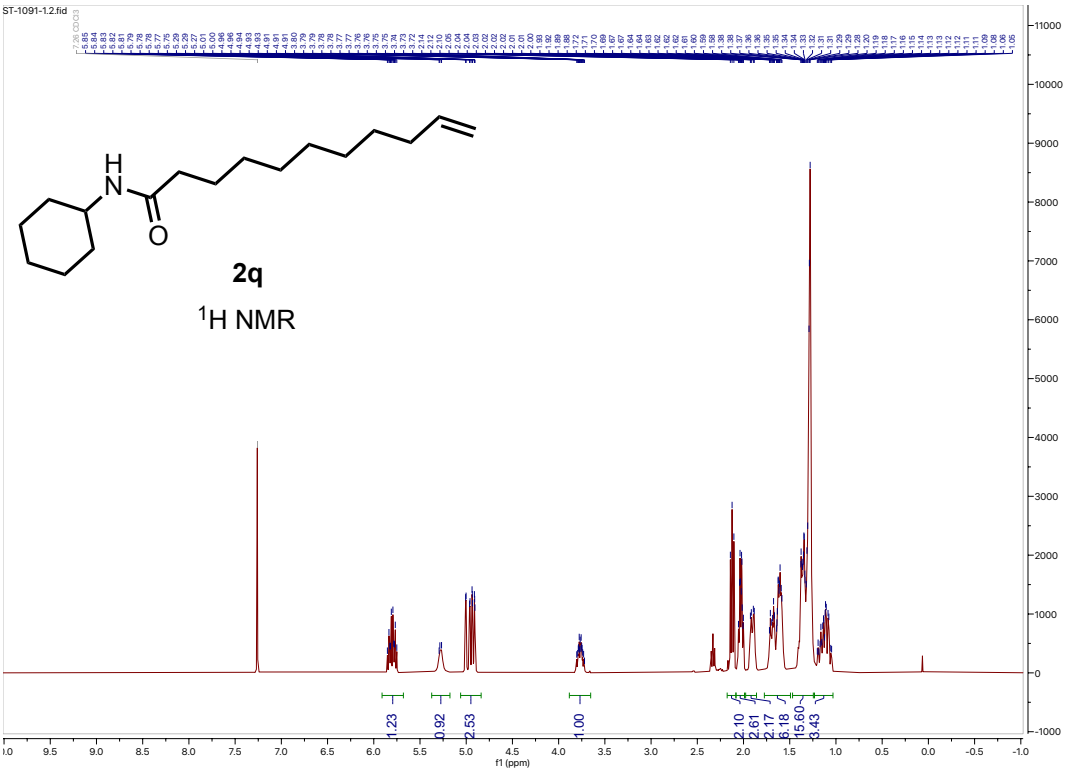
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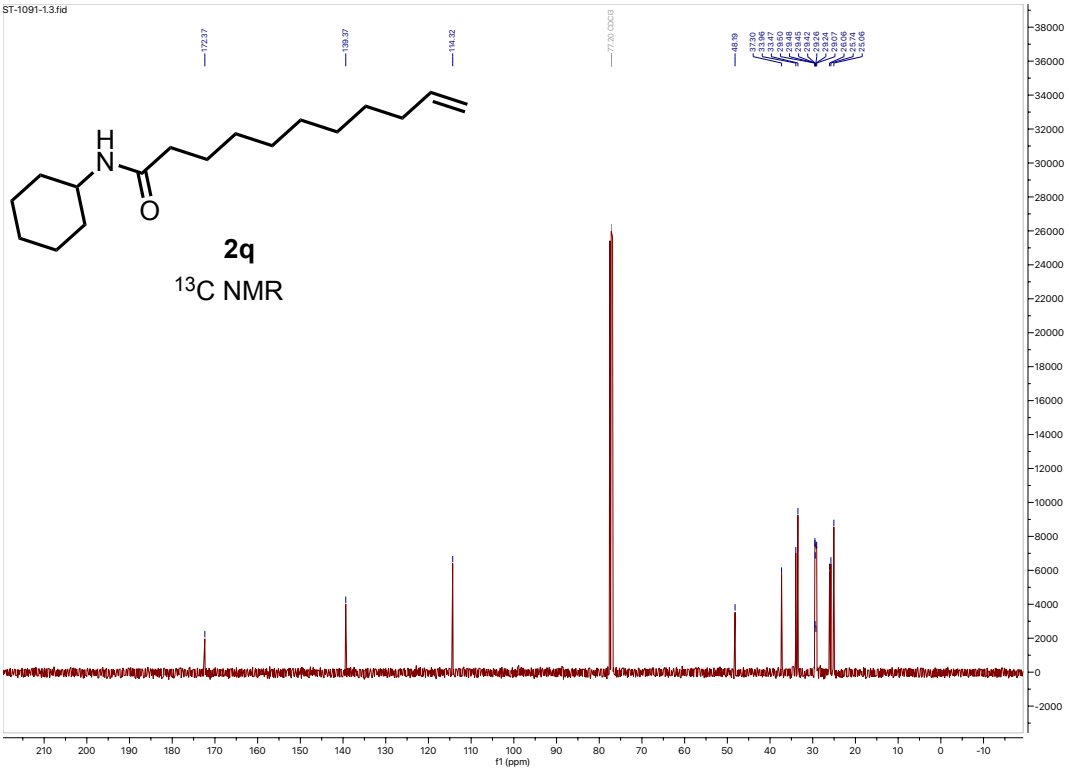
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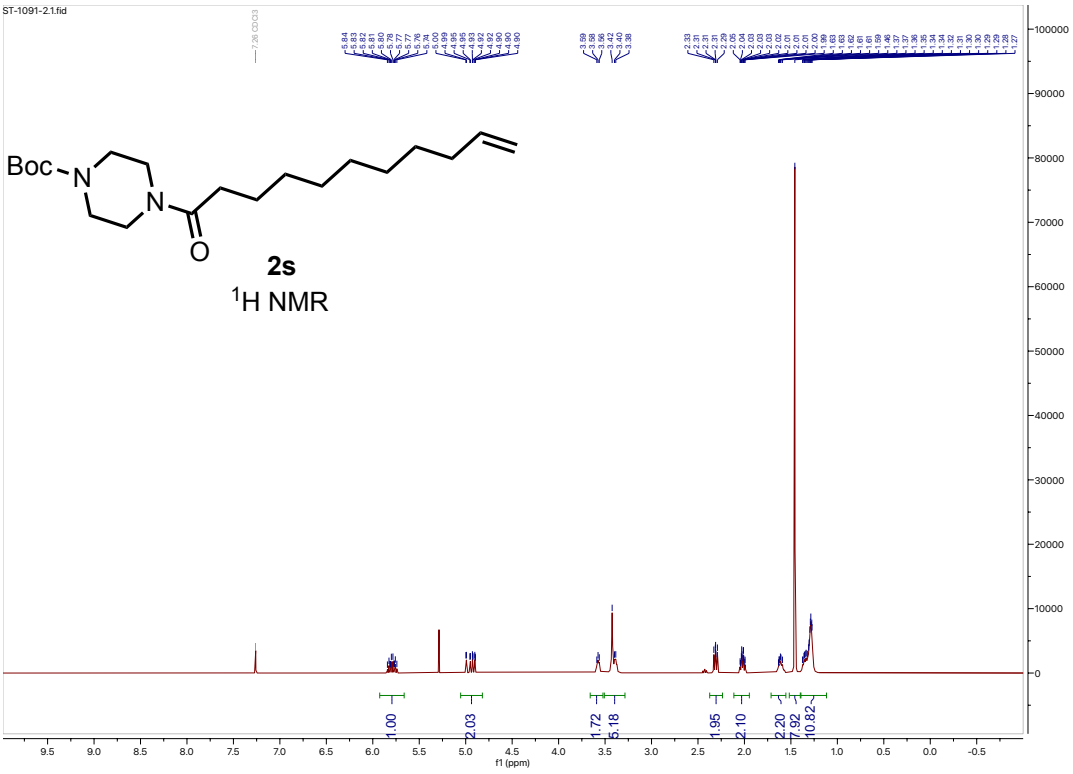
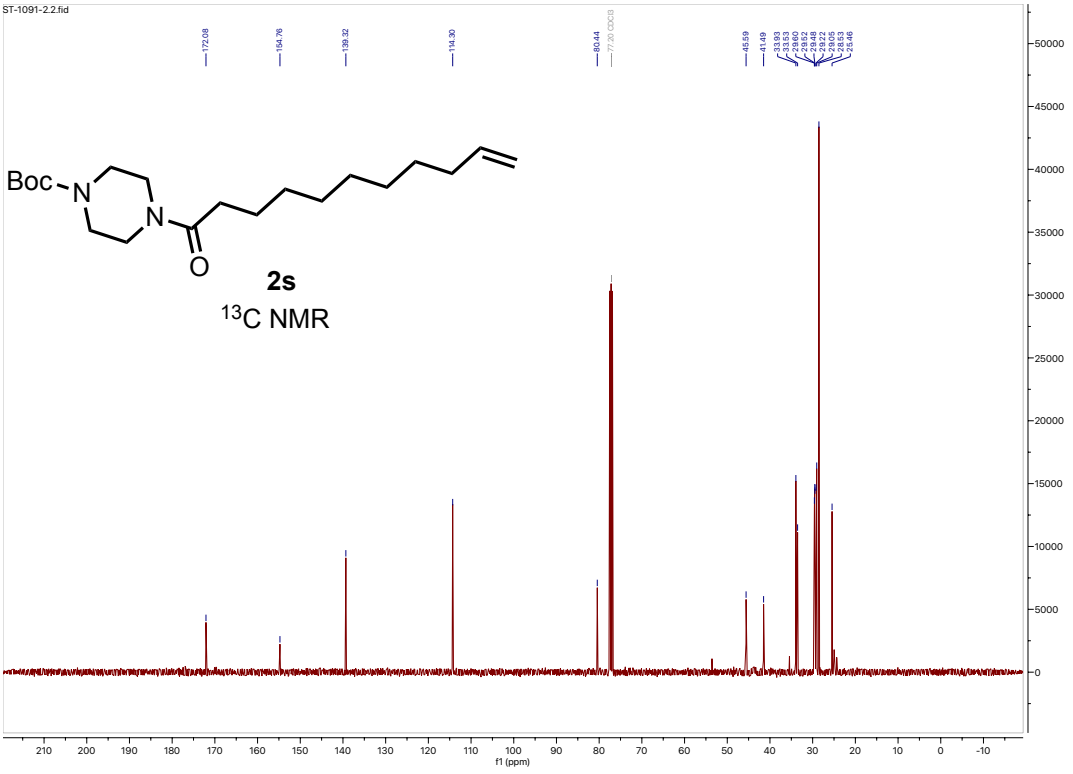
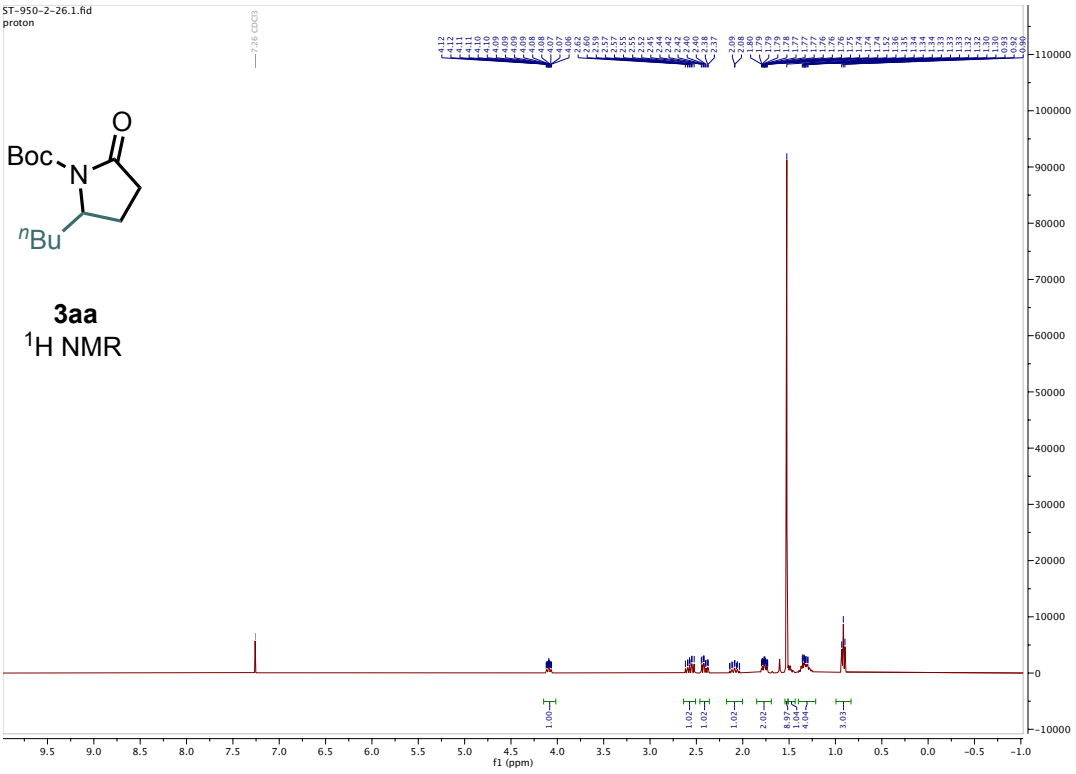
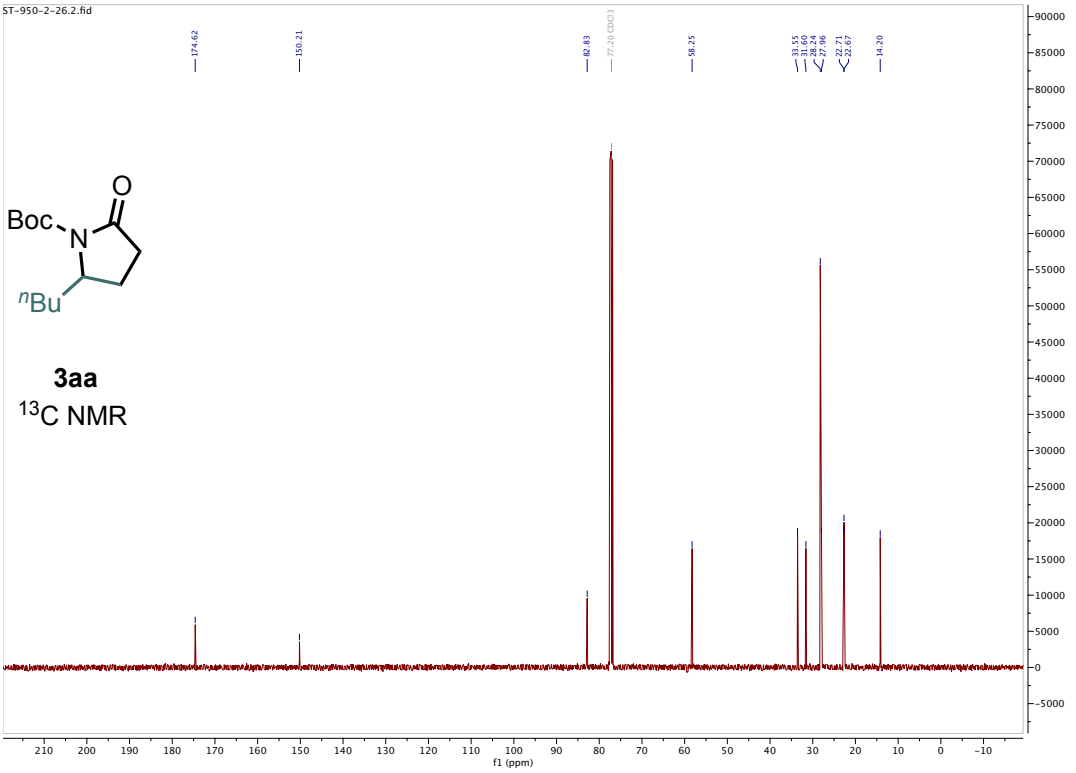
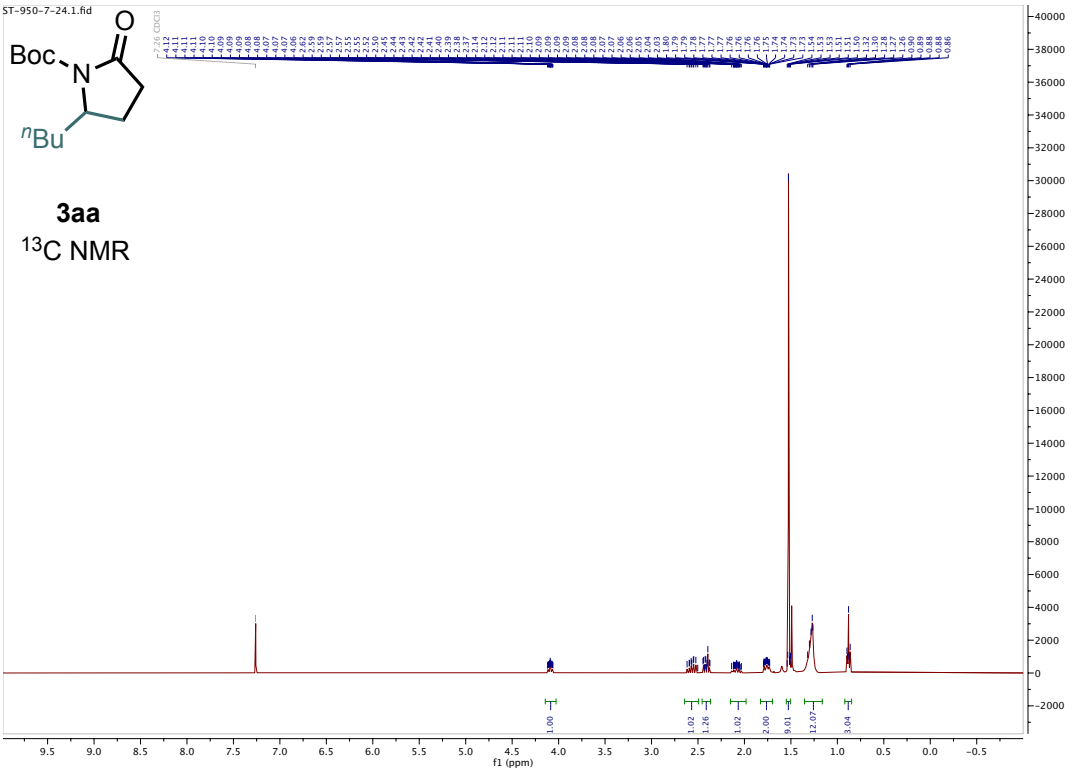
13C NMR
f1 (ppm)

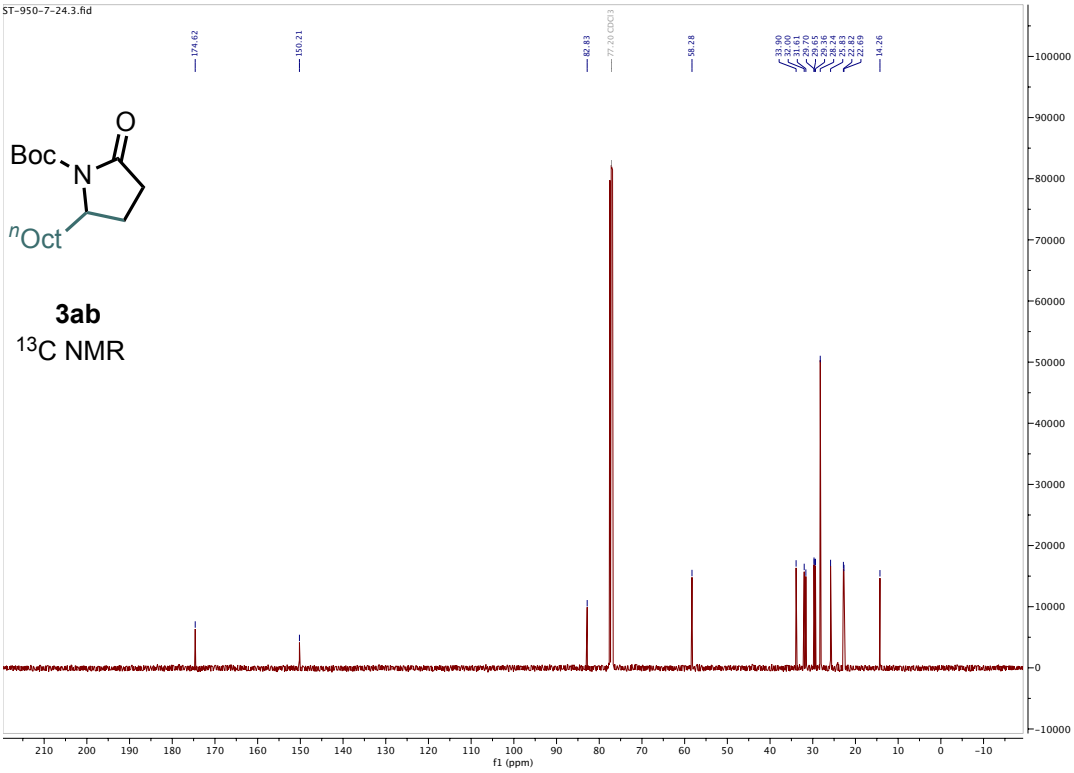

ST-1091-2.1.fid
Boc
N
N
O
2s
1H NMR
1.00
2.03
1.72
5.18
1.95
2.10
2.20
7.92
10.82
f1 (ppm)

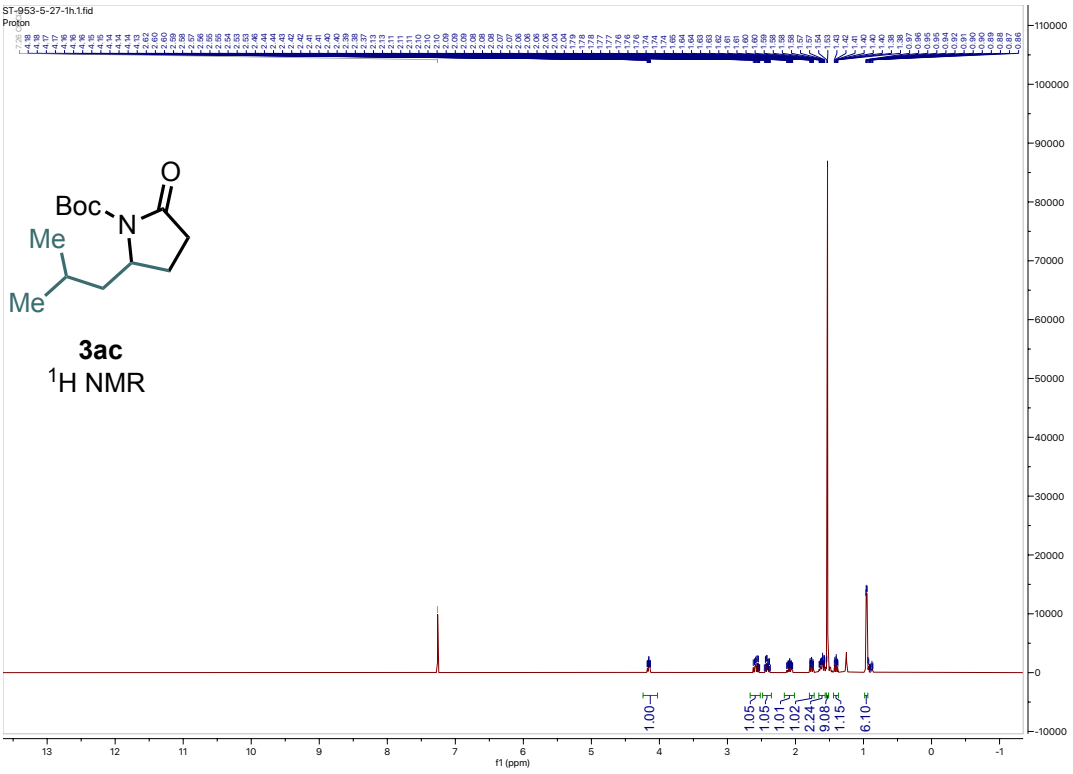

ST-1091-2.2.fid
Boc
N
N
O
2s
13C NMR
f1 (ppm)


ST-950-2-26.1.fid
proton
Boc
N
O
nBu
3aa
1H NMR
f1 (ppm)


ST-950-2-26.2.fid
174.62
150.21
82.83
77.20 CDCl3
58.25
31.55
31.40
28.24
27.96
22.71
22.67
14.20
Boc
N
O
nBu
3aa
13C NMR
f1 (ppm)


ST-950-7-24.1.fid
Boc
N
O
nBu
3aa
13C NMR
f1 (ppm)


ST-950-7-24.3.fid
174.62
150.21
82.83
77.20 CDCl3
58.28
14.26
Boc
N
O
nOct
3ab
13C NMR
f1 (ppm)


