Electronic Supplementary Material (ESI) for Green Chemistry.

Electronic Supplementary Information

Facile, green, and functional group-tolerant reductions of carboxylic acids…*in, or with, water*

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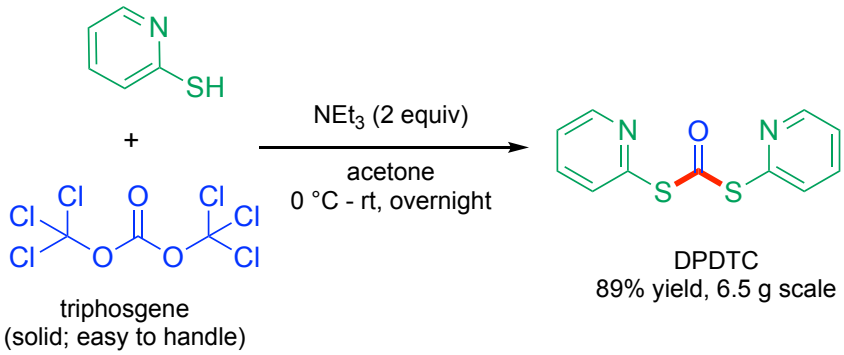
1. General Information

Reagents and chemicals were purchased from Sigma-Aldrich, Combi-Blocks, Alfa Aesar, Acros Organics, or TCI Chemicals and used without further purification. Carbonyl iron powder (CIP) used for reductions was 99.9% purity, R10 grade, with average particle size of 2.5-3.5 μm. This material was stored in air with no special precautions. Deuterated solvents were purchased from Cambridge Isotopes Laboratories. TPGS-750-M[1] and Coolade[2] are either prepared or supplied by PHT International (TPGS-750-M also available from Sigma-Aldrich catalog #733857). All commercially available metal salts, organic compounds were used as received from commercial suppliers: Sigma Aldrich, Strem, Combi-Blocks, Enamine, Fluorochem, Alfa Aesar, Acros and ChemBridge. $NiCl_2$(dme), $NiBr_2$(dme) (98%), 4,4′-di-*t*-butyl-2,2′-dipyridyl (dtbbpy, 98%) and diphenylsilane (Ph_2SiH_2, 97%) were purchased from Combi Blocks Inc., TCI Chemicals and Sigma Aldrich and used as received. The desired 2 wt % of surfactant solution in HPLC water was prepared by dissolving 2 g of surfactant to 98 g of HPLC water (which was degassed with argon prior to use) and stored under argon. Thin-layer chromatography (TLC) was performed using Silica Gel 60 F254 plates (Merck, 0.25 mm thick). Flash chromatography is performed either manually or in an automated Biotage system using Silica Gel 60 (Silicycle, 40-63 nm). GCMS data were recorded on a 5975C Mass Selective Detector, coupled with a 7890A Gas Chromatograph (Agilent Technologies). ^1H, ^{13}C and ^{19}F NMR spectra were recorded on either a Bruker Avance III HD 400 MHz (400 MHz for ^1H, 100 MHz for ^{13}C), a Bruker Avance NEO 500 MHz (500 MHz for ^1H, 125 MHz for ^{13}C, 470 MHz for ^{19}F) or on a Varian Unity Inova 500 MHz (500 MHz for ^1H, 125 MHz for ^{13}C); DMSO-d_6, $CDCl_3$, or CD_3OD was used as NMR solvent. Residual peaks for DMSO in DMSO-d_6 (^1H = 2.50 ppm, ^{13}C = 39.51 ppm), $CHCl_3$ in $CDCl_3$ (^1H = 7.26 ppm, 13C = 77.26 ppm), CH_3OH in CD_3OD (^1H = 3.31 ppm, ^{13}C = 49.00 ppm) have been assigned. The chemical shifts are reported in parts per million (ppm), the coupling constants *J* values are given in Hertz (Hz). The peak patterns are indicated as follows: bs, broad singlet; s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet. HRMS were recorded on a Waters Micromass LCT TOF ES+ Premier mass spectrometer using ESI ionization.

2. Synthesis of di-2-pyridyldithiocarbonate (DPDTC)



Scheme S1. Synthesis of DPDTC.

All glassware was flame dried. To a 500 ml round-bottom flask equipped with a PTFE-coated magnetic stir bar was added 2-mercaptopyridine (6 equiv, 60 mmol, 6.67 g), then the flask was sealed with a rubber septum and anhydrous acetone (100 mL) was added via syringe under a positive flow of argon, followed by anhydrous Et_3N (6 equiv, 60 mmol, 8.36 mL) and the solution was stirred until all components were fully dissolved. An ice bath was used to cool the resulting solution, an argon balloon was affixed to the septum via a needle, then a solution of triphosgene (1 equiv, 10 mmol, 2.967 g) in acetone (12.5 mL) was slowly added over the course of 15 min. The ice was replaced as needed to keep the solution cool during addition of triphosgene to prevent excessive generation of phosgene gas. Upon full addition, triethylammonium chloride was observed to precipitate. The reaction was allowed to warm to room temperature and stir overnight. Upon completion, the septum was removed inside a fume hood and allowed to expel any excess phosgene gas, then the reaction mixture was filtered to remove triethylammonium chloride, and the filtrate was concentrated in vacuo to afford a crude oil containing crystals of remaining triethylammonium chloride. The crude residue was redissolved in EtOAc in 10 mL portions and filtered into a separatory funnel. The combined organic extracts were washed with saturated aqueous $NaHCO_3$ (100 mL), followed by DI water (100 mL), followed by saturated brine (100 mL). The organic layer was separated and dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo and residual solvent was removed under high vacuum overnight to afford DPDTC as a light-yellow solid (6.596 g, 89% yield).

Caution: Triphosgene is acutely toxic and releases toxic phosgene gas on contact with moisture. It should be handled on small scale in a fume hood or glove box and weighed out using a pre-weighed, tightly sealed container.

Note: Commercial sources of inexpensive 2-mercaptopyridine may require purification prior to use due to the presence of the derived disulfide. This can be accomplished via recrystallization from EtOAc.

3. General Procedures

3.1. Synthesis of S-2-pyridyl thioesters from carboxylic acids: Procedure 1

3.1.1. Procedure 1A

Di-2-Pyridyl-di-thio Carbonate (DPDTC)

Scheme S2. Synthesis of thioesters from carboxylic acids.

i. For reduction to aldehydes

Reaction set up: In a 1-dram vial with a magnetic stir bar was added the carboxylic acid (1 mmol) and DPDTC (1.1 mmol) and the resulting mixture was allowed to stir at 65 °C until full consumption of the acid was observed by TLC or GC-MS. After completion of the reaction, a small amount of EtOAc was added to the reaction mixture and it was subjected to column chromatography with EtOAc/hexanes or acetone/hexanes as eluent to obtain the pure thioester.

ii. For reduction to alcohols

Reaction set up: In a 1-dram vial with a magnetic stir bar was added the carboxylic acid (1 equiv) and DPDTC (1.1 equiv) and the mixture was allowed to stir at 65 °C until full consumption of the

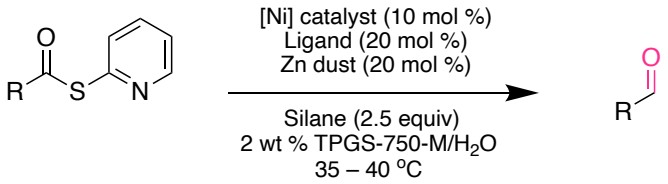
acid was observed by TLC or GC-MS. After completion, the crude thioester was carried forward as is to the alcohol in a 1-pot fashion (*see Procedure-7*).

(Note: In certain cases, {for syntheses of compounds **49, 50 and 51**} EtOAc (2 M global concentration) and 10 mol % DMAP were added for solubility reasons along with activation of DPDTC to facilitate formation of the thioester).

3.1.2. Procedure 1B

The procedure was adapted from Brown and coworkers.[3] In a 3-dram vial with a magnetic stir-bar was charged with dry acetonitrile (0.25 M global concentration), 2,2'-dipyridyl disulfide (1.1 equiv) and triphenylphosphine (1.5 equiv) and the solution was allowed to stir at rt for 10-15 min. To this solution was added the carboxylic acid (1 mmol, 1 equiv) and the mixture was allowed to stir at rt for 16 h. The reaction mixture was then concentrated *in vacuo*, after which the crude material was purified by flash chromatography to give pure thioester.

3.2. Optimization for reduction to aldehydes: Procedure 2



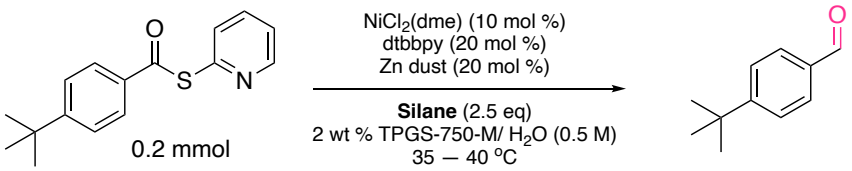
Scheme S3. Synthesis of aldehydes from thioesters.

Reaction set up: In a 1-dram vial with a magnetic stir bar was added the thioester (0.2 mmol) and ligand (20 mol %). The vial was capped and taken to the glovebox wherein the Ni catalyst (10 mol %) and Zn dust (20 mol %) were added. After removal from the glovebox, the vial was purged with argon for 10 min during which the silane (2.5 equiv) was added followed by the addition of 2 wt % TPGS-750-M/H_2O solution (0.4 mL). The vial was sealed with Parafilm, and the reaction was stirred at 1200 rpm at 35-40 °C until completion (determined by TLC or GC-MS) after which the reaction mixture was extracted 3 times with EtOAc and then concentrated *in vacuo*.

Subsequently, 1 mL of $CDCl_3$ was added followed by the addition of 1,3,5-trimethoxybenzene (5-7 mg) as internal standard and the sample analyzed by ^1H NMR (20s relaxation delay; aldehyde peak integrated *vs*. internal standard).

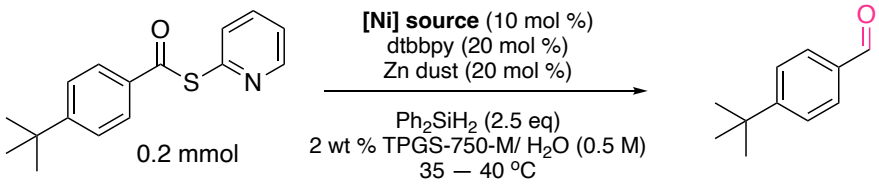
3.2.1. Optimization of Reaction conditions using Procedure 2

Table S1. Screening of Silane Sources.



Entry	Silane	Yield (NMR)
1.	triethylsilane	trace
2.	polymethylhydrosiloxane (PMHS)	trace
3.	triisopropylsilane	15%
4.	**diphenylsilane**	**48%**
5.	phenylsilane	26%
6.	TMDS	trace

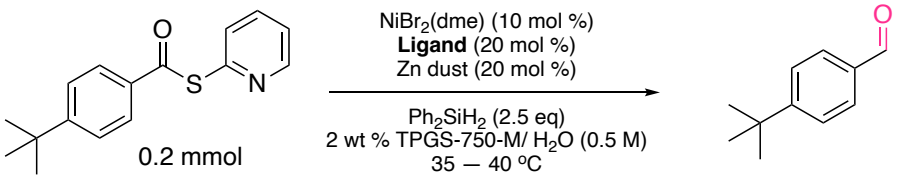
Table S2. Screening of Nickel catalyst.



Entry	Nickel	Yield (NMR)
1.	$Ni(acac)_2$	traces
2.	$NiCl_2(dme)$	43%
3.	$Ni(COD)_2$	15%
4.	**$NiBr_2(dme)$**	**52%[a]**
5.	$NiBr_2(Diglyme)$	46%
6.	$NiCl_2.6H_2O$	38%

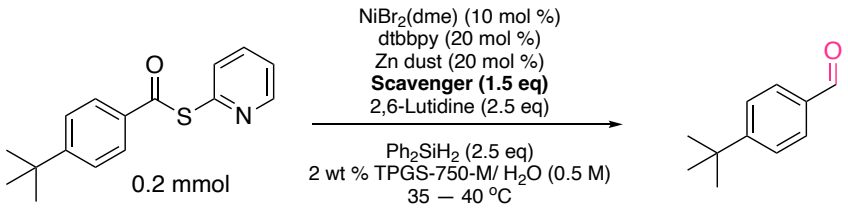
[a] The reaction was isolated with 5-10% EtOAc/Hexanes to give 46% of the aldehyde.

Table S3. Ligand Screening.



Entry	Ligand	Yield (NMR)
1.	bipy	30 %
2.	**dtbbpy**	**52%**
3.	1,10-phenanthroline	13%
4.	bathophenanthroline	15%
5.	4,4'-dimethoxybipyridine	40%
6.	Terpy	10%
7.	1,2,7,8-tetramethylphenanthroline	16%

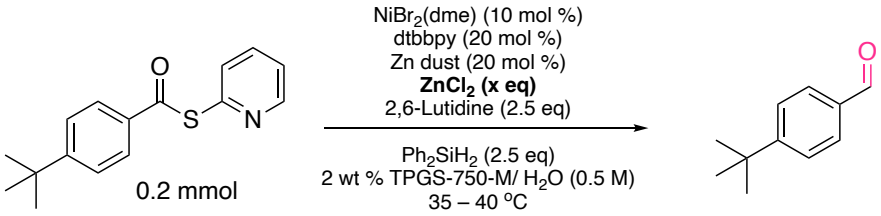
Table S4. Scavenger Screening.



Entry	Scavenger	Yield (NMR)
1	**$ZnCl_2$**	**95%[a]**
2	$ZnBr_2$	95%
3	$Zn(OAc)_2$	80%
4	CuTC[b]	no product
5	CuMeSal	20%
6	*N*-ethyl maleimide	no product
7	Phenyl Isocyanate	no product

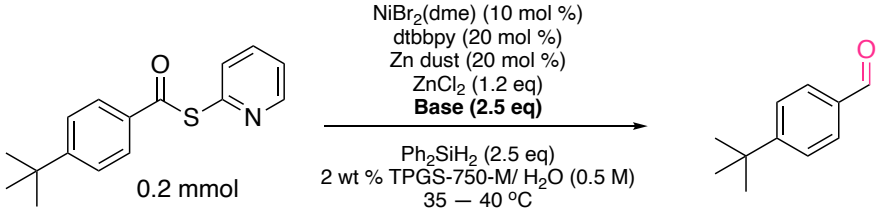
[a] The product was purified to give 92% isolated yield of the aldehyde. [b] CuTC: copper thiophene-2-carboxylate.

Table S5. Screening of Scavenger Loading.



Entry	Scavenger loading	Yield (NMR)
1.	1.5 equiv	95%
2.	**1.2 equiv**	**94%**
3.	1 equiv	87%
4	0.7 equiv	82%

Table S6. Base Screening.

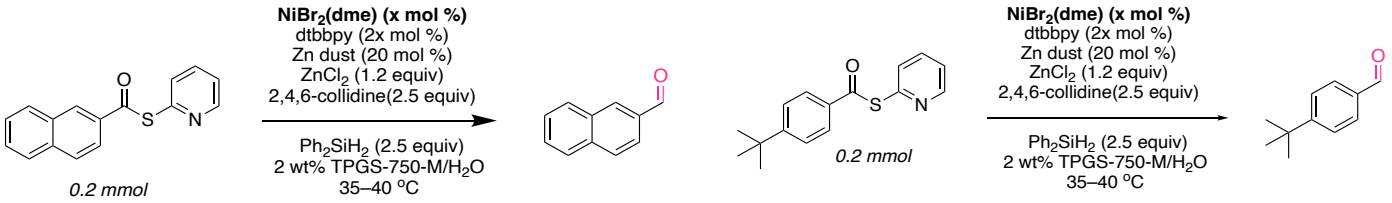


Entry	Base	Yield (NMR)
1.	2,6-lutidine	95%
2.	**2,4,6-collidine**	**95%**[a]
3.	Et_3N	88%
4.	DIPEA	80%
5.	*N*-methylmorpholine	90%

[a] Use of collidine led to slightly better stirring and emulsification as compared to lutidine.

Note: Inorganic bases were not screened because they formed a precipitate with the byproduct, 2 mercaptopyridine, resulting in poor stirring of the reaction mixture.

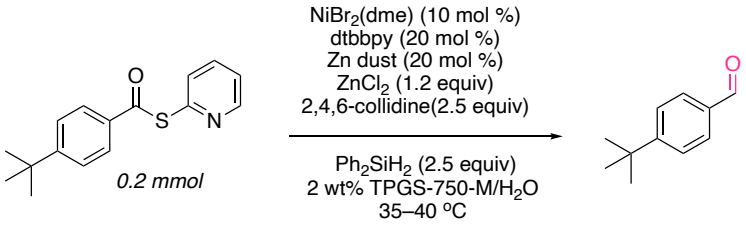
Table S7. Screening of Catalyst Loading (on 2 different substrates).



Entry	$NiBr_2$(dme) (mol%)	Yield (2-NpCHO)*	Yield (4-*t*-butyl PhCHO)*
1	**10**	**90%**	**96%**
2	7.5	81%	93%
3	**5**	**76%**	**92%**
4	2.5	~60%	78%

*NMR yields using 1,3,5-trimethoxybenzene as internal standard.

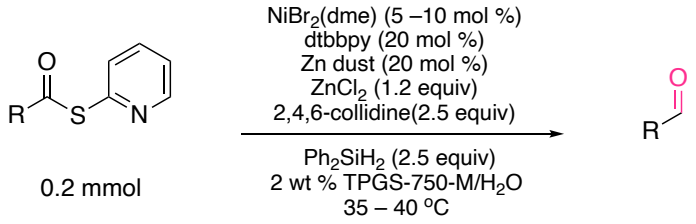
Table S8. Additional control experiments.



Entry	Deviation from optimized conditions	Yield (%)[a]
1.	none	92
2.	without $NiBr_2$(dme) / Dtbbpy	0
3.	no Dtbbpy	0
4.	no Zn	0
5.	no $ZnCl_2$	46
6.	without 2,4,6-collidine (hydrolysis of the thioester was observed)	75
7.	at 60 °C	85

[a] Isolated yields.

3.3. General procedure for reduction of thioesters to aldehydes: Procedure 3



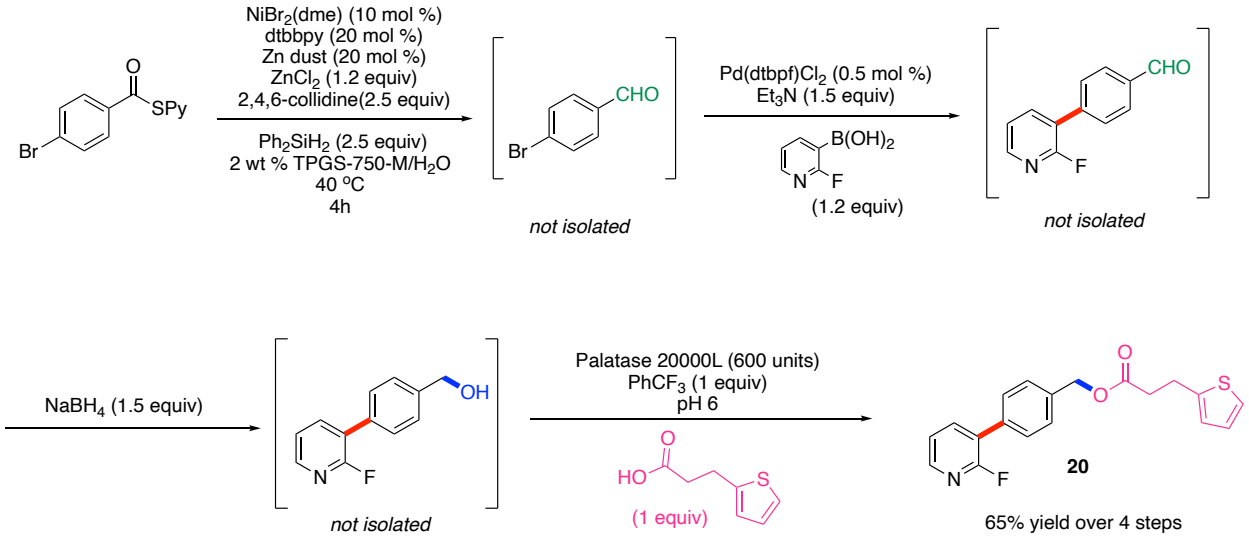
Scheme S4. Reduction of thioesters to aldehydes.

Reaction set up: In a 1-dram vial with a magnetic stir bar was added the thioester (0.2 mmol) and 4,4'-di-*t*-butyl bipyridine (20 mol %). The vial was capped and was taken to the glovebox wherein $NiBr_2$(dme) (5-10 mol % based on the substrate), Zn dust (20 mol %) and zinc chloride (1.2 equiv) were added. After removal from the glovebox, the vial was purged with argon for 10 min during which diphenylsilane (2.5 equiv) was added followed by the addition of 2,4,6-collidine (2.5 equiv) and 2 wt % TPGS-750-M/H_2O solution (0.4 mL; 0.5 M). The vial was sealed with Parafilm and the reaction was stirred at 1200 rpm at 35-40 °C until completion (determined by TLC or GC-MS). Upon completion, the reaction mixture was diluted with 1 M HCl and extracted 3 times with EtOAc, after which the mixture was concentrated *in vacuo*. Column chromatography using EtOAc/hexanes as eluent afforded the pure product.

Note:

- *For non-polar aldehydes, the first 2 column volumes were run in 10-20% DCM/hexanes or just pure hexanes to separate the non-polar impurities and then the desired solvent system was used.*
- *$NiBr_2$(dme) is bench stable and can also be used outside of the glove box.*

3.4. General procedure for the 4-step, 1-pot sequence: Procedure 4



Scheme S5. 4-step-1-pot sequence to synthesize **20**.

Step 1: Reduction of a thioester to the corresponding aldehyde

In a 2-dram vial with a magnetic stir bar was added *S*-(12yridine-2-yl) 4-bromobenzothioate (0.5 mmol, 147 mg) and dtbbpy (20 mol %, 27 mg). The vial was capped and was taken to the glovebox into which was added $NiBr_2$(dme) (10 mol %, 15.4 mg), Zn dust (20 mol %, 6.5 mg) and zinc chloride (1.2 eq, 82 mg) were added. The vial was removed from the glovebox after which it was purged with argon for 10 min during which time diphenylsilane (2.5 equiv, 232 µL) was added followed by the addition of 2,4,6-collidine (2.5 equiv, 165 µL) and 2 wt % TPGS-750-M/H_2O solution (1 mL, 0.5 M). The vial was sealed with Parafilm and the reaction was stirred at 1200 rpm at 35-40 °C until completion (determined by TLC or GC-MS). Upon completion, the reaction mixture was used as it is without further purification.

Step 2: Suzuki-Miyaura Reaction

To the reaction mixture from step 1 was added Pd(dtbpf)Cl_2 (0.5 mol %, 1.6 mg), (2-fluoropyridin-3-yl)boronic acid (1.5 equiv, 105.7 mg), Et_3N (2 equiv, 140 µL), and the mixture was allowed to stir at 45 °C (ca. 1 h) until completion as observed by TLC or GC-MS. The crude mixture was used in the next step without further purification.

Step 3: Reduction to the alcohol

The crude reaction mixture from step 2 was cooled in an ice bath and then treated with sodium borohydride (1.5 equiv, 28.5 mg). Evolution of gas was observed. The reaction was then allowed to stir at rt for 20 min until completion, as monitored by TLC.

Step 4: Lipase-catalyzed esterification

The reaction mixture from step 3 was acidified to pH 6 using conc. HCl, after which 2-thiophene propanoic acid (1 equiv, 78.1 mg) was added. The lipase (600 units) was added followed by the addition of trifluoromethyltoluene ($PhCF_3$, 1 equiv, 61 μL) and the reaction was allowed to stir at 30 ^oC for 24 h. After completion, the reaction was extracted with 2 x 1 mL of EtOAc. The organic layer was dried with anhydrous Na_2SO_4, filtered through a pad of Celite to remove residual surfactant and salts, and concentrated *in vacuo*. It was subjected to column chromatography using 25% EtOAc/hexanes to obtain the final product (**20**) as a light brown oil (yield: 65%; 110 mg).

3.5. General procedure for 2-step, 1-pot synthesis of products 15-19: Procedure 5

Step 1: Suzuki-Miyaura reaction

In a 1-dram vial with a magnetic stir bar was added *S*-(pyridine-2-yl) 3-iodobenzothioate (0.5 mmol, 170.5 mg), boronic acid (1.2 equiv) and $Pd(dtbpf)Cl_2$ (0.5 mol %, 1.6 mg). The vial was evacuated and backfilled 3 times with argon. To the vial was added Et_3N (1.5 equiv) followed by the addition of 2 wt % TPGS-750-M/H_2O solution (0.5 M, 1 mL). The reaction was stirred at 40 ^oC until completion (by TLC or GC-MS). The crude product was then extracted with EtOAc and washed with 1 M HCl followed by washing with 10% Na_2CO_3 to remove residual hydrolyzed thioester. The organic phase was then concentrated under reduced pressure and used directly in the next step.