ST-953-5-27-1h.1.fid
Proton
Boc
N
O
Me
Me
3ac
1H NMR
1.00
1.05
1.05
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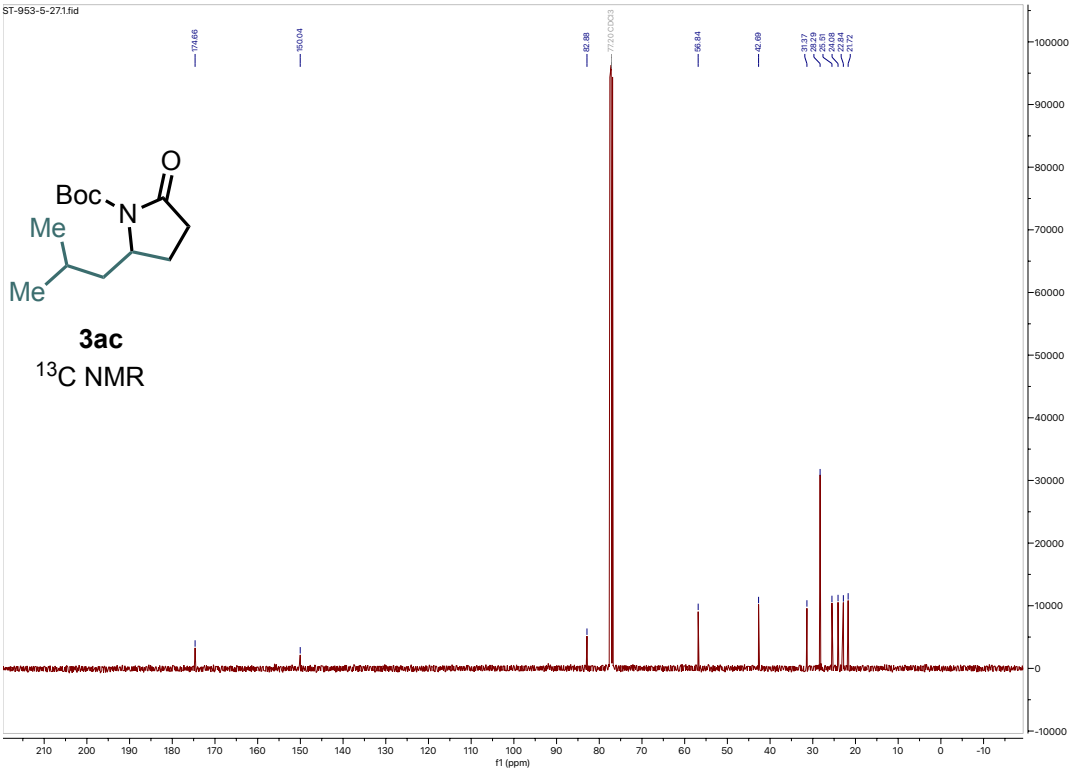
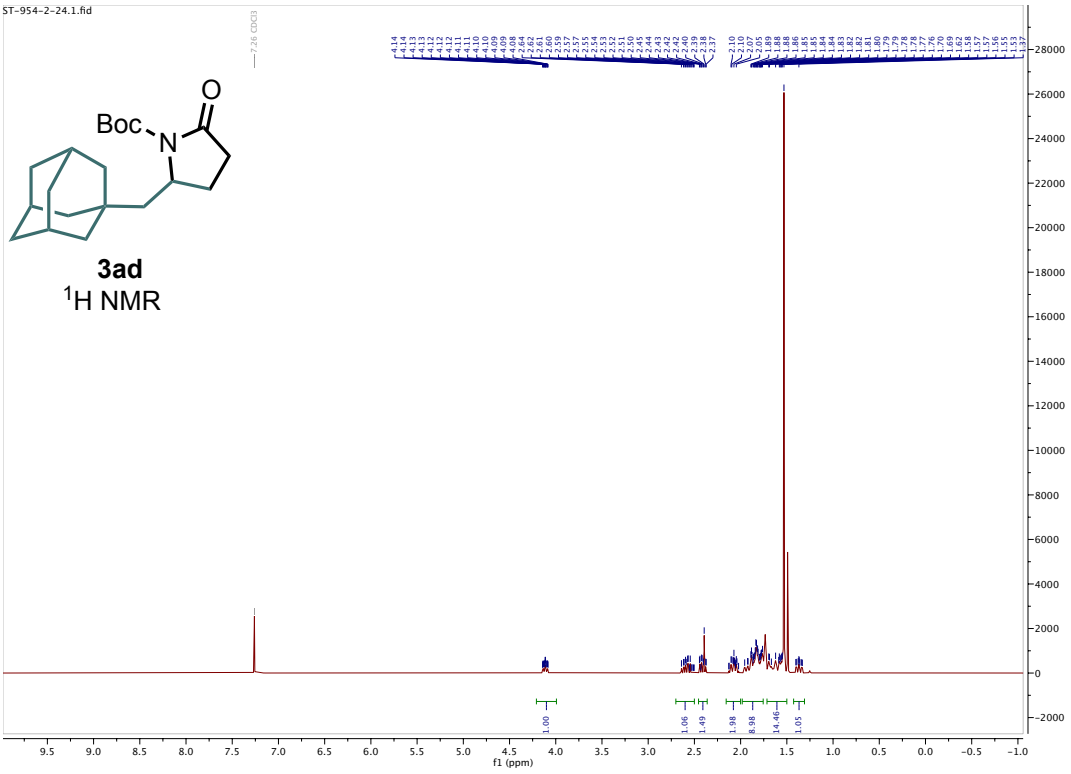
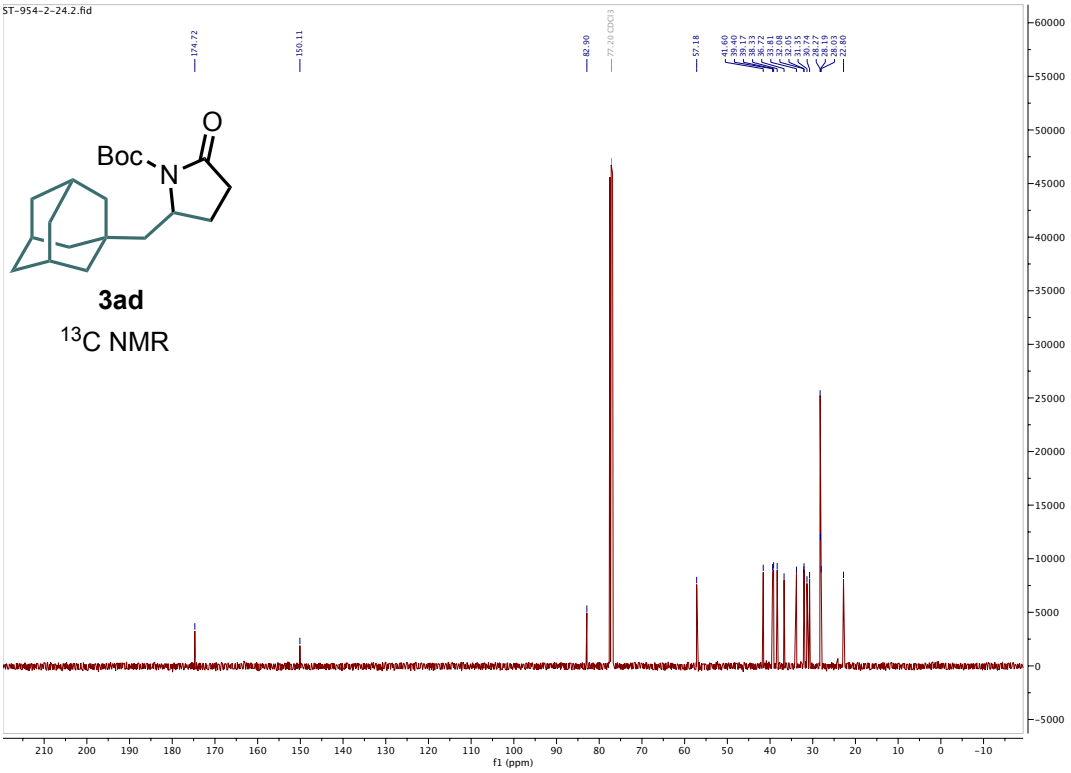
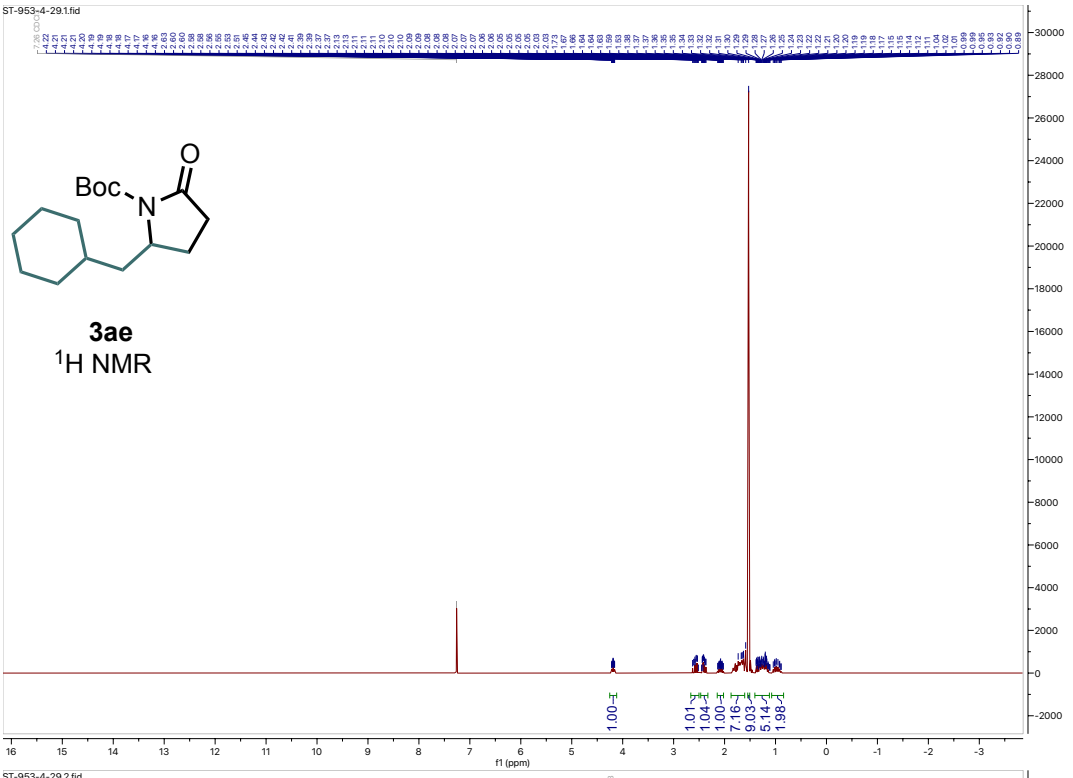
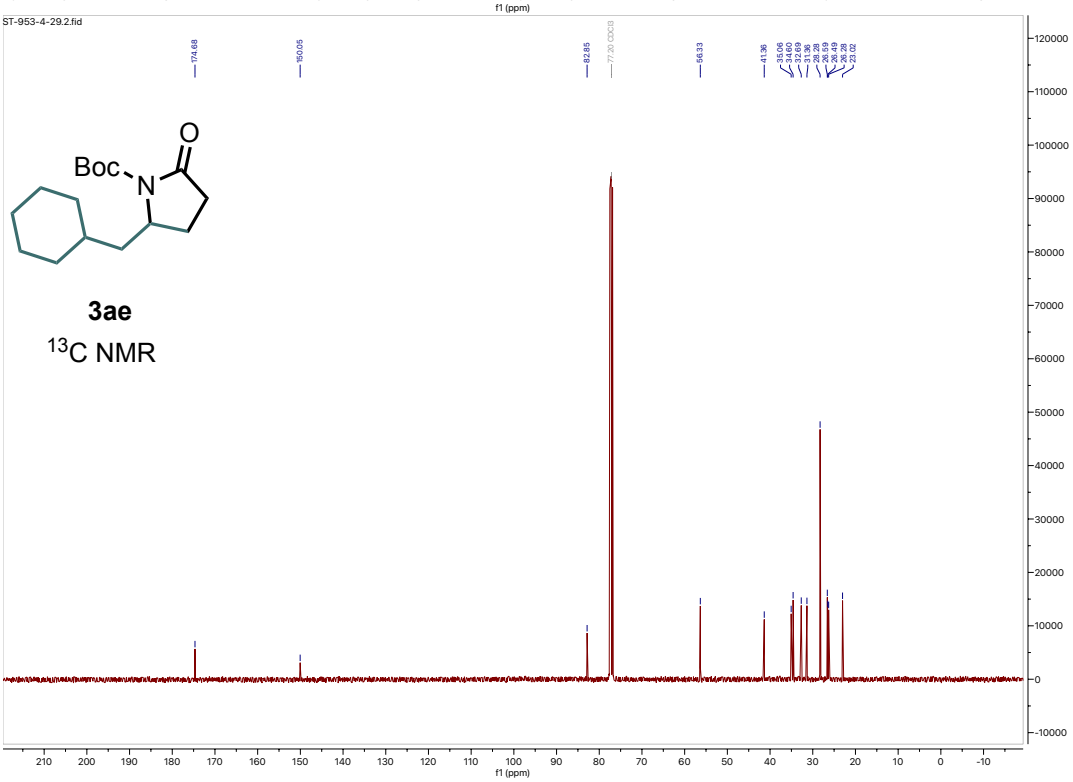
1.02
2.24
9.08
1.15
6.10
f1 (ppm)

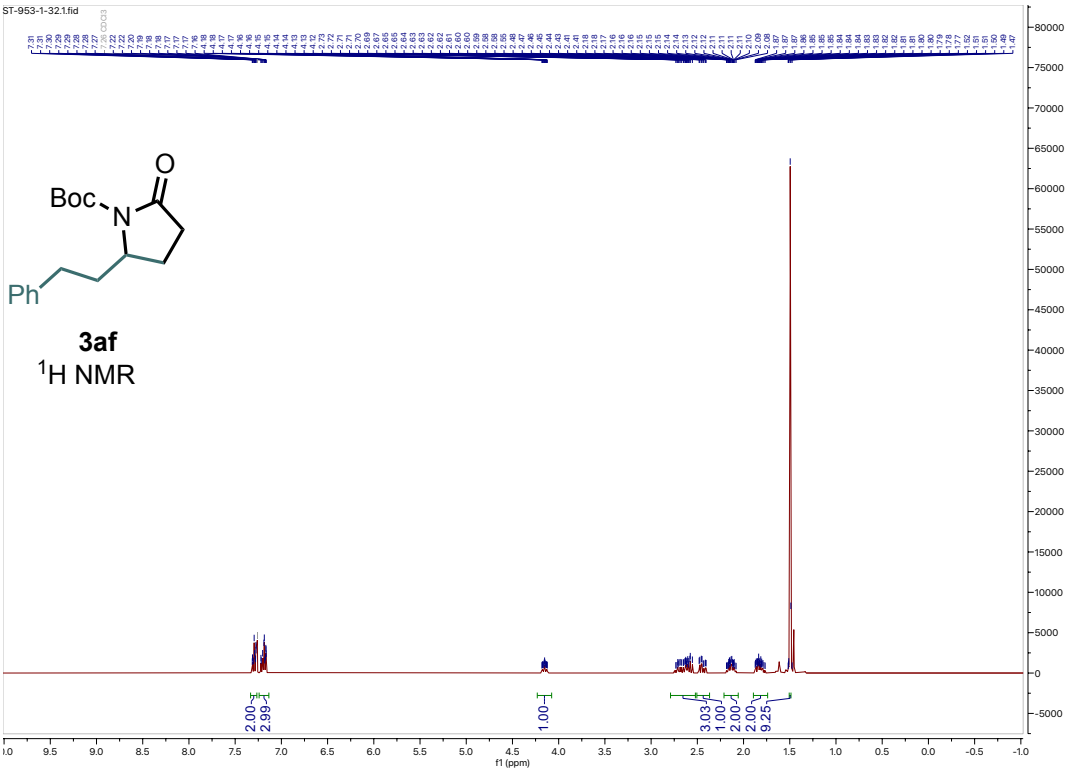

ST-953-5-27.1.fid
174.66
150.04
82.88
77.20 CDCl3
56.84
42.69
31.37
28.29
26.31
24.06
22.84
21.72
Boc
N
O
Me
Me
3ac
13C NMR
f1 (ppm)

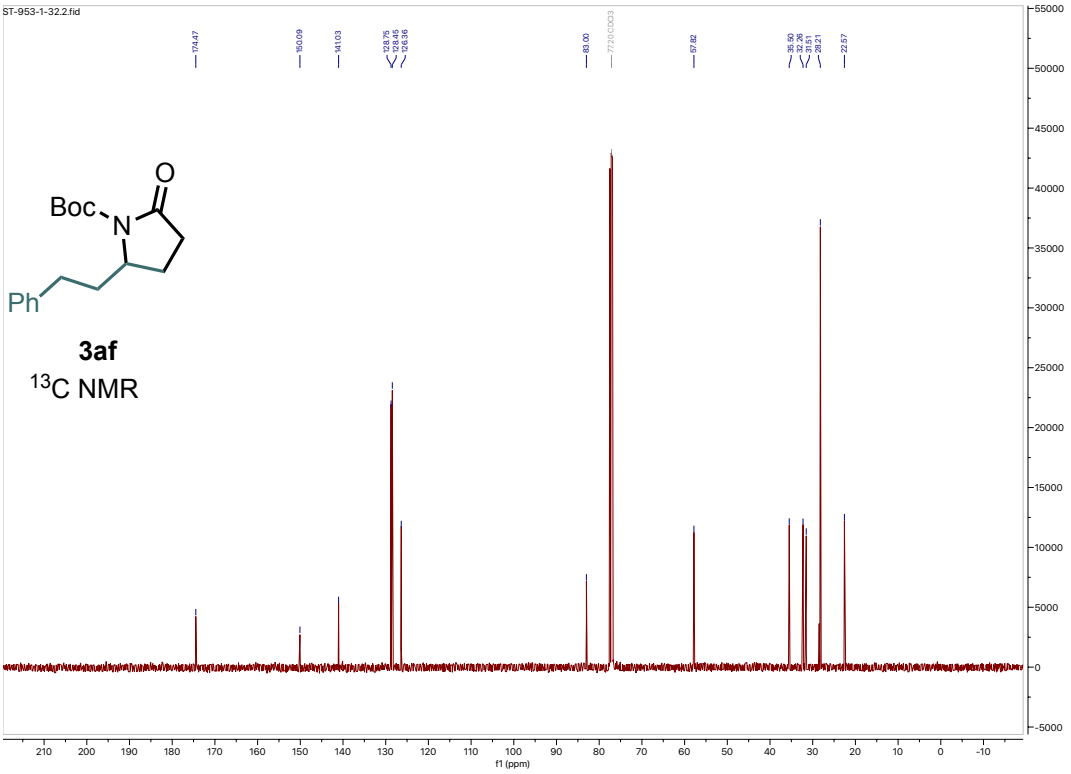

ST-954-2-24.1.fid
Boc
N
O
3ad
1H NMR
f1 (ppm)

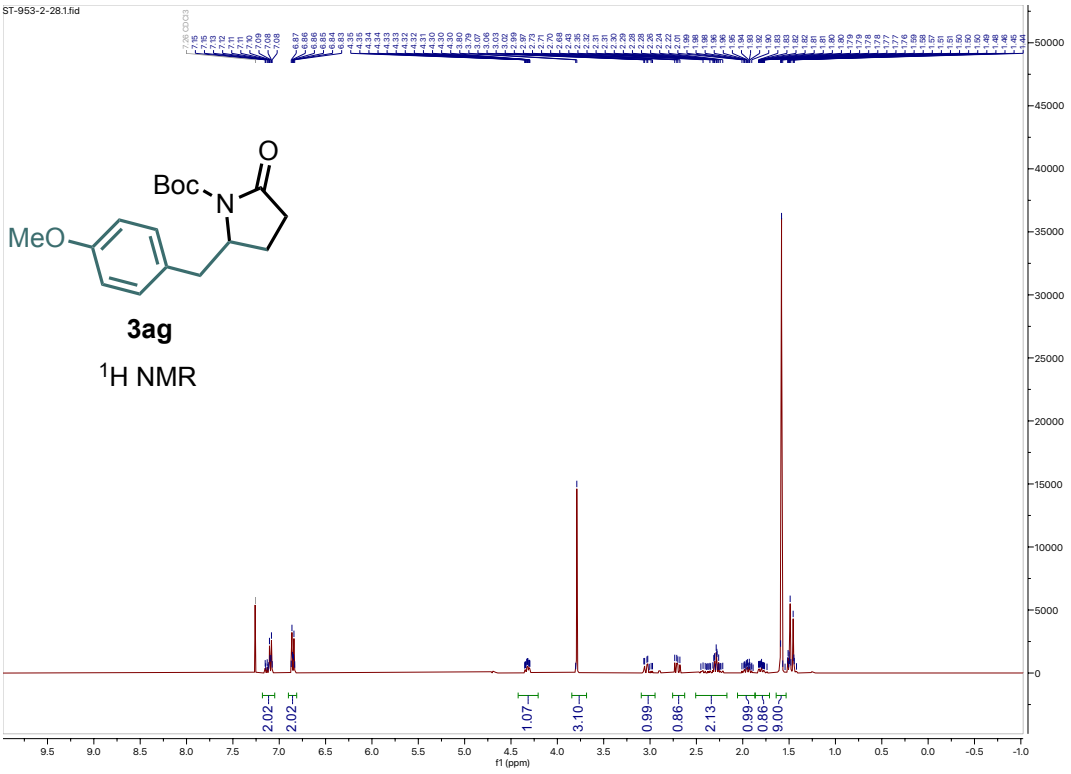

ST-954-2-24.2.fid
Boc
N
O
3ad
13C NMR
f1 (ppm)

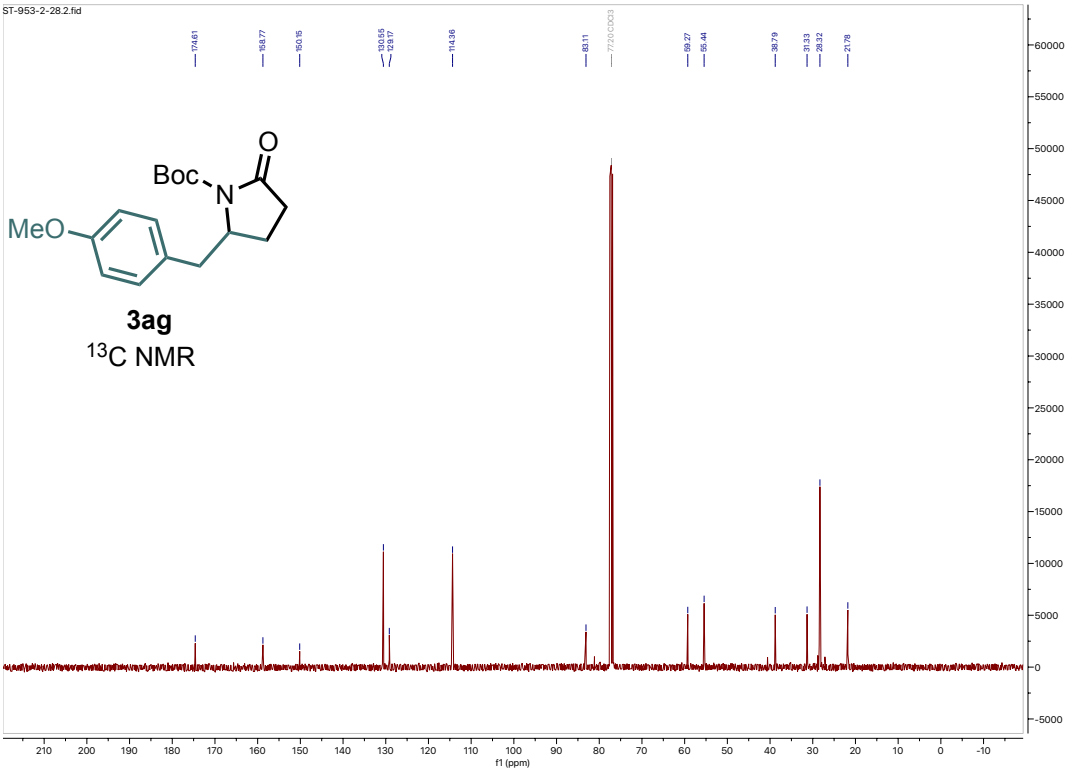

ST-953-4-29.1.fid
Boc
N
O
3ae
1H NMR
1.00
1.01
1.04
1.00
7.16
9.03
5.14
1.98
f1 (ppm)