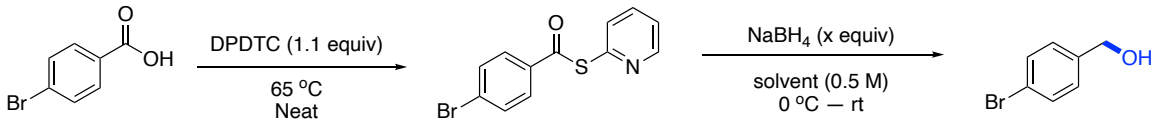
Step 2: Reduction to the aldehyde

To the crude product from step 1 was added 4,4'-di-*t*-butylbipyridine (20 mol %, 27 mg) and taken into the glovebox wherein $NiBr_2$(dme) (10 mol %, 15.4 mg), Zn dust (20 mol %, 6.5 mg) and zinc

chloride (1.2 equiv, 82 mg) were added. The vial was removed from the glovebox and purged with argon for 10-15 min after which time diphenylsilane (2.5 equiv, 232 µL) and 2,4,6-collidine (2.5 equiv, 165 µL) were added to the vial, followed by the addition of 2 wt % TPGS-750-M/H_2O solution (0.5 M). The reaction was allowed to stir at 40 °C at 1200 rpm until completion as determined by TLC or GC-MS. The crude product was extracted 3 times with EtOAc and then concentrated *in vacuo* and then subjected to flash chromatography using EtOAc/hexanes as eluent.

3.6. Optimization of reduction of carboxylic acids to alcohols: Procedure 6

4-Bromobenzoic acid was chosen as the model substrate to optimize conditions.



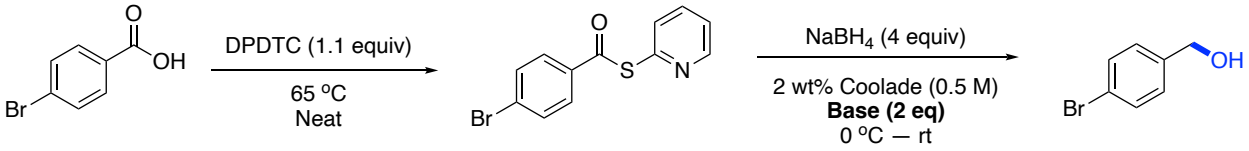
Scheme S6. Model reaction for reduction of acids to alcohols.

Reaction set up: All optimization reactions were carried out on a 0.25 mmol scale. *S*-(Pyridin-2-yl)-4-bromobenzothioate was synthesized from 4-bromobenzoic acid according to general procedure 1A (ii). Upon completion, the reaction vial was cooled to rt and solvent (0.5 M) was added. The vial was cooled to 0 °C using an ice bath after which sodium borohydride (4 equiv) was added slowly as a powder. Evolution of gas was observed. The reaction was warmed to rt and stirred until completion (by TLC or GC-MS), after which the reaction mixture was concentrated *in vacuo* to remove most of the solvent and 1 mL of EtOAc was then added. The reaction mixture was then neutralized with 1 M HCl solution, and the organic phase was separated and concentrated *in vacuo*, to which 1 mL of $CDCl_3$ was added followed by the addition of trimethoxybenzene as internal standard. The yields were determined by quantitative NMR using 20 s relaxation time (alcohol CH_2 peak was integrated with respect to the internal standard peak).

Note: Initial experiments were done using 2 wt % TPGS 750-M/H_2O[1] as the reaction medium. However, considerable foaming was observed from generation of hydrogen gas. Hence, the

medium was switched to 2 wt % Coolade[2] in water, after which the foaming was greatly reduced. 2 wt % Coolade solution was selected for preliminary screening experiments.

Table S8. Base screen using 2 wt % Coolade as the reaction medium.

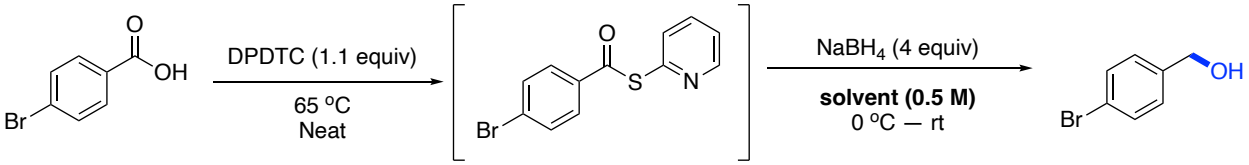


Entry[a]	Base (2 equiv)	Yield[b]
1.	Et_3N	85%
2.	$NaHCO_3$	72%
3.	*N*-methylmorpholine	76%
4.	DIPEA	86%
5.	2,6-lutidine	83%

[a] Reactions carried out on a 0.25 mmol scale; global concentration: 0.5 M. [b] NMR yields.

Note: *Formation of unwanted side products was observed when reactions were carried out under basic conditions in water. One of the side products was the hydrolysis of the thioester back to the carboxylic acid, which could not be avoided. Therefore, several green solvents were screened.*

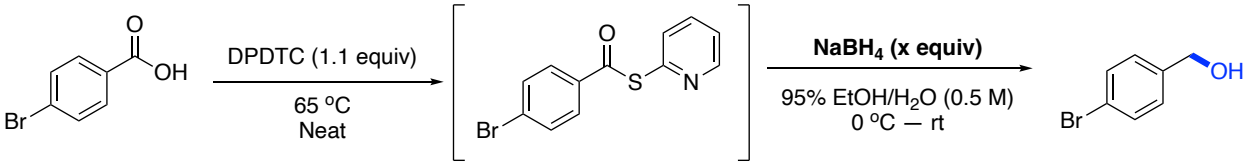
Table S9. Screening of green solvents.



Entry[a]	Solvent	Yield[b]
1.	MeOH	45%
2.	**95% EtOH/H_2O**	**94%**
3.	isopropanol	82%
4.	absolute EtOH	91%

[a] Reactions carried out on a 0.25 mmol scale, Reaction time: 3 h.; [b] Isolated yields.

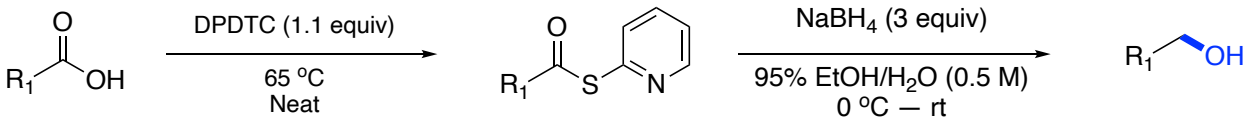
Table S10. Effect of equivalents of sodium borohydride.



Entry	Equivalents of $NaBH_4$	Yield*
1.	1.5	50%
2.	2	80%
3.	2.5	92%
4.	**3**	**98% (94%)** [a]
5.	4	quantitative

*NMR yields. [a]isolated yield.

3.7. General Procedure for reduction of carboxylic acids to alcohols: Procedure 7



Scheme S7. Reduction of carboxylic acids to alcohols.

Step 1; Thioester synthesis: In a 1-dram vial with a magnetic stir bar was added the carboxylic acid (1 equiv) and DPDTC (1.1 equiv) and the mixture was allowed to stir at 65 °C until full consumption of the acid was observed by TLC or GC-MS. After completion, the crude reaction mixture was carried forward without further purification for reduction to the alcohol in a 1-pot fashion.

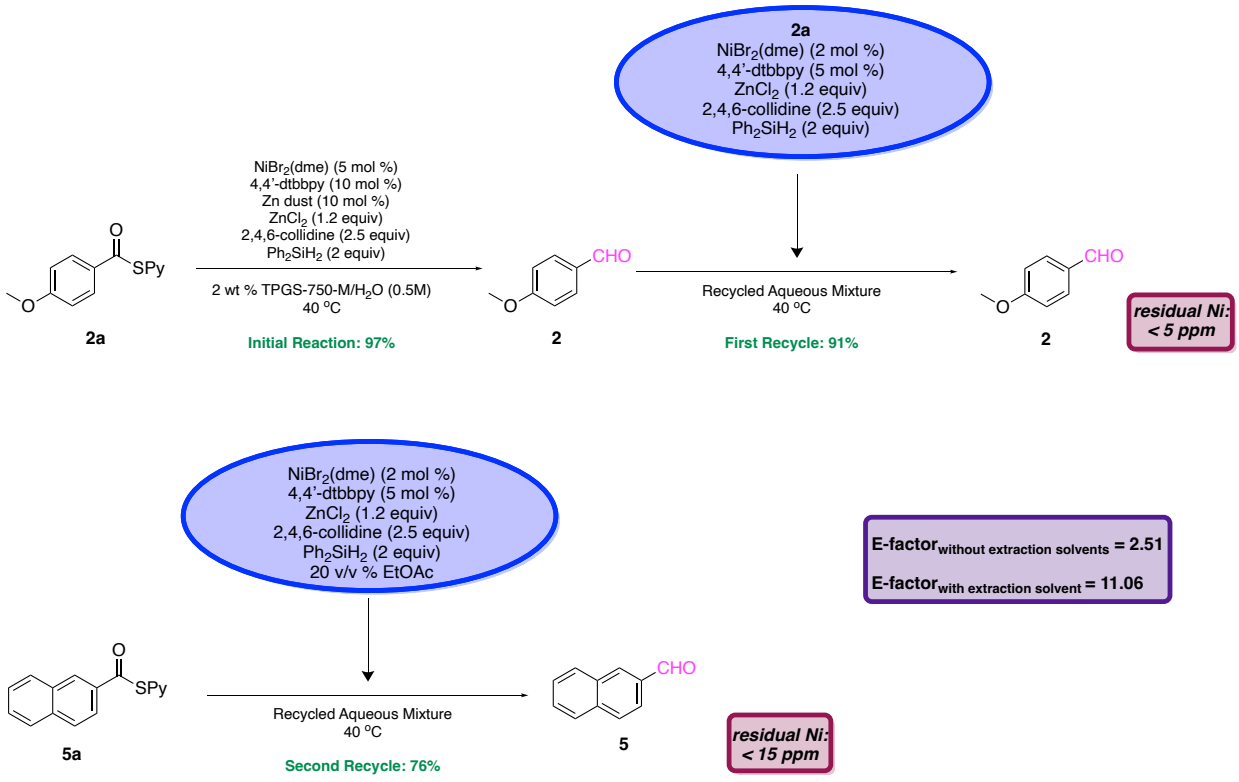
Note: *In some cases, EtOAc (2 M global concentration) and 10 mol % DMAP were added for solubility reasons and conversion of the carboxylic acid to the thioester (compounds* ***49, 50, 51****).*

Step 2; Reduction to the alcohol: To the crude product from the previous step was added 95% EtOH (0.5 M) and the vial was cooled to 0 °C using an ice bath. Sodium borohydride (3 equiv) was added (caution: slight evolution of gas was observed). The reaction vial was kept in the ice bath for 10 min, after which it was warmed to rt and allowed to stir until completion (by TLC or GC-MS). Typically, the reaction takes about 30 min but may take longer depending on the substrate. Upon completion, the reaction mixture was concentrated *in vacuo* to remove most of the ethanol. It was then neutralized with 1 M HCl, and the aqueous phase was extracted with EtOAc

(3 x 0.5 mL). The extracts were then concentrated *in vacuo* and the crude material purified using silica gel column chromatography using EtOAc/hexanes or acetone/hexanes as eluent.

4. E Factor calculations and aqueous recycling studies for reduction of thioesters to aldehydes.

4.1. Aqueous recycling studies



Scheme S8. Aqueous recycling studies.

Initial Reaction: To a 1-dram vial equipped with a magnetic stir bar was added thioester **2a** (0.5 mmol, 122.65 mg) and 4,4'-di-*t*-butylbipyridine (10 mol %, 13.4 mg). The vial was capped and was taken to the glovebox where $NiBr_2$(dme) (5 mol %, 7.7 mg), Zn dust (20 mol %, 6.5 mg), and zinc chloride (1.2 equiv, 81.8 mg) were added. The vial was then removed from the glovebox and purged with argon for 10 min during which diphenylsilane (2 equiv, 186 µL) was added followed by the addition of 2,4,6-collidine (2.5 equiv, 165 µL) and then 2 wt % TPGS-750-M/H_2O solution (0.5 M, 1 mL) was added. The vial was sealed with Parafilm and the reaction was stirred at 1200 rpm at 35-40 °C until completion (determined by TLC or GC-MS). Upon completion, the reaction

mixture was extracted two times with EtOAc (0.3 mL) and was then concentrated *in vacuo*, after which it was subjected to column chromatography using 33% EtOAc/hexanes as eluent to give 4-methoxybenzaldehyde (**2**) as a colorless liquid (66 mg, 97%).

First Recycle: To the 1-dram vial containing the 2 wt % TPGS-750-M aqueous phase used for the previous reaction (and extracted with EtOAc) was added fresh thioester **2a** (0.5 mmol, 122.65 mg) and fresh 4,4'-di-*t*-butylbipyridine (5 mol %, 6.7 mg). The vial was then capped and placed into a glovebox in which $NiBr_2$(dme) (2 mol %, 3.1 mg) and zinc chloride (1.2 equiv, 81.8 mg), were added. The vial was then purged with argon for 10 min during which diphenylsilane (2 equiv, 186 μL) was added followed by the addition of 2,4,6-collidine (2 equiv, 132 μL). The vial was then removed from the glovebox and sealed with Parafilm, after which the reaction was stirred at 1200 rpm at 35-40 ^oC until completion (determined by TLC or GC-MS). Upon completion, the reaction mixture was extracted two times with EtOAc (0.3 mL) and then concentrated *in vacuo*, and the crude material then subjected to column chromatography using 33% EtOAc/hexanes as eluent to give 4-methoxy benzaldehyde (**2**) as a colorless liquid (62 mg, 91%).

Second Recycle: To the 1-dram vial containing the 2 wt % TPGS-750-M aqueous phase used for the previous two reactions (and extracted with EtOAc twice) was added fresh thioester **5a** (0.5 mmol, 132.6 mg) and fresh 4,4'-di-*t*-butylbipyridine (5 mol %, 6.7 mg). The vial was capped and taken to the glovebox in which $NiBr_2$(dme) (2 mol %, 3.1 mg) and zinc chloride (1.2 equiv, 81.8 mg) were added. The vial was removed from the glovebox and then purged with argon for 10 min during which diphenylsilane (2 equiv, 186 μL) was added followed by the addition of 2,4,6-collidine (2 equiv, 132 μL) and EtOAc (20 v/v%, 0.2 mL). The vial was sealed with Parafilm and the reaction was stirred at 1200 rpm at 35-40 ^oC until completion (determined by TLC or GC-MS). Upon completion, the reaction mixture was extracted two times with EtOAc (0.3 mL) and was then concentrated *in vacuo*, and the crude material then subjected to column chromatography using 15% EtOAc/hexanes as eluent to give 2-naphthaldehyde (**5**) as a white solid (59.3 mg, 76%). At this point, the aqueous phase had become too saturated with salts and much of the water had been lost due to small scale extractions. Further recycling studies, therefore, were not pursued.

4.2. E Factor calculations

E Factor = (mass waste organics) / (mass product)

1. Ph_2SiH_2: 2 equiv per reaction. Therefore, 1 equiv extra over 3 reactions
   Total 3 equiv

   $Mass_{diphenylsilane}$ = *276 mg*

2. 2,4,6-collidine – 2.5 equiv per reaction. Therefore, 0.1 equiv extra reagent (1.2 equiv zinc chloride needs 2.4 equiv collidine to quench the HCl released).
   Total 0.3 equiv extra reagent.

   $Mass_{2,4,6\text{-}collidine}$ = *18.17 mg*

3. 2 Mercaptopyridine- 1 equiv released as a byproduct per reaction.
   Total 3 equiv

   $Mass_{2\text{-}Py\text{-}SH}$ = 166.5 mg

4. $Mass_{EtOAc}$ = 8.81 mg

5. $mass_{extraction\text{-}solvent}$ = 1620 mg

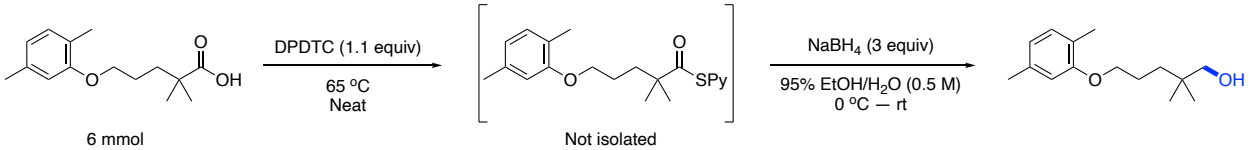
E $Factor_{without\ extraction\ solvent}$ = (*$Mass_{diphenylsilane}$ + $Mass_{2,4,6\text{-}collidine}$ + $Mass_{2\text{-}Py\text{-}SH}$) / $Mass_{product}$*

= (276 + 18.17 + 166.5 + 8.81) / 66 + 62 + 59.3 = *2.51*

E $Factor_{with\ extraction\ solvent}$ = (*$Mass_{diphenylsilane}$ + $Mass_{2,4,6\text{-}collidine}$ + $Mass_{2\text{-}Py\text{-}SH}$* + $Mass_{extraction\text{-}solvent}$*) / $Mass_{product}$*

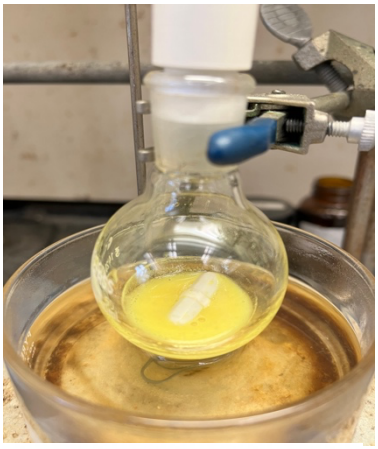
= (276 + 18.17 + 166.5 + 8.81 + 1620) / 66 + 62 + 59.3 = *11.06*

5. Solvent recovery studies and E Factor calculations for reduction of carboxylic acids to alcohols



6 mmol Not isolated

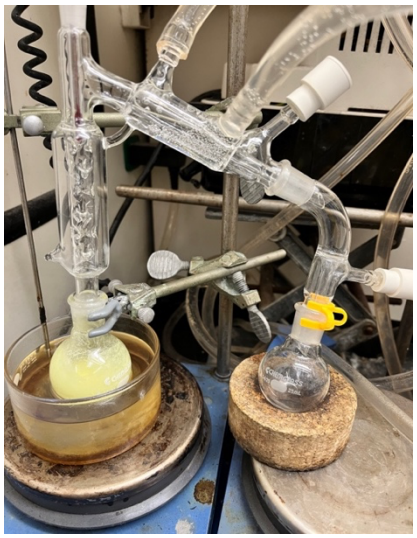
At the start of the reaction



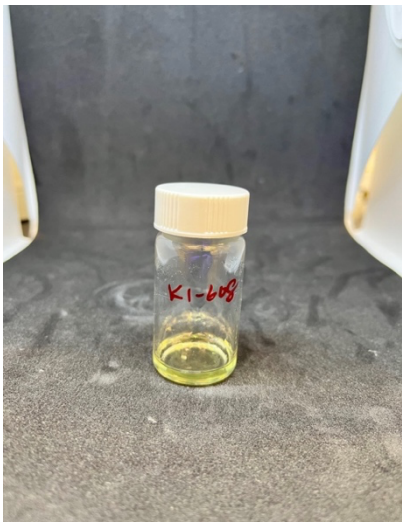
After formation of thioester

Scheme S9. Large-scale reaction and solvent recovery studies.

Reaction setup: To an oven-dried 100 mL round bottom flask with a magnetic stir bar was added gemfibrozil (6 mmol, 1.5 g) followed by the addition of DPDTC (1.1 equiv, 1.64 g). The flask was sealed with a septum and a vent needle was positioned therein. The reaction mixture was allowed to stir at 65 ºC for 3-4 h until complete (formation of a thick cloudy emulsion from a clear viscous oil was observed; see Figure above). Upon completion, the reaction was allowed to cool to rt and 95% EtOH (0.5 M, 12 mL) was added. Subsequently, the flask was placed in an ice bath and sodium borohydride (3 equiv, 684 mg) was added portion wise over 5 min to contain the bubbling of H_2 gas. Once all the $NaBH_4$ had been added, the reaction was stirred at rt for ca. 45 min until complete reduction was observed (by TLC or GC-MS). The crude reaction mixture was then arranged for solvent recovery using the setup as shown in the Figure below. Once most of the solvent was recovered (see below), the mixture was subjected to a short filtration column using 15-20% EtOAc/hexanes to give the corresponding alcohol as a light-yellow oil (yield: 89%; 1.26 g).



solvent recovery setup



product after filtration column

Solvent recovery calculations:

Amount of solvent added to the reaction: 12 mL

Amount of solvent recovered after distillation: 10 mL

Percentage of solvent recovered: (10/12) *100 = **83.3%**

E Factor calculations

E Factor = (mass waste organics) / (mass product)

1. *2-mercaptopyridine*

   1 equiv generated in the reaction = 6 mmol * 1 * 111.2 = 667.2 mg.

2. *Sodium borohydride*

   1 equiv extra was used = 6 mmol * 1 * 37.84 = 227.04.

3. *Ethanol*

   17% was lost during the recovery process = 2 mL = 2 * 0.789 g/mL = 1.578 g

   Total waste generated = 1.578 g + 0.227 g + 0.667 g = 2.47 g

E Factor = 2.47 / 1.26 = 1.96

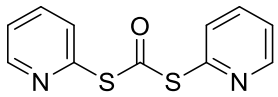
6. ICP-MS data for residual nickel

ICP-MS data for residual Ni was obtained from the University of California, Los Angeles, ICP-MS Core Facility.

		Nickel		**Source**
		[µg/g]		
Sample #	Sample weight in analysis [mg]	Average*	stdev	
K1- 305	4.30	4.687	0.171	1st recycle product
K1- 317	5.20	13.250	0.140	2nd recycle product

**Each sample was done in triplicated measurements with background correction.*
n/a represents below detection limit.

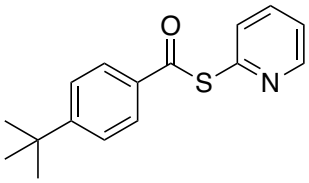
7. Analytical data



***S,S*-Di(pyridin-2-yl) carbonodithioate (DPDTC): ^{1}H NMR (400 MHz, $CDCl_3$) δ** 8.58 (ddd, *J* = 4.9, 2.0, 0.9 Hz, 1H), 7.78 – 7.59 (m, 2H), 7.27 (ddd, *J* = 7.3, 5.4, 1.4 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$) δ** 185.7, 150.7, 150.7, 137.5, 130.5, 124.2. **R_f**: 0.13 (30% EtOAc/Hexanes). Spectral data matched those previously reported.[37]

7.1. Analytical data for S-2-pyridyl thioesters

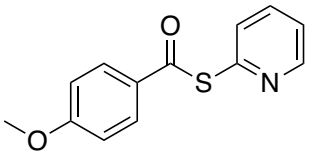
***S*-(Pyridin-2-yl) 4-(*t*-butyl)benzothioate (1a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.70 – 8.63 (m, 1H), 8.00 – 7.92 (m, 2H), 7.82 – 7.69 (m, 2H), 7.54 – 7.47 (m, 2H), 7.31 (ddt, *J* = 7.6, 4.9, 1.3 Hz, 1H), 1.35 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 189.0, 157.9, 151.7, 150.6, 137.2, 134.0, 130.9, 127.6, 125.9, 123.6, 35.4, 31.2. Yield: 91%; 247 mg; white solid; $\mathbf{R_f}$ = 0.30 (15% EtOAc/hexanes). Spectral data matched those previously reported.[4]

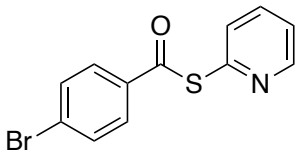
***S*-(Pyridin-2-yl) 4-methoxybenzothioate (2a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (ddd, *J* = 4.9, 1.9, 1.0 Hz, 1H), 8.05 – 7.96 (m, 2H), 7.78 (td, *J* = 7.6, 1.9 Hz, 1H), 7.72 (dt, *J* = 7.9, 1.2 Hz, 1H), 7.32 (ddd, *J* = 7.3, 4.9, 1.4 Hz, 1H), 7.02 – 6.93 (m, 2H), 3.89 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 187.7, 164.2, 151.7, 150.5, 137.1, 130.9, 129.9, 129.3, 123.5, 114.0, 55.6. Yield: 90%, 220.7 mg; yellow solid; $\mathbf{R_f}$ = 0.30 (30% EtOAc/hexanes). Spectral data matched those previously reported.[5]

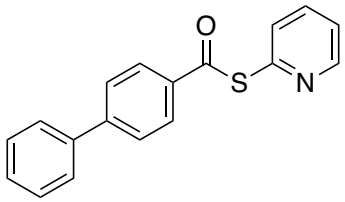
***S*-(Pyridin-2-yl) 4-bromobenzothioate (3a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.68 (ddd, *J* = 4.8, 2.0, 0.9 Hz, 1H), 7.92 – 7.83 (m, 2H), 7.79 (td, *J* = 7.7, 1.9 Hz, 1H), 7.71 (dt, *J* = 7.9, 1.1 Hz, 1H), 7.67 – 7.59 (m, 2H), 7.34 (ddd, *J* = 7.5, 4.9, 1.2 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 188.5, 150.9, 150.7, 137.3, 135.3, 132.2, 130.9, 129.1, 129.0, 123.8. Yield: 89%, 261.81; white flaky crystalline solid; **R_f** = 0.31 (15% EtOAc/hexanes). Spectral data matched those previously reported.[5]

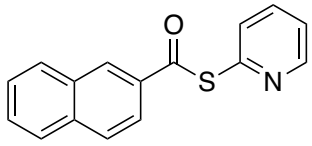
***S*-(Pyridin-2-yl) [1,1'-biphenyl]-4-carbothioate (4a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.70 (ddd, *J* = 4.8, 1.9, 0.9 Hz, 1H), 8.14 – 8.06 (m, 2H), 7.81 (td, *J* = 7.6, 1.9 Hz, 1H), 7.76 (dt, *J* = 7.9, 1.2 Hz, 1H), 7.74 – 7.69 (m, 2H), 7.66 – 7.62 (m, 2H), 7.53 – 7.45 (m, 2H), 7.45 – 7.38 (m, 1H), 7.35 (ddd, *J* = 7.3, 4.8, 1.4 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 188.9, 151.4, 150.5, 146.7, 139.7, 137.2, 135.3, 130.9, 129.0, 128.4, 128.2, 127.5, 127.3, 123.7. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{18}H_{14}NOS$ 292.0796; Found: 292.0805. Yield: 93%; 270.97 mg; white solid; **R_f** = 0.30 (12% EtOAc/hexanes).