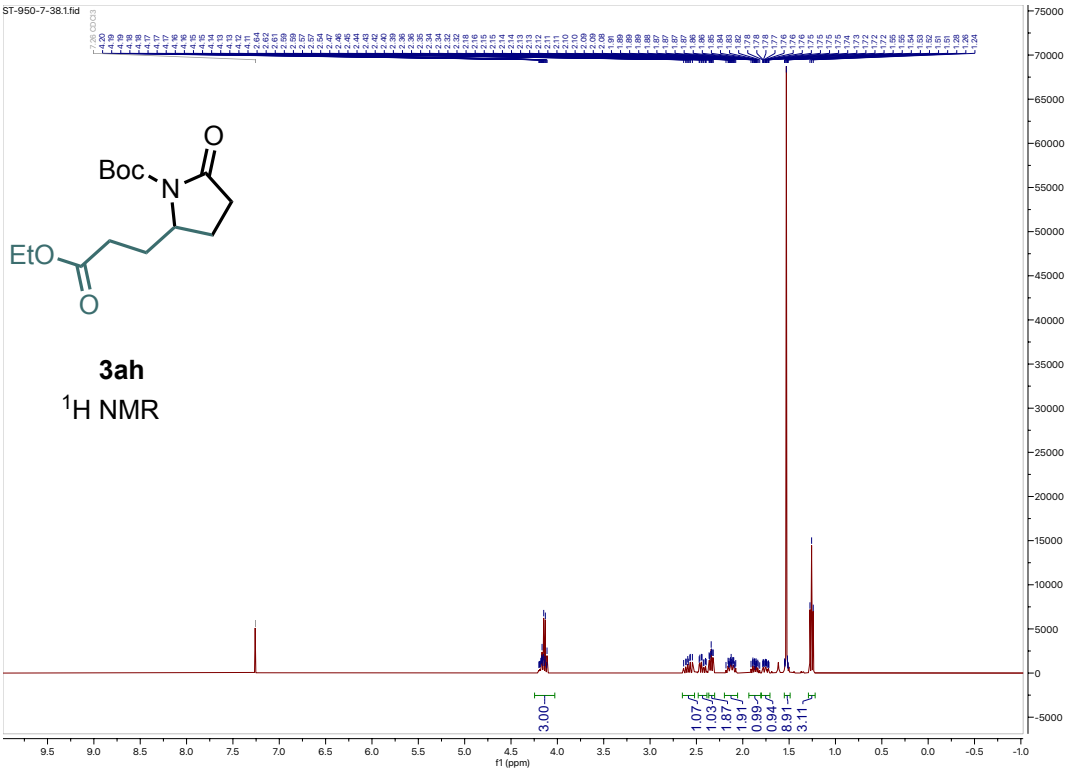

ST-953-4-29.2.fid
174.68
150.05
82.85
77.20 CDCl3
56.33
41.36
35.06
34.60
32.69
31.36
29.28
26.59
26.49
26.39
23.02
Boc
N
O
3ae
13C NMR
f1 (ppm)

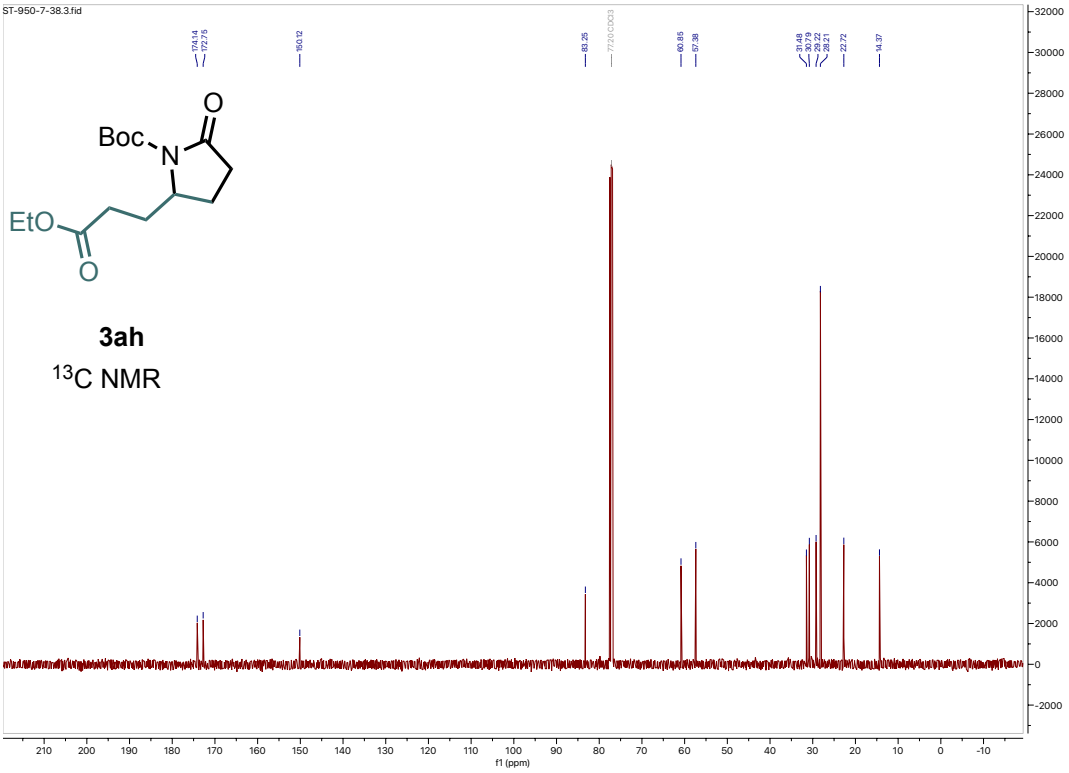

ST-953-1-32.1.fid
Boc
N
O
Ph
3af
1H NMR
f1 (ppm)


ST-953-1-32.2.fid
Boc
N
O
Ph
3af
13C NMR
f1 (ppm)


ST-953-2-28.1.fid
Boc
N
O
MeO
3ag
1H NMR
2.02
2.02
1.07
3.10
0.99
0.86
2.13
0.99
0.86
9.00
f1 (ppm)


ST-953-2-28.2.fid
174.61
158.77
150.15
130.65
129.17
114.36
83.11
77.20 CDCl3
59.27
55.44
38.79
31.33
28.32
21.78
Boc
N
O
MeO
3ag
13C NMR
f1 (ppm)


ST-950-7-38.1.fid
Boc
N
O
EtO
O
3ah
1H NMR
f1 (ppm)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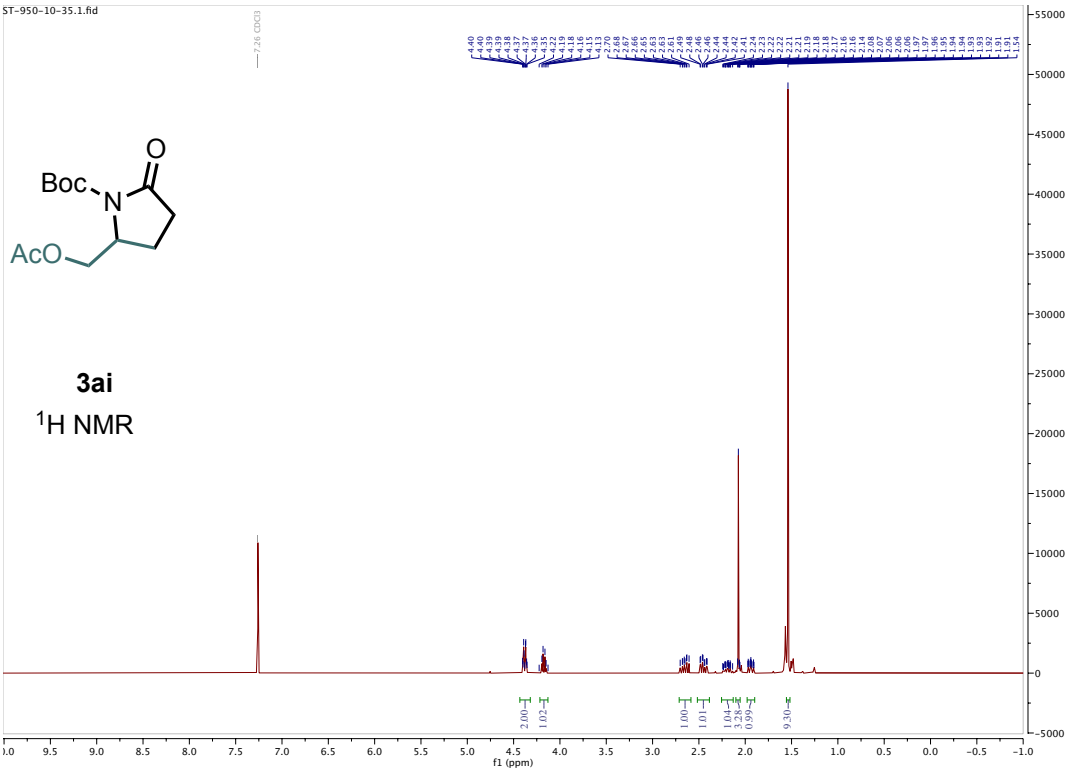
Boc
N
O
EtO
O
3ah
13C NMR
f1 (ppm)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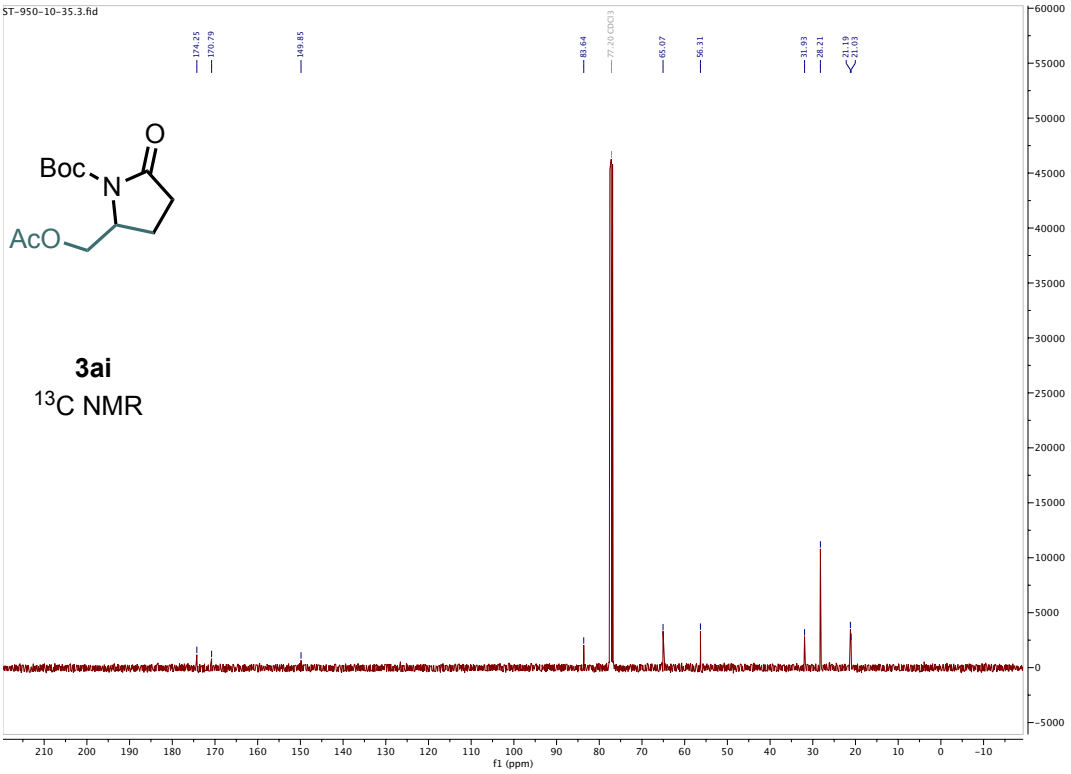
Boc
N
O
AcO
3ai
1H NMR
f1 (ppm)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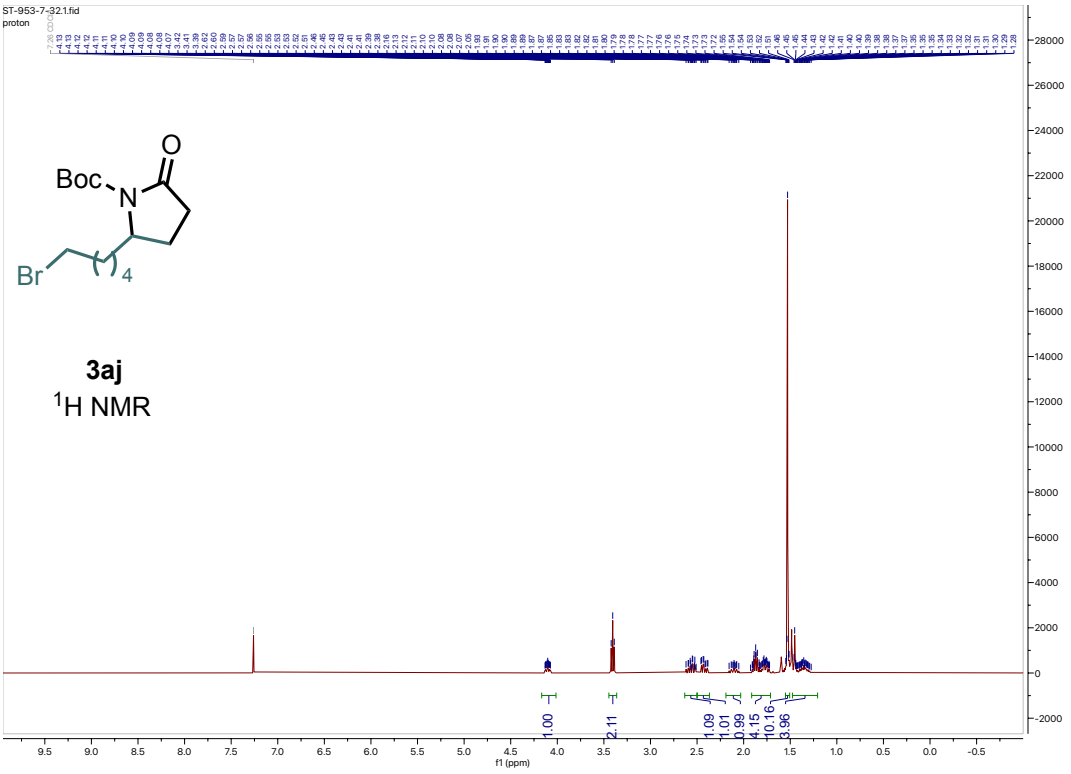
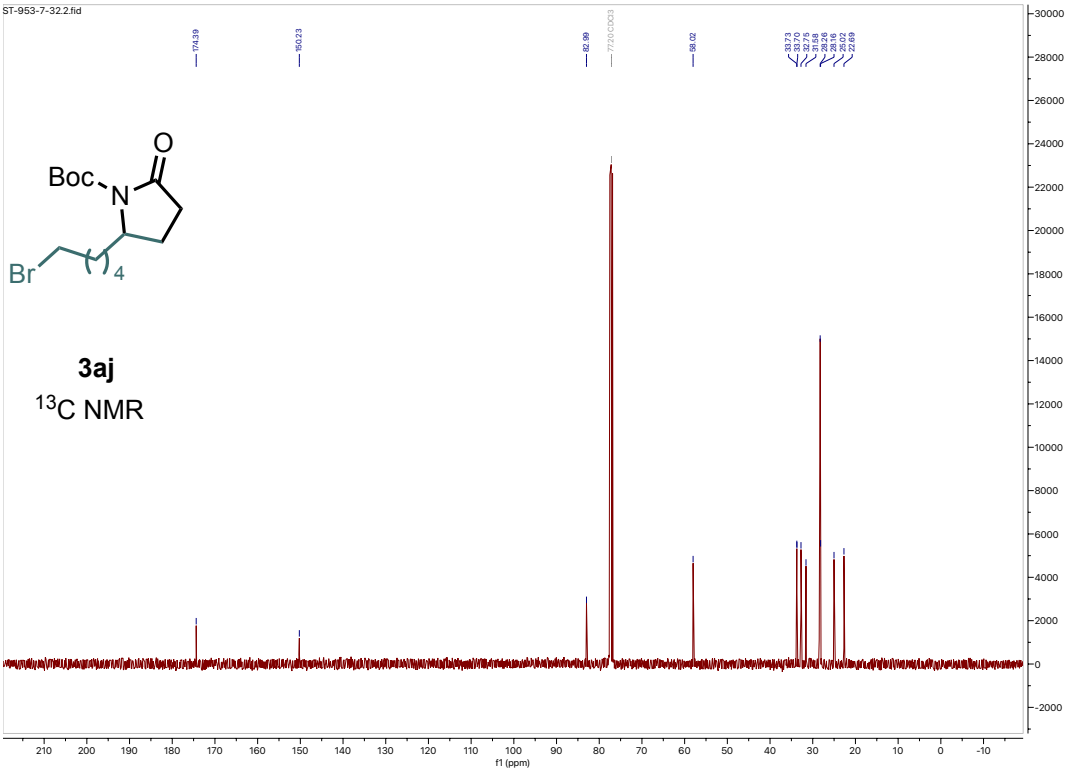
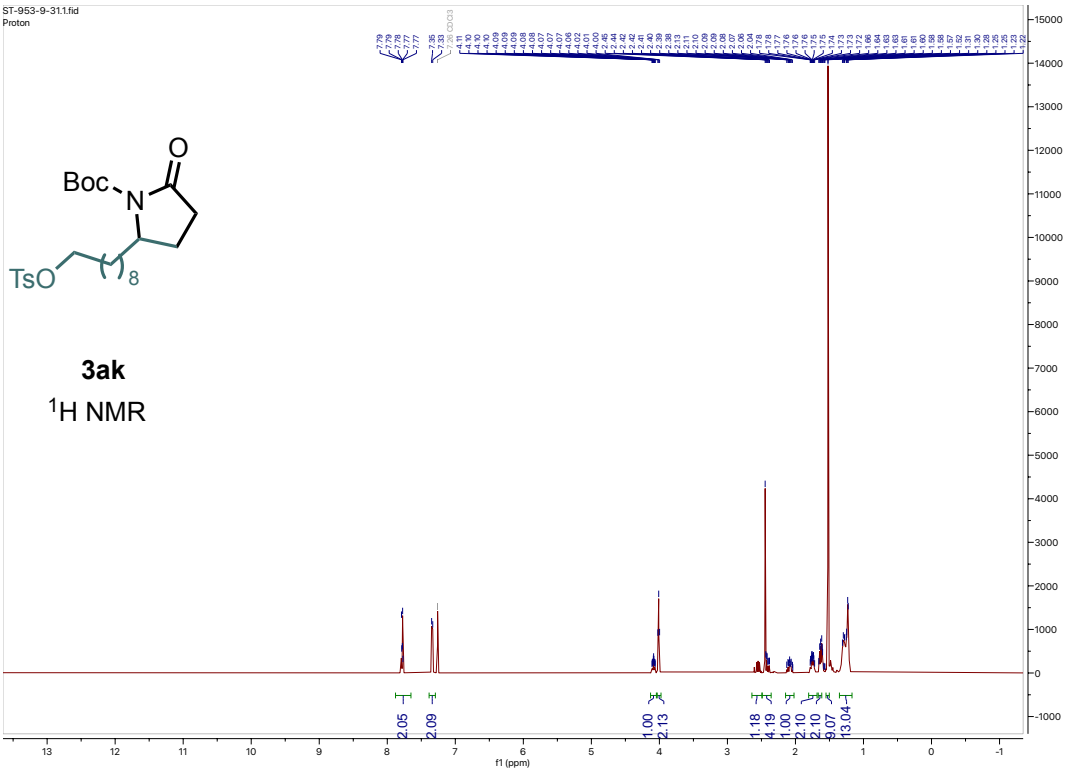
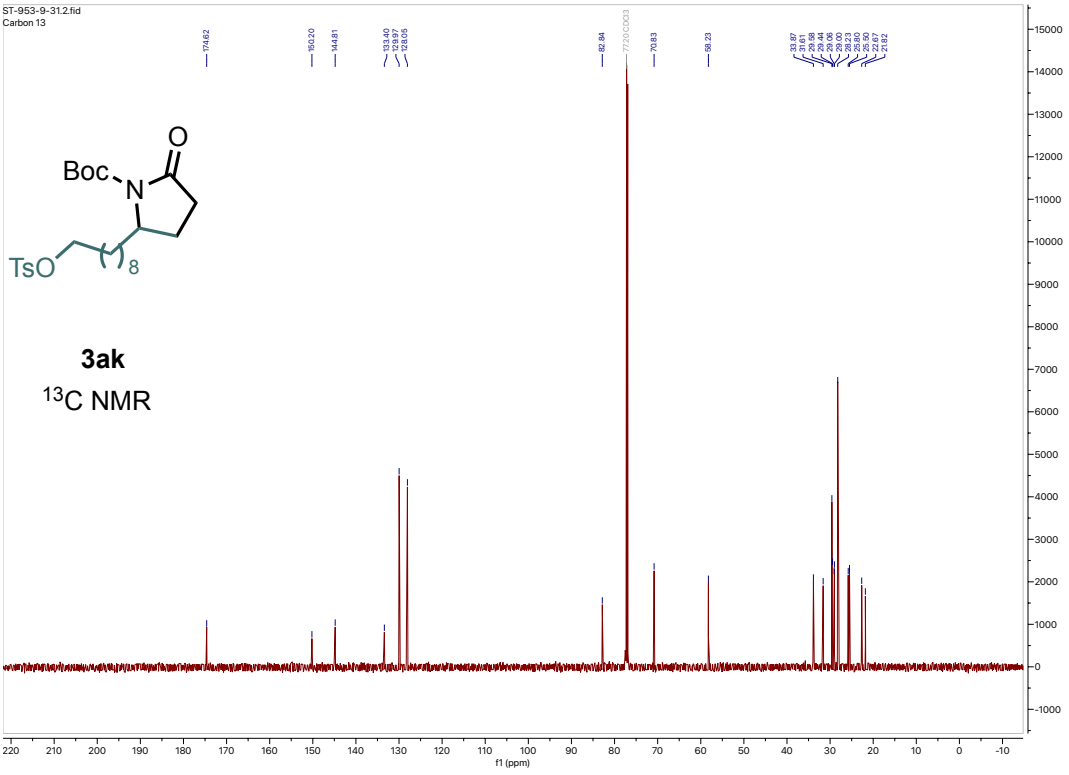
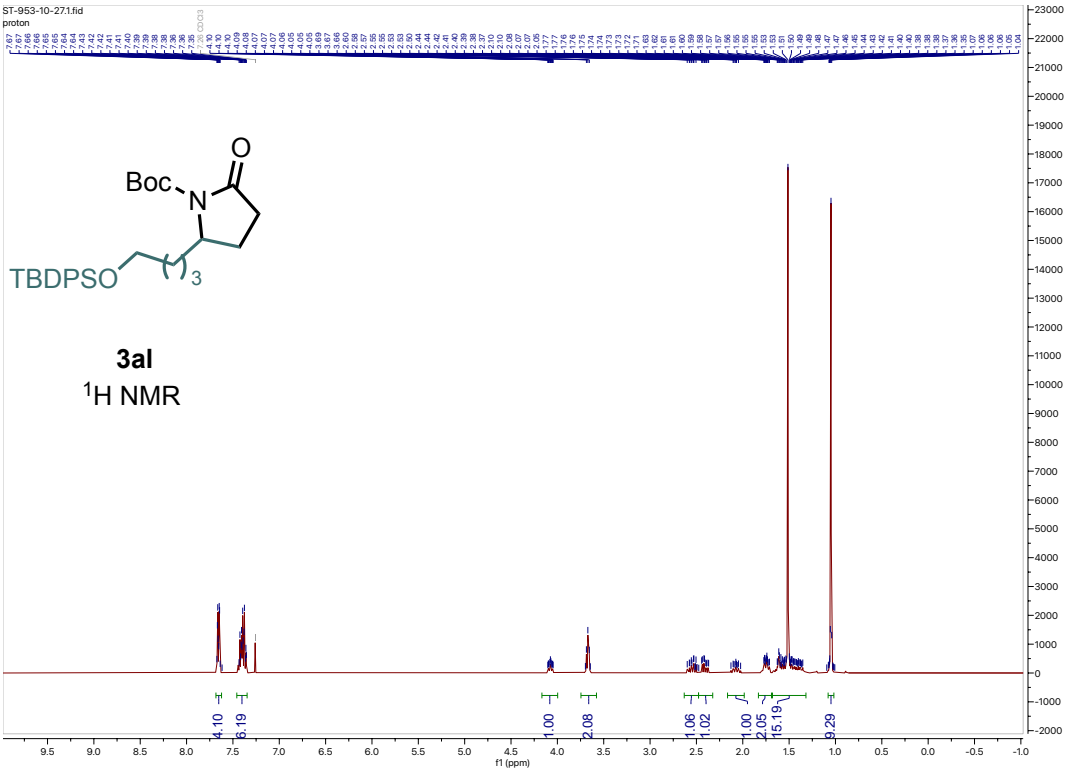
Boc
N
O
AcO
3ai
13C NMR
f1 (ppm)

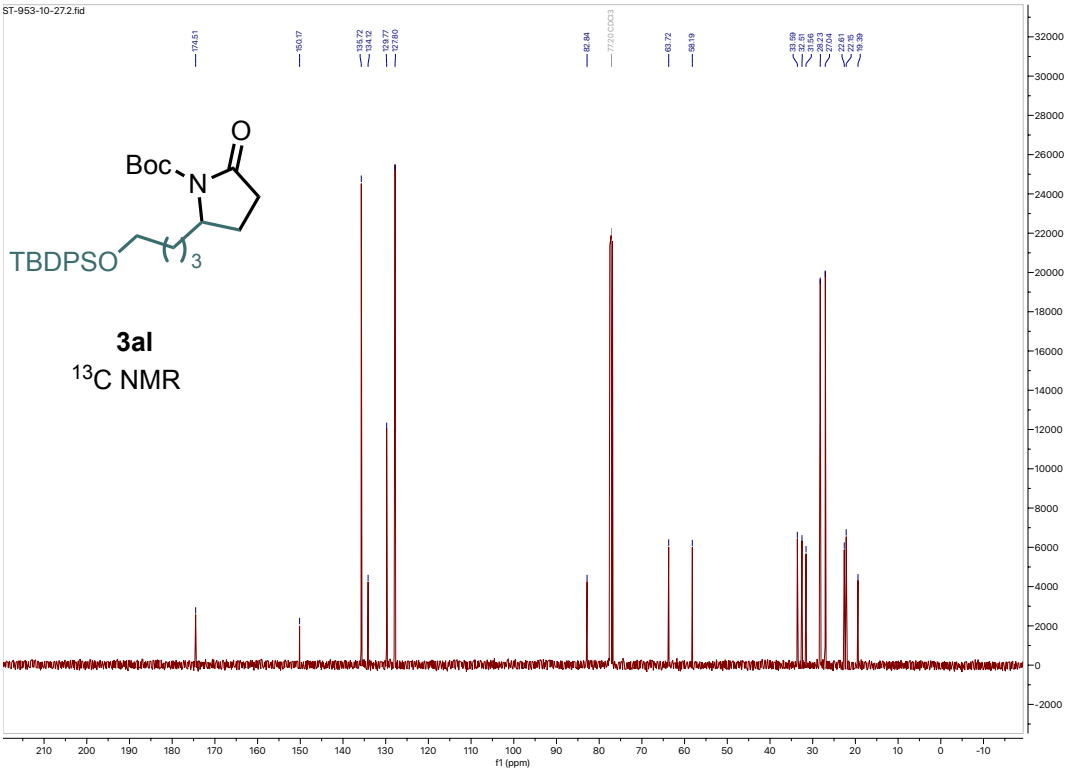

ST-953-7-32.1.fid
proton
Boc
N
O
Br
4
3aj
1H NMR
1.00
2.11
1.09
1.01
0.99
4.15
10.16
3.96
f1 (ppm)