***S*-(Pyridin-2-yl) naphthalene-2-carbothioate (5a)**

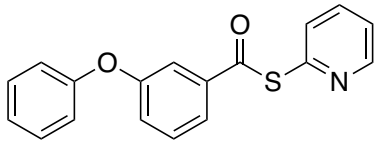


Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 – 8.67 (m, 1H), 8.62 (d, *J* = 1.8 Hz, 1H), 8.01 (td, *J* = 8.2, 1.6 Hz, 2H), 7.95 – 7.87 (m, 2H), 7.84 – 7.76 (m, 2H), 7.67 – 7.53 (m, 2H), 7.35 (ddd, *J* = 6.7, 4.8, 1.9 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 189.3, 151.5, 150.6, 137.2, 136.0, 133.9, 132.5, 130.9, 129.7, 129.3, 128.8,

128.8, 127.9, 127.1, 123.7, 123.2, 77.4, 77.0, 76.7. Yield: 91%, 241.45 mg; white solid; $\mathbf{R_f}$ = 0.29 (10% EtOAc/hexanes). Spectral data matched those previously reported.[6]

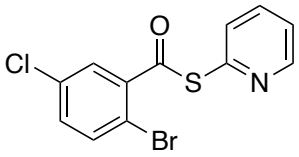
***S*-(Pyridin-2-yl) 3-phenoxybenzothioate (6a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.69 (dd, *J* = 5.1, 1.9 Hz, 1H), 7.83 – 7.76 (m, 2H), 7.73 (dt, *J* = 7.9, 1.1 Hz, 1H), 7.63 (t, *J* = 2.1 Hz, 1H), 7.47 (t, *J* = 7.9 Hz, 1H), 7.43 – 7.36 (m, 2H), 7.36 – 7.31 (m, 1H), 7.31 – 7.25 (m, 1H), 7.22 – 7.14 (m, 1H), 7.10 – 7.04 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 188.9, 151.4, 150.5, 146.7, 139.7, 137.2, 135.3, 130.9, 129.0, 128.4, 128.2, 127.5, 127.3, 123.7, 77.4, 77.3, 77.0, 76.7. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{18}H_{14}NO_2S$ 308.0745; Found: 308.0735. Yield: 70%; 215.15 mg; light yellow oil; $\mathbf{R_f}$ = 0.30 (30% EtOAc/hexanes).

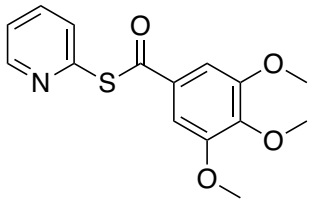
***S*-(Pyridin-2-yl) 2-bromo-5-chlorobenzothioate (7a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.71 – 8.65 (m, 1H), 7.86 – 7.74 (m, 2H), 7.72 (d, *J* = 2.5 Hz, 1H), 7.60 (d, *J* = 8.5 Hz, 1H), 7.40 – 7.30 (m, 2H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 189.1, 150.7, 150.7, 140.1, 137.5, 135.3, 133.7, 132.6, 130.4, 129.2, 124.1, 117.1. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{12}H_8BrClNOS$ 327.9198; Found: 327.9190. Yield: 80%, 262.88 mg; white solid; $\mathbf{R_f}$ = 0.30 (15% acetone/hexanes).

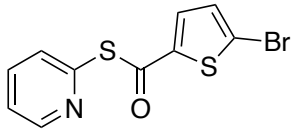
***S*-(Pyridin-2-yl) 3,4,5-trimethoxybenzothioate (8a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (ddd, *J* = 4.8, 1.9, 1.0 Hz, 1H), 7.82 – 7.68 (m, 2H), 7.32 (ddt, *J* = 7.4, 4.9, 1.6 Hz, 1H), 7.26 (d, *J* = 0.9 Hz, 2H), 3.96 – 3.84 (m, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 188.4, 153.2, 151.5, 150.5, 143.1, 137.2, 131.7, 130.8, 123.7, 105.0, 61.0, 56.3. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{15}H_{16}NO_4S$ 306.0800; Found: 306.0797. Yield: 90%, 274.81 mg; white solid; $\mathbf{R_f}$ = 0.23 (30% EtOAc/hexanes).

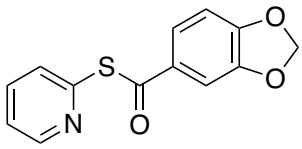
***S*-(Pyridin-2-yl) 5-bromothiophene-2-carbothioate (9a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 (ddd, *J* = 4.8, 1.9, 1.0 Hz, 1H), 7.78 (td, *J* = 7.6, 1.9 Hz, 1H), 7.73 (dt, *J* = 7.9, 1.2 Hz, 1H), 7.67 (d, *J* = 4.1 Hz, 1H), 7.33 (ddd, *J* = 7.3, 4.9, 1.4 Hz, 1H), 7.14 (d, *J* = 4.1 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 180.1, 150.6, 142.4, 137.3, 132.2, 131.3, 130.7, 123.9, 122.8. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{10}H_7BrNOS_2$ 299.9152; Found: 299.9138. Yield: 86%, 234 mg; white solid; $\mathbf{R_f}$ = 0.31 (30% EtOAc/hexanes).

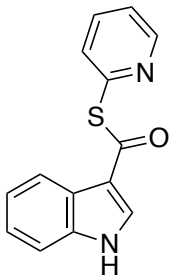
***S*-(Pyridin-2-yl) benzo[*d*][1,3]dioxole-5-carbothioate (10a)**



Reaction carried out on a 1 mmol scale using General procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (ddd, *J* = 4.8, 1.9, 0.9 Hz, 1H), 7.78 (td, *J* = 7.7, 1.9 Hz, 1H), 7.74 – 7.63 (m, 2H), 7.44 (d, *J* = 1.8 Hz, 1H), 7.32 (ddd, *J* = 7.4, 4.9, 1.3 Hz, 1H), 6.88 (d, *J* = 8.2 Hz, 1H), 6.07 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 187.5, 152.5, 151.5, 150.5, 148.3, 137.1, 131.0, 130.9, 124.0, 123.6, 108.2, 107.5, 102.1. Yield: 85%; 220.15 mg; white solid; $\mathbf{R_f}$ = 0.30 (30% EtOAc/hexanes). Spectral data matched those previously reported.[6]

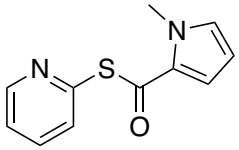
***S*-(Pyridin-2-yl) 1*H*-indole-3-carbothioate (11a)**



Reaction carried out on a 1 mmol scale using General Procedure 1B.

^{1}H NMR (400 MHz, DMSO) δ 8.61 (ddd, *J* = 4.8, 1.9, 0.9 Hz, 1H), 8.43 (s, 1H), 8.06 – 7.96 (m, 1H), 7.89 (td, *J* = 7.7, 1.9 Hz, 1H), 7.75 (dt, *J* = 7.9, 1.0 Hz, 1H), 7.57 – 7.47 (m, 1H), 7.42 (ddd, *J* = 7.6, 4.9, 1.2 Hz, 1H), 7.23 (pd, *J* = 7.1, 1.5 Hz, 2H). **^{13}C NMR (101 MHz, DMSO)** δ 180.9, 152.1, 150.7, 137.8, 137.1, 133.9, 131.2, 125.1, 124.0, 123.8, 122.8, 121.0, 114.9, 113.2. **HRMS (**ESI**)** *m/z*: $[M+Na]^+$ calcd. for $C_{14}H_{10}N_2OSNa$ 277.0411; Found: 277.0448. Yield: 50%; 127 mg; white solid; R_f = 0.15 (30% EtOAc/hexanes).

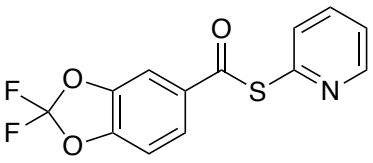
***S*-(Pyridin-2-yl) 1-methyl-1*H*-pyrrole-2-carbothioate (12a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.66 (dd, *J* = 4.9, 1.9 Hz, 1H), 7.75 (td, *J* = 7.7, 1.9 Hz, 1H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.29 (ddd, *J* = 7.5, 4.8, 1.2 Hz, 1H), 7.23 (dd, *J* = 4.3, 1.7 Hz, 1H), 6.86 (t, *J* = 2.1 Hz, 1H), 6.17 (dd, *J* = 4.3, 2.5 Hz, 1H), 3.87 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 178.8, 151.9, 150.5, 137.0, 131.5, 130.9, 128.4, 123.3, 119.5, 108.9, 37.2. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{11}H_{11}N_2OS$ 219.0592; Found: 219.0601. Yield: 65%, 141.8 mg; yellow oil; R_f = 0.30 (20% EtOAc/hexanes).

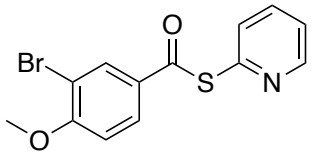
***S*-(Pyridin-2-yl) 2,2-difluorobenzo[*d*][1,3]dioxole-5-carbothioate (13a)**



Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.71 (dd, *J* = 5.0, 1.9 Hz, 1H), 7.91 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.83 (td, *J* = 7.7, 1.9 Hz, 1H), 7.74 (dt, *J* = 4.9, 2.1 Hz, 2H), 7.38 (ddd, *J* = 7.6, 4.9, 1.2 Hz, 1H), 7.20 (d, *J* = 8.4 Hz, 1H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 187.4, 150.7, 147.6, 144.2, 137.4, 132.9, 131.8 (t, *J* = 259.3 Hz), 130.9, 124.8, 123.9, 109.5, 108.8, 77.3, 77.2, 77.0, 76.8. **^{19}F NMR (471 MHz, $CDCl_3$)** δ -49.6. **HRMS (**ESI) *m/z*: $[M+H]^+$ calcd. for $C_{13}H_8F_2NO_3S$ 296.0192; Found: 296.0205. Yield: 82%; 212.58 mg; white solid; **R_f** = 0.31 (30% EtOAc/hexanes).

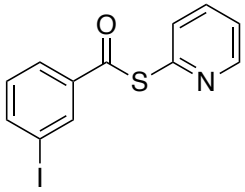
***S*-(Pyridin-2-yl) 3-bromo-4-methoxybenzothioate (14a)**



Reaction carried out on a 1 mmol scale using General procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (dd, *J* = 4.9, 1.9 Hz, 1H), 8.22 (d, *J* = 2.2 Hz, 1H), 8.00 (dd, *J* = 8.6, 2.2 Hz, 1H), 7.79 (td, *J* = 7.7, 1.9 Hz, 1H), 7.71 (d, *J* = 7.8 Hz, 1H), 7.37 – 7.30 (m, 1H), 6.96 (d, *J* = 8.7 Hz, 1H), 3.98 (s, 3H), 1.25 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 186.9, 160.3, 151.2, 150.6, 137.2, 132.9, 130.9, 130.3, 128.8, 123.7, 112.2, 111.3, 56.6. **HRMS (**ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{13}H_{10}BrNO_2SNa$ 345.9513; Found: 345.9512. Yield: 86%; 279.5 mg; white solid; R_f = 0.29 (30% EtOAc/hexanes).

***S*-(Pyridin-2-yl) 3-iodobenzothioate (15a)**

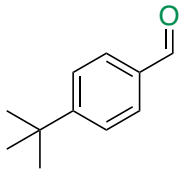


Reaction carried out on a 1 mmol scale using General Procedure 1A.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (ddd, *J* = 4.8, 1.9, 0.9 Hz, 1H), 8.31 (t, *J* = 1.8 Hz, 1H), 7.95 (dddd, *J* = 16.6, 7.9, 1.8, 1.1 Hz, 2H), 7.79 (td, *J* = 7.7, 1.9 Hz, 1H), 7.70 (dt, *J* = 8.0, 1.1 Hz, 1H), 7.34 (ddd, *J* = 7.5, 4.9, 1.2 Hz, 1H), 7.23 (t, *J* = 7.9 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 188.0, 150.8, 150.6, 142.6, 138.2, 137.3, 136.3, 130.8, 130.5, 126.7, 123.9, 94.4. Yield: 88%; 300 mg; off white solid; **R_f** = 0.27 (30% EtOAc/hexanes). Spectral data matched those previously reported.[7]

7.2. Analytical data for product aldehydes

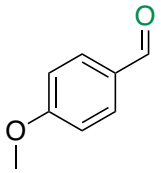
4-(*t*-Butyl)benzaldehyde (1)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.86 – 7.78 (m, 2H), 7.59 – 7.52 (m, 2H), 1.36 (s, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 192.2, 129.8, 126.1, 31.2. Yield: 92%; 30 mg; colorless oil, $\mathbf{R_f}$ = 0.60 (10% EtOAc/hexanes). Spectral data matched those previously reported.[8]

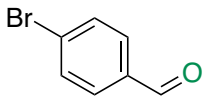
4-Methoxybenzaldehyde (2)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.88 (s, 1H), 7.88 – 7.79 (m, 2H), 7.04 – 6.96 (m, 2H), 3.88 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 190.8, 164.6, 132.0, 130.0, 114.3, 55.6. Yield: 97%, 26.3 mg; colorless oil. $\mathbf{R_f}$ = 0.31 (33% EtOAc/hexanes). Spectral data matched those previously reported.[8]

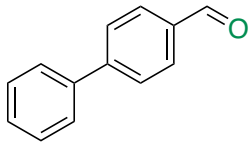
4-Bromobenzaldehyde (3)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.00 (d, J = 1.8 Hz, 1H), 7.77 (dd, J = 8.5, 1.9 Hz, 2H), 7.74 – 7.69 (m, 2H).**^{13}C NMR (126 MHz, $CDCl_3$)** δ 191.1, 135.1, 132.5, 131.0, 129.8. Yield- 91%, 34 mg; crystalline white solid. $\mathbf{R_f}$ = 0.30 (20% EtOAc/hexanes). Spectral data matched those previously reported.[8]

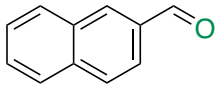
Biphenyl-4-carboxaldehyde (4)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1H), 8.00 – 7.92 (m, 2H), 7.80 – 7.72 (m, 2H), 7.68 – 7.60 (m, 2H), 7.53 – 7.46 (m, 2H), 7.46 – 7.38 (m, 1H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 192.1, 168.2, 147.4, 139.9, 135.3, 130.4, 129.2, 128.6, 127.8, 127.5. Yield: 70%, 25.5 mg; white powder. $\mathbf{R_f}$ = 0.30 (10% EtOAc/hexanes). Spectral data matched those previously reported.[8]

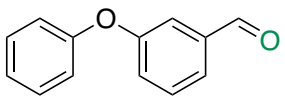
2-Naphthaldehyde (5)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.17 (s, 1H), 8.35 (d, J = 1.3 Hz, 1H), 8.05 – 7.89 (m, 4H), 7.69 – 7.57 (m, 2H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 192.3, 136.5, 134.6, 134.1, 132.7, 129.6, 129.1, 129.1, 128.1, 127.1, 122.8. Yield: 82%, 25.61 mg; white solid. $\mathbf{R_f}$ = 0.33 (15% EtOAc/hexanes). Spectral data matched those previously reported.[8]

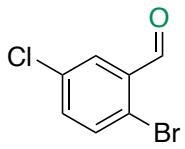
3-Phenoxybenzaldehyde (6)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (s, 1H), 7.60 (dt, J = 7.5, 1.3 Hz, 1H), 7.53 – 7.42 (m, 2H), 7.42 – 7.32 (m, 2H), 7.32 – 7.23 (m, 1H), 7.23 – 7.12 (m, 1H), 7.08 – 7.00 (m, 2H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 191.5, 158.4, 156.2, 138.1, 137.8, 130.7, 130.5, 130.3, 130.1, 130.0, 129.9, 129.9, 129.7, 128.5, 124.7, 124.6, 124.2, 119.5, 118.2, 114.7. Yield: 93%, 36.86 mg; yellow oil; $\mathbf{R_f}$ = 0.33 (30% EtOAc/hexanes). Spectral data matched those previously reported.[9]

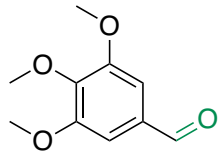
2-Bromo-5-chlorobenzaldehyde (7)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.28 (s, 1H), 7.86 (d, *J* = 2.6 Hz, 1H), 7.59 (d, *J* = 8.5 Hz, 1H), 7.41 (dd, *J* = 8.5, 2.7 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 190.5, 135.1, 135.1, 134.6, 134.4, 129.7, 124.6. Yield: 82%, 35.11 mg; light yellow crystalline solid; **R_f** = 0.30 (10% EtOAc/hexanes). Spectral data matched those previously reported.[10]

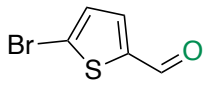
3,4,5-Trimethoxybenzaldehyde (8)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 7.13 (s, 2H), 3.93 (d, *J* = 2.9 Hz, 9H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 191.1, 153.7, 143.6, 131.7, 106.7, 61.0, 56.3. Yield: 84%, 33 mg; light yellowish-brown powder. **R_f** = 0.25 (30% EtOAc/hexanes). Spectral data matched those previously reported.[11]

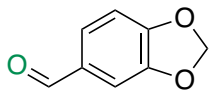
5-Bromothiophene-2-carbaldehyde (9)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 1H), 7.52 (d, *J* = 4.0 Hz, 1H), 7.19 (d, *J* = 4.0 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 181.8, 145.2, 136.5, 131.5, 125.0. Yield: 82%, 31.32 mg; reddish brown liquid. **R_f** = 0.34 (33% EtOAc/hexanes). Spectral data matched those previously reported.[12]

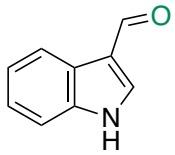
Piperonal (10)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.80 (s, 1H), 7.40 (dd, *J* = 7.9, 1.6 Hz, 1H), 7.32 (d, *J* = 1.6 Hz, 1H), 6.92 (d, *J* = 7.9 Hz, 1H), 6.07 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 190.3, 153.1, 148.7, 131.9, 128.7, 108.4, 107.0, 102.1. Yield: 88%, 26.4 mg; white crystalline solid. **R_f** = 0.31 (20% EtOAc/hexanes). Spectral data matched those previously reported.[13]

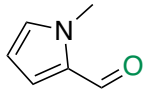
Indole-3-carboxaldehyde (11)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, DMSO) δ 9.91 (s, 1H), 8.27 (s, 1H), 8.11 – 8.04 (m, 1H), 7.53 – 7.46 (m, 1H), 7.22 (dtd, *J* = 17.8, 7.2, 1.3 Hz, 2H). **^{13}C NMR (101 MHz, DMSO)** δ 185.4, 138.9, 137.5, 124.6, 123.9, 122.6, 121.3, 118.6, 112.9. Yield: 76%, 23 mg; yellow crystalline solid. **R_f** = 0.25 (40% EtOAc/hexanes). Spectral data matched those previously reported.[14]

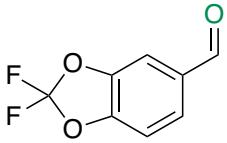
1-Methyl-1*H*-pyrrole-2-carbaldehyde (12)



Reaction carried out on a 0.4 mmol scale using General Procedure 3.

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.55 (d, *J* = 1.0 Hz, 1H), 6.94 – 6.85 (m, 2H), 6.21 (dd, *J* = 4.0, 2.4 Hz, 1H), 3.96 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 179.6, 132.1, 132.0, 124.1, 109.5, 36.5. Yield: 78%, 34.04 mg; reddish-orange liquid. **R_f** = 0.35 (25% EtOAc/hexanes). Spectral data matched those previously reported.[15]

2,2-Difluorobenzo[*d*][1,3] dioxole-5-carbaldehyde (13)

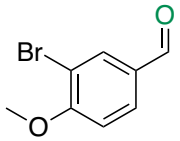


Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.96 (s, 1H), 7.71 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.64 (d, *J* = 1.5 Hz, 1H), 7.28 (s, 1H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 189.7, 148.1, 144.6, 133.7, 133.2, 131.6 (t, *J* = 259.0, 257.0 Hz),

130.0, 129.,6 128.7, 119.5, 109.8, 109.2, 108.8. **^{19}F NMR (471 MHz, $CDCl_3$)** δ -49.7. Yield: 80%, 30 mg; colorless liquid; **R_f** = 0.40 (30% EtOAc/hexanes). Spectral data matched those previously reported.[16]

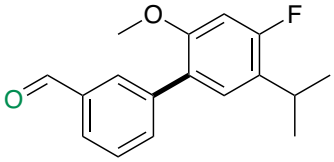
3-Bromo-4-methoxybenzaldehyde (14)



Reaction carried out on a 0.2 mmol scale using General Procedure 3.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.84 (s, 1H), 8.08 (d, *J* = 2.0 Hz, 1H), 7.82 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.01 (d, *J* = 8.4 Hz, 1H), 3.99 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 189.7, 160.8, 134.7, 131.3, 130.9, 112.8, 111.7, 111.7, 56.8. Yield: 91%, 39 mg; off white crystalline powder; **R_f** = 0.32 (30% EtOAc/hexanes). Spectral data matched those previously reported.[17]

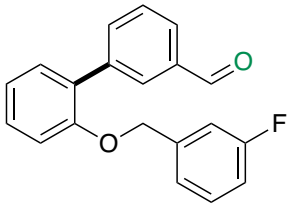
4'-Fluoro-5'-isopropyl-2'-methoxy-[1,1'-biphenyl]-3-carbaldehyde (15)



Reaction carried out on a 0.5 mmol scale using General Procedure 5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.09 (s, 1H), 8.02 (t, *J* = 1.8 Hz, 1H), 7.86 (dt, *J* = 7.6, 1.5 Hz, 1H), 7.79 (dt, *J* = 7.8, 1.5 Hz, 1H), 7.59 (t, *J* = 7.7 Hz, 1H), 7.20 (d, *J* = 8.6 Hz, 1H), 6.72 (d, *J* = 12.1 Hz, 1H), 3.81 (s, 3H), 3.24 (m, *J* = 7.0 Hz, 1H), 1.30 (d, *J* = 7.0 Hz, 6H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 192.5, 161.7, 159.8, 155.4 (d, *J* = 10.0 Hz), 139.2, 136.4, 135.7, 130.9, 129.2 (d, *J* = 7.3 Hz), 128.7, 128.2, 127.4 (d, *J* = 15.3 Hz), 125.0 (d, *J* = 3.3 Hz), 99.7 (d, *J* = 27.5 Hz), 55.9, 26.8, 26.8, 22.8. **^{19}F NMR (471 MHz, $CDCl_3$)** δ -116.6. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{17}H_{18}FO_2$ 271.1290; Found: 273.1281. Yield: 71%, 97 mg; reddish brown oil; **R_f** = 0.31 (15% EtOAc/hexanes).

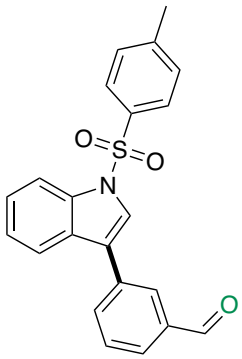
2'-((3-Fluorobenzyl)oxy)-[1,1'-biphenyl]-3-carbaldehyde (16)



Reaction carried out on a 0.5 mmol scale using General Procedure 5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.08 (s, 1H), 8.13 (t, *J* = 1.8 Hz, 1H), 7.92 – 7.85 (m, 2H), 7.61 (t, *J* = 7.7 Hz, 1H), 7.42 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.38 (td, *J* = 7.7, 1.8 Hz, 1H), 7.31 (td, *J* = 7.9, 5.8 Hz, 1H), 7.17 – 6.94 (m, 5H), 5.12 (s, 2H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 192.4, 163.9, 162.0, 155.3, 139.6 (d, *J* = 7.4 Hz), 139.4, 136.3, 135.7, 131.1, 130.9, 130.2 (d, *J* = 8.2 Hz), 129.8, 129.4, 128.7, 128.3, 122.3, 122.3, 121.8, 121.1, 114.8 (d, *J* = 21.1 Hz), 113.9 (d, *J* = 22.4 Hz),113.2, 112.8, 69.8, 69.7. **^{19}F NMR (471 MHz, $CDCl_3$)** δ -112.7. **HRMS (**ESI) *m/z*: $[M+H]^+$ calcd. for $C_{20}H_{16}FO_2$ 307.1134; Found: 307.1150. Yield: 72%, 110 mg; yellow oil; $\mathbf{R_f}$ = 0.30 (15% EtOAc/hexanes).

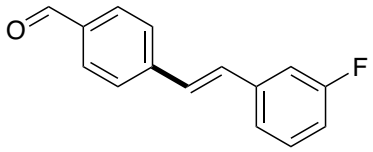
3-(1-Tosyl-1*H*-indol-3-yl)benzaldehyde (17)



Reaction carried out on a 0.5 mmol scale using General Procedure 5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.12 (s, 1H), 8.16 – 8.07 (m, 2H), 7.90 (dd, *J* = 7.6, 1.7 Hz, 2H), 7.87 – 7.83 (m, 2H), 7.79 (d, *J* = 9.3 Hz, 2H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.42 (ddd, *J* = 8.3, 7.1, 1.2 Hz, 1H), 7.37 – 7.30 (m, 1H), 7.27 (d, *J* = 9.5 Hz, 2H), 2.37 (s, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 192.1, 145.3, 137.0, 135.5, 135.1, 134.3, 133.7, 130.1, 130.0, 129.7, 129.0, 128.8, 128.6, 127.0, 126.9, 125.2, 123.8, 123.6, 122.5, 120.1, 114.0, 21.6. **HRMS (**ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{22}H_{17}NO_3SNa$ 398.0826; Found: 398.0826. Yield: 65%, 122 mg; off white - light yellow solid; $\mathbf{R_f}$ = 0.33 (20% EtOAc/hexanes).

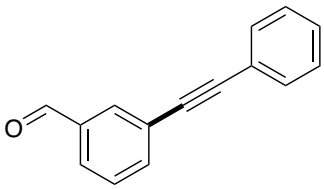
(*E*)-4-(3-Fluorostyryl)benzaldehyde (18)



Reaction carried out on a 0.5 mmol scale using General Procedure 5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 9.99 (s, 1H), 7.90 – 7.84 (m, 2H), 7.67 – 7.61 (m, 2H), 7.37 – 7.17 (m, 4H), 7.12 (d, *J* = 16.3 Hz, 1H), 7.00 (tdd, *J* = 8.2, 2.6, 1.1 Hz, 1H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 191.6, 164.2, 162.2, 142.8, 139.0 (d, *J* = 7.5 Hz), 135.6, 131.0 (d, *J* = 2.8 Hz), 130.4, 130.3, 130.3, 130.0, 128.7, 127.3, 127.1, 126.8, 122.9, 122.9, 115.4 (d, *J* = 21.4 Hz), 113.2 (d, *J* = 22.0 Hz). **^{19}F NMR (471 MHz, $CDCl_3$)** δ -112.9. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{15}H_{12}FO$ 227.0872; Found: 227.0858. Yield: 67%, 76 mg, off white crystalline solid; $\mathbf{R_f}$ = 0.31 (15% EtOAc/hexanes).

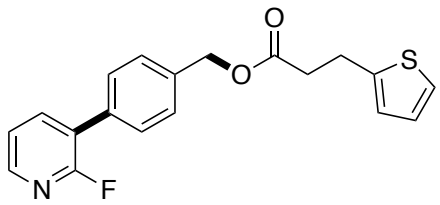
3-(Phenylethynyl)benzaldehyde (19)



Reaction carried out on a 0.5 mmol scale using general procedure 5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.04 (s, 1H), 8.05 (d, *J* = 1.7 Hz, 1H), 7.87 (dt, *J* = 7.8, 1.4 Hz, 1H), 7.80 (dt, *J* = 7.6, 1.5 Hz, 1H), 7.61 – 7.51 (m, 3H), 7.39 (p, *J* = 3.3 Hz, 3H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 191.6, 137.1, 136.5, 133.0, 132.0, 131.7, 131.5, 129.1, 128.9, 128.8, 128.6, 128.5, 128.5, 128.5, 124.6, 122.7, 91.0, 87.9, 29.7. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{15}H_{11}O$ 207.0809; Found: 207.0821. Yield: 60%, 62 mg; yellow oil; $\mathbf{R_f}$ = 0.33 (15% EtOAc/hexanes).

4-(2-Fluoropyridin-3-yl)benzyl 3-(thiophen-2-yl)propanoate (20)

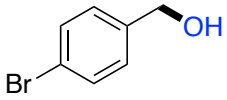


Reaction carried out on a 1 mmol scale using General Procedure 4.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (dt, *J* = 4.9, 1.6 Hz, 1H), 7.87 (ddd, *J* = 9.7, 7.4, 2.0 Hz, 1H), 7.56 (dq, *J* = 8.4, 2.1 Hz, 2H), 7.45 – 7.39 (m, 2H), 7.29 (ddd, *J* = 7.1, 4.8, 1.8 Hz, 1H), 7.15 – 7.10 (m, 1H), 6.91 (dd, *J* = 5.2, 3.4 Hz, 1H), 6.82 (dd, *J* = 3.4, 1.1 Hz, 1H), 5.18 (s, 2H), 3.24 – 3.17 (m, 2H), 2.77 (t, *J* = 7.5 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 172.2, 161.6, 159.2, 146.7 (d, *J* = 14.6 Hz), 142.9, 140.8 (d, *J* = 4.4 Hz), 136.2, 133.8, 129.2 (d, *J* = 3.1 Hz), 128.5, 126.9, 124.8, 123.6, 123.3, 122.0 (d, *J* = 4.4 Hz), 65.9, 36.2, 25.2. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -70.9. **HRMS** (ESI) *m/z*: $[M+H]^+$ calcd. for $C_{19}H_{17}FNO_2S$ 342.0964; Found: 342.0949. Yield: 65%, 222 mg; light brown oil; $\mathbf{R_f}$ = 0.33 (30% EtOAc/hexanes).

7.3. Analytical data for product alcohols

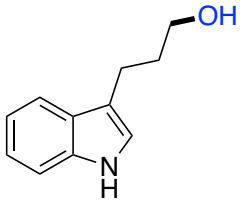
(4-Bromophenyl)methanol (21)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.45 (m, 2H), 7.26 – 7.21 (m, 2H), 4.66 (s, 2H), 1.69 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 139.8, 131.6, 128.6, 121.4, 64.4. Yield: 93%; 43.5 mg; white crystalline solid; $\mathbf{R_f}$ = 0.39 (30% EtOAc/hexanes). Spectral data matched those previously reported.[18]

3-(1*H*-Indol-3-yl)propan-1-ol (22)

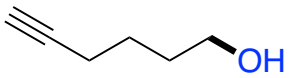


Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.72 – 7.65 (m, 1H), 7.40 (dd, *J* = 8.0, 1.1 Hz, 1H), 7.30 – 7.22 (m, 1H), 7.19 (td, *J* = 7.4, 1.1 Hz, 1H), 7.02 (d, *J* = 2.5 Hz, 1H), 3.79 (t, *J* = 6.4 Hz, 2H), 2.97 – 2.88

(m, 2H), 2.11 – 2.00 (m, 2H), 1.68 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 136.5, 127.6, 122.0, 121.4, 119.3, 119.0, 116.0, 111.2, 62.7, 33.0, 21.5. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_{11}H_{14}NO$ 176.1075; Found: 176.1085. Yield: 89%, 39 mg; colorless oil; **R_f** = 0.29 (35% EtOAc/hexanes).

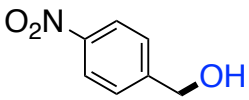
Hex-5-yn-1-ol (23)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.57 (t, *J* = 6.3 Hz, 2H), 2.70 (d, *J* = 3.2 Hz, 1H), 2.16 (td, *J* = 6.8, 2.6 Hz, 2H), 1.91 (t, *J* = 2.6 Hz, 1H), 1.67 – 1.47 (m, 4H).**^{13}C NMR (101 MHz, $CDCl_3$)** δ 84.3, 68.6, 62.0, 31.6, 24.7, 18.2. Yield: 82%, 40.2 mg; colorless oil; **R_f** = 0.33 (30% EtOAc/hexanes, $KMnO_4$ stain). Spectral data matched those previously reported.[19]

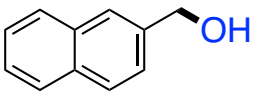
(4-Nitrophenyl)methanol (24)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 – 8.14 (m, 2H), 7.51 (d, *J* = 8.4 Hz, 2H), 4.81 (d, *J* = 5.7 Hz, 2H), 2.37 (t, *J* = 5.8 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 148.4, 147.3, 127.1, 123.8, 64.0. Yield: 90%, 69 mg; light yellow solid; **R_f** = 0.15 (30 % EtOAc/hexanes). Spectral data matched those previously reported.[20]

Naphthalen-2-ylmethanol (25)

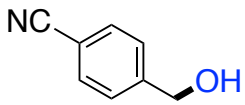


Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 – 7.77 (m, 4H), 7.49 (tp, *J* = 6.0, 2.0 Hz, 3H), 4.84 (s, 2H), 2.05 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 138.4, 133.4, 133.0, 128.3, 127.9, 127.8, 126.2, 125.9, 125.5, 125.2,

65.4. Yield: 95%; 75.15 mg; white solid; $\mathbf{R_f}$ = 0.31 (20% EtOAc/hexanes). Spectral data matched those previously reported.[20]

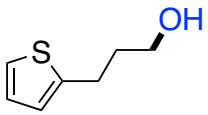
4-(Hydroxymethyl)benzonitrile (26)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 – 7.60 (m, 2H), 7.50 – 7.44 (m, 2H), 4.77 (s, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 146.4, 132.4, 127.1, 119.0, 111.3, 64.3. Yield: 78%; 26 mg; white solid; $\mathbf{R_f}$ = 0.40 (30% EtOAc/hexanes). Spectral data matched those previously reported.[22]

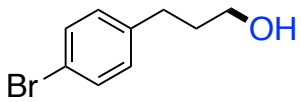
3-(Thiophen-2-yl)propan-1-ol (27)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (dd, J = 5.1, 1.2 Hz, 1H), 6.92 (dd, J = 5.1, 3.4 Hz, 1H), 6.81 (dq, J = 3.3, 1.1 Hz, 1H), 3.70 (t, J = 6.4 Hz, 2H), 2.98 – 2.90 (m, 2H), 1.95 (ddt, J = 8.4, 7.5, 6.4 Hz, 2H), 1.81 – 1.69 (m, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 144.7, 126.9, 124.4, 123.2, 61.9, 34.6, 26.2. Yield: 85%; 60.4 mg; colorless to light yellow oil; $\mathbf{R_f}$ = 0.21 (10% acetone/hexanes). Spectral data matched those previously reported.[23]

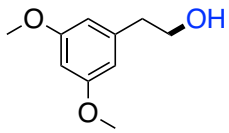
3-(4-Bromophenyl)propan-1-ol (28)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.35 (m, 2H), 7.10 – 7.02 (m, 2H), 3.64 (t, J = 6.4 Hz, 2H), 2.65 (dd, J = 8.7, 6.7 Hz, 2H), 1.84 (ddt, J = 14.0, 11.7, 5.8 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.9, 131.5, 130.3, 126.1, 119.7, 62.0, 34.1, 31.5. Yield: 88%; 94.6 mg; colorless to light yellow oil; $\mathbf{R_f}$ = 0.35 (30% EtOAc/hexanes). Spectral data matched those previously reported.[24]

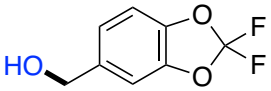
2-(3,5-Dimethoxyphenyl)ethan-1-ol (29)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.39 (d, *J* = 2.3 Hz, 2H), 6.34 (t, *J* = 2.3 Hz, 1H), 3.85 (t, *J* = 6.5 Hz, 2H), 3.78 (s, 6H), 2.81 (t, *J* = 6.5 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.1, 141.0, 107.2, 98.5, 63.6, 55.4, 39.6. Yield: 91%; 41.4 mg; colorless oil; **R_f** = 0.31 (50% EtOAc/hexanes). Spectral data matched those previously reported.[25]

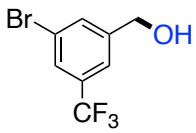
(2,2-Difluorobenzo[*d*][1,3]dioxol-5-yl)methanol (30)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, *J* = 1.5 Hz, 1H), 7.08 – 6.98 (m, 2H), 4.66 (s, 2H), 1.89 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 144.1, 143.3, 137.2, 131.8 (t, *J* = 253.14 Hz), 122.1, 109.4, 108.6, 64.9, 28.4, 14.3. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -50.1. Yield: 80%; 37.6 mg; colorless liquid; **R_f** = 0.31 (30% EtOAc/hexanes). Spectral data matched those previously reported.[26]

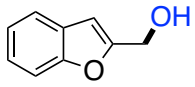
(3-Bromo-5-(trifluoromethyl)phenyl)methanol (31)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 11.0 Hz, 2H), 7.56 (s, 1H), 4.75 (s, 2H), 2.00 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 144.1, 133.1, 132.6 (q, *J* = 33 Hz), 127.6 (q, *J* = 3.8 Hz), 124.6, 123.0, 122.2 (q, *J* = 3.7 Hz), 60.6. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -62.8. Yield: 89%; 57 mg; colorless oil; **R_f** = 0.31 (20% EtOAc/hexanes). Spectral data matched those previously reported.[27]

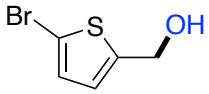
Benzofuran-2-ylmethanol (32)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.50 – 7.43 (m, 1H), 7.32 – 7.26 (m, 1H), 7.22 (td, *J* = 7.4, 1.1 Hz, 1H), 6.65 (s, 1H), 4.77 (s, 2H), 2.13 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 156.6, 155.2, 128.3, 124.5, 122.9, 121.3, 111.4, 104.3, 77.5, 58.3. Yield: 83%; 31 mg; yellow oil; **R_f** = 0.29 (30% EtOAc/hexanes). Spectral data matched those previously reported.[28]

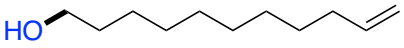
(5-Bromothiophen-2-yl)methanol (33)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.91 (d, *J* = 3.7 Hz, 1H), 6.75 (d, *J* = 3.7 Hz, 1H), 4.74 (s, 2H), 1.93 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 145.8, 129.7, 125.8, 112.4, 60.3. Yield: 85%; 41 mg; light yellow oil; **R_f** = 0.30 (10% acetone/hexanes). Spectral data matched those previously reported.[29]

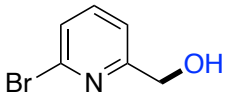
Undec-10-en-1-ol (34)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.80 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 4.98 (dq, *J* = 17.1, 1.7 Hz, 1H), 4.92 (ddt, *J* = 10.3, 2.4, 1.3 Hz, 1H), 3.62 (t, *J* = 6.7 Hz, 2H), 2.03 (tdd, *J* = 8.0, 6.0, 1.5 Hz, 2H), 1.67 (s, 1H), 1.61 – 1.49 (m, 2H), 1.38 – 1.22 (m, 12H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 139.3, 114.2, 63.1, 33.9, 32.9, 29.7, 29.5, 29.2, 29.0, 25.9. Yield: 93%; 79.2 mg; colorless liquid; **R_f** = 0.40 (30% EtOAc/hexanes, $KMnO_4$ stain). Spectral data matched those previously reported.[30]

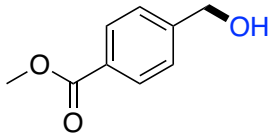
(6-Bromopyridin-2-yl)methanol (35)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (t, *J* = 7.7 Hz, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.28 (d, *J* = 7.6 Hz, 1H), 4.72 (s, 2H), 3.50 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 161.5, 141.4, 139.2, 126.7, 119.5, 64.3. Yield: 64%; 60 mg; white solid; **R_f** = 0.31 (30% EtOAc/hexanes). Spectral data matched those previously reported.[31]

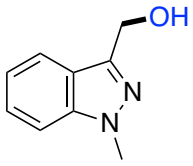
Methyl 4-(hydroxymethyl)benzoate (36)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 7.97 (m, 2H), 7.46 – 7.38 (m, 2H), 4.76 (s, 2H), 3.91 (s, 3H), 2.02 (d, *J* = 7.3 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 167.1, 146.1, 130.0, 129.4, 126.6, 64.8, 52.3. Yield: 86%; 77.5 mg; white solid; **R_f** = 0.21 (30% EtOAc/hexanes). Spectral data matched those previously reported.[21]

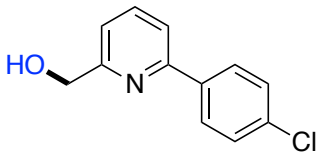
(1-Methyl-1*H*-indazol-3-yl)methanol (37)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (dt, *J* = 8.1, 1.1 Hz, 1H), 7.35 (ddd, *J* = 8.0, 6.8, 1.1 Hz, 1H), 7.31 – 7.20 (m, 1H), 7.11 (ddd, *J* = 8.0, 6.8, 1.0 Hz, 1H), 4.97 (s, 2H), 3.90 (s, 3H), 2.98 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 144.2, 141.1, 126.7, 122.0, 120.6, 120.6, 109.1, 58.0, 35.3, 29.8. **HRMS** (ESI) *m/z*: $[M+Na]^+$ calcd. for $C_9H_{11}N_2ONa$ 185.0690; Found: 185.0679. Yield: 72%; 58 mg; colorless oil; **R_f** = 0.33 (30% acetone/hexanes).

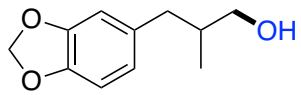
(6-(4-Chlorophenyl)pyridin-2-yl)methanol (38)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (ddt, *J* = 7.9, 4.5, 2.5 Hz, 2H), 7.74 (tdd, *J* = 7.8, 2.9, 1.9 Hz, 1H), 7.60 (td, *J* = 5.4, 2.7 Hz, 1H), 7.44 (ddd, *J* = 8.6, 4.0, 2.0 Hz, 2H), 7.18 (d, *J* = 7.6 Hz, 1H), 4.81 (d, *J* = 2.1 Hz, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 158.9, 155.0, 137.7, 137.3, 135.4, 129.1, 128.2, 119.2, 118.9, 64.1. **HRMS (**ESI) *m/z*: $[M+H]^+$ calcd. for $C_{12}H_{11}ClNO$ 220.0529; Found: 220.0518. Yield: 82%; 90 mg; white solid; R_f = 0.31 (30% EtOAc/hexanes).

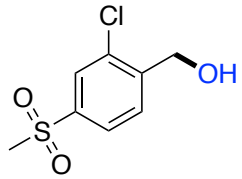
3-(Benzo[*d*][1,3]dioxol-5-yl)-2-methylpropan-1-ol (39)



Reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.72 (d, *J* = 7.9 Hz, 1H), 6.67 (d, *J* = 1.7 Hz, 1H), 6.61 (dd, *J* = 7.8, 1.8 Hz, 1H), 5.91 (s, 2H), 3.48 (qd, *J* = 10.6, 5.9 Hz, 2H), 2.67 (dd, *J* = 13.6, 6.4 Hz, 1H), 2.34 (dd, *J* = 13.6, 8.0 Hz, 1H), 1.88 (dp, *J* = 8.0, 6.3 Hz, 1H), 1.56 (s, 1H), 0.90 (d, *J* = 6.7 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 147.6, 145.8, 134.5, 122.4, 122.0, 109.8, 109.6, 108.2, 100.9, 67.6, 39.5, 38.0, 16.5. **HRMS (**ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{11}H_{15}O_3Na$ 217.0840; Found: 217.0829. Yield: 75%; 78.8 mg; colorless oil; **R_f** = 0.40 (20% acetone/hexanes).

(2-Chloro-4-(methylsulfonyl)phenyl)methanol (40)

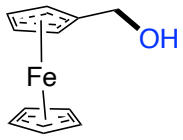


Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, *J* = 1.8 Hz, 1H), 7.82 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.77 (d, *J* = 8.1 Hz, 1H), 4.85 (d, *J* = 4.5 Hz, 2H), 3.06 (s, 3H), 2.35 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 144.9, 140.6,

133.2, 128.8, 128.1, 125.9, 62.1, 44.7. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. for $C_8H_{10}ClO_3S$ 221.0039; Found: 221.0050. Yield: 85%; white solid; $\mathbf{R_f}$ = 0.35 (50% EtOAc/hexanes).

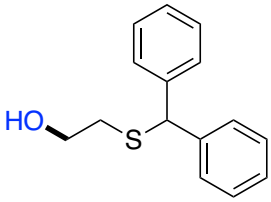
Hydroxymethylferrocene (41)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.31 (d, *J* = 5.3 Hz, 2H), 4.26 (s, 2H), 4.20 (s, 7H), 1.52 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 88.9, 69.5, 68.8, 68.3, 68.1, 60.9. Yield: 87%; 47 mg; orange solid; R_f = 0.29 (30% acetone/hexanes). Spectral data matched those previously reported.[32]

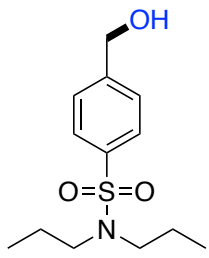
2-(Benzhydrylthio)ethan-1-ol (42)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.42 (m, 4H), 7.37 – 7.30 (m, 4H), 7.28 – 7.22 (m, 2H), 5.22 (s, 1H), 3.67 (t, *J* = 6.0 Hz, 2H), 2.63 (t, *J* = 6.0 Hz, 2H), 2.09 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 141.2, 128.8, 128.4, 127.5, 60.5, 53.8, 35.5. Yield: 88%; 54 mg; yellow oil; $\mathbf{R_f}$ = 0.30 (20% EtOAc/hexanes). Spectral data matched those previously reported.[33]

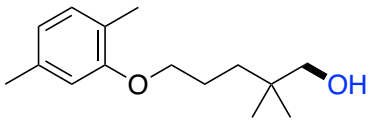
4-(Hydroxymethyl)-*N*,*N*-dipropylbenzenesulfonamide (43)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 – 7.68 (m, 2H), 7.45 (d, *J* = 8.2 Hz, 2H), 4.74 (s, 2H), 3.07 – 3.00 (m, 4H), 1.58 – 1.47 (m, 4H), 0.85 (t, *J* = 7.4 Hz, 6H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 145.8, 138.9, 127.3, 127.1, 64.3, 50.1, 22.1, 11.3. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. For $C_{13}H_{22}NO_3S$ 272.1320; Found: 272.1313. Yield: 90%; 61 mg; colorless oil; **R_f** = 0.35 (35% EtOAc/hexanes).

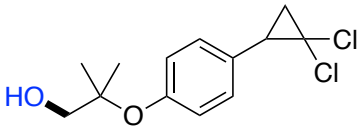
5-(2,5-Dimethylphenoxy)-2,2-dimethylpentan-1-ol (44)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.02 (d, *J* = 7.4 Hz, 1H), 6.70 – 6.62 (m, 2H), 3.94 (t, *J* = 6.4 Hz, 2H), 3.37 (s, 2H), 2.32 (s, 3H), 2.20 (s, 3H), 1.83 – 1.74 (m, 2H), 1.58 (s, 1H), 1.47 – 1.40 (m, 2H), 0.94 (s, 6H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 157.2, 136.6, 130.4, 123.7, 120.8, 112.2, 71.9, 68.7, 35.0, 34.9, 24.2, 24.0, 21.5, 15.9. **HRMS (**ESI**)** *m/z*: $[M+Na]^+$ calcd. For $C_{15}H_{24}O_2Na$ 259.1674; Found: 259.1681. Yield: 91%; 54 mg; light yellow oil; **R_f** = 0.38 (20% EtOAc/hexanes)

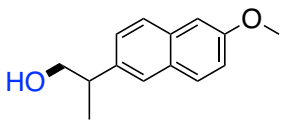
2-(4-(2,2-Dichlorocyclopropyl)phenoxy)-2-methylpropan-1-ol (45)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.18 – 7.12 (m, 2H), 6.99 – 6.93 (m, 2H), 3.59 (s, 2H), 2.86 (dd, *J* = 10.7, 8.3 Hz, 1H), 2.23 (s, 1H), 1.96 (dd, *J* = 10.7, 7.4 Hz, 1H), 1.80 (dd, *J* = 8.3, 7.4 Hz, 1H), 1.27 (s, 6H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 154.2, 149.6, 130.1, 129.7, 123.7, 81.0, 80.8, 70.4, 60.9, 35.0, 26.0, 23.2. **HRMS (**ESI**)** *m/z*: $[M+H]^+$ calcd. For $C_{13}H_{17}O_2Cl_2$ 275.0605; Found: 275.0620. Yield: 91%; 62.5 mg; yellow oil; **R_f** = 0.30 (20% EtOAc/hexanes).

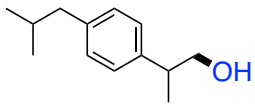
2-(6-Methoxynaphthalen-2-yl)propan-1-ol (46)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (dd, *J* = 8.5, 6.7 Hz, 2H), 7.61 (d, *J* = 1.8 Hz, 1H), 7.35 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.17 – 7.11 (m, 2H), 3.92 (s, 3H), 3.78 (d, *J* = 6.8 Hz, 2H), 3.09 (h, *J* = 7.0 Hz, 1H), 1.36 (d, *J* = 7.0 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 157.6, 138.8, 133.7, 129.3, 129.2, 127.4, 126.4, 126.0, 119.1, 105.7, 68.8, 55.5, 42.5, 17.8. Yield: 90%; 49 mg; white solid; **R_f** = 0.31 (25% EtOAc/hexanes). Spectral data matched those previously reported.[34]

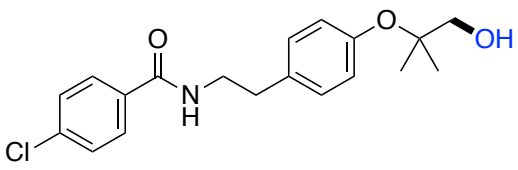
2-(4-Isobutylphenyl)propan-1-ol (47)



Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (q, *J* = 8.2 Hz, 4H), 3.69 (d, *J* = 6.9 Hz, 2H), 2.93 (h, *J* = 7.0 Hz, 1H), 2.45 (d, *J* = 7.2 Hz, 2H), 1.86 (dp, *J* = 13.6, 6.7 Hz, 1H), 1.43 (d, *J* = 2.0 Hz, 1H), 1.27 (d, *J* = 7.0 Hz, 3H), 0.91 (d, *J* = 6.6 Hz, 6H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 140.9, 140.2, 129.5, 127.3, 68.9, 45.2, 42.2, 30.4, 22.6, 17.8. Yield: 85%; 41 mg; colorless oil; **R_f** = 0.38 (20% EtOAc/hexanes). Spectral data matched those previously reported.[30]

4-Chloro-*N*-(4-((1-hydroxy-2-methylpropan-2-yl)oxy)phenethyl)benzamide (48)

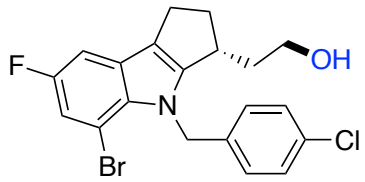


Reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 – 7.58 (m, 2H), 7.39 – 7.33 (m, 2H), 7.15 – 7.09 (m, 2H), 6.97 – 6.90 (m, 2H), 6.20 (t, *J* = 5.8 Hz, 1H), 3.67 (td, *J* = 7.0, 5.8 Hz, 2H), 3.57 (s, 2H), 2.88 (t, *J* = 7.0 Hz, 2H), 1.26

(s, 6H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 166.5, 153.4, 137.8, 134.2, 133.1, 129.5, 128.9, 128.4, 124.3, 80.8, 70.3, 41.4, 35.0, 23.2. **HRMS (**ESI) *m/z*: $[M+H]^+$ calcd. for $C_{19}H_{23}ClNO_3$ 348.1366; Found: 348.1356. Yield: 82%; 72 mg; off white crystalline powder; **R_f** = 0.28 (30% acetone/hexanes).