ST-953-7-32.2.fid
174.39
150.23
82.99
77.20 CDCl3
58.02
53.73
39.70
31.73
31.68
28.26
28.06
25.02
22.69
Boc
N
O
Br
4
3aj
13C NMR
f1 (ppm)


ST-953-9-31.1.fid
Proton
Boc
N
O
TsO
8
3ak
1H NMR
f1 (ppm)


ST-953-9-31.2.fid
Carbon 13
Boc
N
O
TsO
8
3ak
13C NMR
f1 (ppm)


ST-953-10-27.1.fid
proton
Boc
N
O
TBDPSO
3
3al
1H NMR
4.10
6.19
1.00
2.08
1.06
1.02
1.00
2.05
15.19
9.29
f1 (ppm)


ST-953-10-27.2.fid
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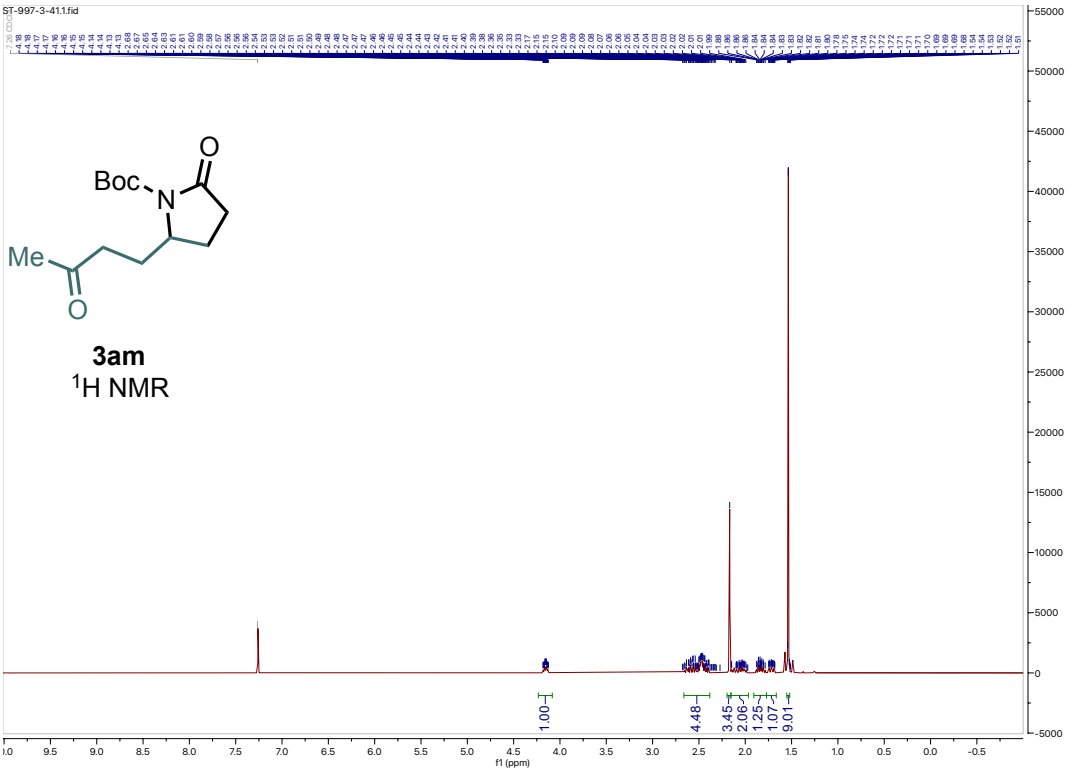
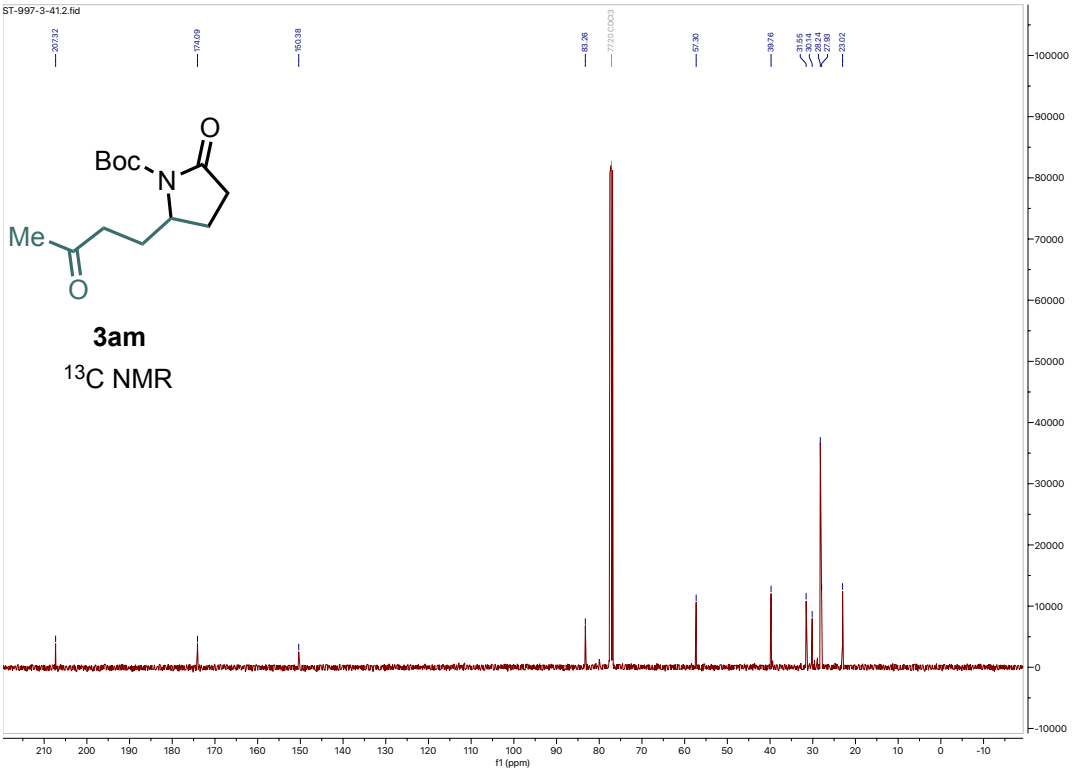
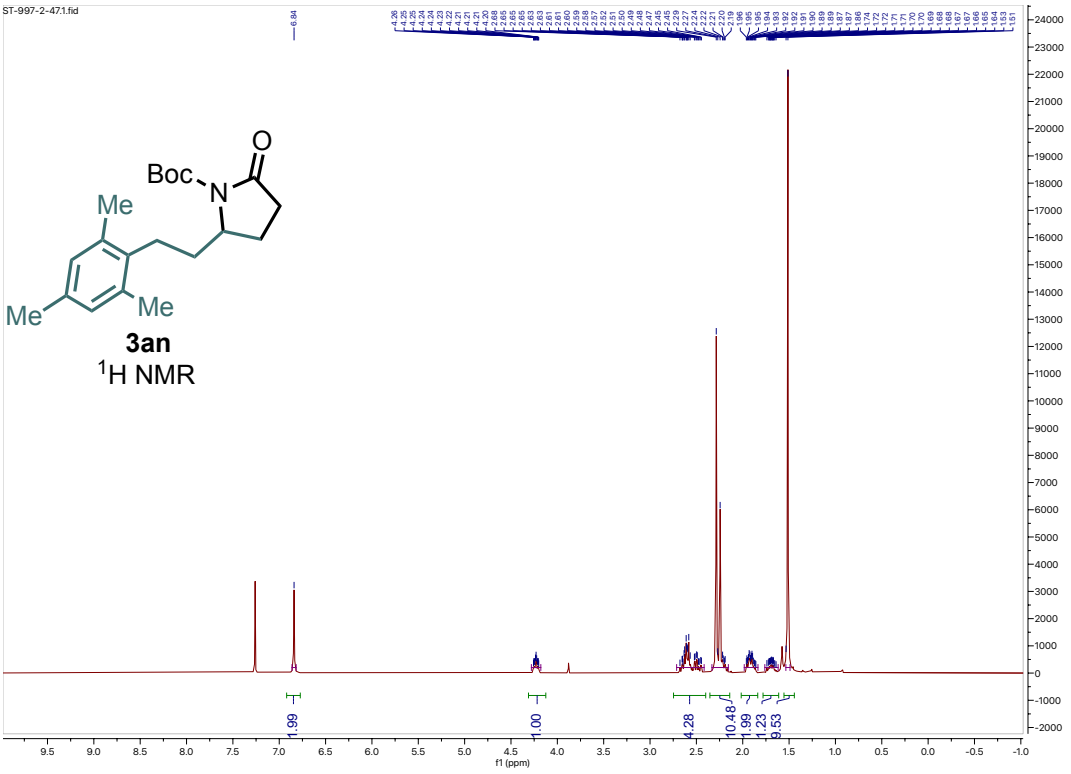
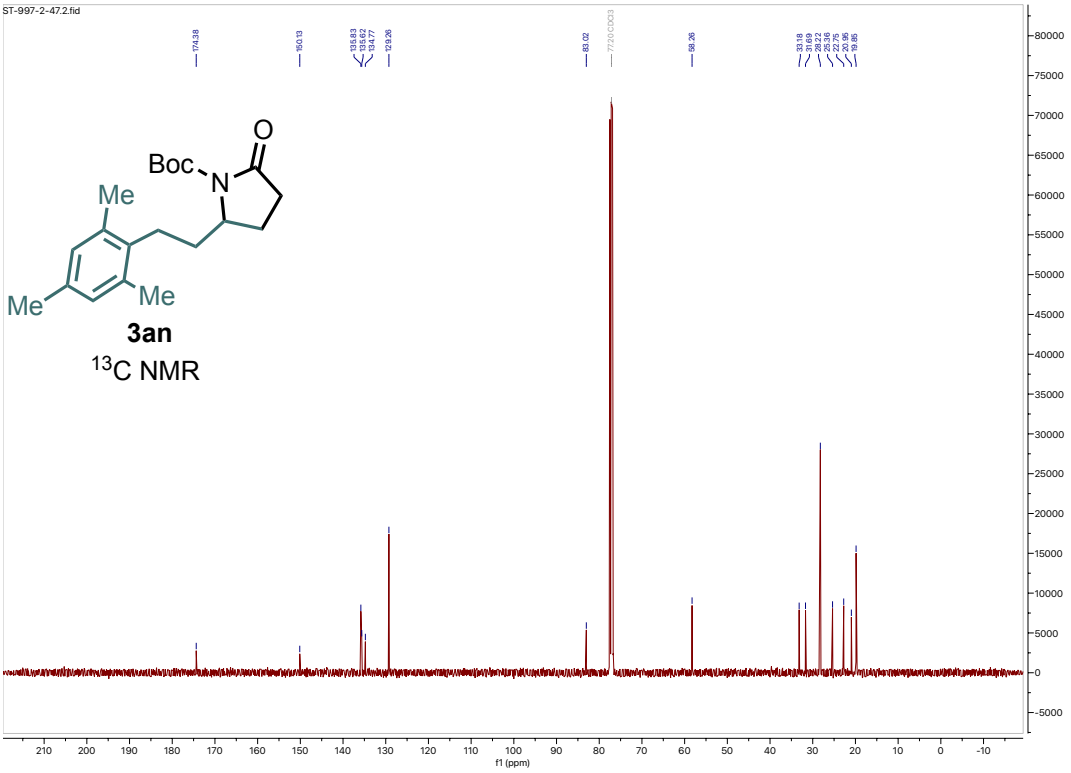
150.17
135.72
134.12
129.77
127.80
82.84
77.20 CDCl3
63.72
58.19
32.89
32.31
31.56
29.33
27.04
22.61
22.15
19.38
Boc
N
O
TBDPSO
3
3al
13C NMR
f1 (ppm)


ST-997-3-41.1.fid
Boc
N
O
Me
O
3am
1H NMR
1.00
4.48
3.45
2.06
1.25
1.07
9.01
f1 (ppm)


ST-997-3-41.2.fid
20.732
174.09
150.38
83.26
77.20 CDCl3
57.30
39.76
31.55
30.14
29.84
27.99
23.02
Boc
N
O
Me
O
3am
13C NMR
f1 (ppm)


ST-997-2-47.1.fid
Boc
N
O
Me
Me
Me
3an
1H NMR
f1 (ppm)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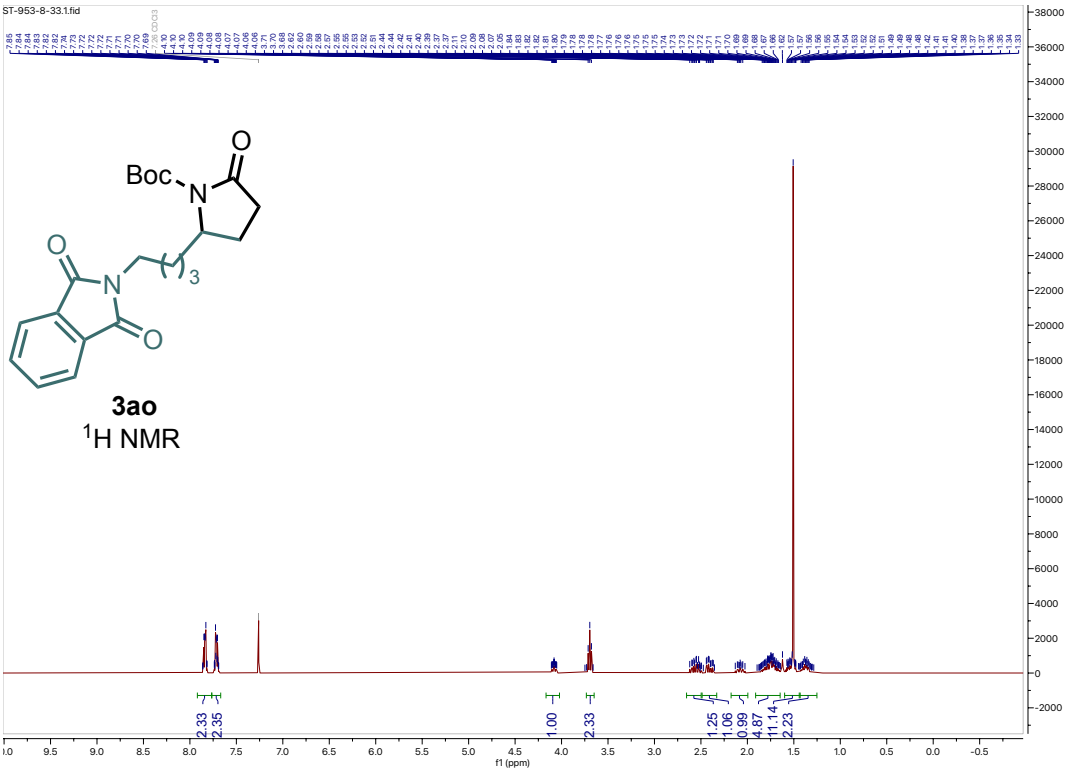
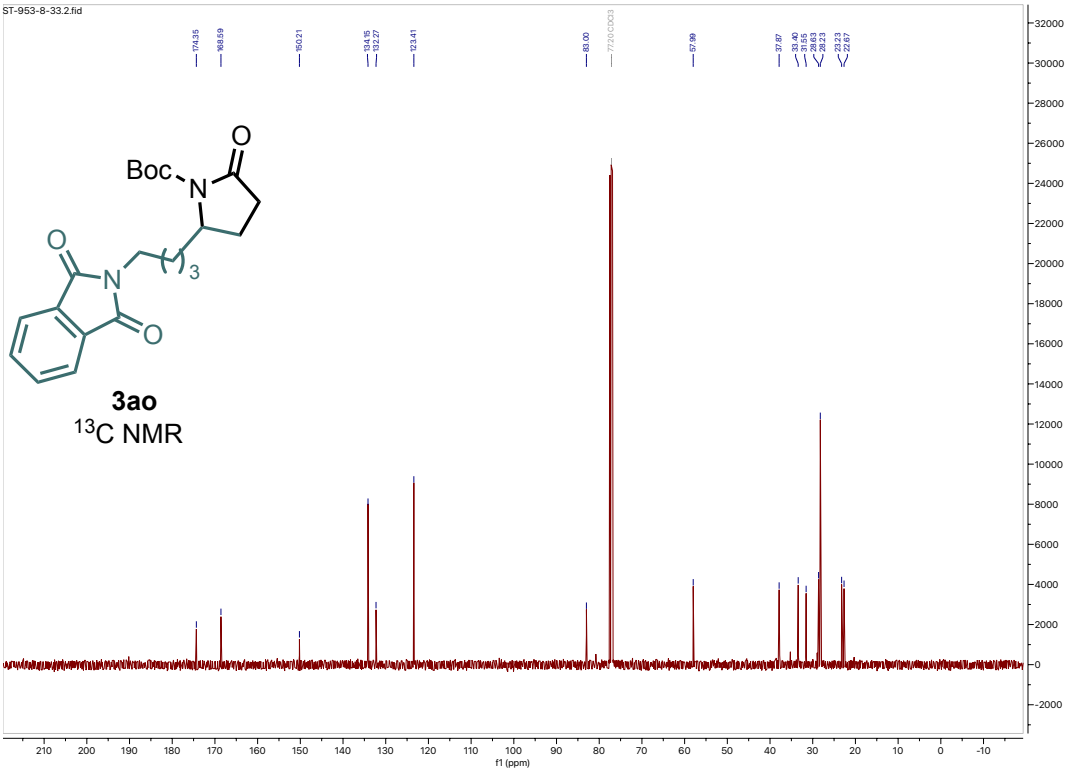
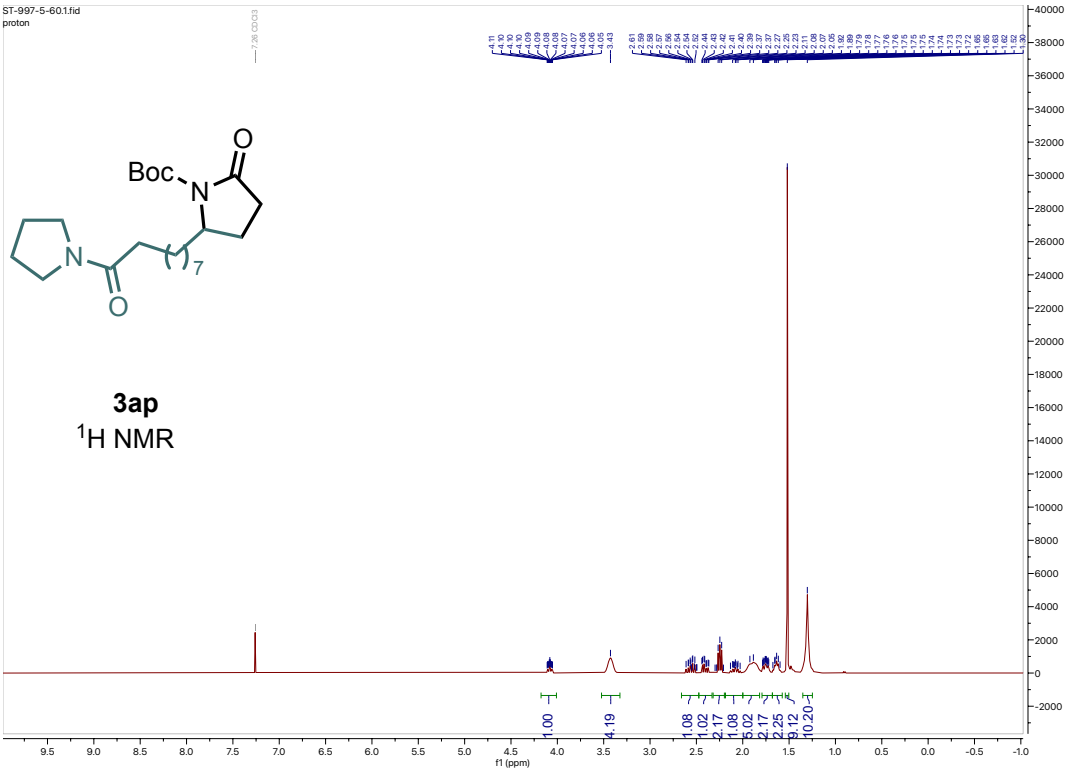
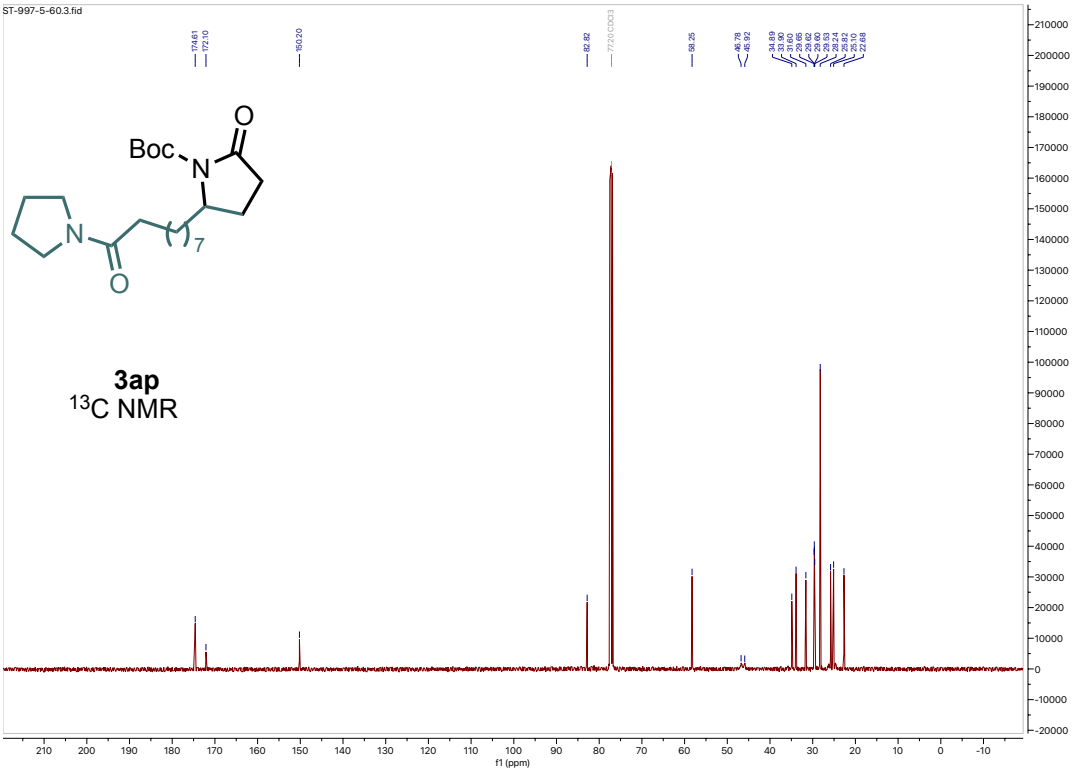
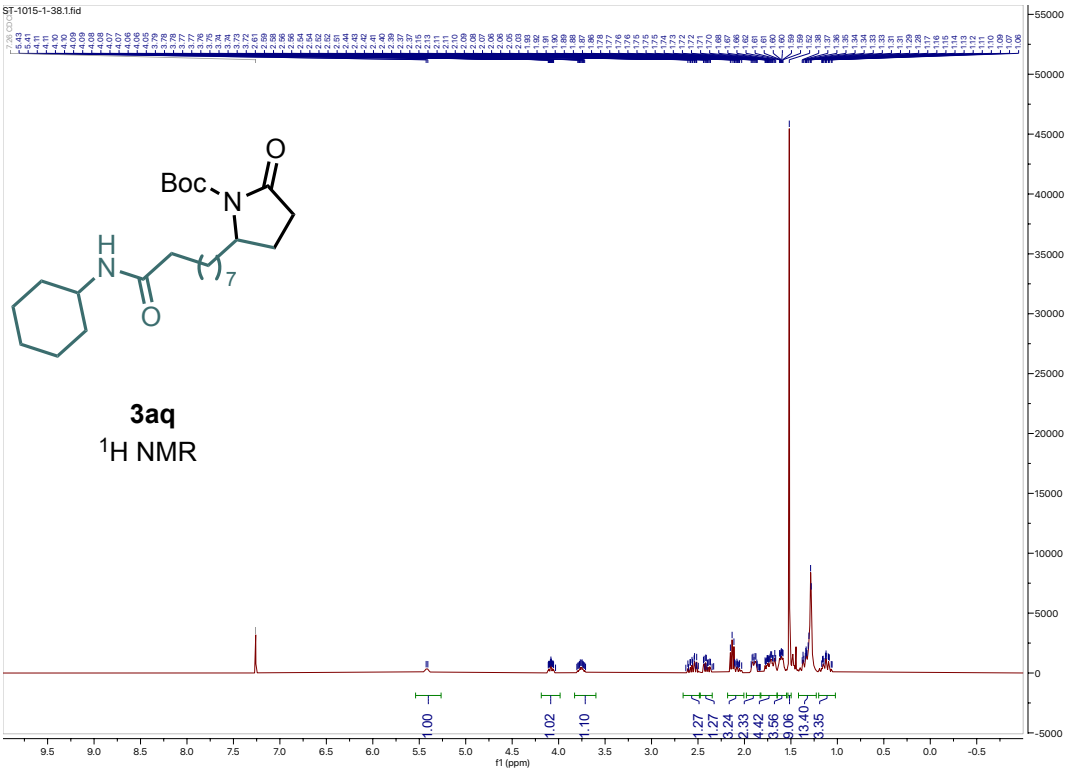
Boc
N
O
Me
Me
Me
3an
13C NMR
f1 (ppm)