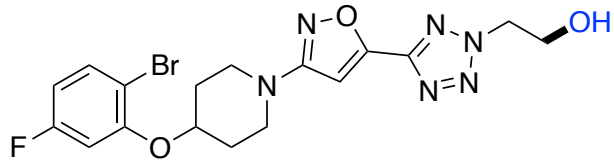
(*R*)-2-(5-Bromo-4-(4-chlorobenzyl)-7-fluoro-1,2,3,4-tetrahydrocyclopenta[*b*]indol-3-yl)ethan-1-ol (49)



Reaction was carried out on a 0.1 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.21 (m, 2H), 7.04 (ddd, *J* = 12.5, 8.8, 2.4 Hz, 2H), 6.82 – 6.78 (m, 2H), 5.84 (d, *J* = 17.3 Hz, 1H), 5.53 (d, *J* = 17.3 Hz, 1H), 3.69 (q, *J* = 6.1 Hz, 2H), 3.31 – 3.23 (m, 1H), 2.91 – 2.81 (m, 1H), 2.79 – 2.64 (m, 2H), 2.29 – 2.17 (m, 1H), 1.87 (dtd, *J* = 14.2, 7.2, 3.7 Hz, 1H), 1.71 – 1.61 (m, 1H), 1.54 (s, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 158.3, 155.9, 152.5, 138.1, 134.2, 133.1, 129.0, 127.0, 119.4 (d, *J* = 4.6 Hz), 114.4, 114.1, 103.6 (d, *J* = 22.4 Hz), 61.0, 48.8, 37.3, 35.6, 35.0, 23.3. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -123.5. **HRMS (**ESI) *m/z*: $[M+H]^+$ calcd. for $C_{20}H_{19}BrClFNO$ 422.0322; Found: 422.0308. Yield: 80%; 34 mg; off white solid; **R_f** = 0.31 (30% EtOAc/hexanes).

2-(5-(3-(4-(2-Bromo-5-fluorophenoxy)piperidin-1-yl)isoxazol-5-yl)-2*H*-tetrazol-2-yl)ethan-1-ol (50)

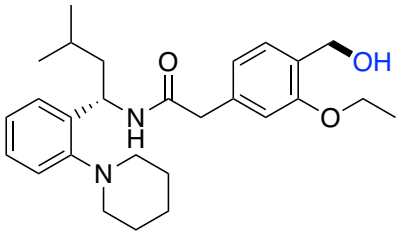


The reaction was carried out on a 0.1 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 (dd, *J* = 8.8, 6.2 Hz, 1H), 6.71 – 6.66 (m, 2H), 6.61 (ddd, *J* = 8.7, 7.8, 2.8 Hz, 1H), 4.88 – 4.83 (m, 2H), 4.61 (tt, *J* = 6.0, 3.3 Hz, 1H), 4.30 – 4.23 (m, 2H), 3.65 (ddd, *J* = 12.8, 8.8, 3.9 Hz, 2H), 3.42 (dt, *J* = 13.1, 5.0 Hz, 2H), 2.03 (tdd, *J* = 13.7, 8.5, 4.4 Hz, 4H), 1.25 (d, *J* = 1.7 Hz, 1H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 166.9, 163.7, 161.8, 158.4, 158.2, 156.2, 154.6 (d, *J* = 8.0 Hz), 149.5, 134.0 (d, *J* = 7.7 Hz), 109.4 (d, *J* = 18.0 Hz), 103.4 (d, *J* = 20.8 Hz), 96.3, 73.1, 60.6, 60.4, 56.0, 44.1, 29.9,

29.3, 22.5, 21.2, 14.3, 14.2. **^{19}F NMR (376 MHz, $CDCl_3$)** δ -112.0. **HRMS (**ESI) *m/z*: $[M+H]^+$ calcd. for $C_{17}H_{19}BrFN_6O_3$ 453.0686; Found: 453.0691. Yield: 61%; 28 mg; off white solid; R_f = 0.14 (40% acetone/hexanes).

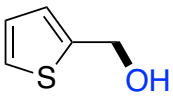
(*S*)-2-(3-Ethoxy-4-(hydroxymethyl)phenyl)-*N*-(3-methyl-1-(2-(piperidin-1-yl)phenyl)butyl)-acetamide (51)



The reaction was carried out on a 0.25 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, DMSO) δ 8.36 (d, *J* = 8.6 Hz, 1H), 7.31 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 7.15 (ddd, *J* = 8.5, 7.2, 1.6 Hz, 1H), 7.12 – 6.98 (m, 2H), 6.83 – 6.74 (m, 2H), 5.37 (td, *J* = 9.3, 4.7 Hz, 1H), 4.88 (t, *J* = 5.6 Hz, 1H), 4.44 (d, *J* = 5.6 Hz, 2H), 3.98 – 3.90 (m, 2H), 3.40 (s, 2H), 3.08 (s, 2H), 2.53 (s, 1H), 1.67 (s, 2H), 1.61 – 1.41 (m, 6H), 1.31 (t, *J* = 7.0 Hz, 4H), 0.90 (dd, *J* = 6.5, 3.5 Hz, 6H). **^{13}C NMR (101 MHz, DMSO)** δ 169.4, 155.0, 151.5, 140.6, 136.1, 128.4, 127.1, 126.8, 126.0, 124.0, 120.6, 120.5, 111.6, 63.0, 57.7, 46.6, 45.9, 42.6, 26.3, 24.9, 23.8, 23.2, 21.8, 14.7. **HRMS (**ESI) *m/z*: $[M+Na]^+$ calcd. for $C_{27}H_{38}N_2O_3Na$ 461.2780; Found: 461.2774. Yield: 96%; 107 mg; white solid; **R_f** = 0.34 (50% EtOAc/hexanes).

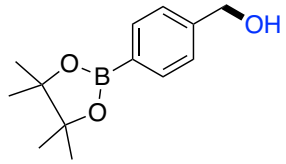
Thiophen-2-ylmethanol



The reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (dd, *J* = 5.0, 1.3 Hz, 1H), 7.03 – 6.95 (m, 2H), 4.81 (d, *J* = 0.8 Hz, 2H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 144.1, 127.0, 125.7, 125.6, 60.0. Yield: 84%; 48 mg; yellow oil; **R_f** = 0.21 (20% EtOAc/hexanes). Spectral data matched those previously reported.[18]

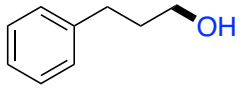
(4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)methanol



The reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.9 Hz, 2H), 7.34 (d, *J* = 7.8 Hz, 2H), 4.68 (s, 2H), 1.34 (s, 12H). **^{13}C NMR (126 MHz, $CDCl_3$)** δ 144.2, 135.1, 126.2, 83.9, 65.2, 24.9. Yield: 68%; 80 mg; colorless oil; $\mathbf{R_f}$ = 0.23 (20% EtOAc/hexanes). Spectral data matched those previously reported.[19]

3-Phenylpropan-1-ol



The reaction was carried out on a 0.5 mmol scale using Procedure 7.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 2H), 7.23 – 7.16 (m, 3H), 3.69 (t, *J* = 6.4 Hz, 2H), 2.72 (dd, *J* = 8.6, 6.8 Hz, 2H), 1.96 – 1.85 (m, 2H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 142.0, 128.5, 128.5, 126.0, 77.5, 62.3, 34.3, 32.2. Yield: 90%; 61 mg; colorless oil; $\mathbf{R_f}$ = 0.32 (30% EtOAc/ hexanes). Spectral data matched those previously reported.[31]

8. References

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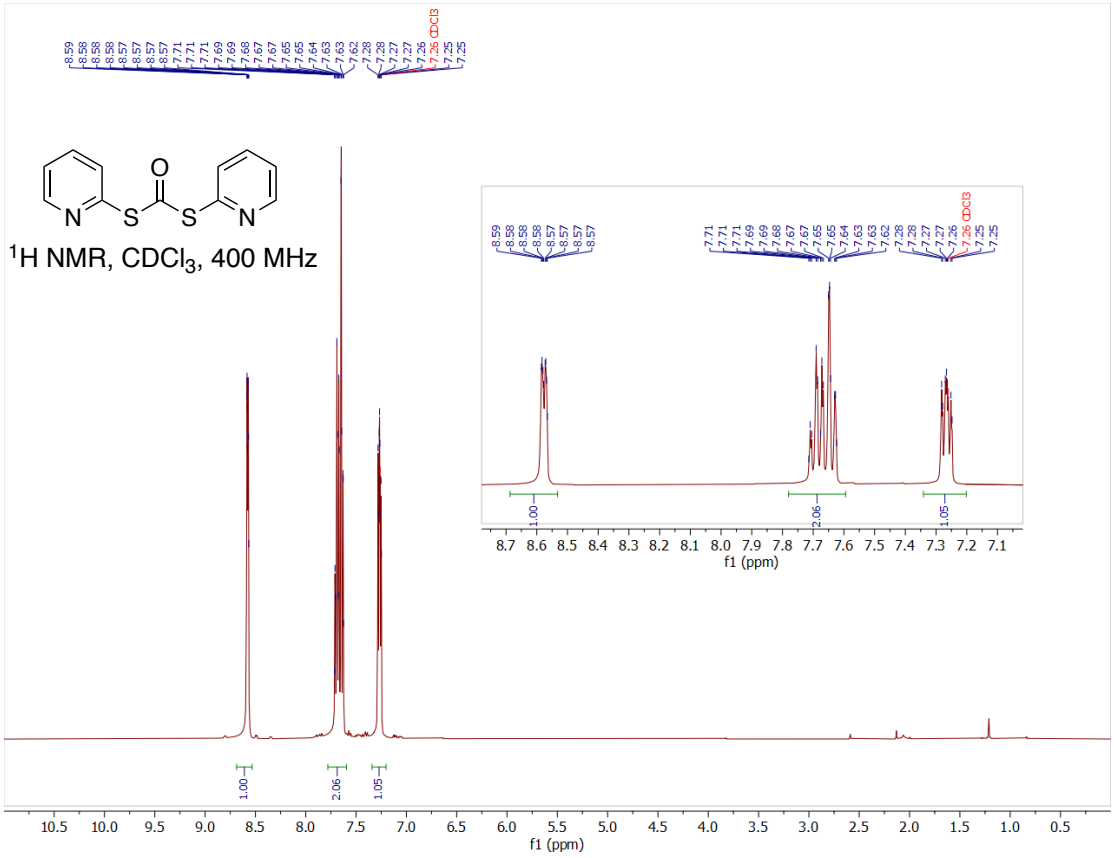
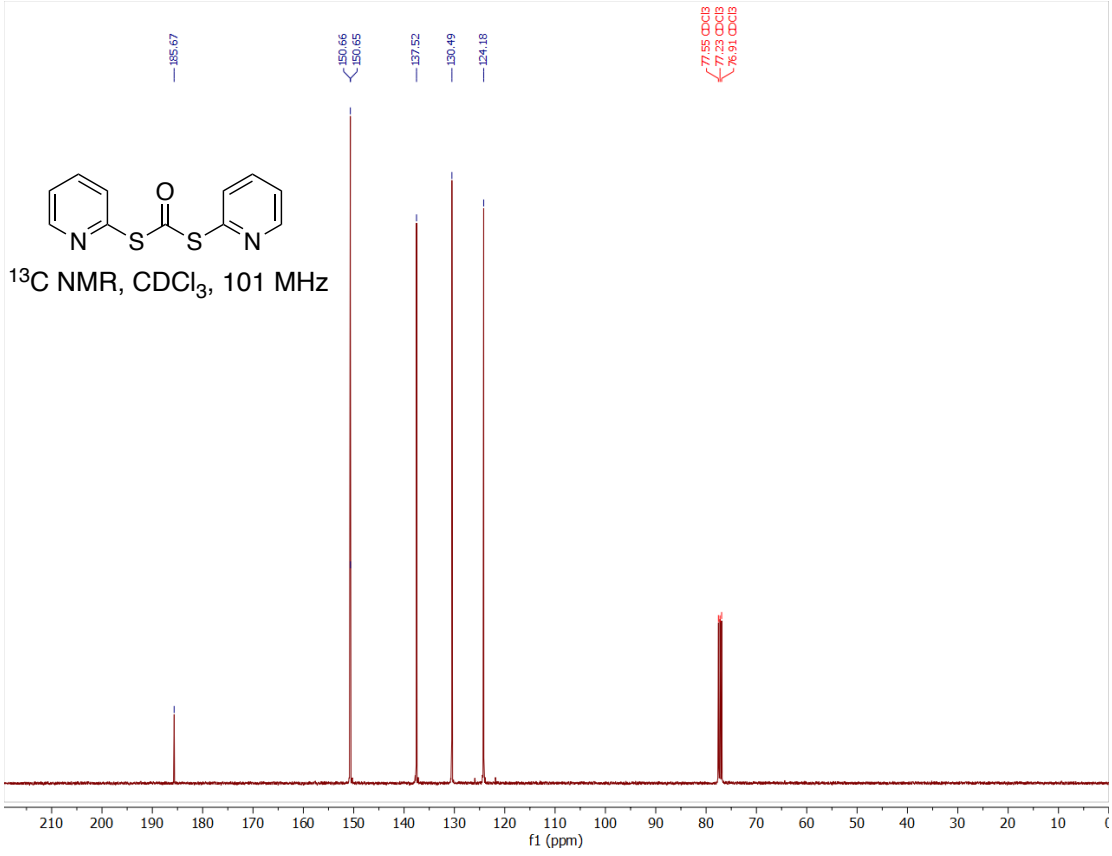
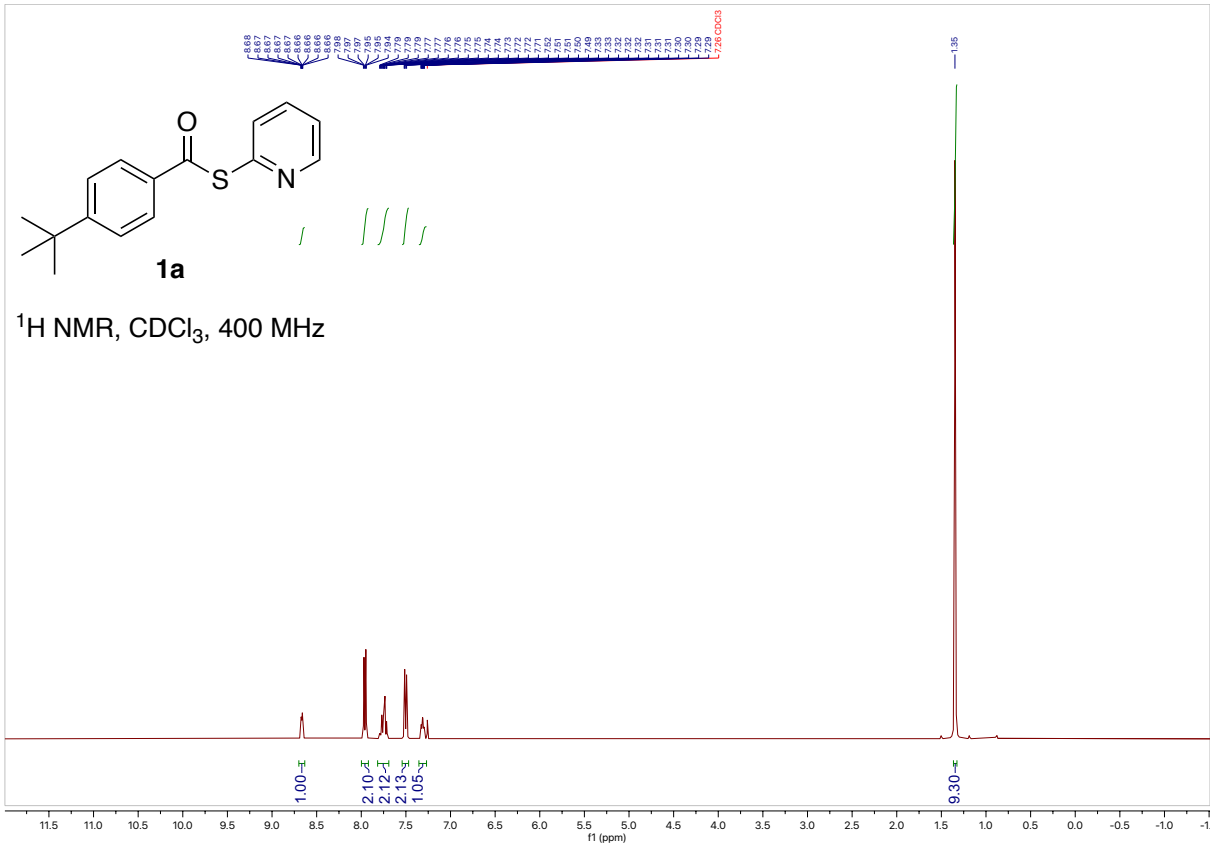
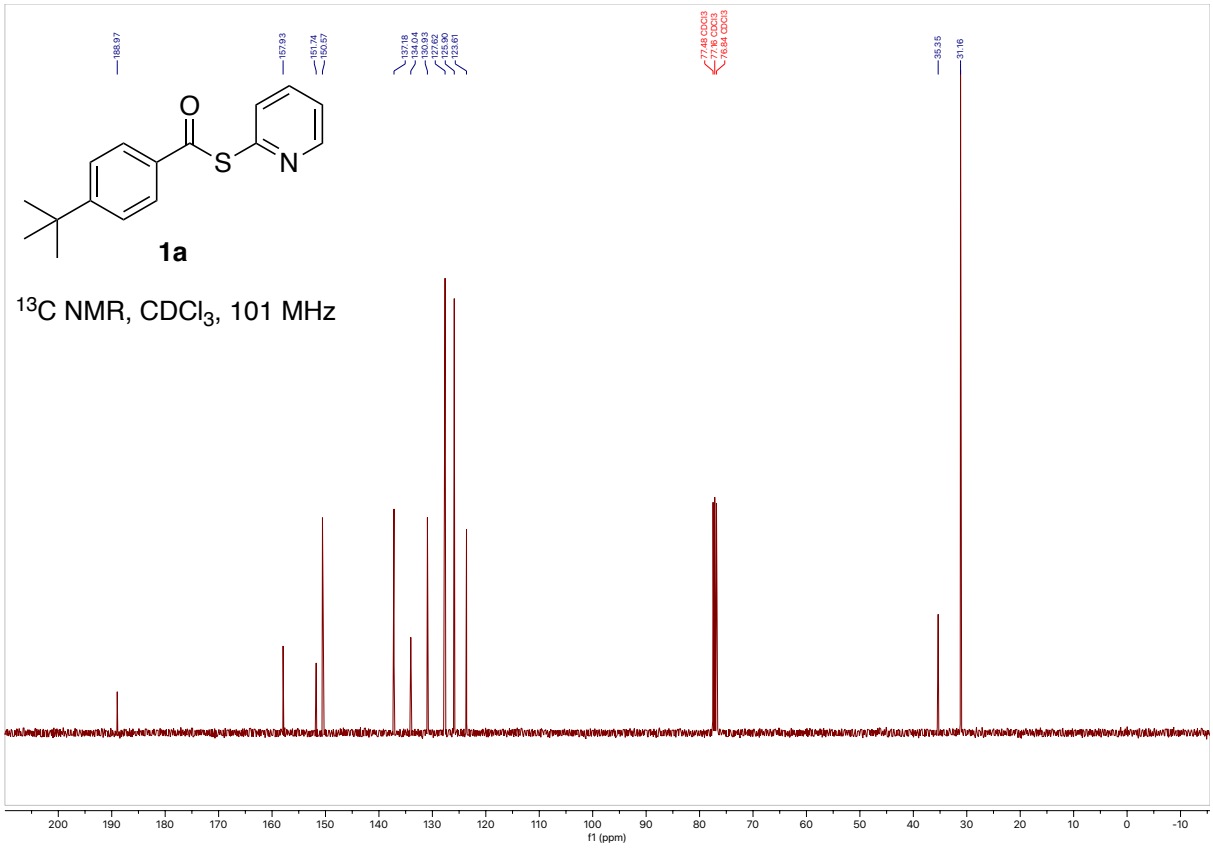
^{1}H, ^{13}C, ^{19}F NMR Spectra of synthesized products


^{1}H NMR, $CDCl_3$, 400 MHz
f1 (ppm)

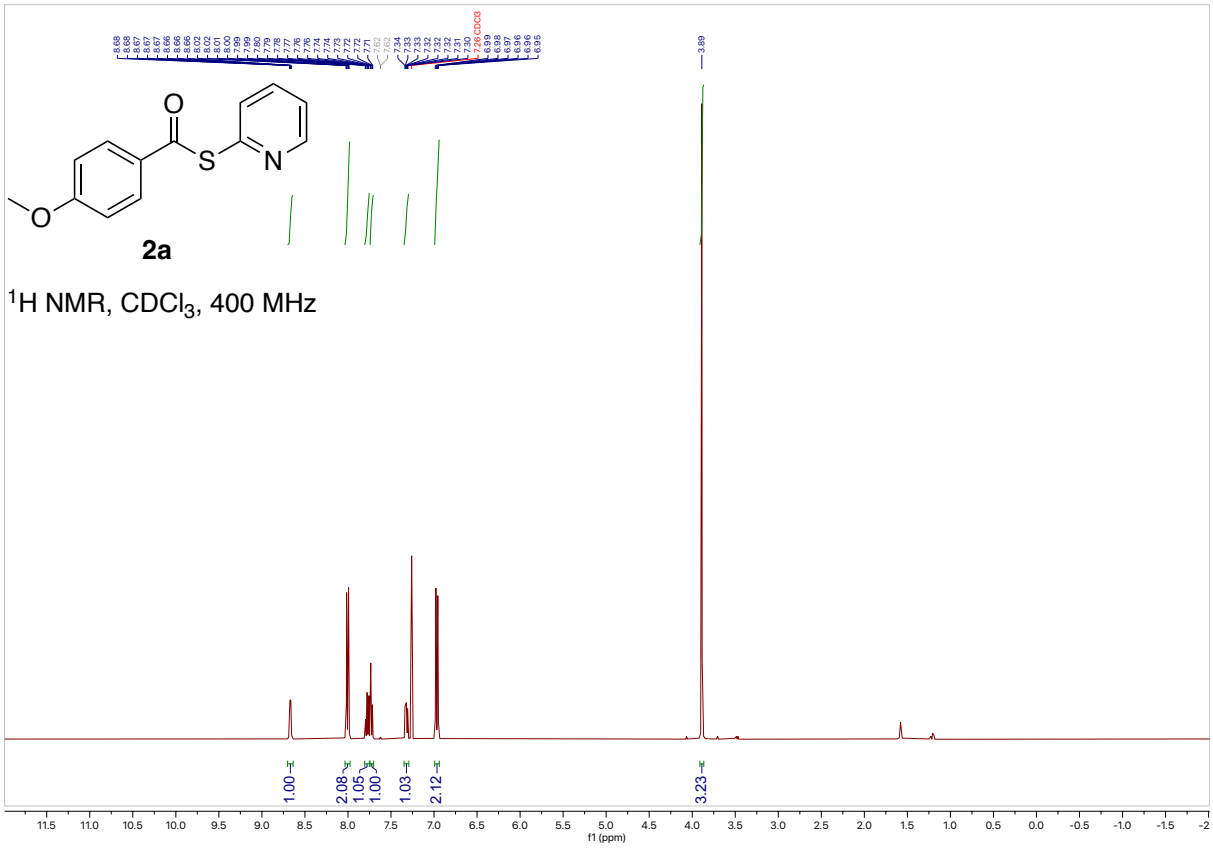

^{13}C NMR, $CDCl_3$, 101 MHz
f1 (ppm)