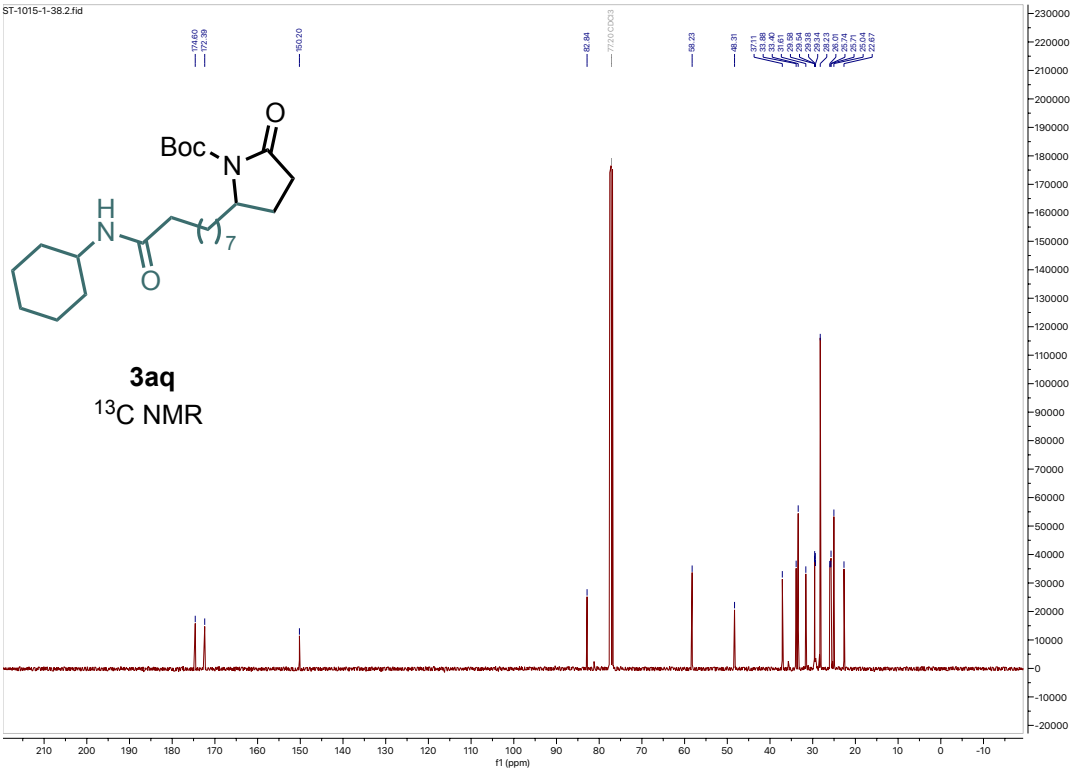

ST-953-8-33.1.fid
Boc
N
O
O
N
O
O
3
3ao
1H NMR
f1 (ppm)


ST-953-8-33.2.fid
Boc
N
O
O
N
O
O
3
3ao
13C NMR
f1 (ppm)


ST-997-5-60.1.fid
proton
Boc
N
O
O
N
7
3ap
1H NMR
1.00
4.19
1.08
1.02
2.17
1.08
5.02
2.17
2.25
9.12
10.20
f1 (ppm)


ST-997-5-60.3.fid
Boc
N
O
O
N
7
3ap
13C NMR
f1 (ppm)


ST-1015-1-38.1.fid
Boc
N
O
H
N
O
7
3aq
1H NMR
1.00
1.02
1.10
1.27
1.27
3.24
2.33
4.42
3.56
9.06
13.40
3.35
f1 (ppm)


ST-1015-1-38.2.fid
174.60
172.39
150.20
82.84
77.20 CDCl3
59.23
48.31
Boc
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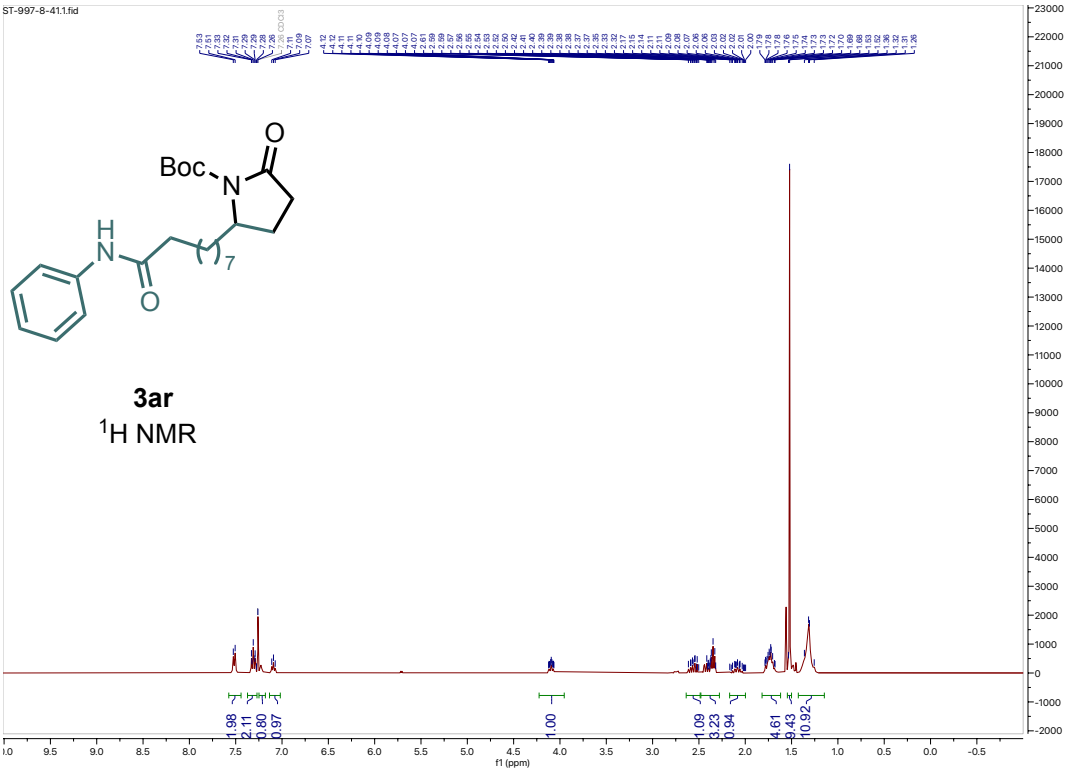
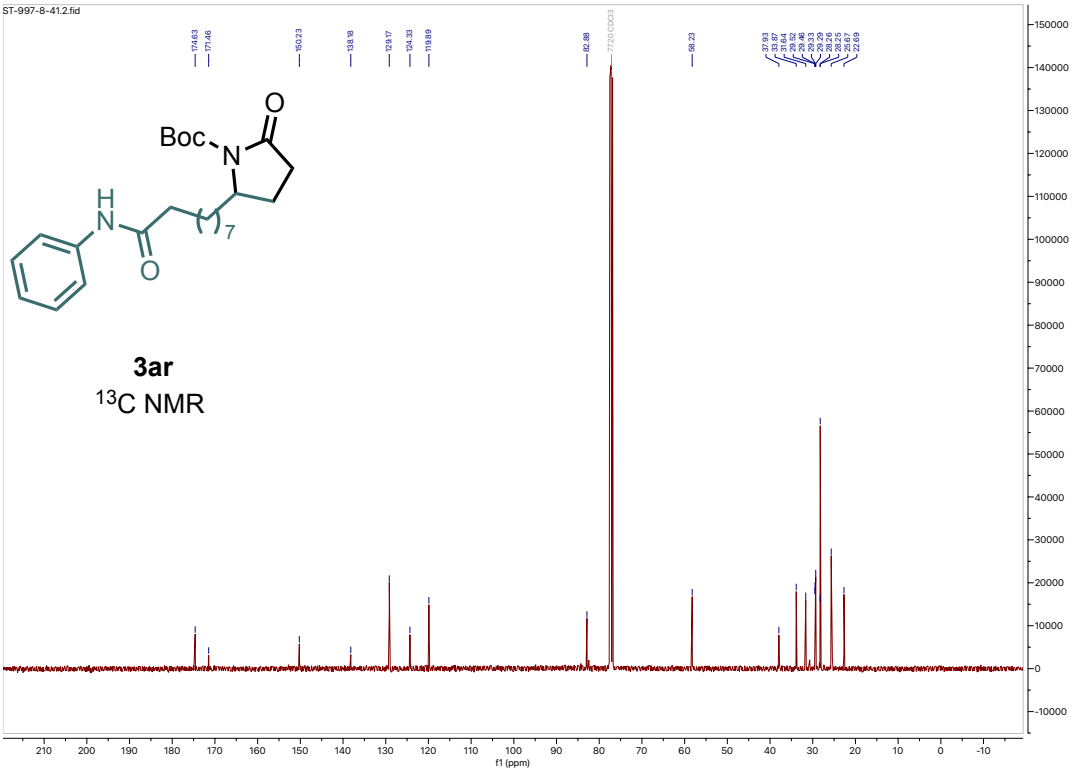
O
H
N
O
7
3aq
13C NMR
f1 (ppm)


ST-997-8-41.1.fid
Boc
N
O
H
N
O
7
3ar
1H NMR
1.98
2.11
0.80
0.97
1.00
1.09
3.23
0.94
4.61
9.43
10.92
f1 (ppm)


ST-997-8-41.2.fid
174.63
171.46
150.23
138.18
129.17
124.33
119.89
82.88
77.20 CDCl3
59.23
37.93
31.87
31.64
29.42
29.36
29.33
29.29
28.26
28.25
25.67
22.69
Boc
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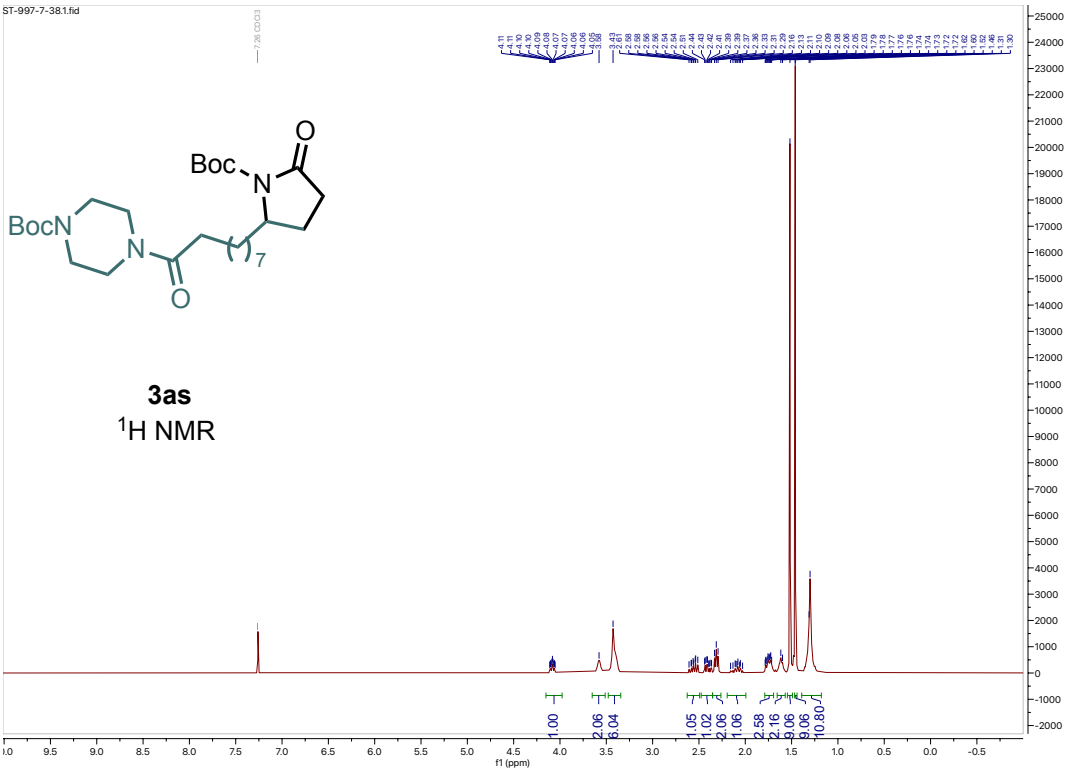
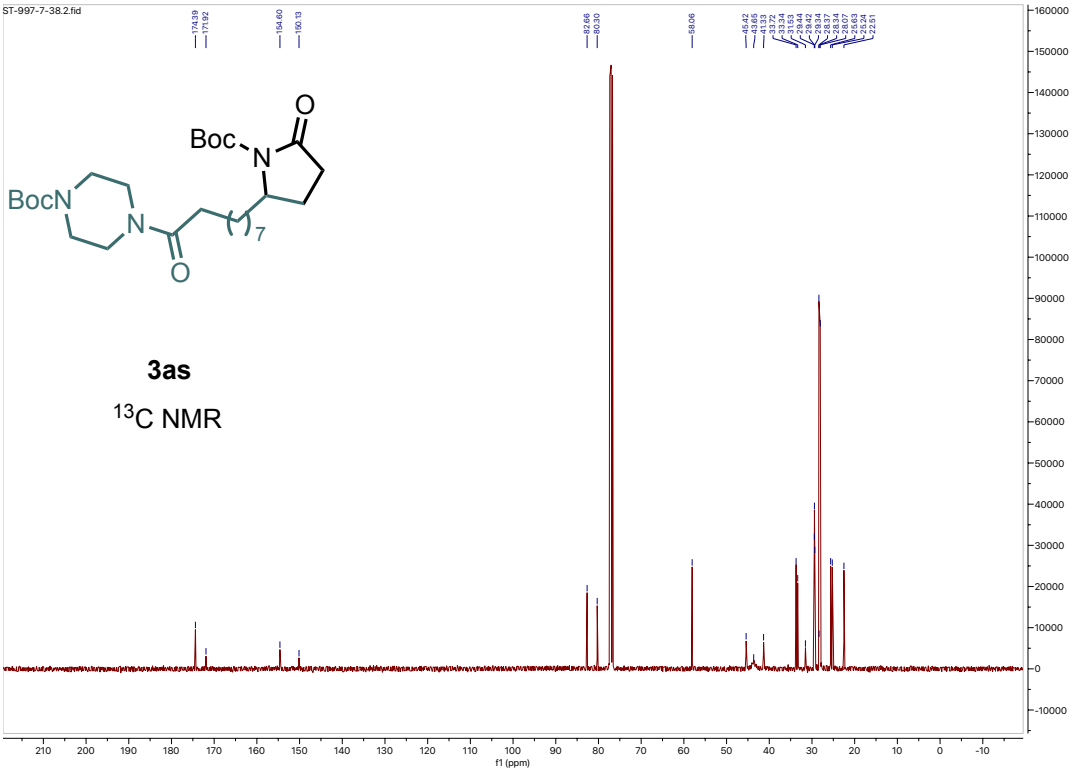
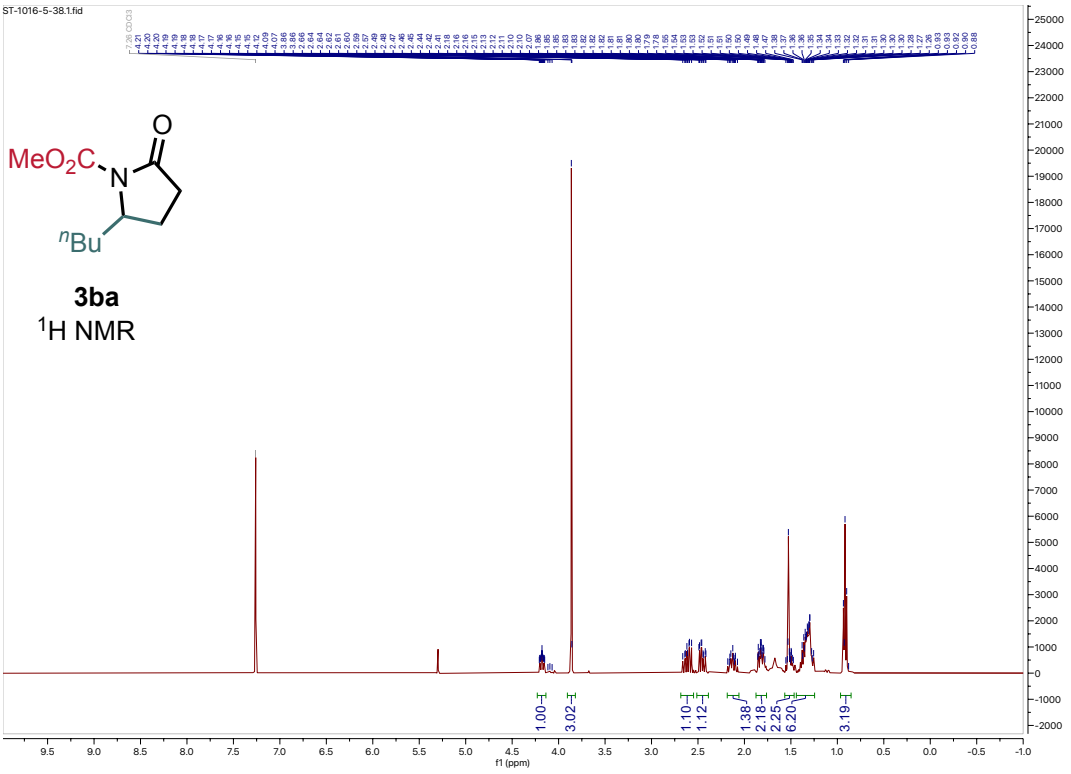
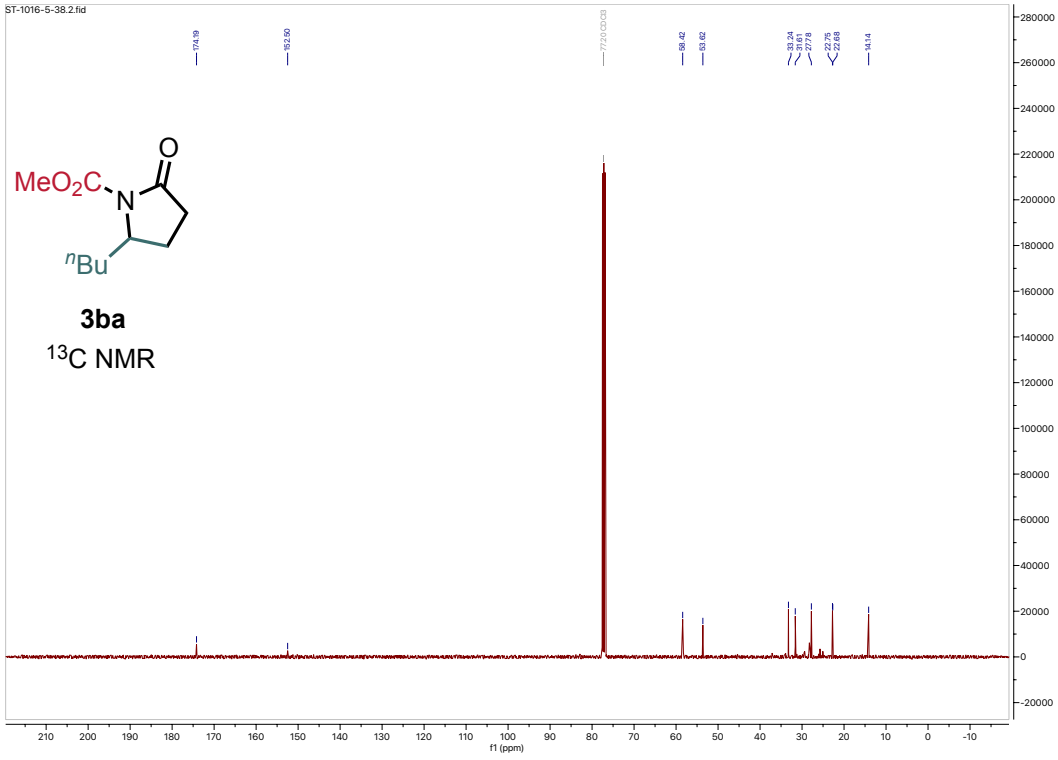
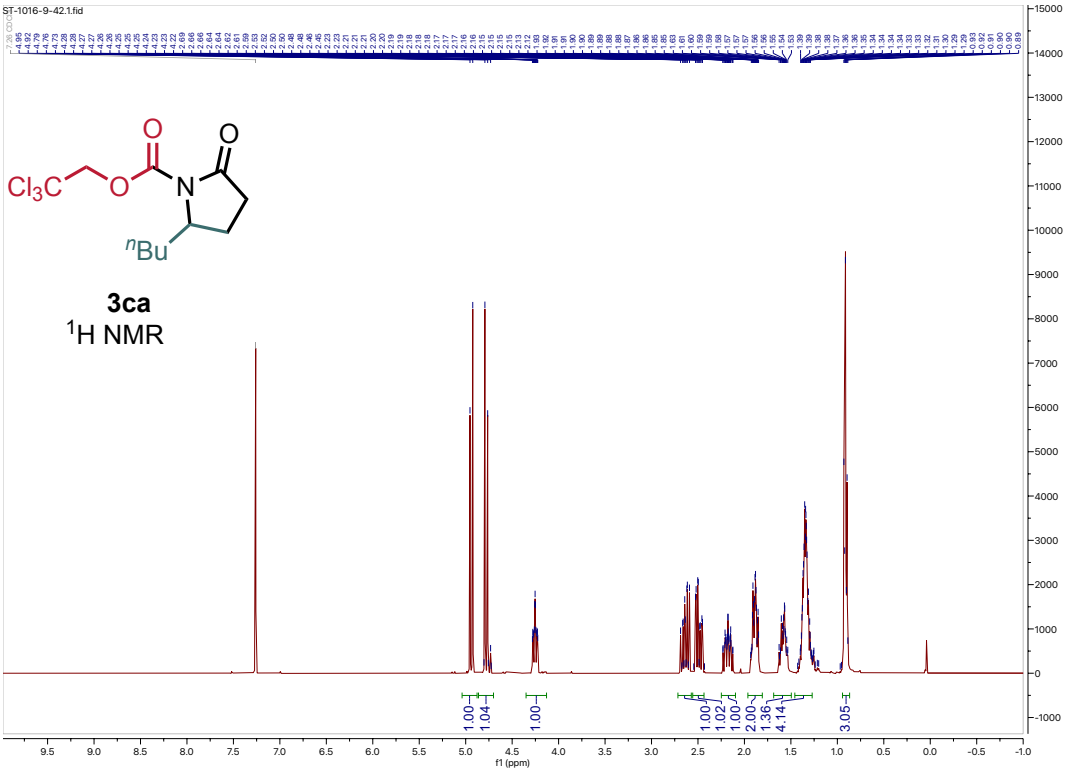
H
N
O
7
3ar
13C NMR
f1 (ppm)

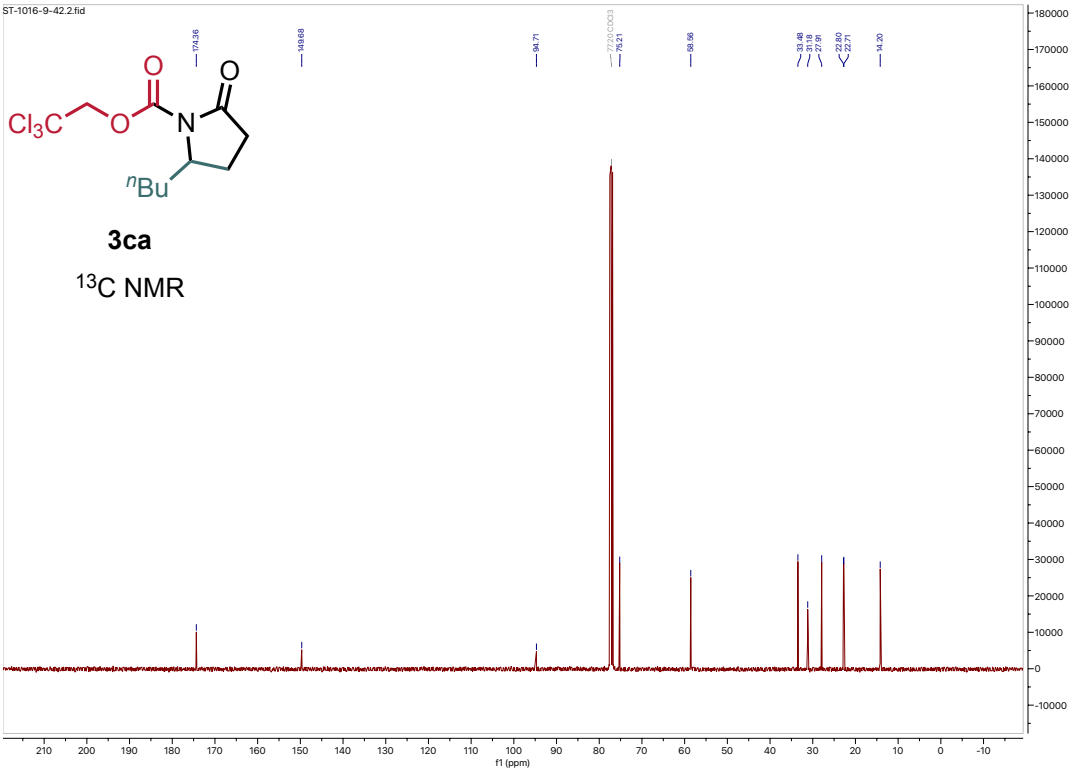

ST-997-7-38.1.fid
Boc
N
O
BocN
N
7
O
3as
1H NMR
f1 (ppm)

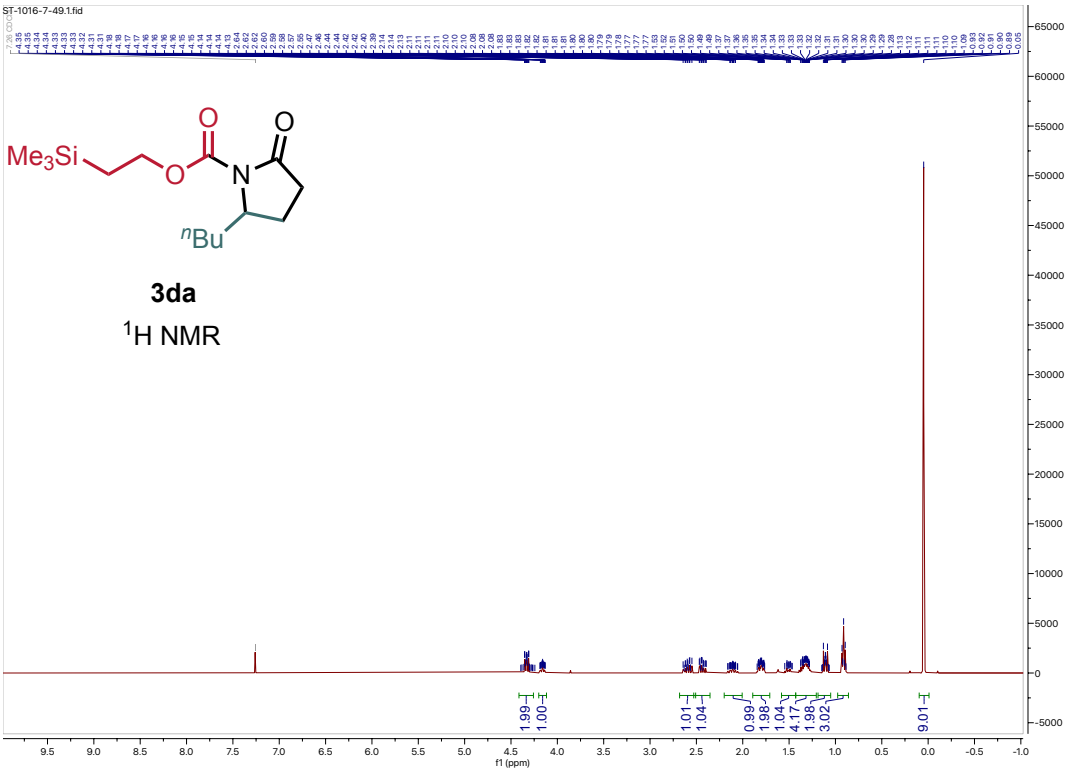

ST-997-7-38.2.fid
Boc
N
O
BocN
N
7
O
3as
13C NMR
f1 (ppm)

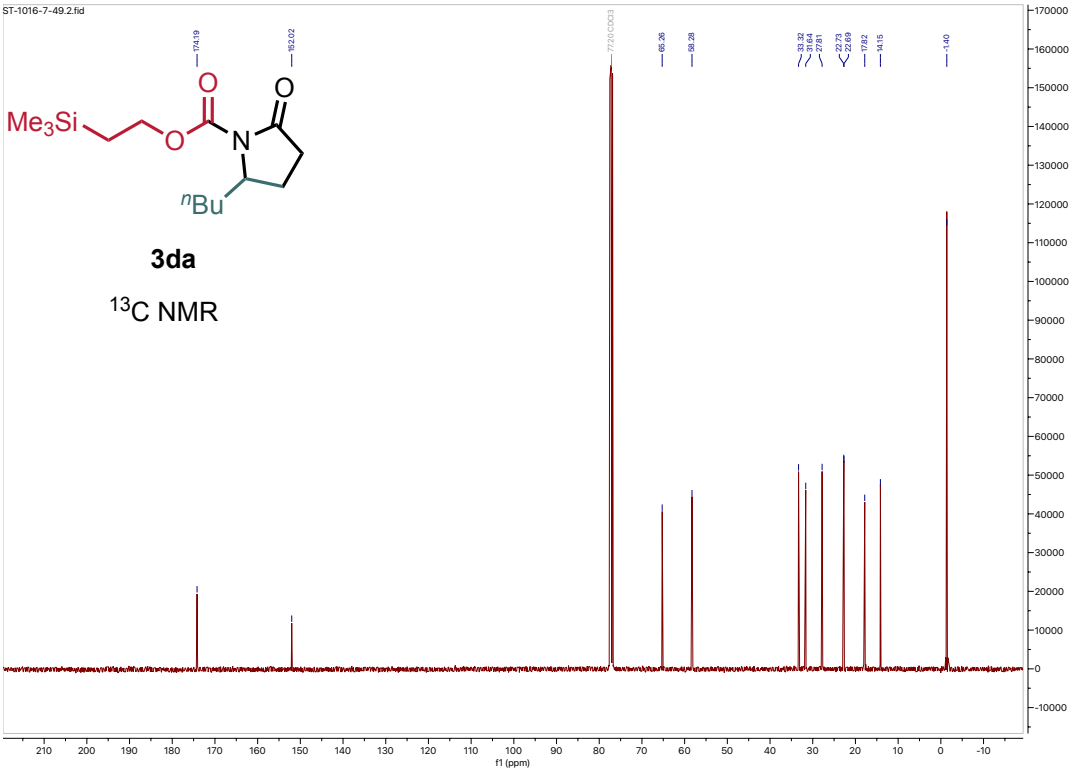

ST-1016-5-38.1.fid
MeO2C
N
O
nBu
3ba
1H NMR
1.00
3.02
1.10
1.12
1.38
2.18
2.25
6.20
3.19
f1 (ppm)

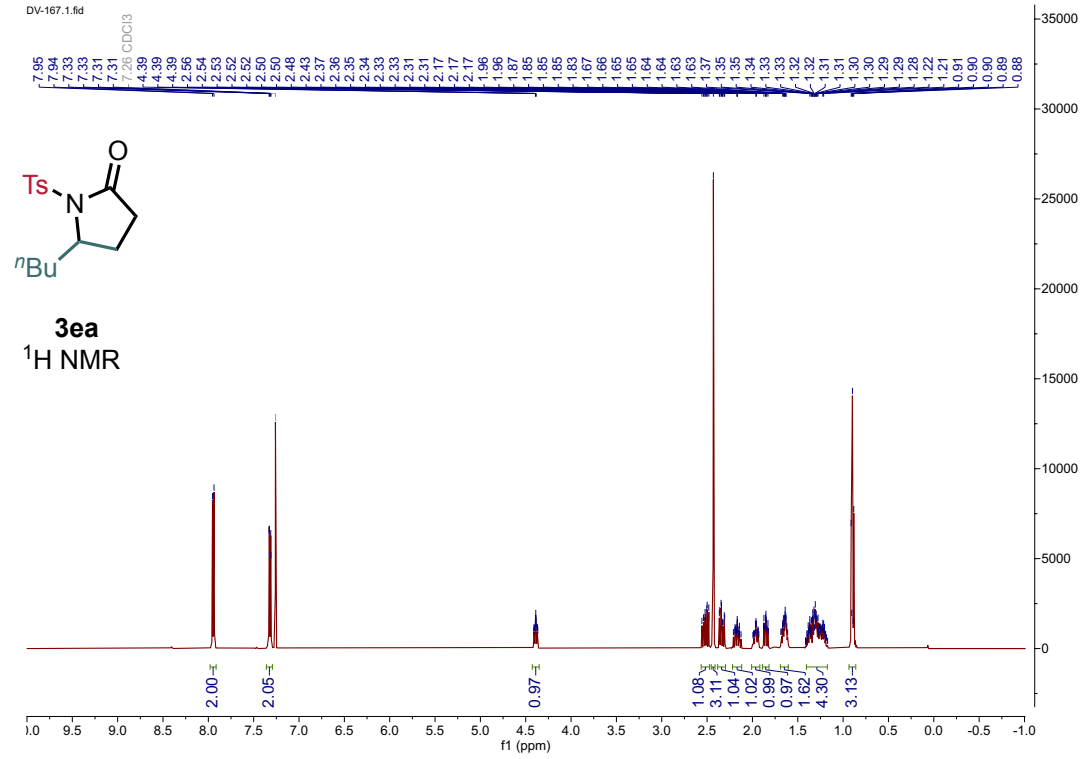

ST-1016-5-38.2.fid
174.19
152.50
77.20 CDCl3
58.42
53.62
30.24
31.61
27.78
22.75
22.68
14.14
MeO2C
N
O
nBu
3ba
13C NMR
f1 (ppm)

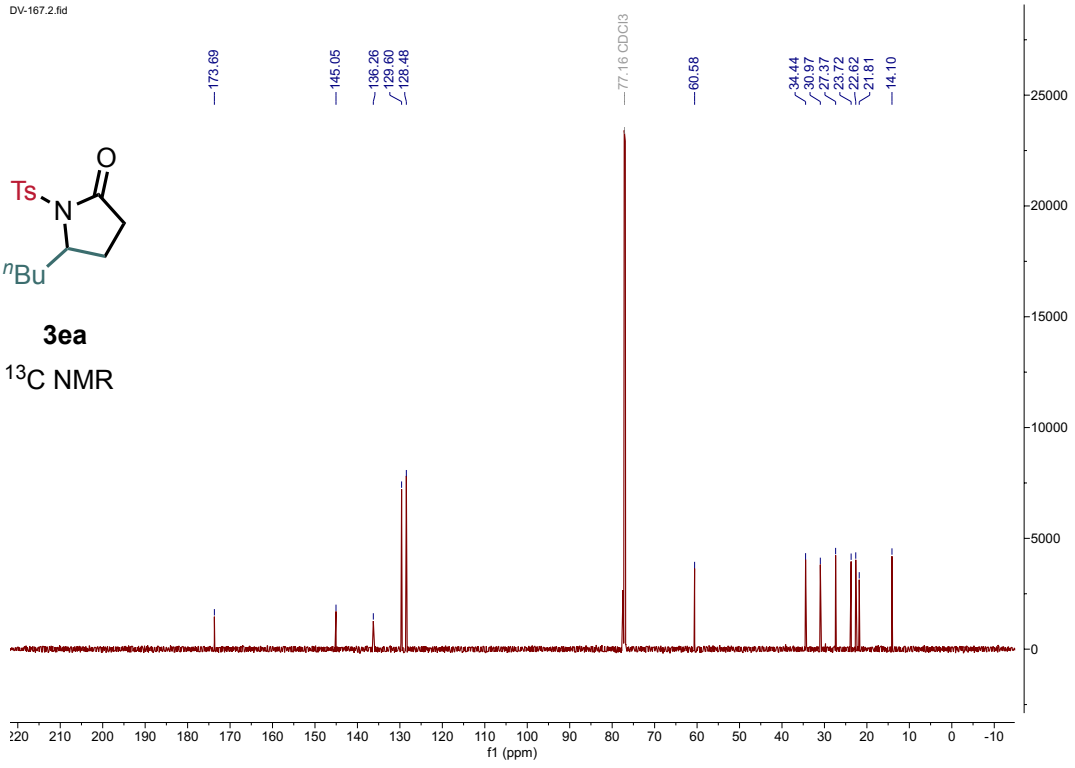

ST-1016-9-42.1.fid
Cl3C
O
O
N
O
nBu
3ca
1H NMR
1.00
1.04
1.00
1.00
1.02
1.00
2.00
1.36
4.14
3.05
f1 (ppm)

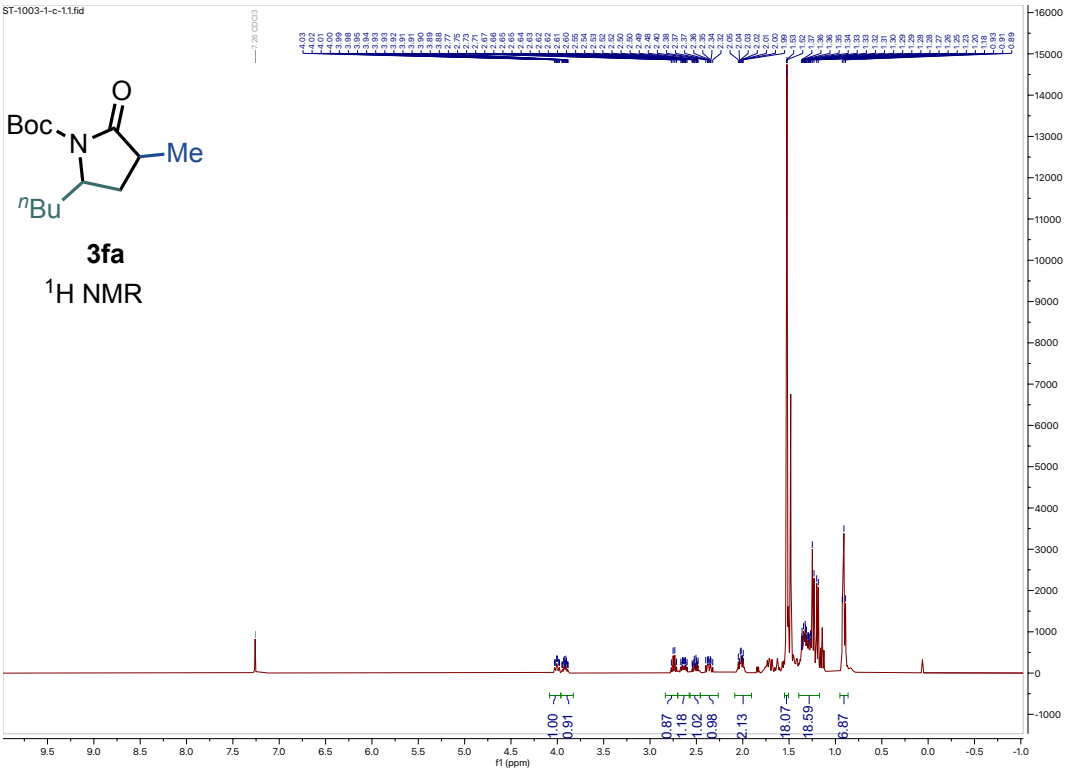

ST-1016-9-42.2.fid
174.36
149.68
94.71
79.21
59.56
33.48
31.18
27.96
22.80
22.71
14.20
Cl3C
O
O
N
O
nBu
3ca
13C NMR
f1 (ppm)

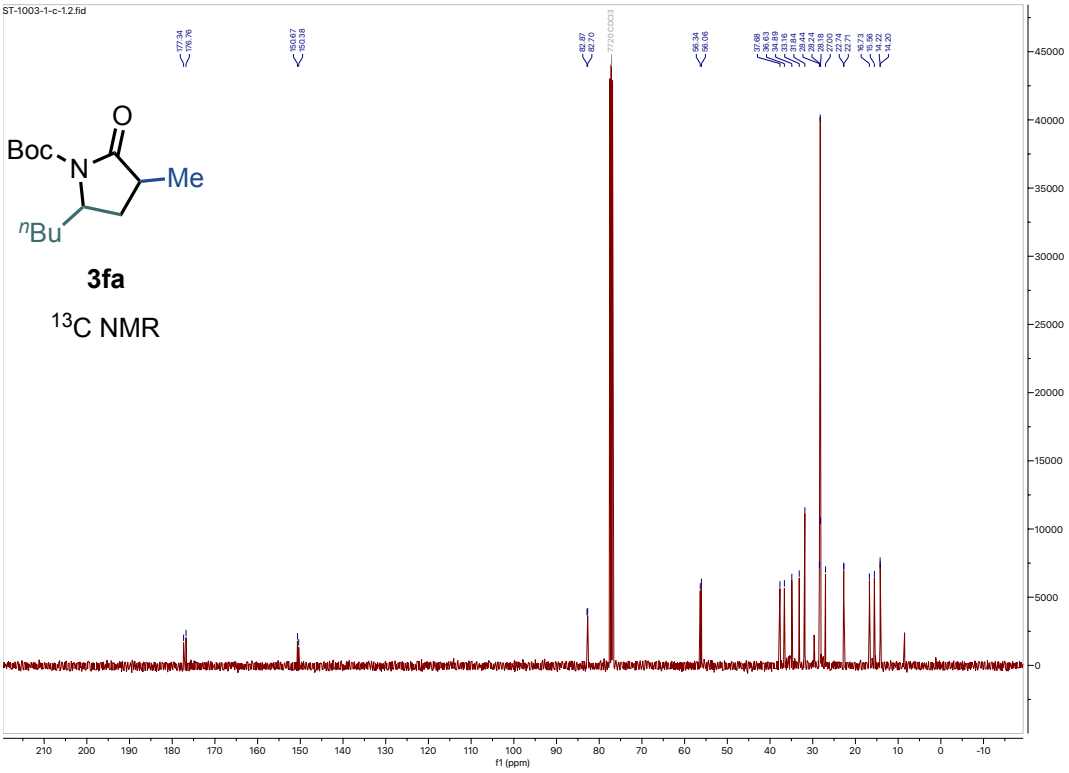

ST-1016-7-49.1.fid
Me3Si
O
O
N
O
nBu
3da
1H NMR
1.99
1.00
1.01
1.04
0.99
1.96
4.17
1.98
3.02
9.01
f1 (ppm)

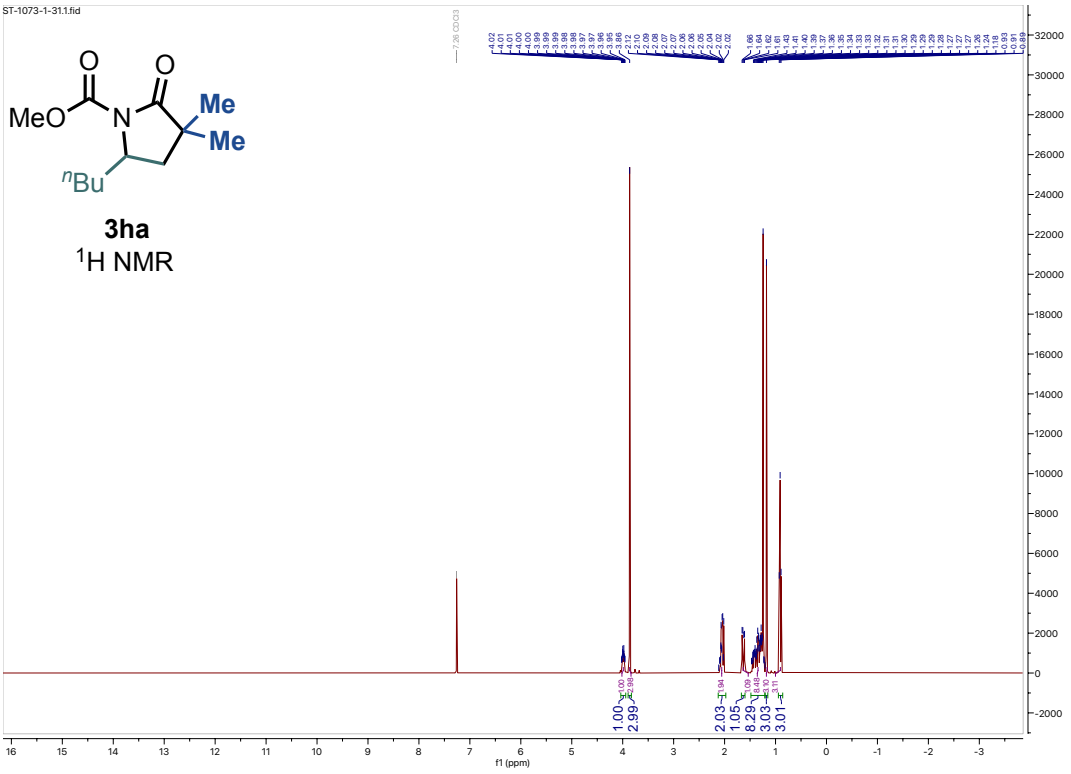

ST-1016-7-49.2.fid
174.19
162.02
77.20 CDCl3
65.26
59.28
33.32
31.64
27.91
22.73
22.69
17.82
14.15
-1.40
Me3Si
O
O
N
O
nBu
3da
13C NMR
f1 (ppm)

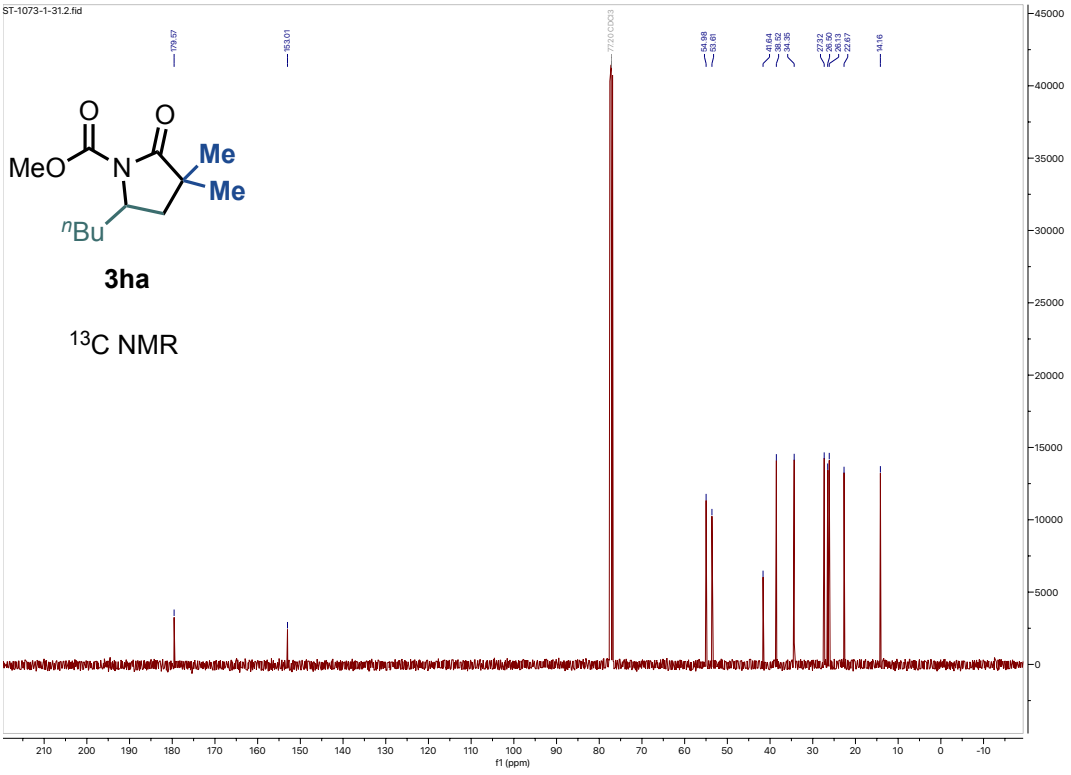

DV-167.1.fid
7.26 CDCl3
Ts
N
O
nBu
3ea
1H NMR
2.00
2.05
0.97
1.08
3.11
1.04
1.02
0.99
0.97
1.62
4.30
3.13
f1 (ppm)