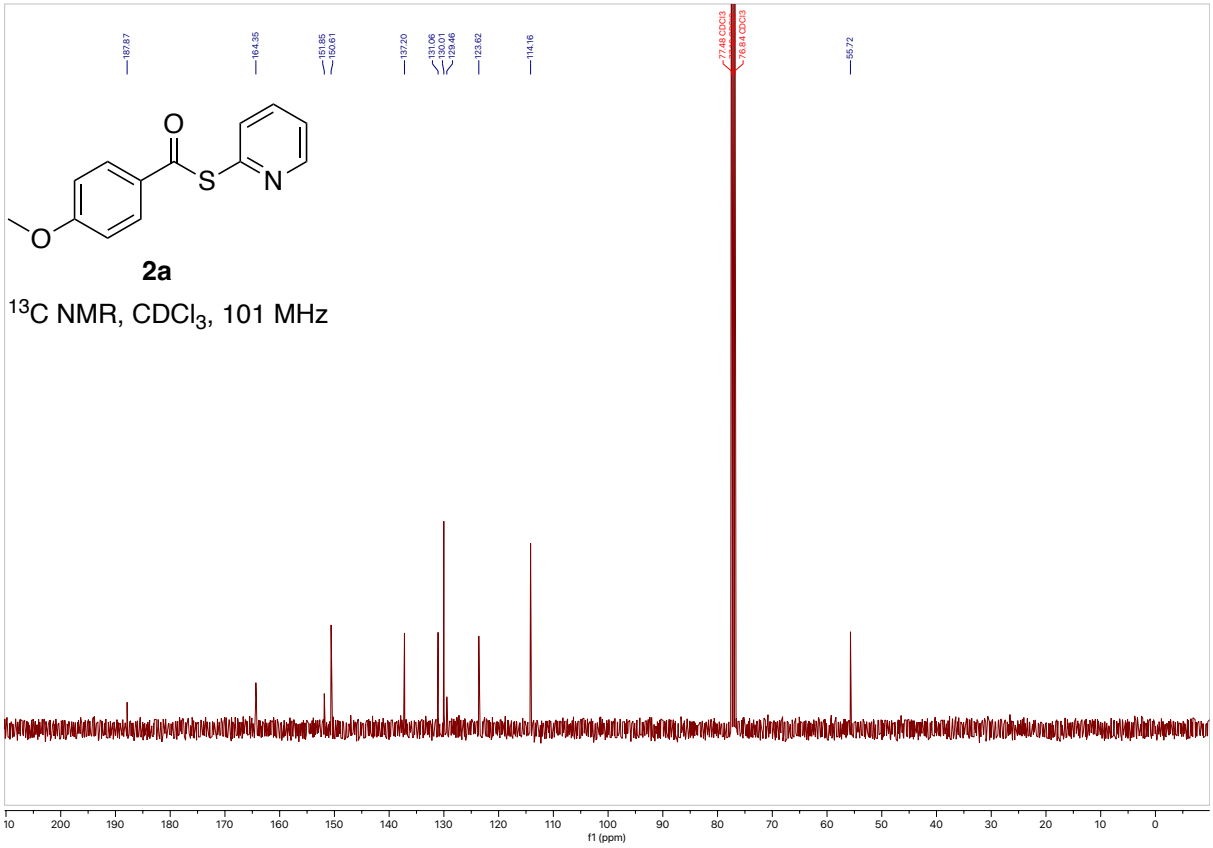


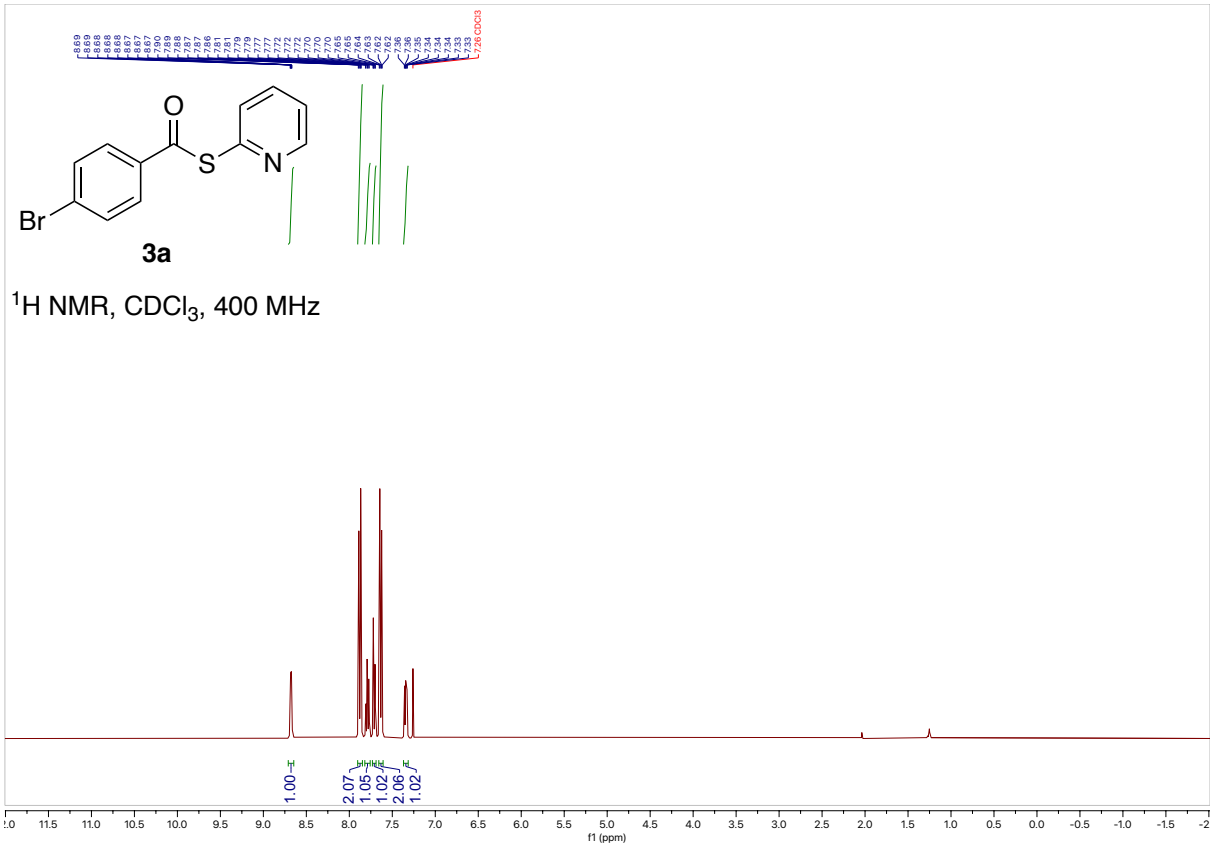
^{1}H NMR, $CDCl_3$, 400 MHz



^{13}C NMR, $CDCl_3$, 101 MHz

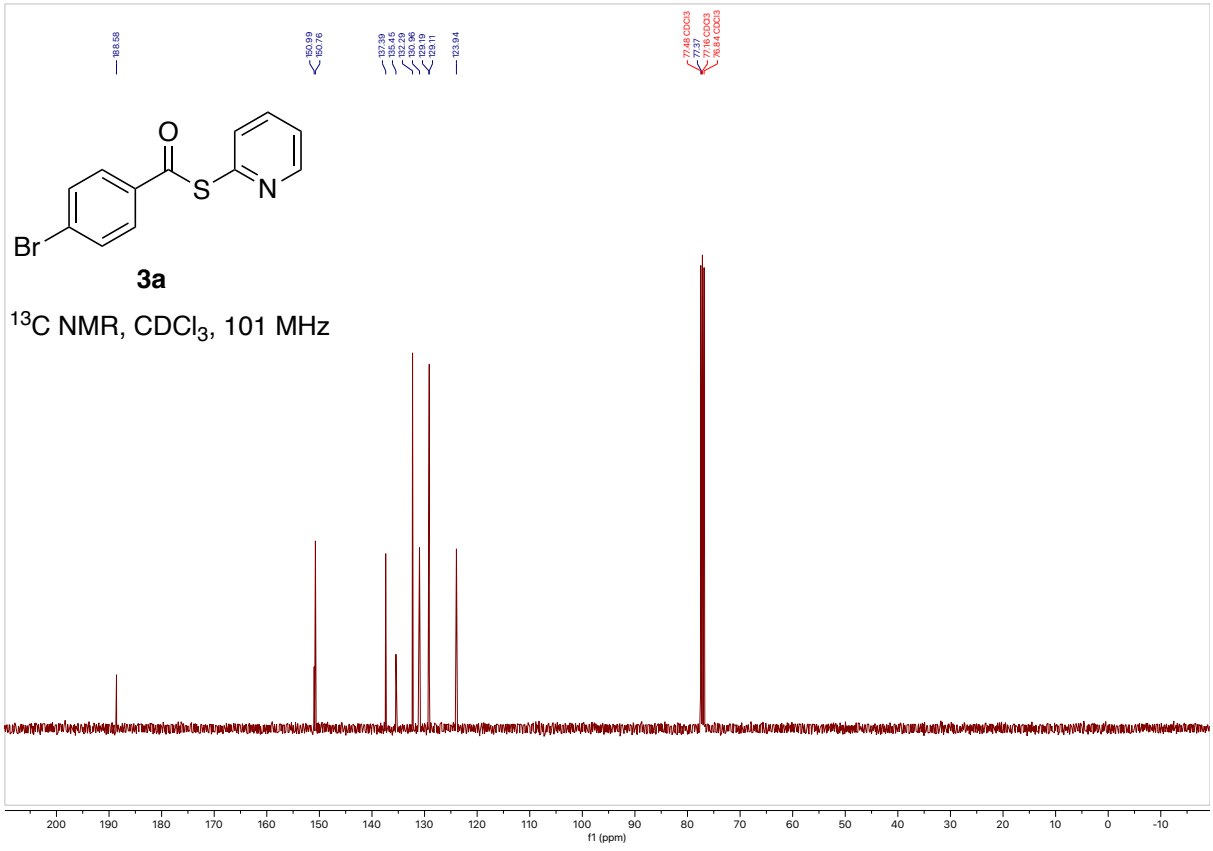

O
S
N
O
2a
1H NMR, CDCl3, 400 MHz
1.00
2.08
1.05
1.00
1.03
2.12
3.23
f1 (ppm)


O
S
N
O
2a
13C NMR, CDCl3, 101 MHz
f1 (ppm)



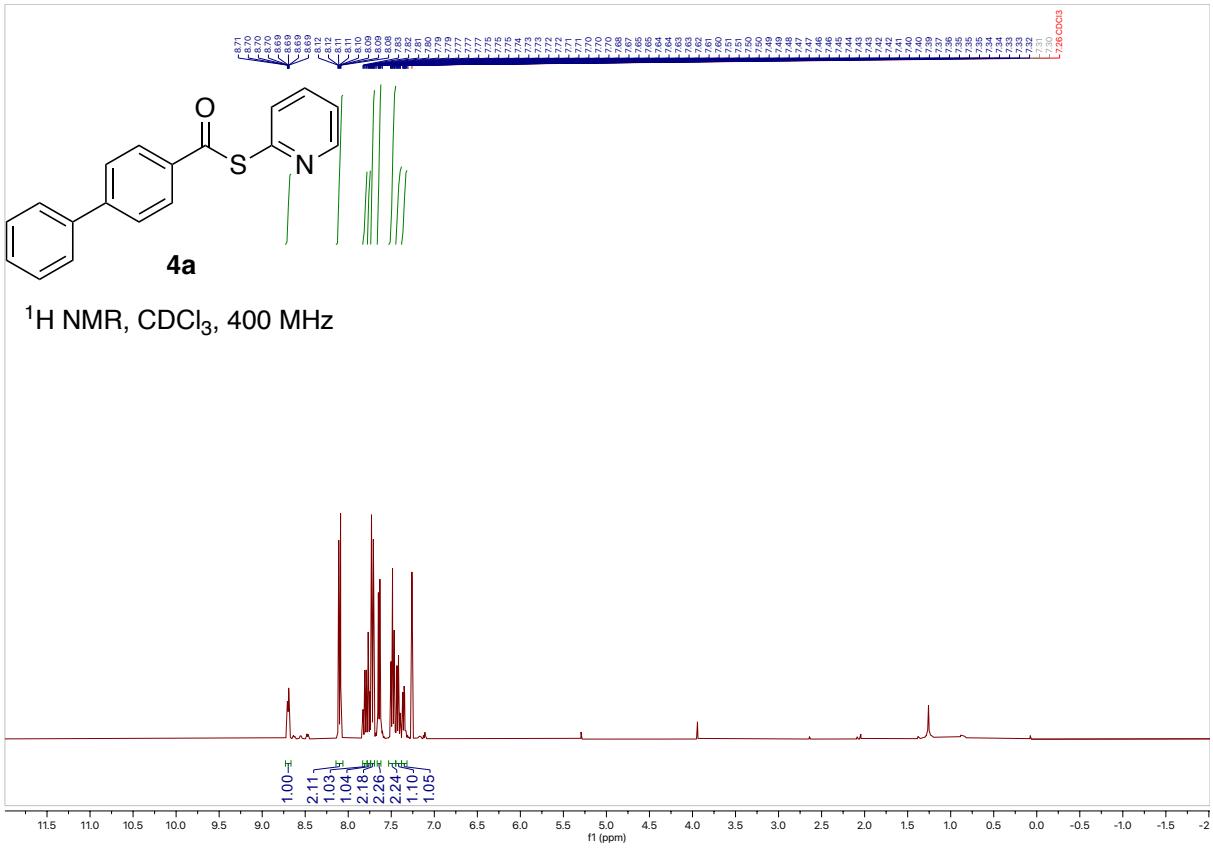
3a

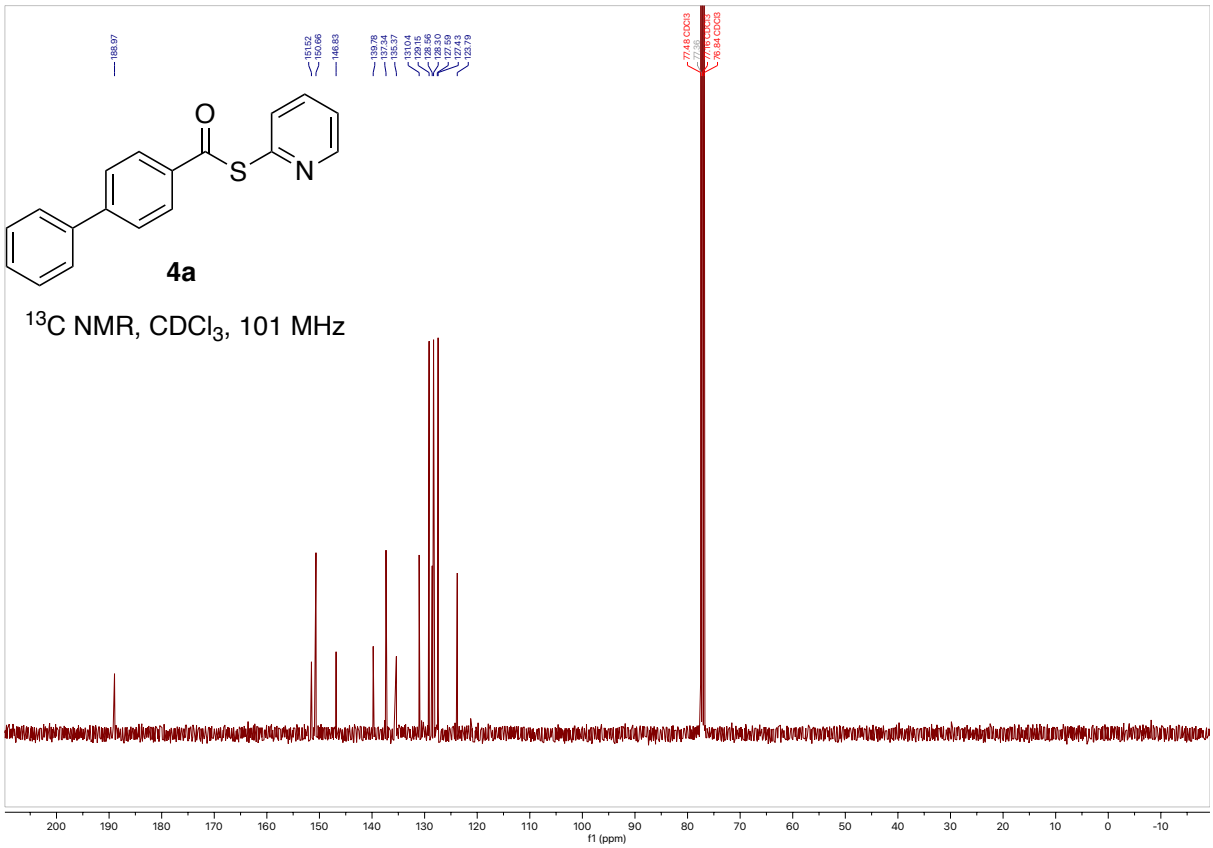
^{1}H NMR, $CDCl_3$, 400 MHz

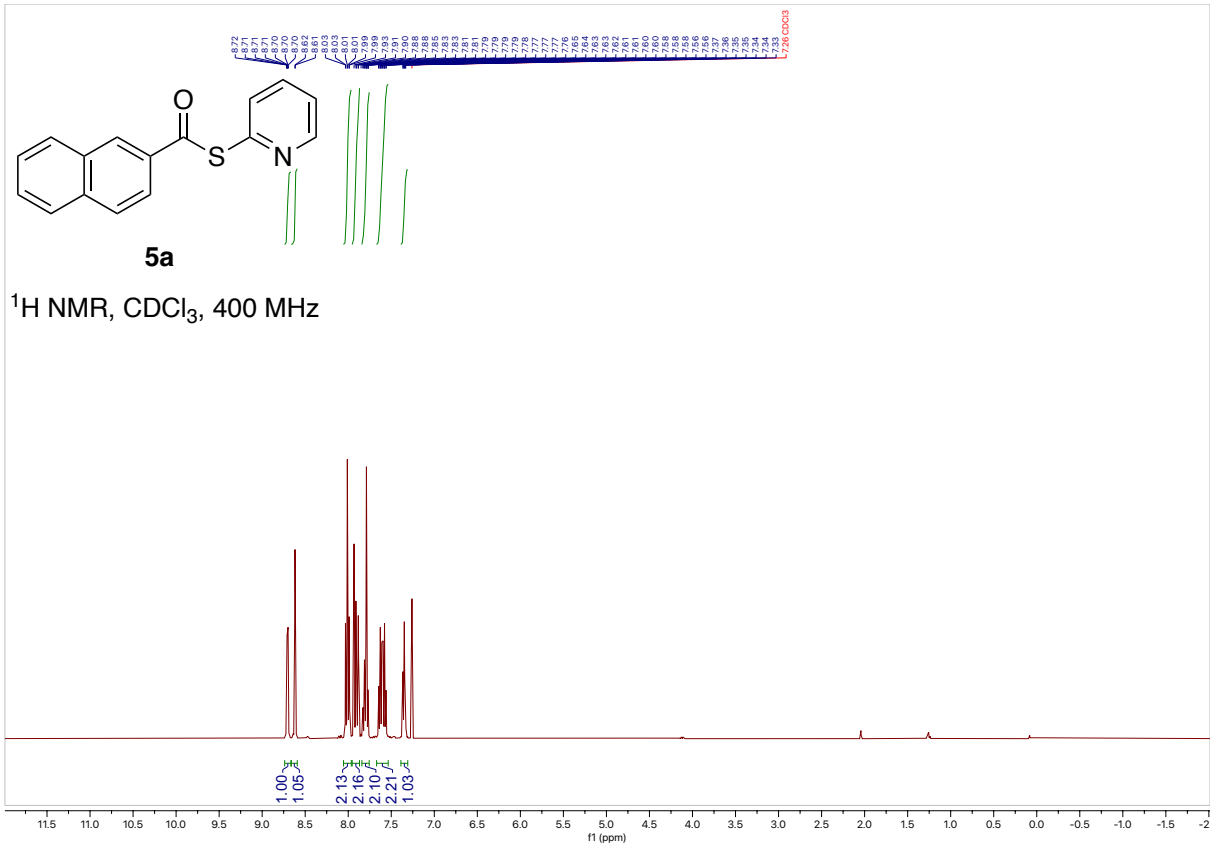


3a

^{13}C NMR, $CDCl_3$, 101 MHz


O
S
N
4a
1H NMR, CDCl3, 400 MHz
1.00
2.11
1.03
1.04
2.18
2.26
2.24
1.10
1.05
f1 (ppm)


O
S
N
4a
13C NMR, CDCl3, 101 MHz
f1 (ppm)


8.72
8.71
8.71
8.71
8.71
8.70
8.70
8.70
8.70
8.62
8.61
8.03
8.03
8.01
8.01
7.99
7.99
7.93
7.91
7.90
7.88
7.88
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7.81
7.81
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7.79
7.79
7.79
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7.75
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7.63
7.63
7.62
7.61
7.61
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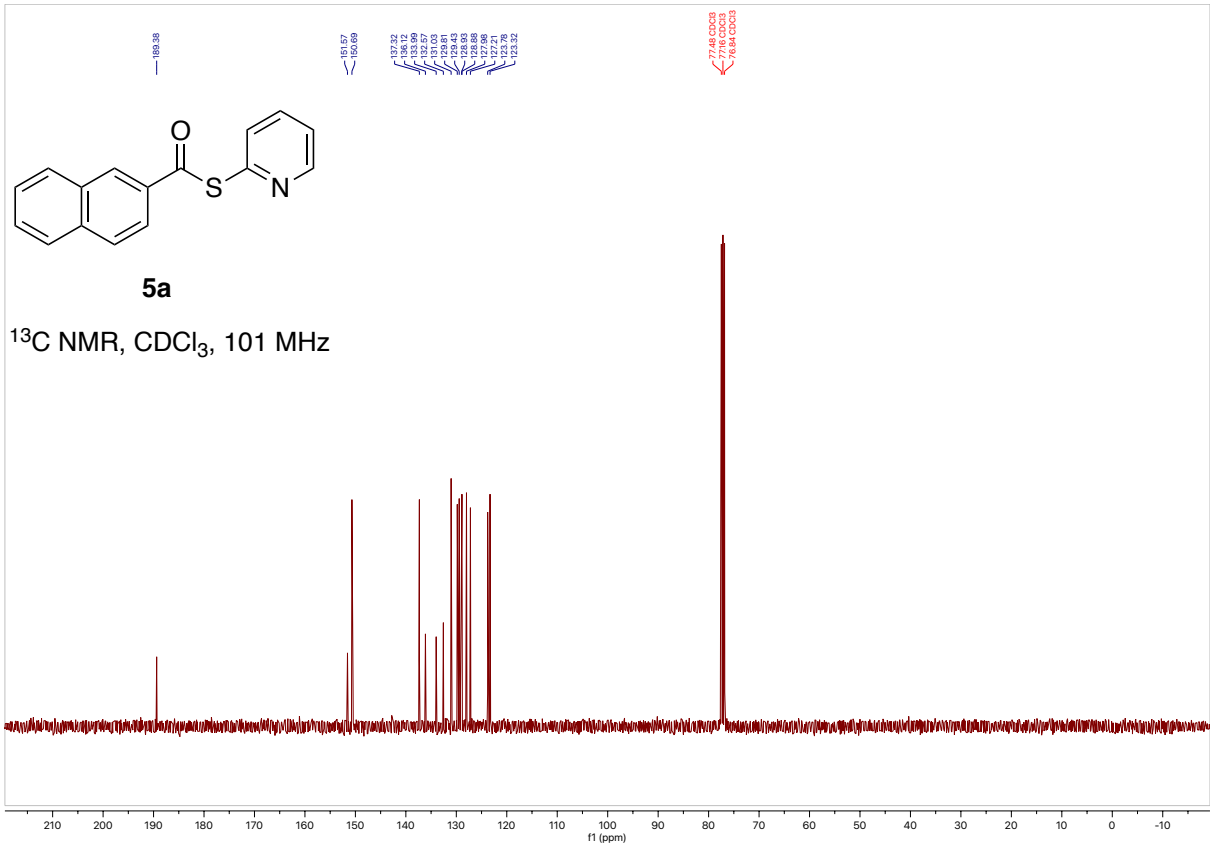
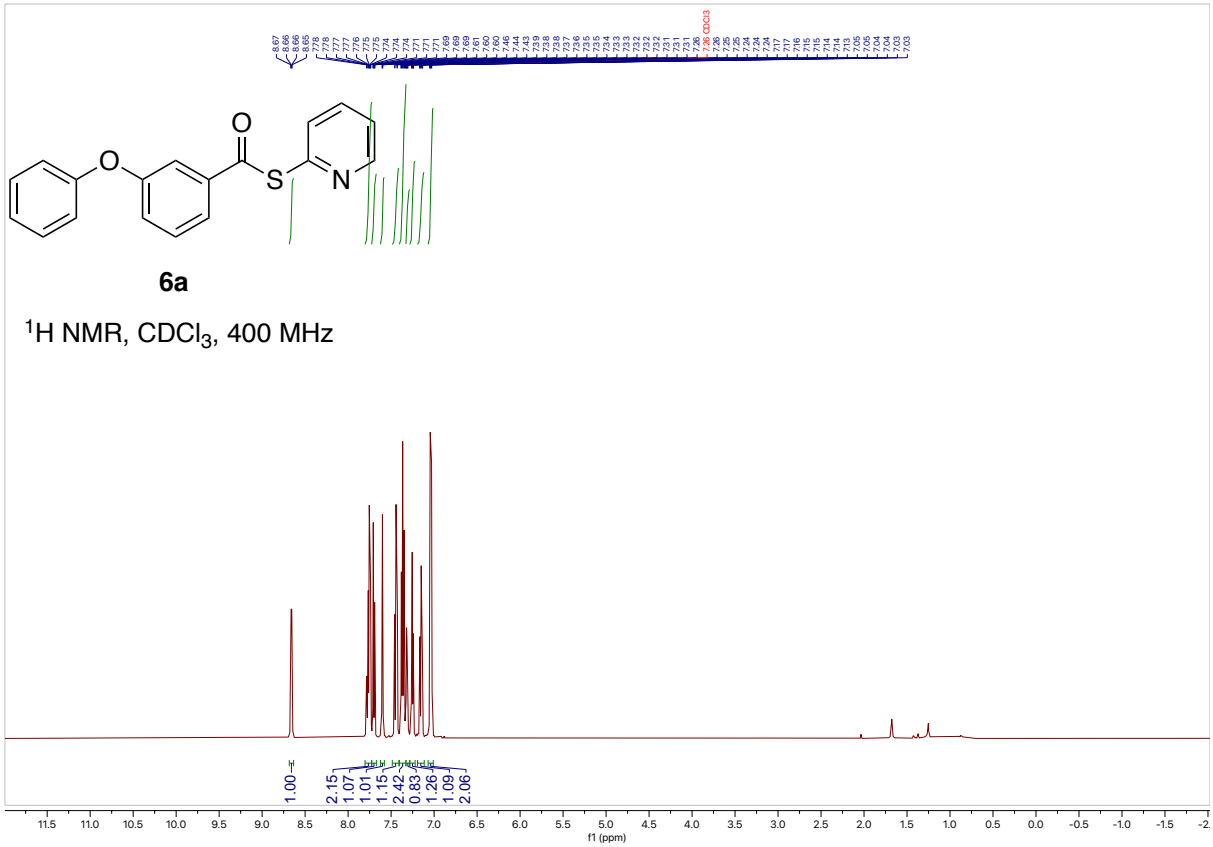
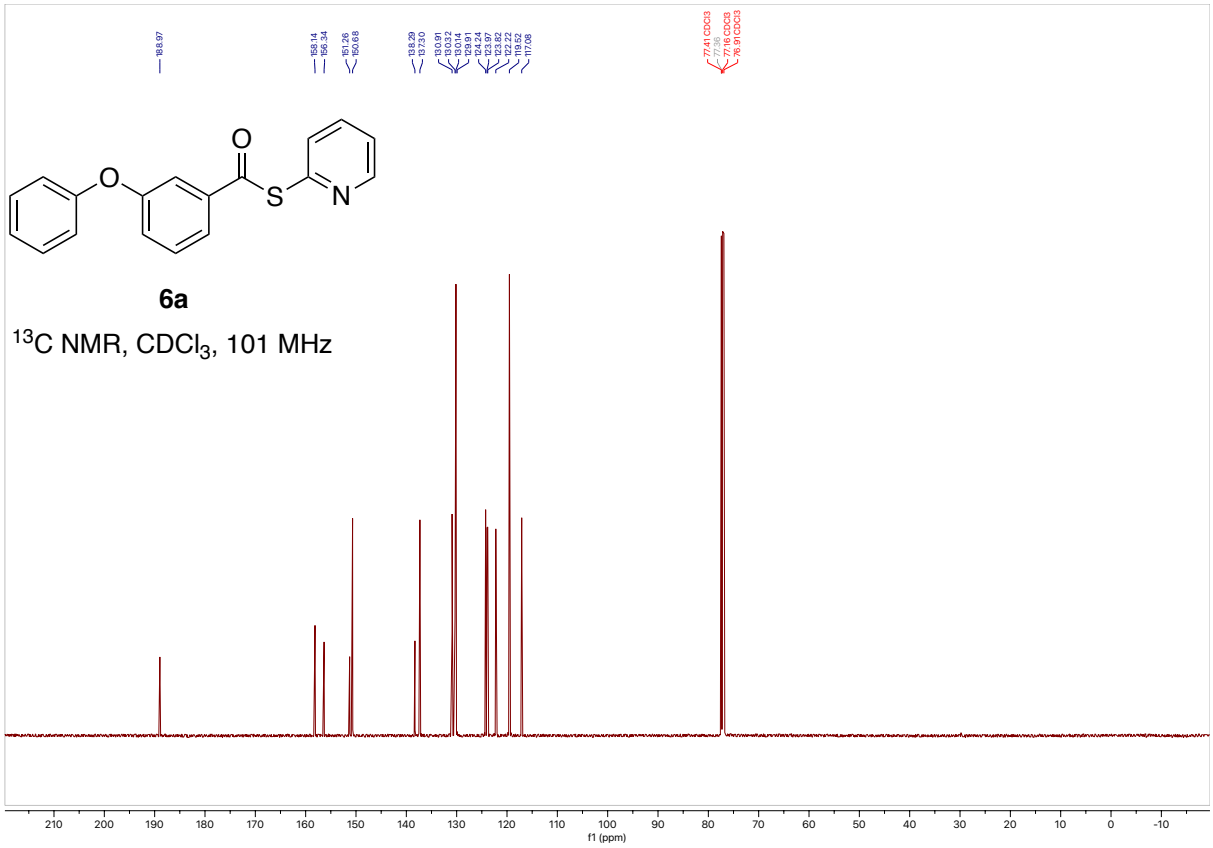
7.58
7.58
7.58
7.58
7.56
7.56
7.37
7.36
7.35
7.35
7.34
7.34
7.33
7.26 CDCl3
O
S
N
5a
1H NMR, CDCl3, 400 MHz
1.00
1.05
2.13
2.16
2.10
2.21
1.03
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2
f1 (ppm)