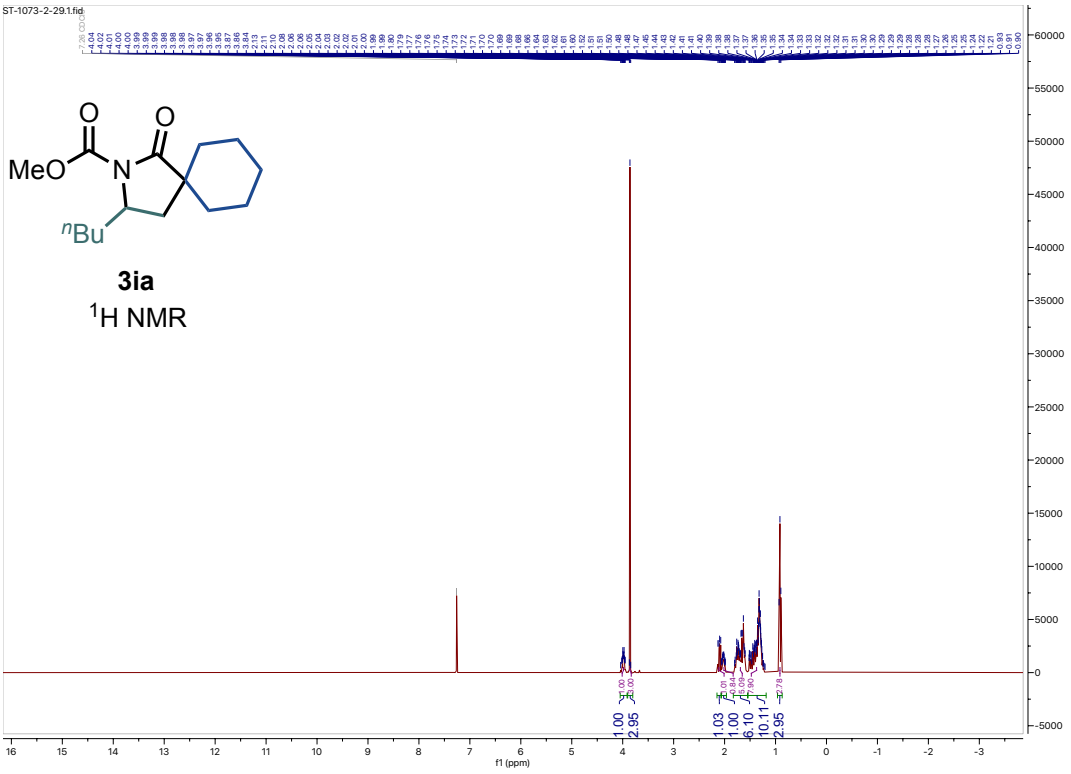

DV-167.2.fid
173.69
145.05
136.26
129.60
128.48
77.16 CDCl3
60.58
34.44
30.97
27.37
23.72
22.62
21.81
14.10
Ts
N
O
nBu
3ea
13C NMR
f1 (ppm)

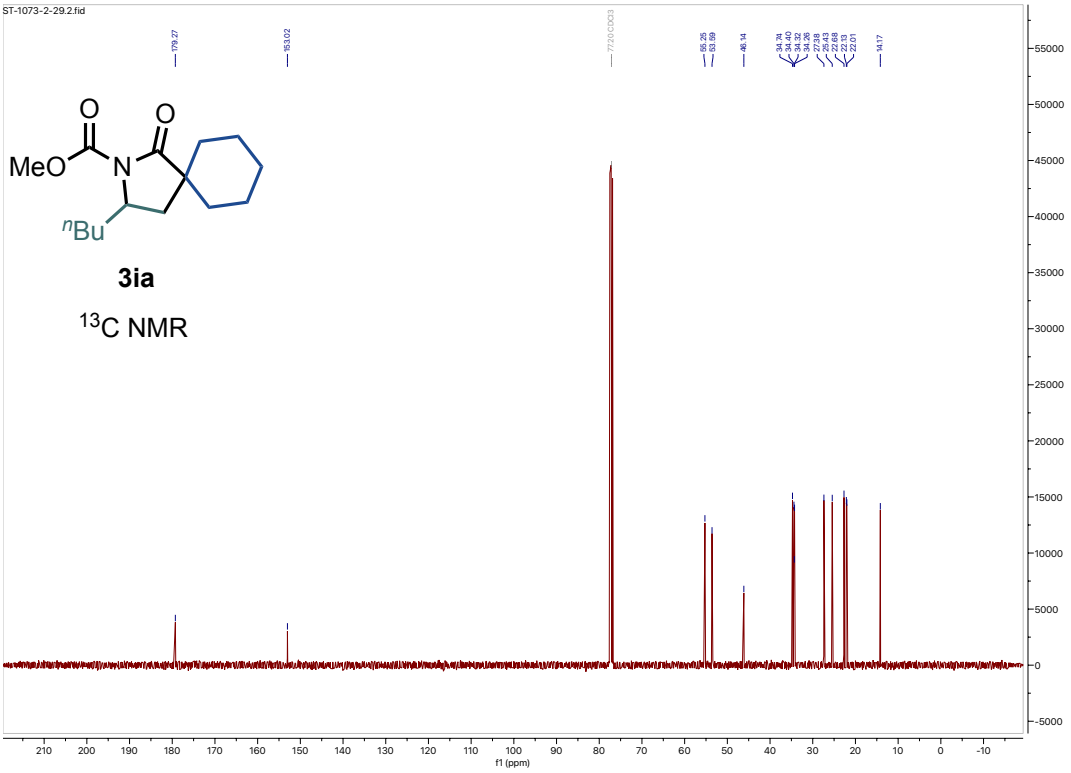

ST-1003-1-c-1.1.fid
Boc
N
O
Me
nBu
3fa
1H NMR
f1 (ppm)

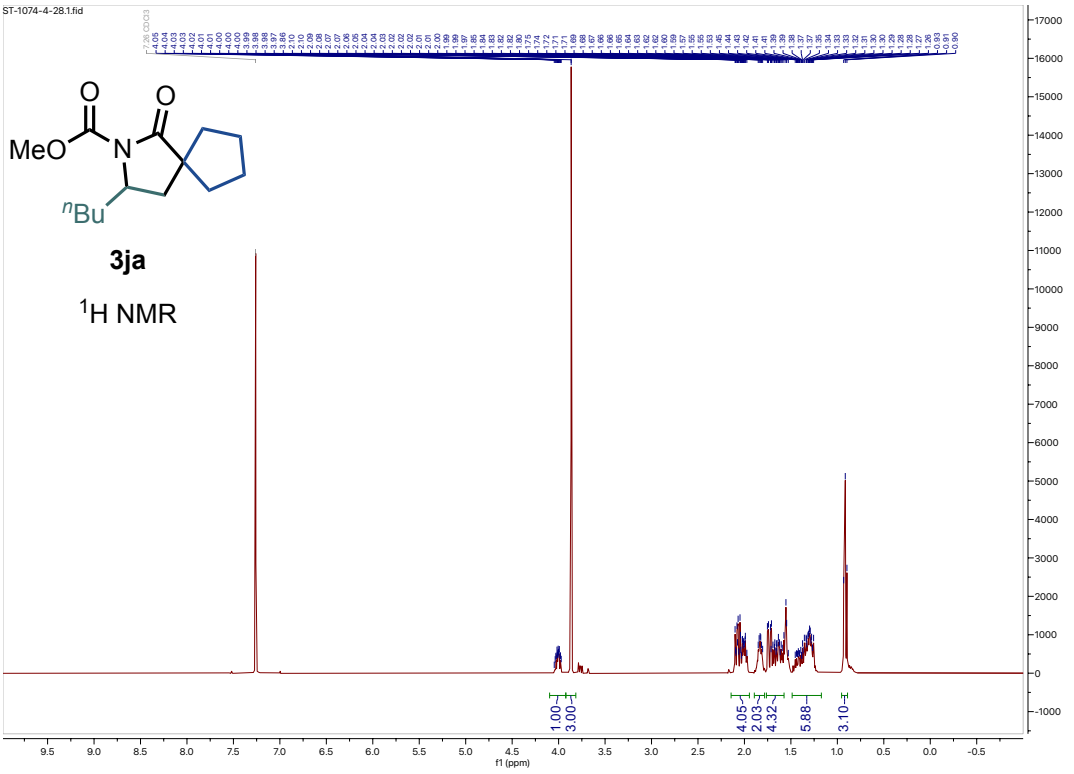

ST-1003-1-c-1.2.fid
Boc
N
O
Me
nBu
3fa
13C NMR
f1 (ppm)

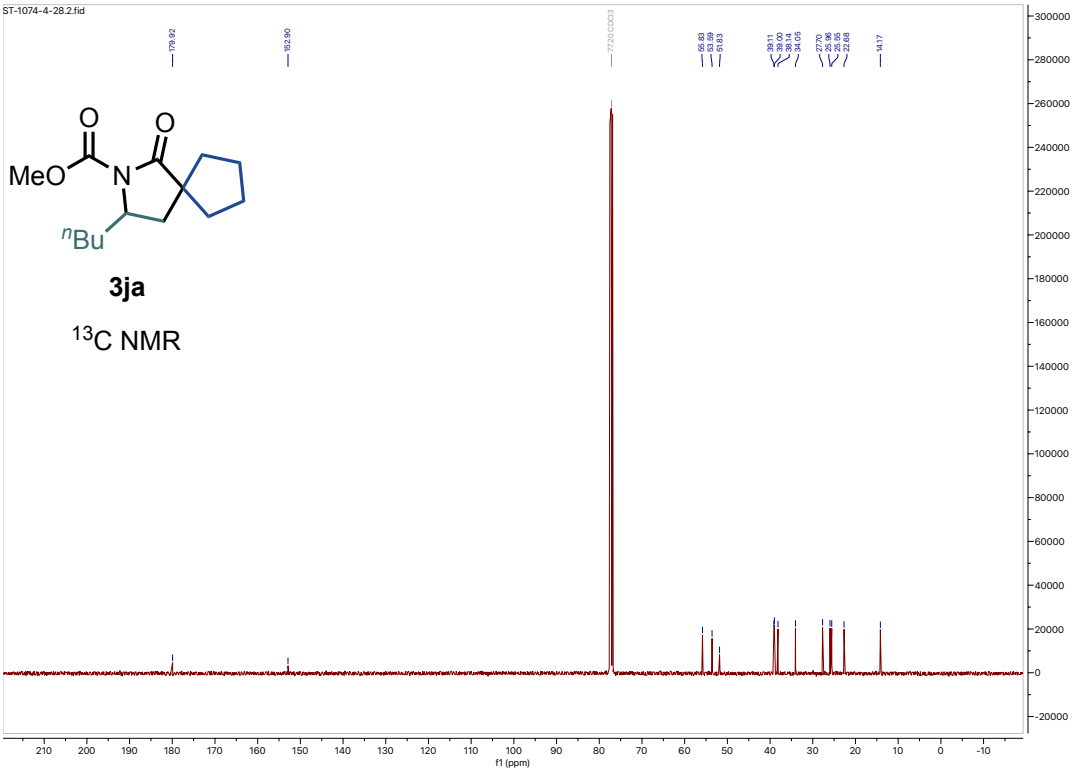

ST-1073-1-31.1.fid
MeO
N
O
O
Me
Me
nBu
3ha
1H NMR
1.00
2.99
2.03
1.05
8.29
3.03
3.01
f1 (ppm)

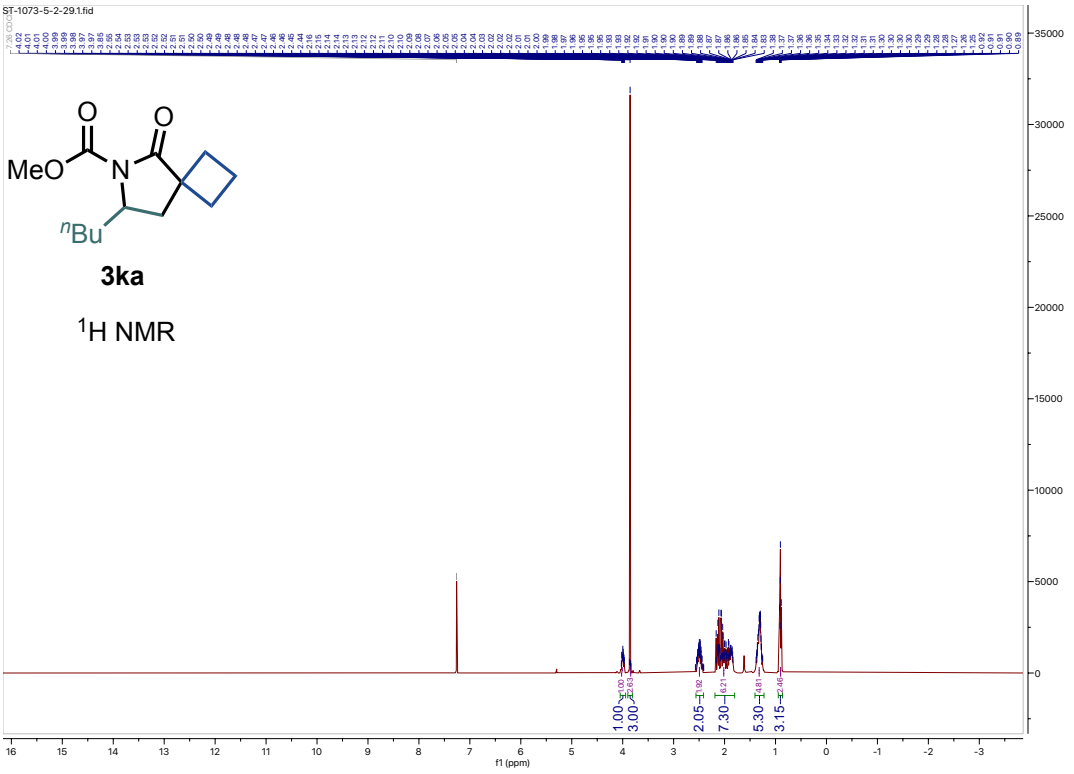

ST-1073-1-31.2.fid
179.57
153.01
77.20 CDCl3
54.98
53.61
41.64
34.52
34.38
27.72
26.92
26.13
22.67
14.16
MeO
N
O
O
Me
Me
nBu
3ha
13C NMR
f1 (ppm)

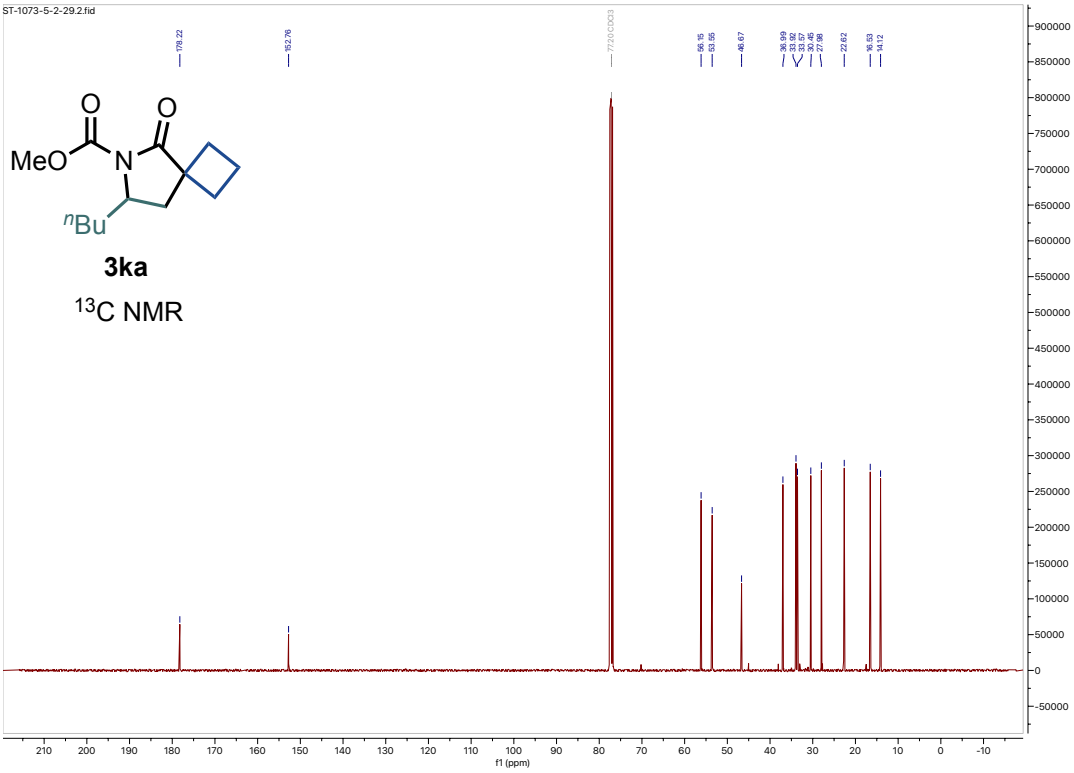

ST-1073-2-29.1.fid
MeO
O
O
N
nBu
3ia
1H NMR
1.00
2.95
1.03
1.00
6.10
10.11
2.95
f1 (ppm)

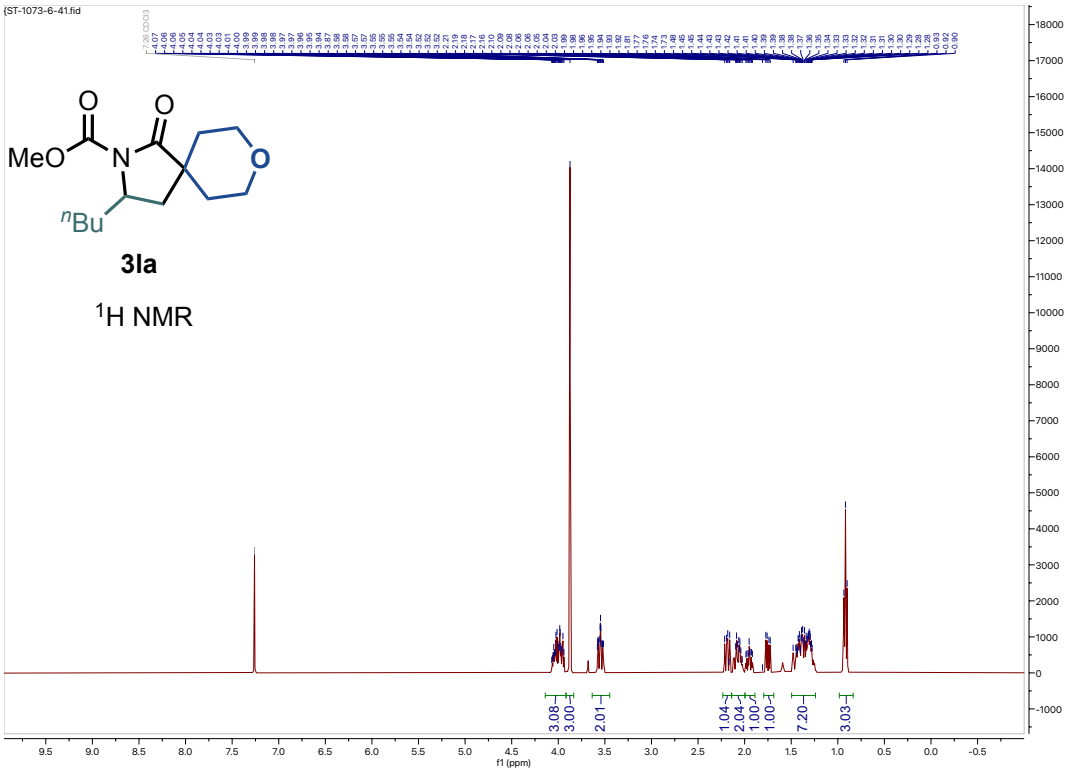

ST-1073-2-29.2.fid
179.27
153.02
77.20 CDCl3
55.25
53.99
46.14
34.20
34.00
34.32
29.26
29.06
26.43
22.68
22.53
22.01
14.17
MeO
O
O
N
nBu
3ia
13C NMR
f1 (ppm)


ST-1074-4-28.1.fid
MeO
O
O
N
nBu
3ja
1H NMR
1.00
3.00
4.05
2.03
4.32
5.88
3.10
f1 (ppm)


ST-1074-4-28.2.fid
179.92
152.90
77.20 CDCl3
66.83
63.39
53.63
39.11
38.00
34.54
34.05
27.70
25.96
25.56
22.66
14.17
MeO
O
O
N
nBu
3ja
13C NMR
f1 (ppm)

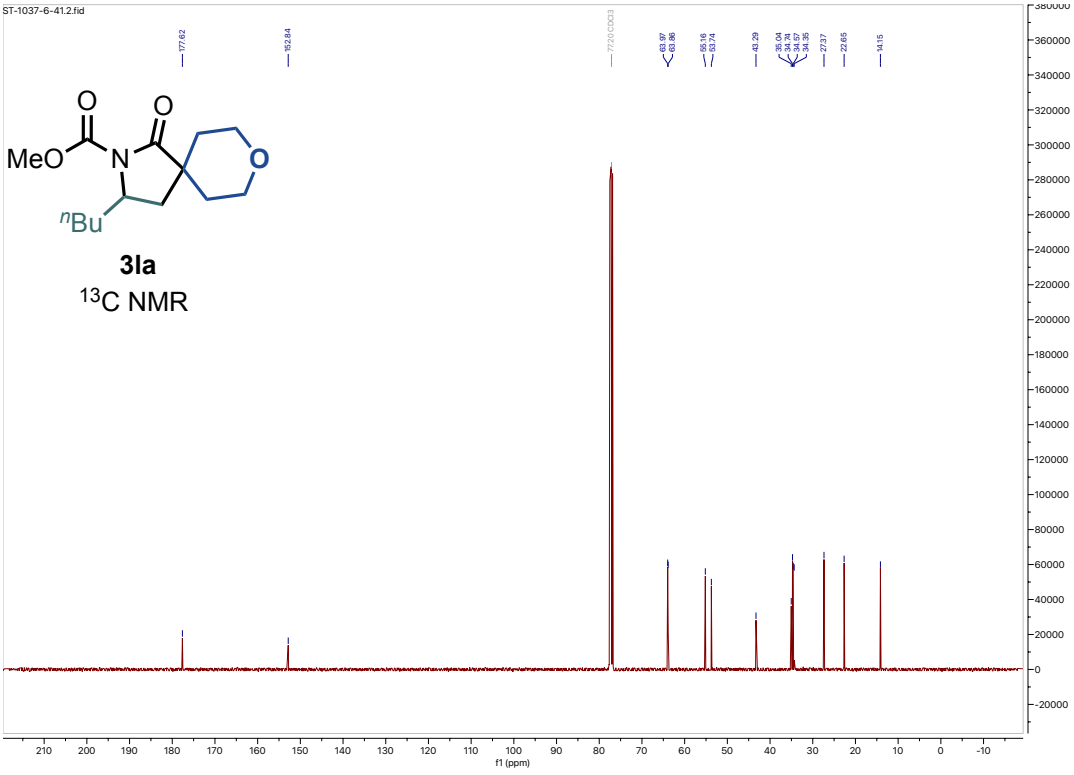

ST-1073-5-2-29.1.fid
MeO
N
O
O
nBu
3ka
1H NMR
1.00
3.00
2.05
7.30
5.30
3.15
f1 (ppm)


ST-1073-5-2-29.2.fid
178.22
152.76
77.20 CDCl3
56.15
53.55
46.67
36.99
33.92
33.55
30.45
27.98
22.62
16.53
14.12
MeO
N
O
O
nBu
3ka
13C NMR
f1 (ppm)