189.38
151.57
150.69
137.32
136.12
133.99
132.57
131.03
129.81
129.43
128.93
128.88
127.98
122.21
123.78
123.32
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
S
N
5a
13C NMR, CDCl3, 101 MHz
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)



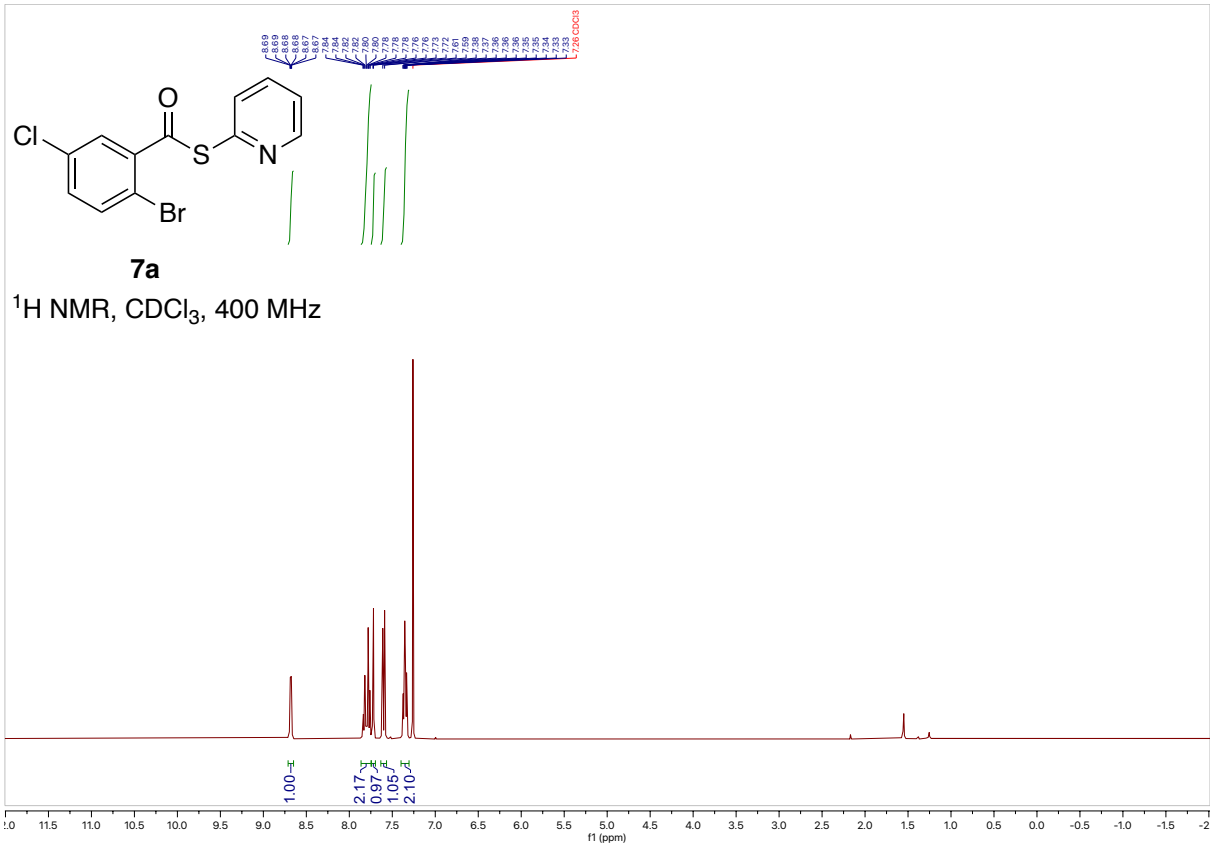
6a

^{1}H NMR, $CDCl_3$, 400 MHz



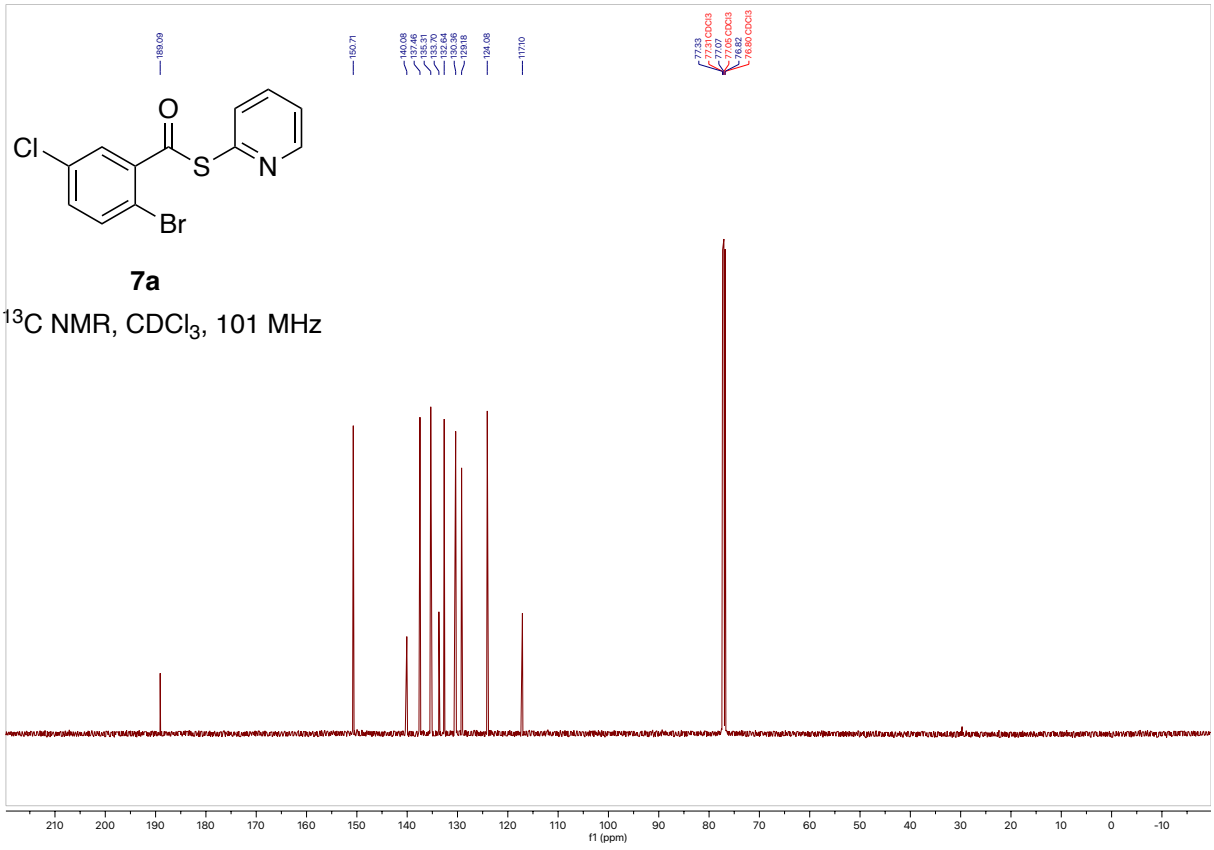
6a

^{13}C NMR, $CDCl_3$, 101 MHz



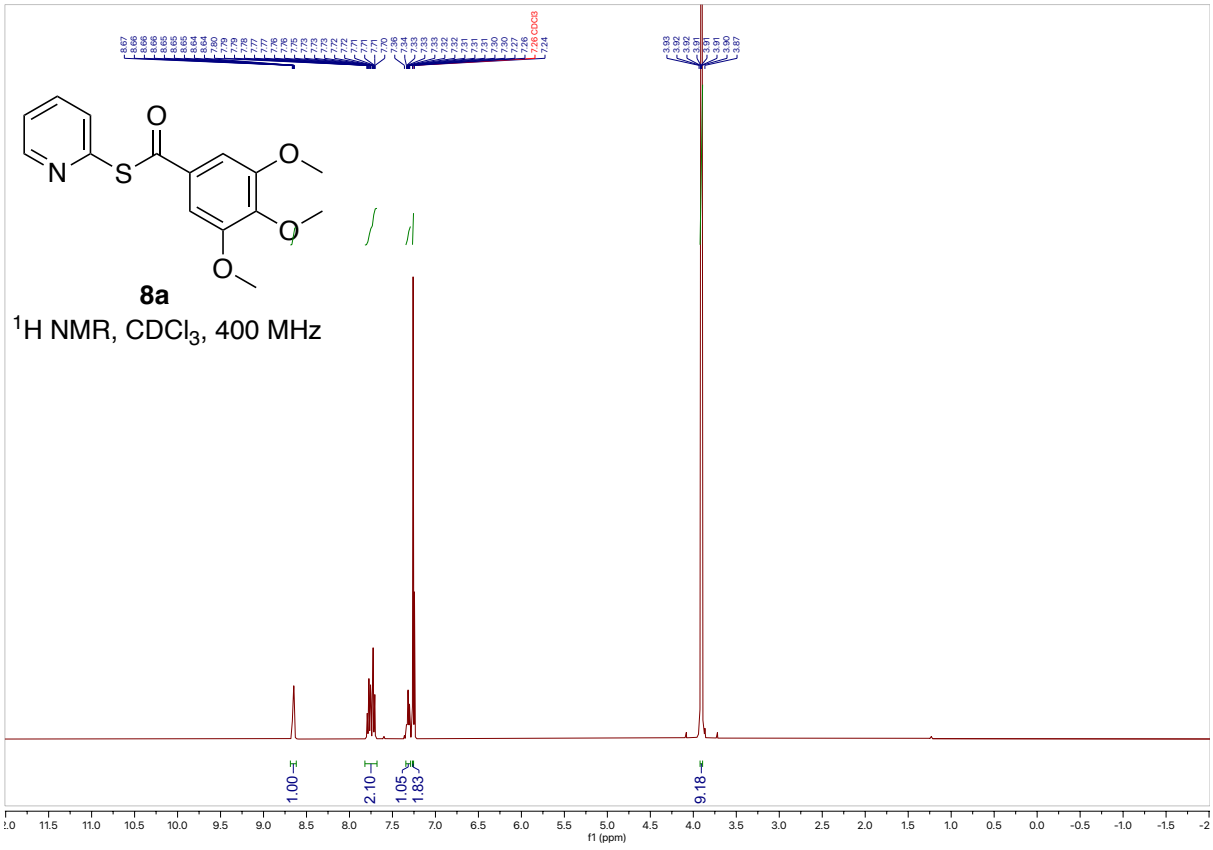
7a

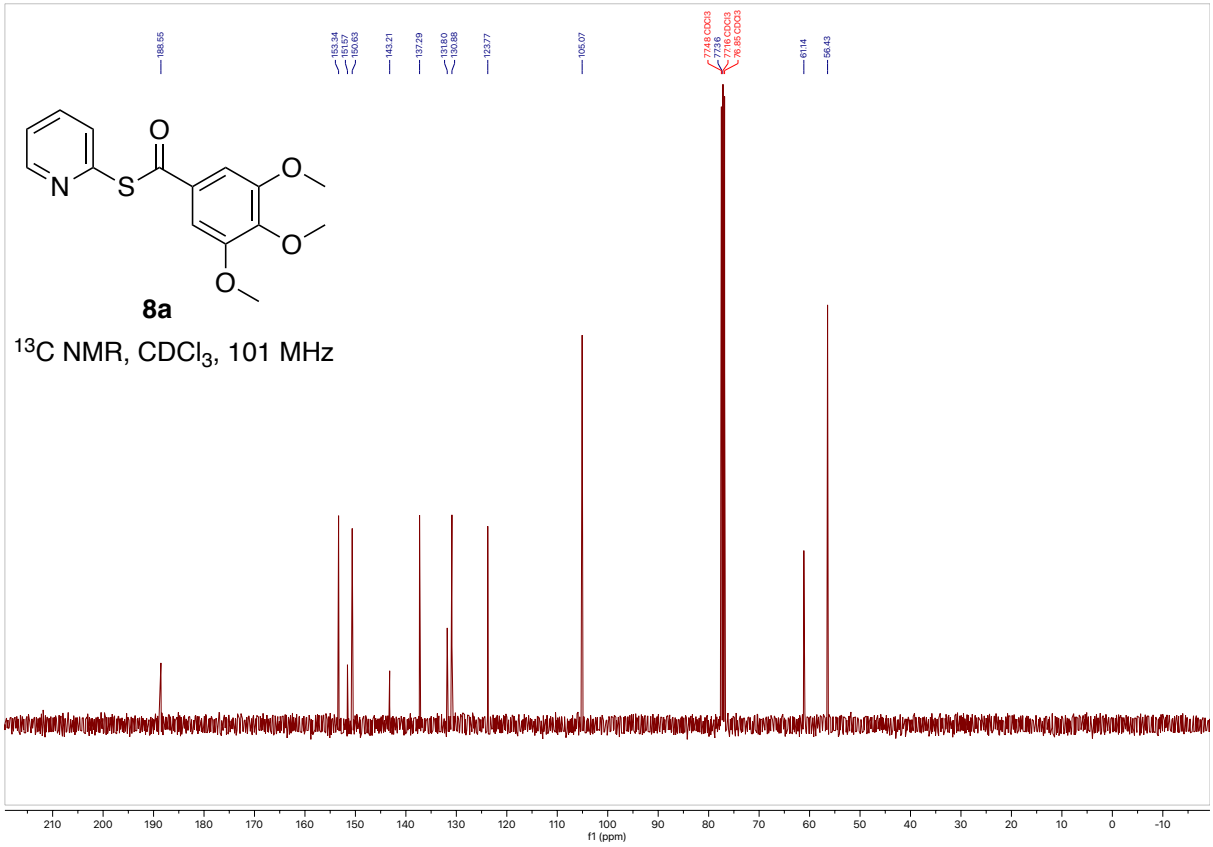
^{1}H NMR, $CDCl_3$, 400 MHz

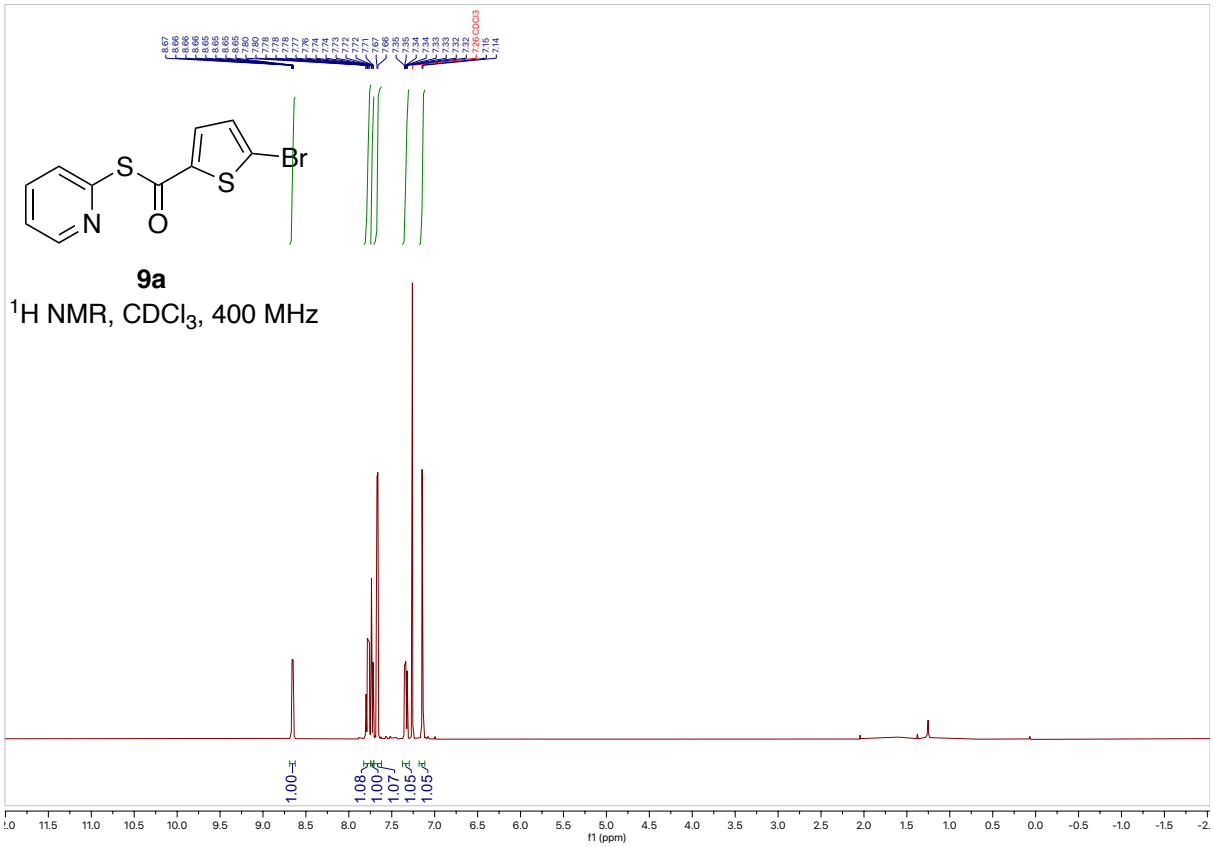


7a

^{13}C NMR, $CDCl_3$, 101 MHz

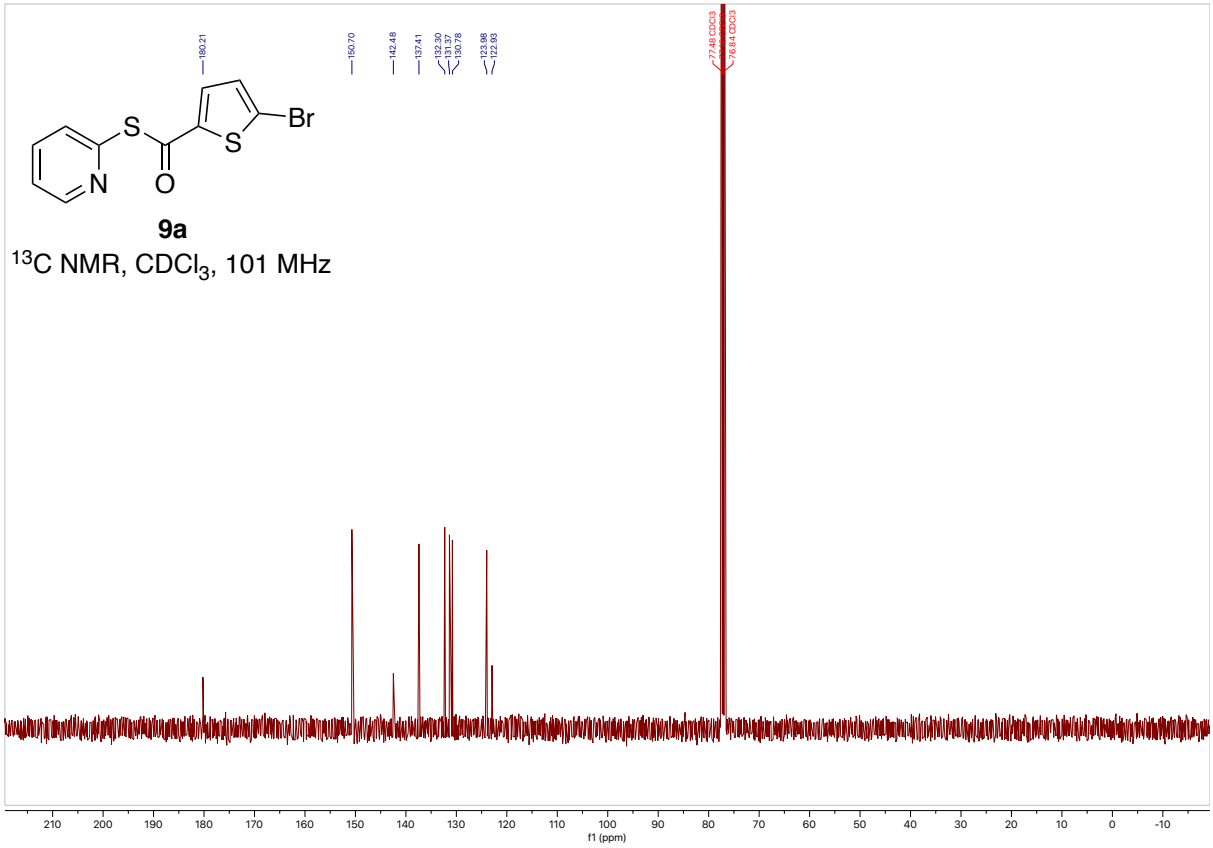

8a
1H NMR, CDCl3, 400 MHz
1.00
2.10
1.05
1.83
9.18
f1 (ppm)


8a
13C NMR, CDCl3, 101 MHz
188.55
153.34
151.57
150.63
143.21
137.29
131.80
130.88
123.77
105.07
77.48 CDCl3
77.16 CDCl3
76.85 CDCl3
61.14
56.43
f1 (ppm)