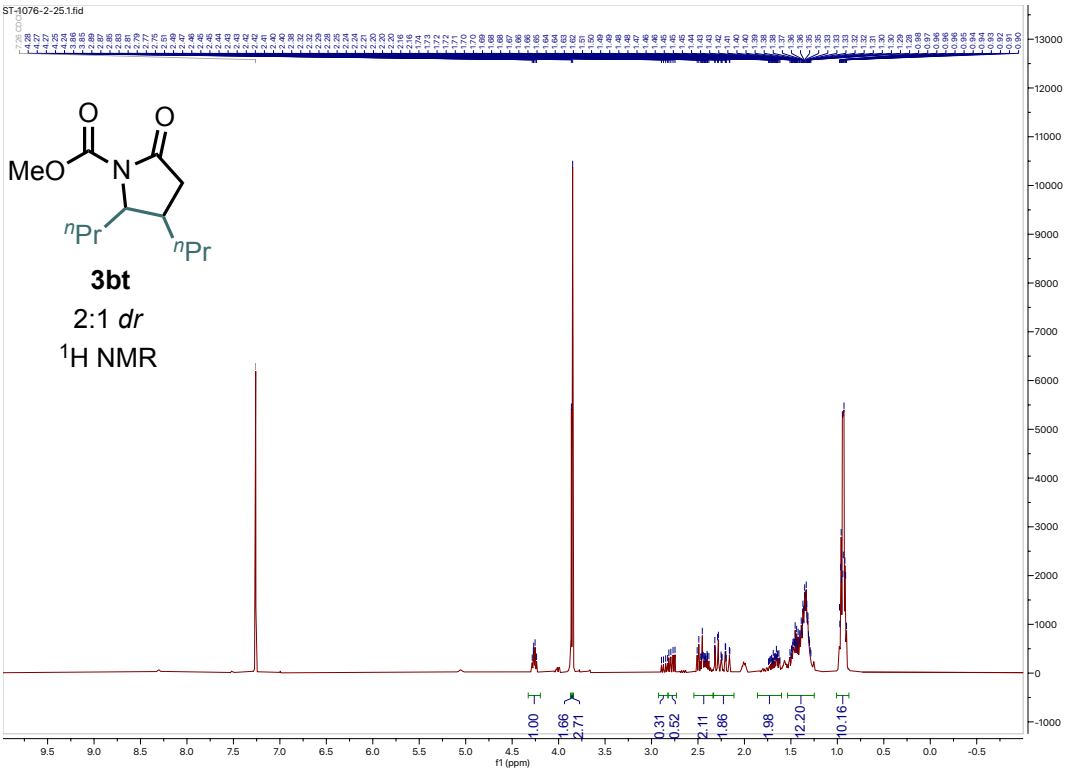
3la

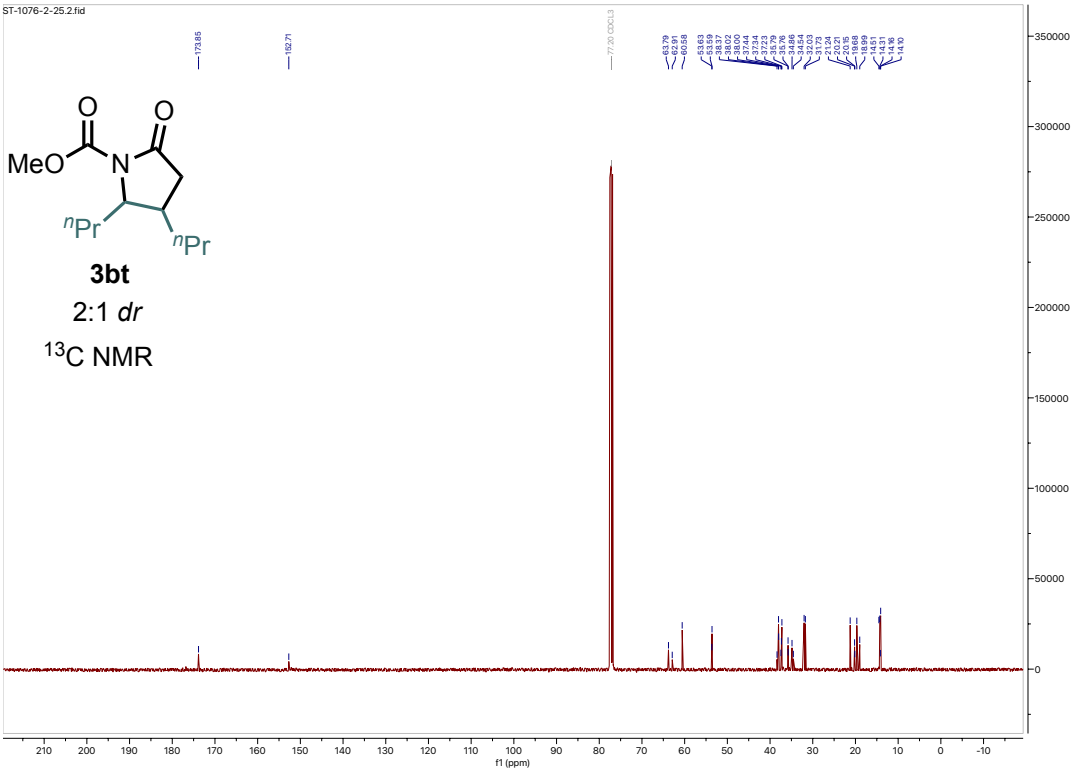
^{1}H NMR

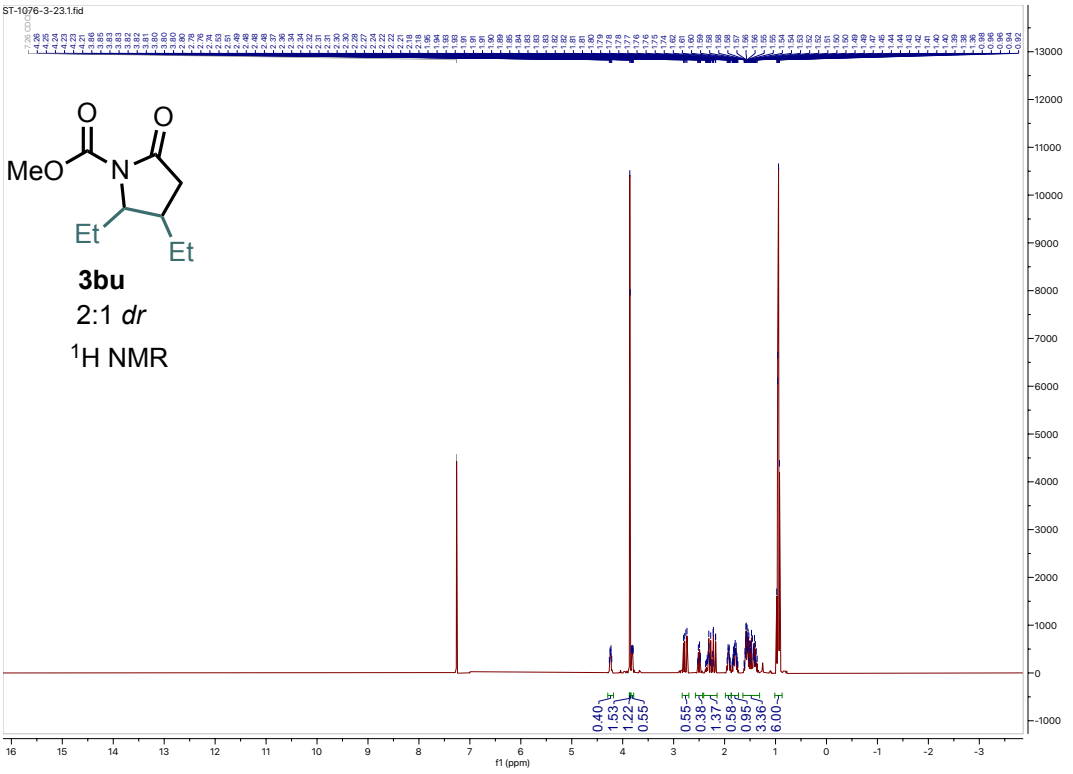


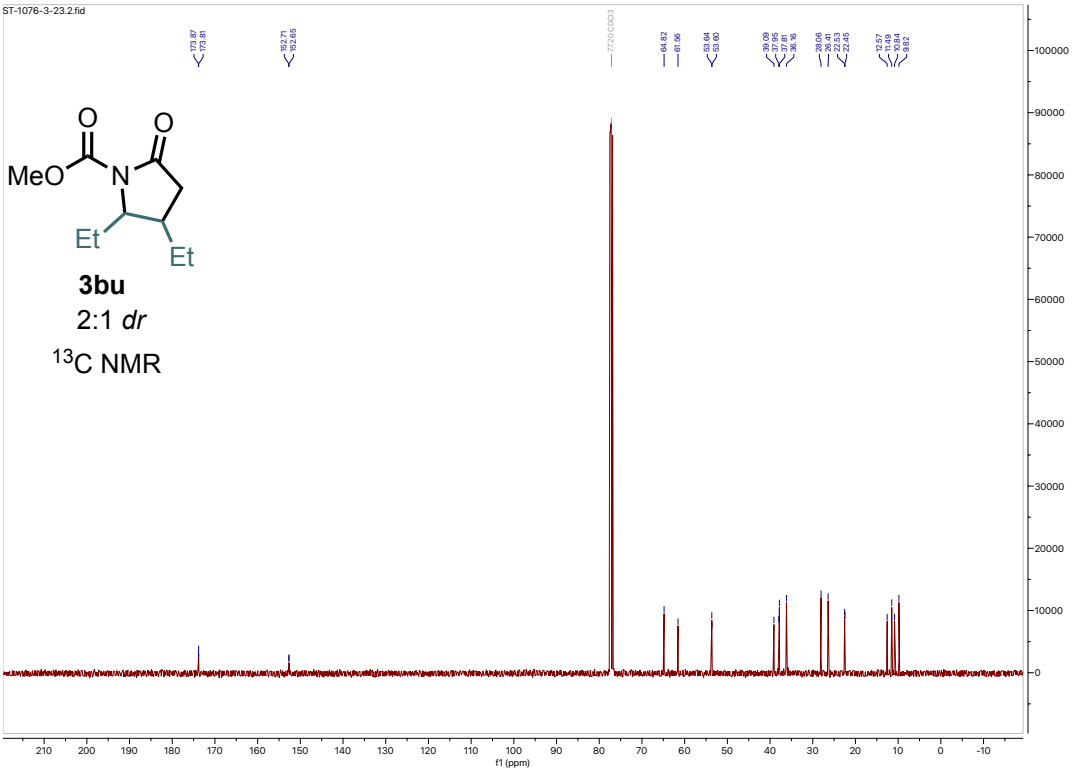
3la

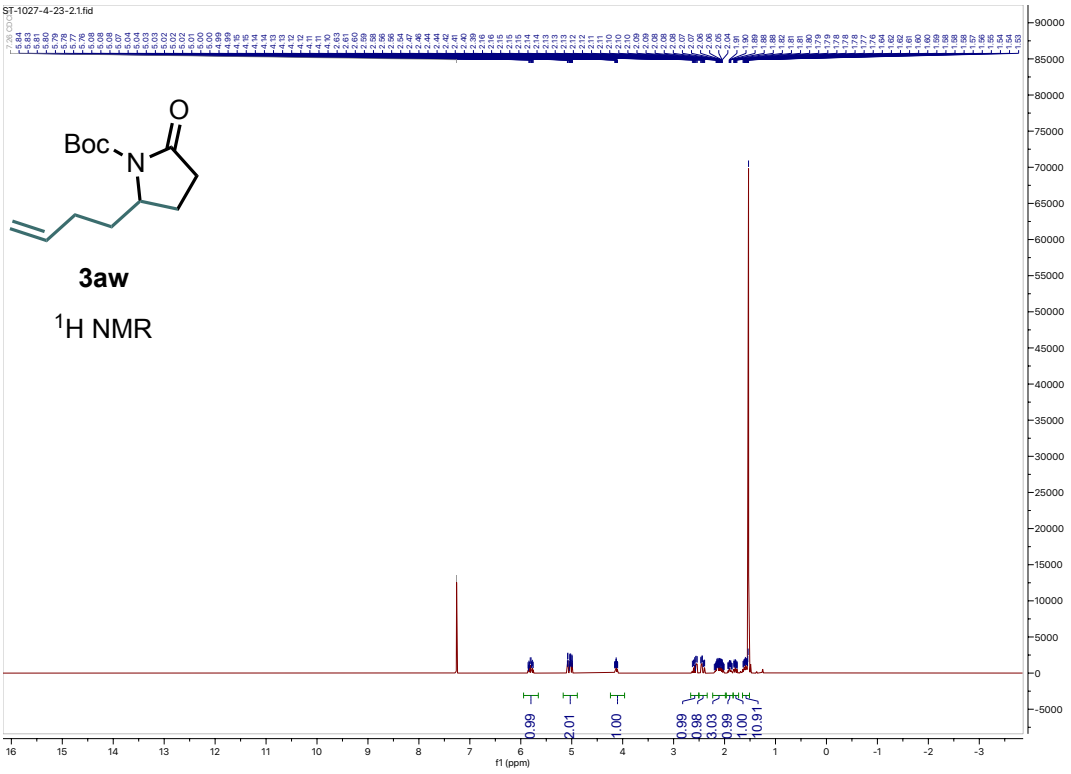
^{13}C NMR

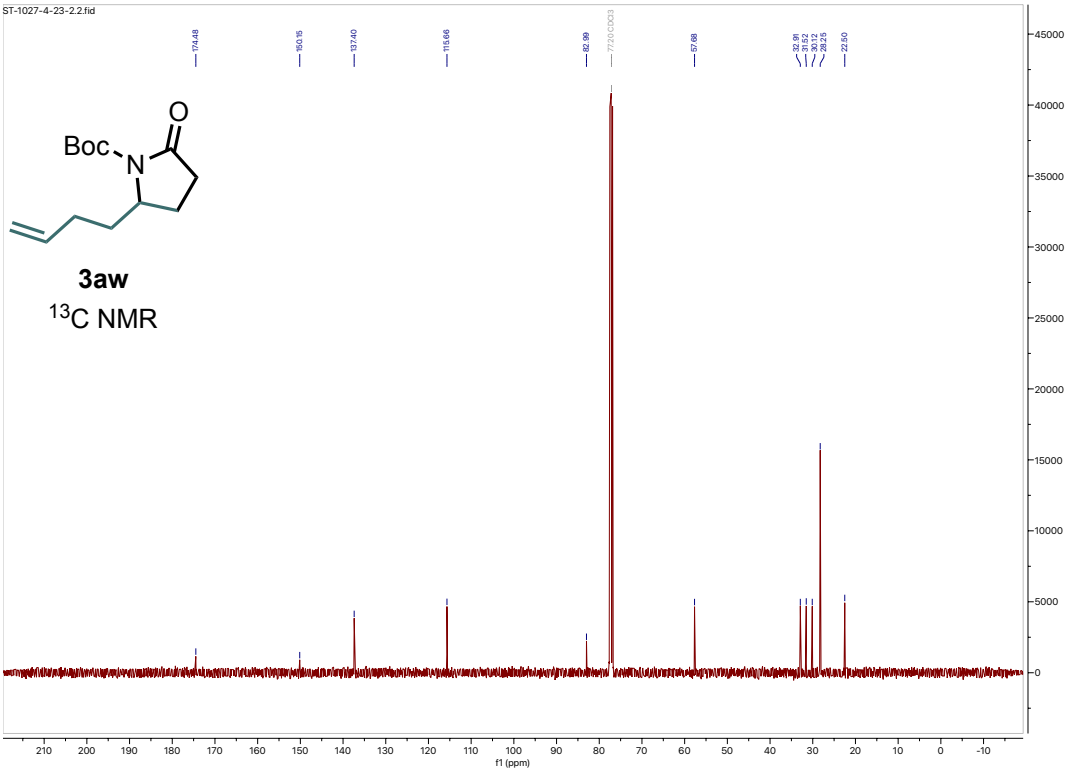

ST-1076-2-25.1.fid
MeO
N
O
O
nPr
nPr
3bt
2:1 dr
1H NMR
f1 (ppm)

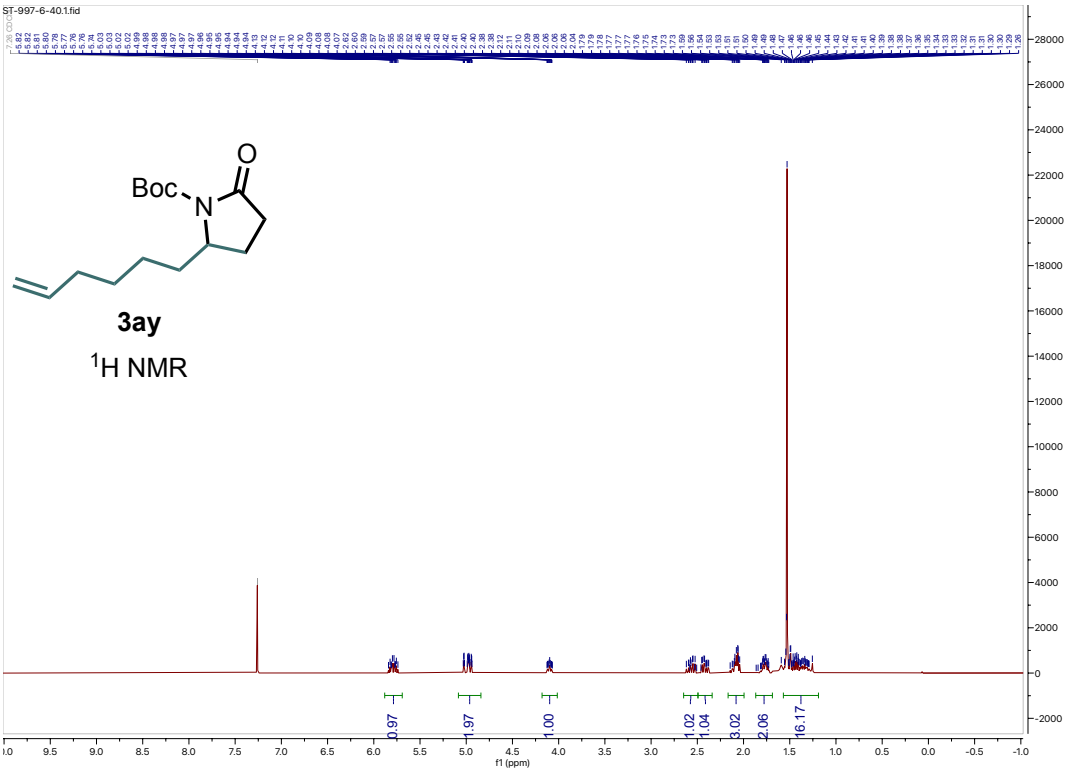

ST-1076-2-25.2.fid
MeO
N
O
O
nPr
nPr
3bt
2:1 dr
13C NMR
f1 (ppm)

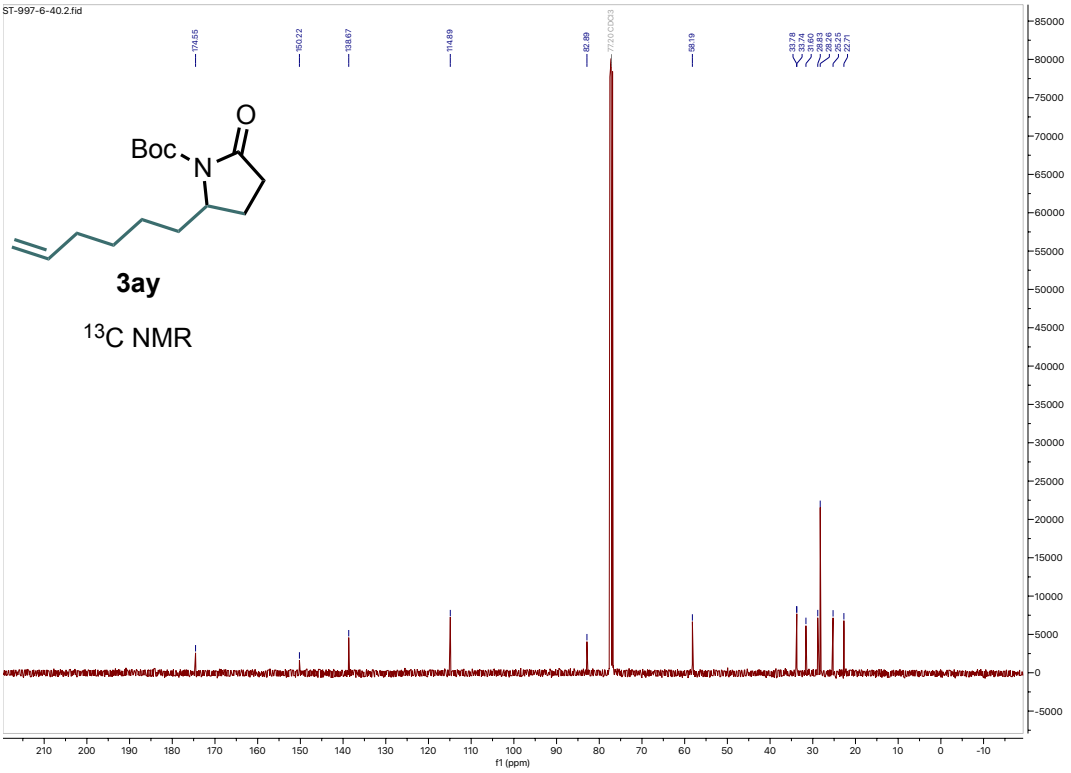

ST-1076-3-23.1.fid
MeO
N
O
O
Et
Et
3bu
2:1 dr
1H NMR
f1 (ppm)


ST-1076-3-23.2.fid
MeO
N
O
O
Et
Et
3bu
2:1 dr
13C NMR
f1 (ppm)


ST-1027-4-23-2.1.fid
Boc
N
O
3aw
1H NMR
0.99
2.01
1.00
0.99
0.98
3.03
0.99
1.00
10.91
f1 (ppm)


ST-1027-4-23-2.2.fid
174.48
160.15
137.40
115.66
82.99
77.20 CDCl3
57.68
32.91
31.52
30.12
28.25
22.50
Boc
N
O
3aw
13C NMR
f1 (ppm)


ST-997-6-40.1.fid
Boc
N
O
3ay
1H NMR
0.97
1.97
1.00
1.02
1.04
3.02
2.06
16.17
f1 (ppm)


ST-997-6-40.2.fid
174.55
150.22
138.67
114.89
82.89
77.20 CDCl3
58.19
33.78
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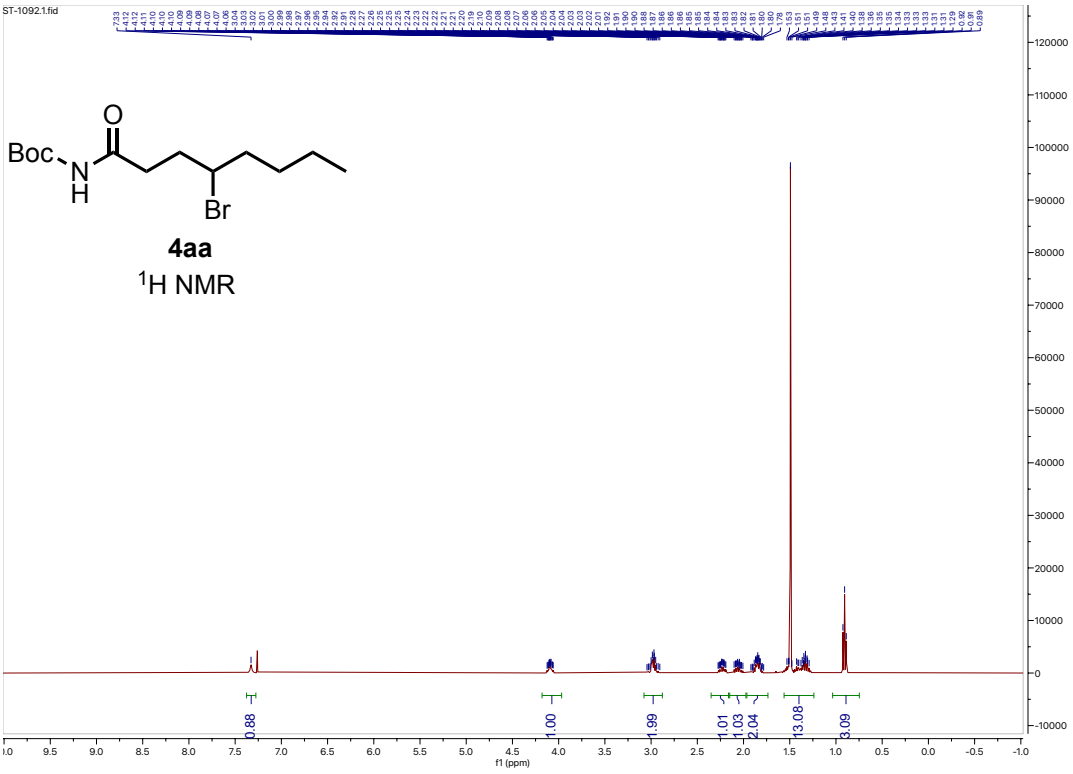
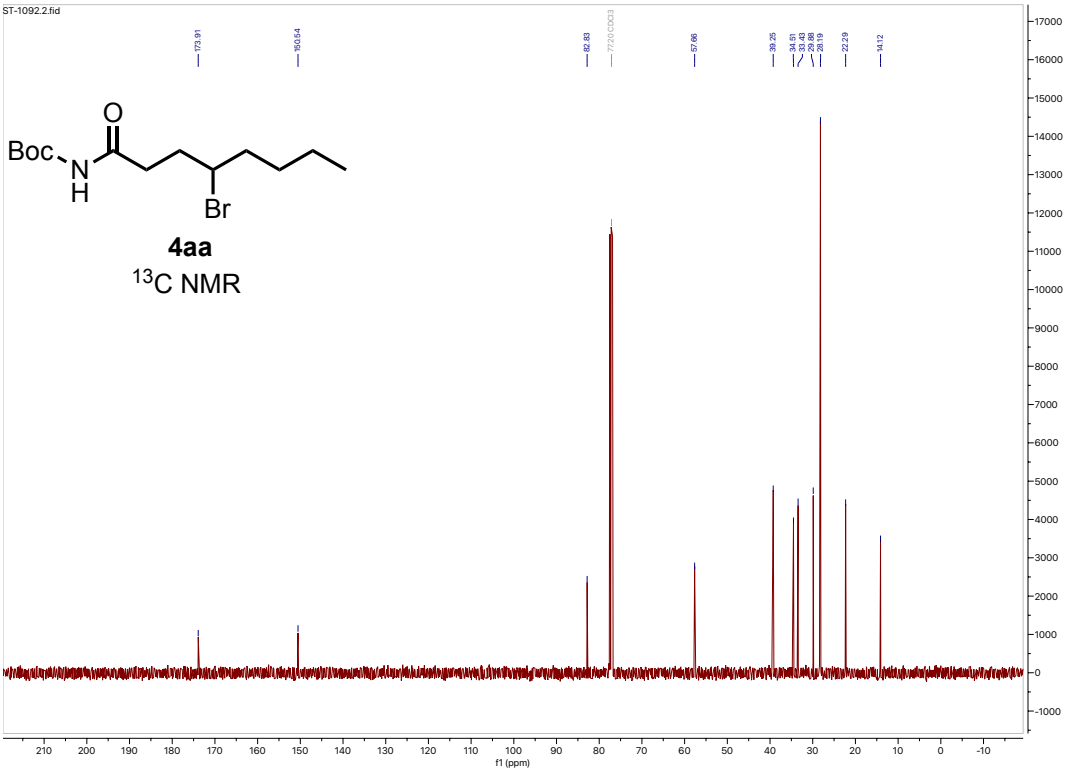
Boc
N
O
3ay
13C NMR
f1 (ppm)


ST-1092.1.fid
Boc
N
H
O
Br
4aa
1H NMR
0.88
1.00
1.99
1.01
1.03
2.04
13.08
3.09
f1 (ppm)


ST-1092.2.fid
173.91
150.54
82.83
77.20 CDCl3
57.66
39.25
34.51
34.43
29.88
28.19
22.29
14.12
Boc
N
H
O
Br
4aa
13C NMR
f1 (ppm)