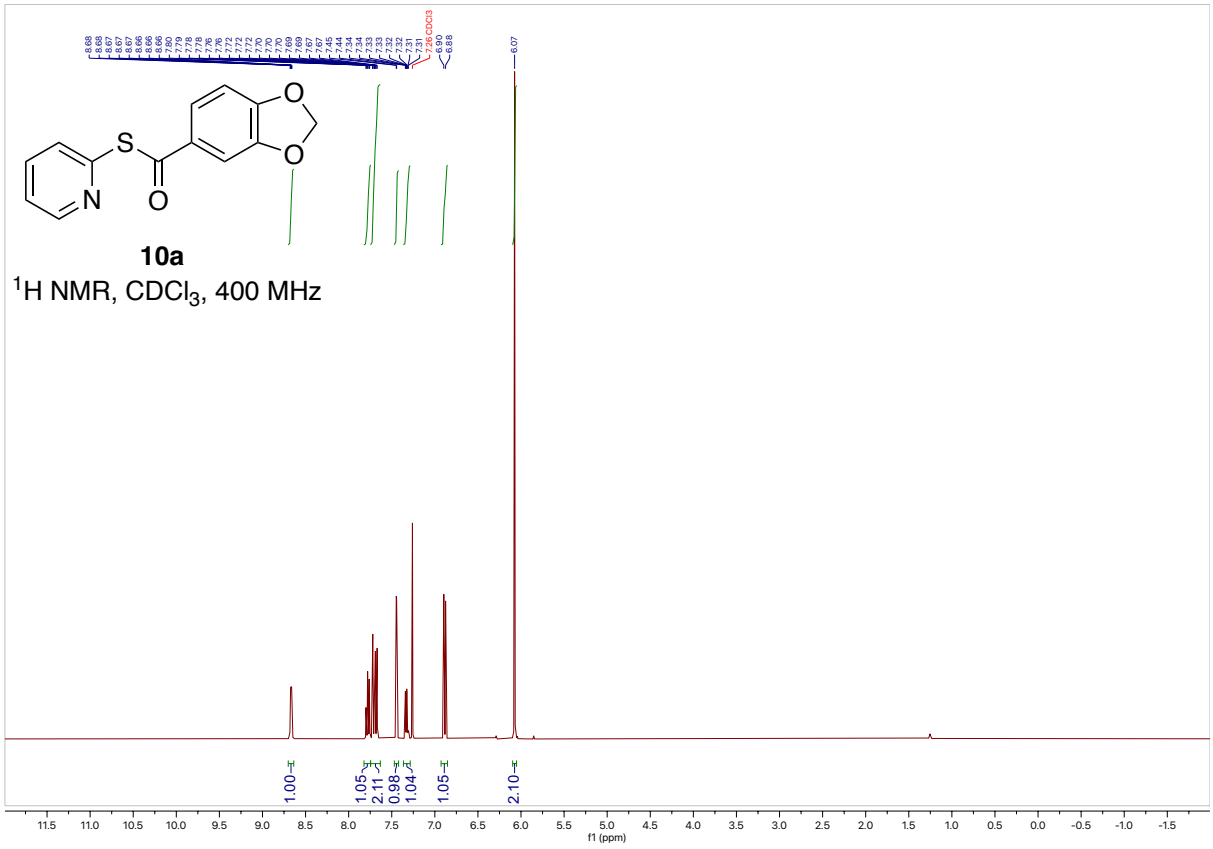
9a

^{1}H NMR, $CDCl_3$, 400 MHz



9a

^{13}C NMR, $CDCl_3$, 101 MHz



10a

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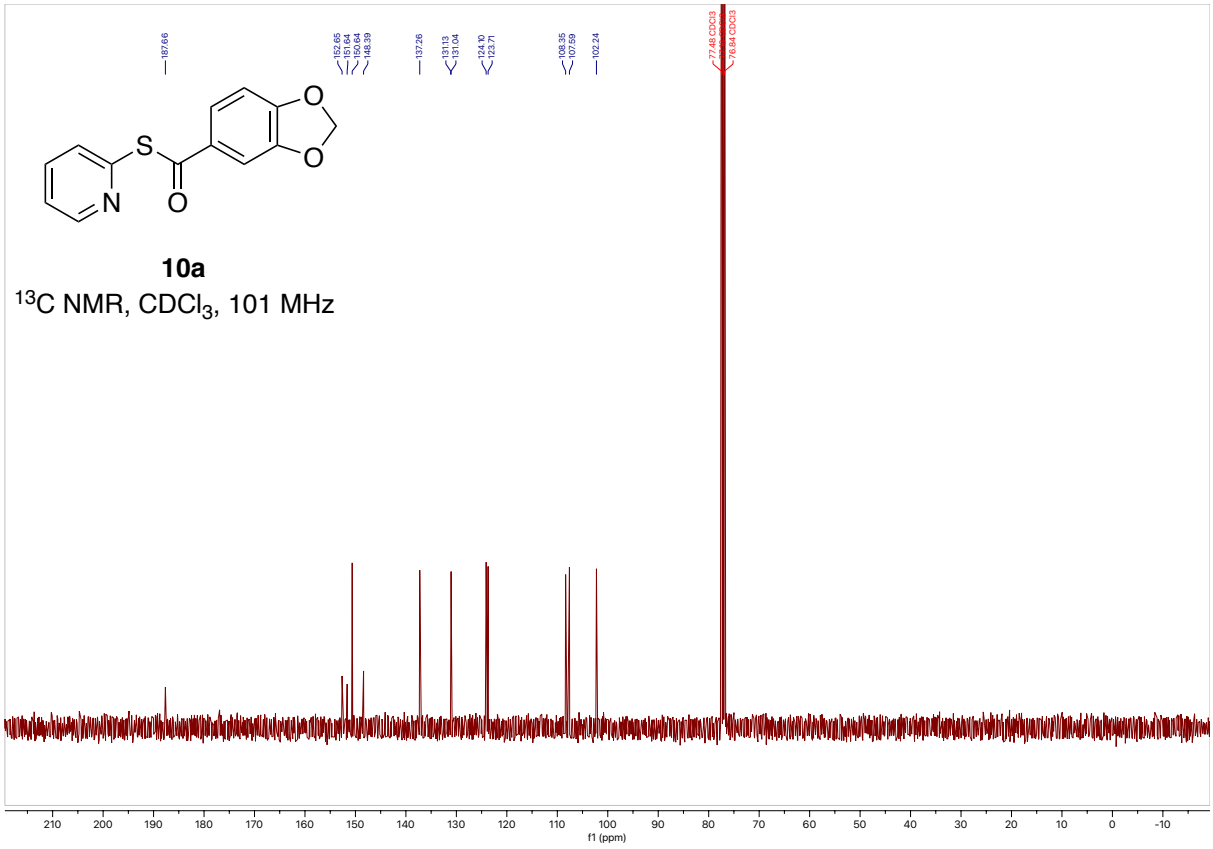

10a

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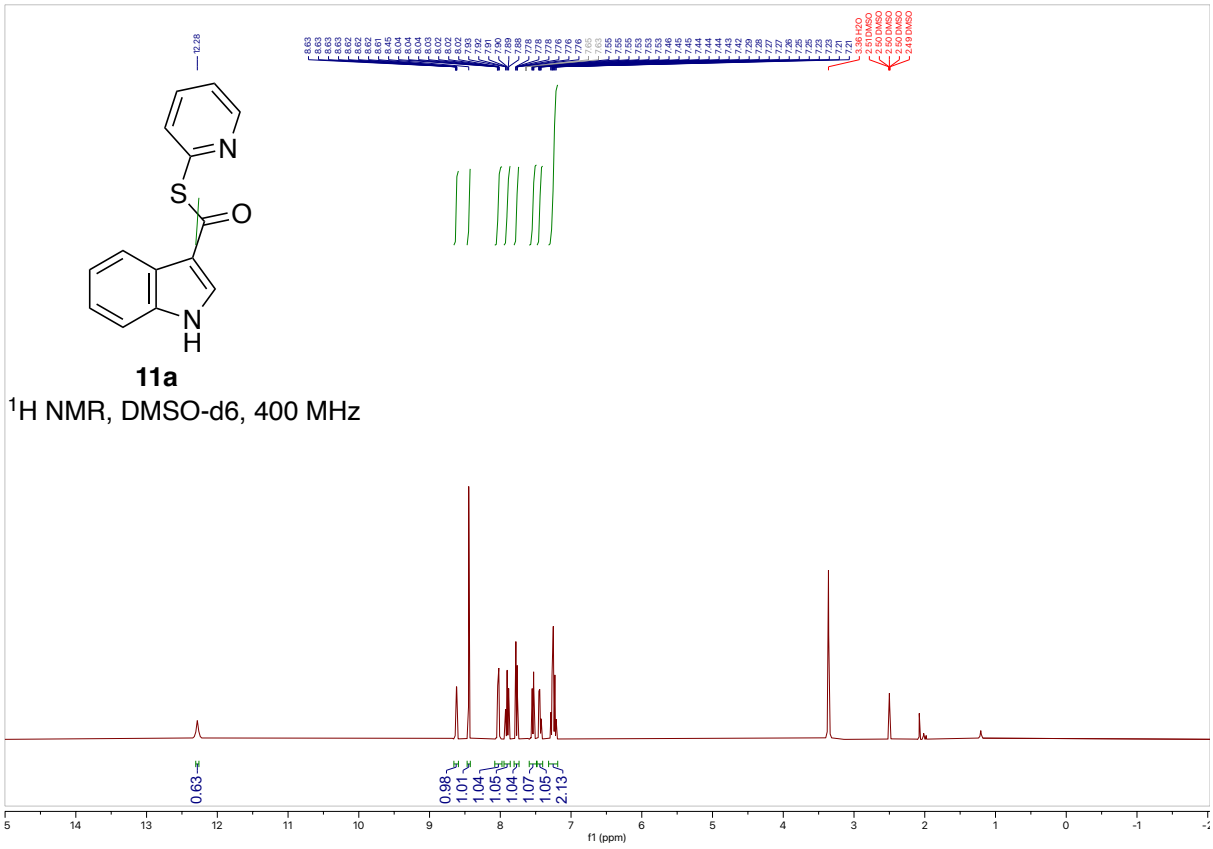
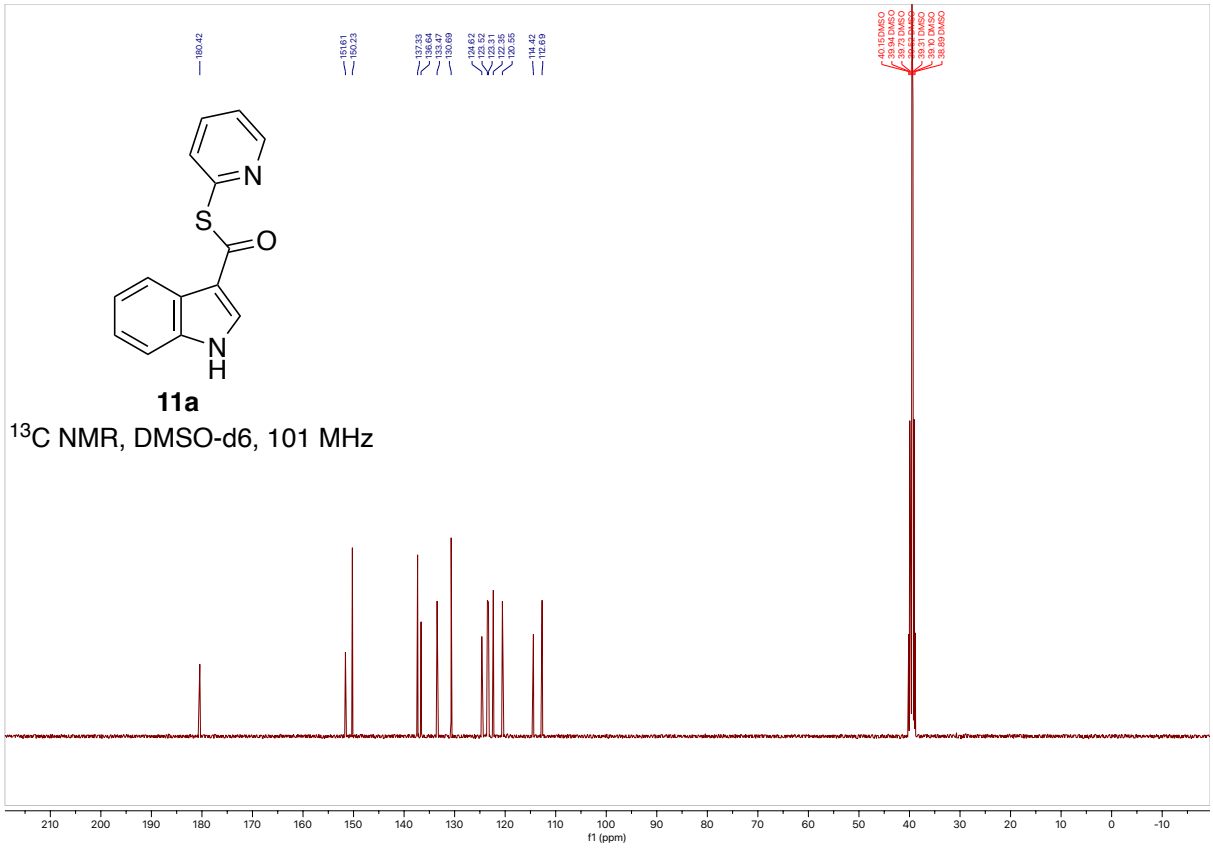


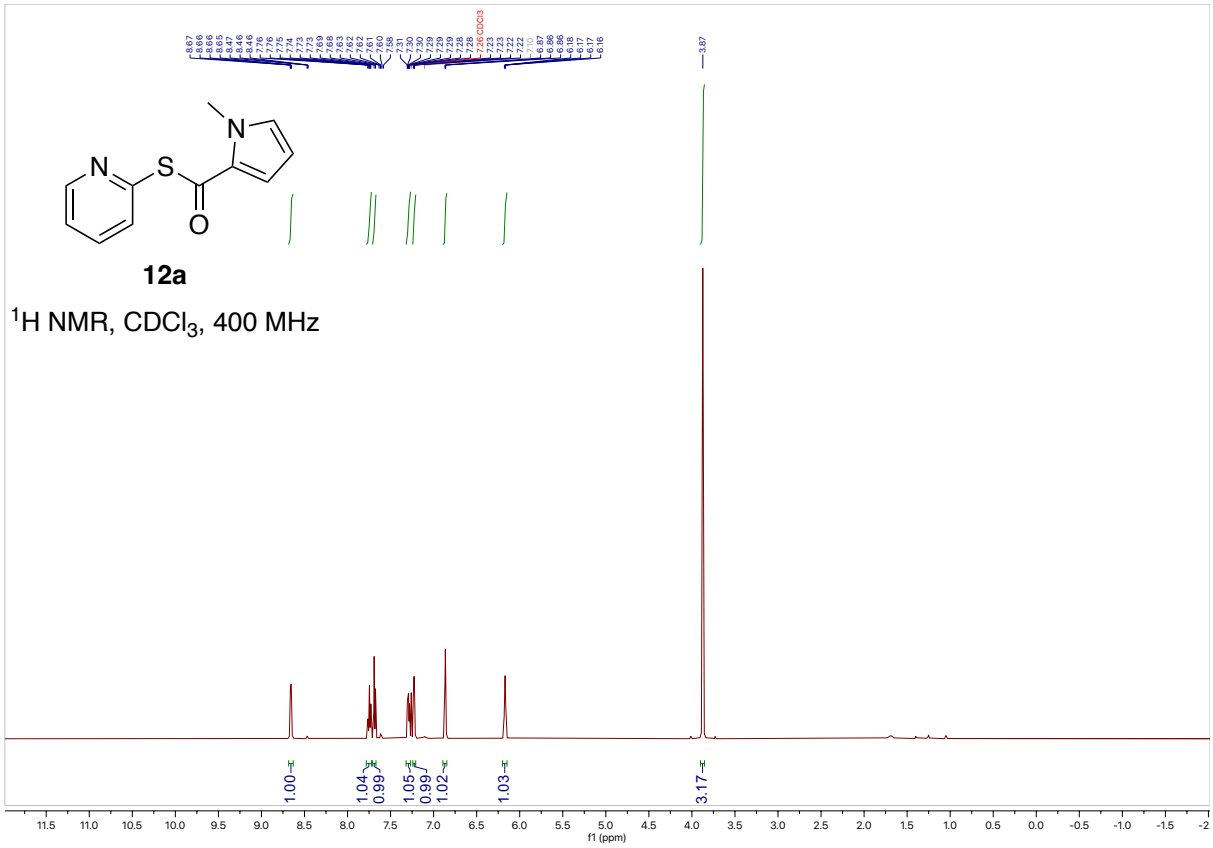
11a

^{1}H NMR, DMSO-d6, 400 MHz



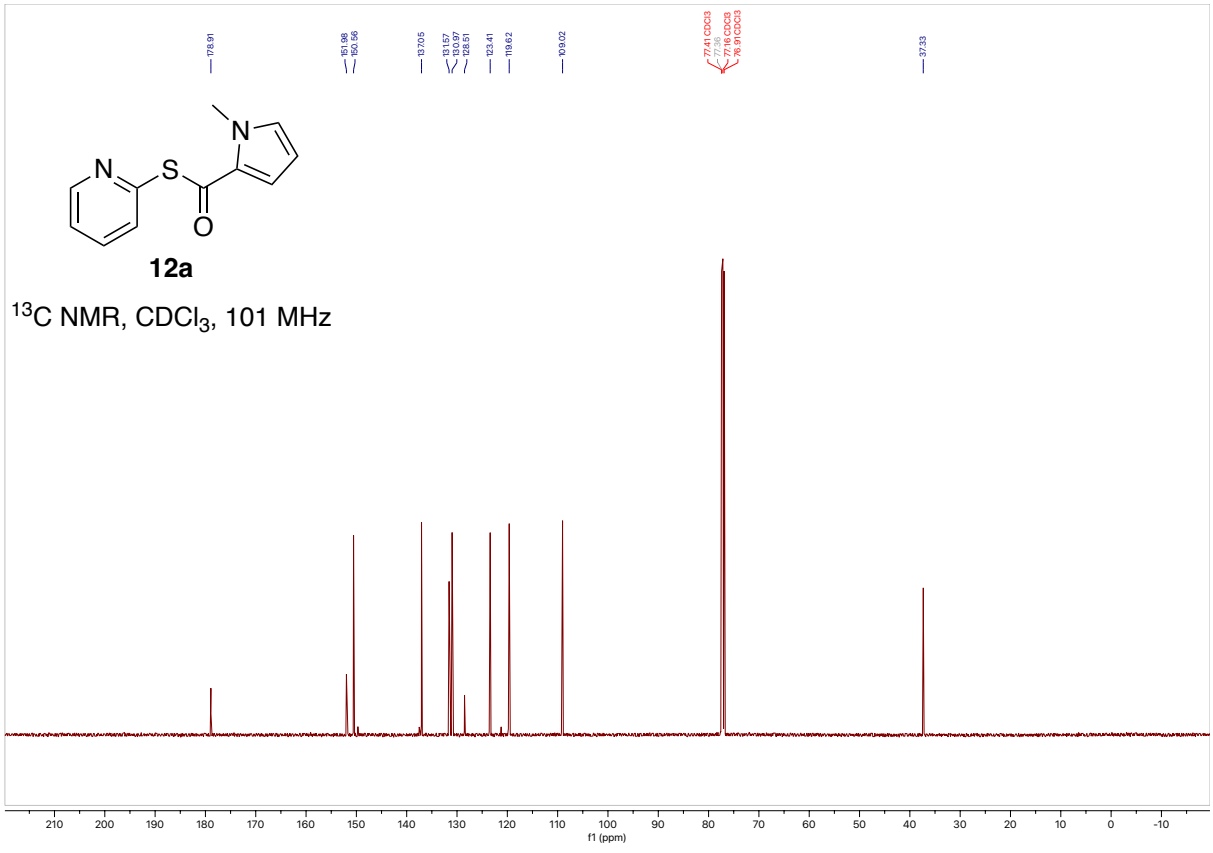
11a

^{13}C NMR, DMSO-d6, 101 MHz



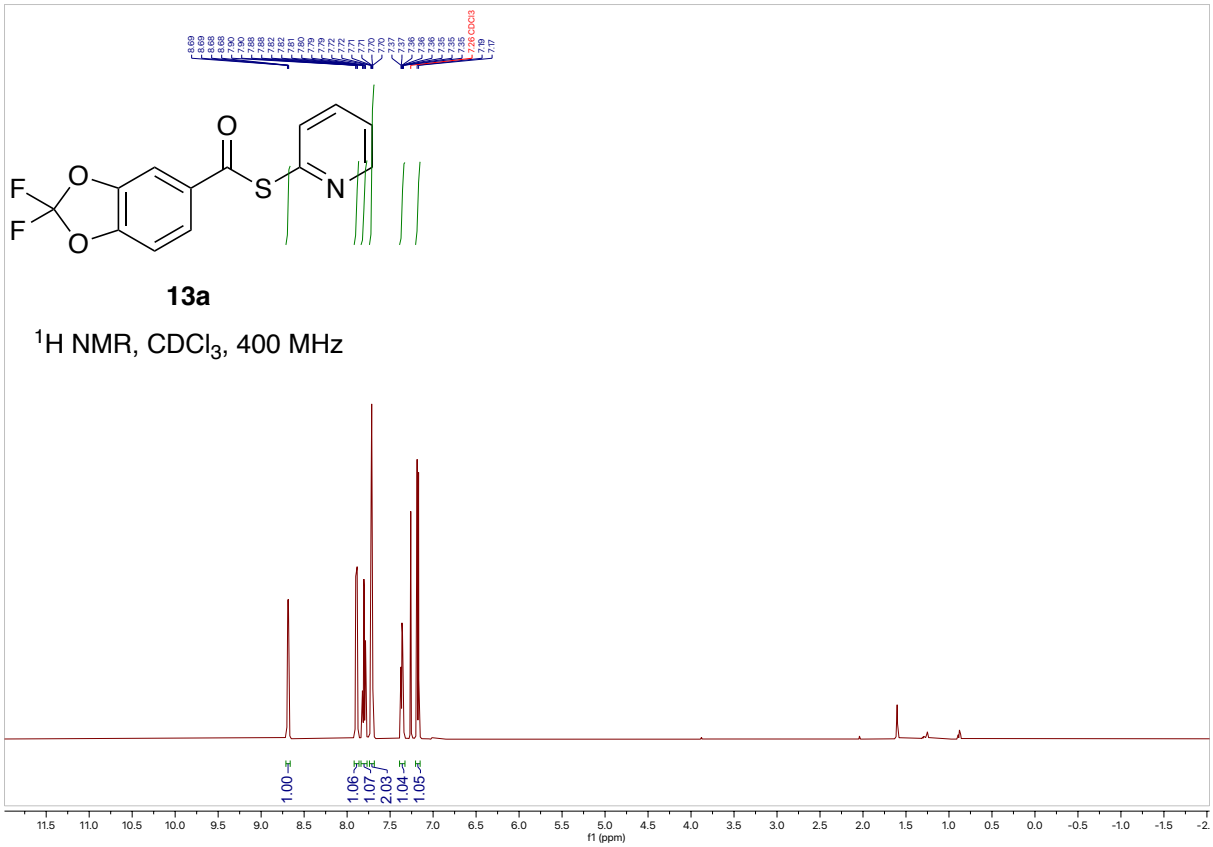
12a

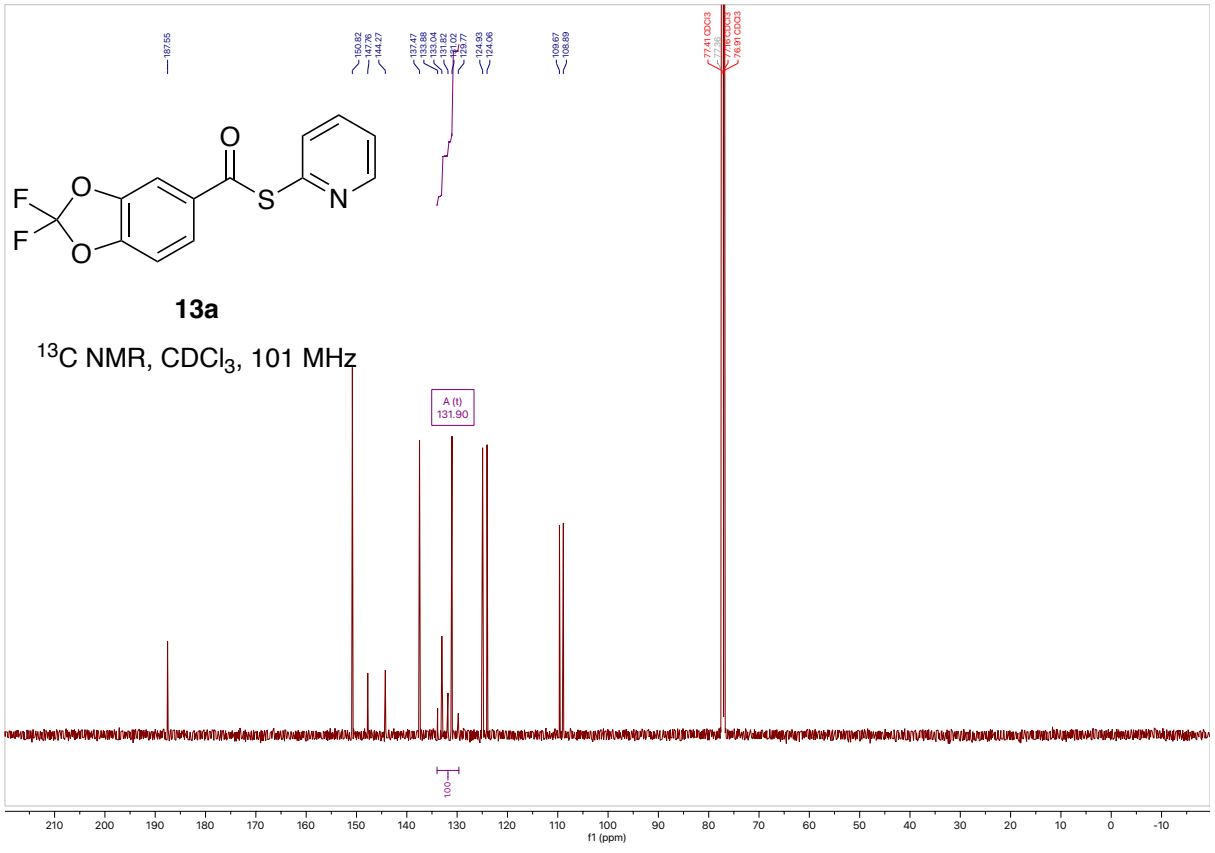
^{1}H NMR, $CDCl_3$, 400 MHz

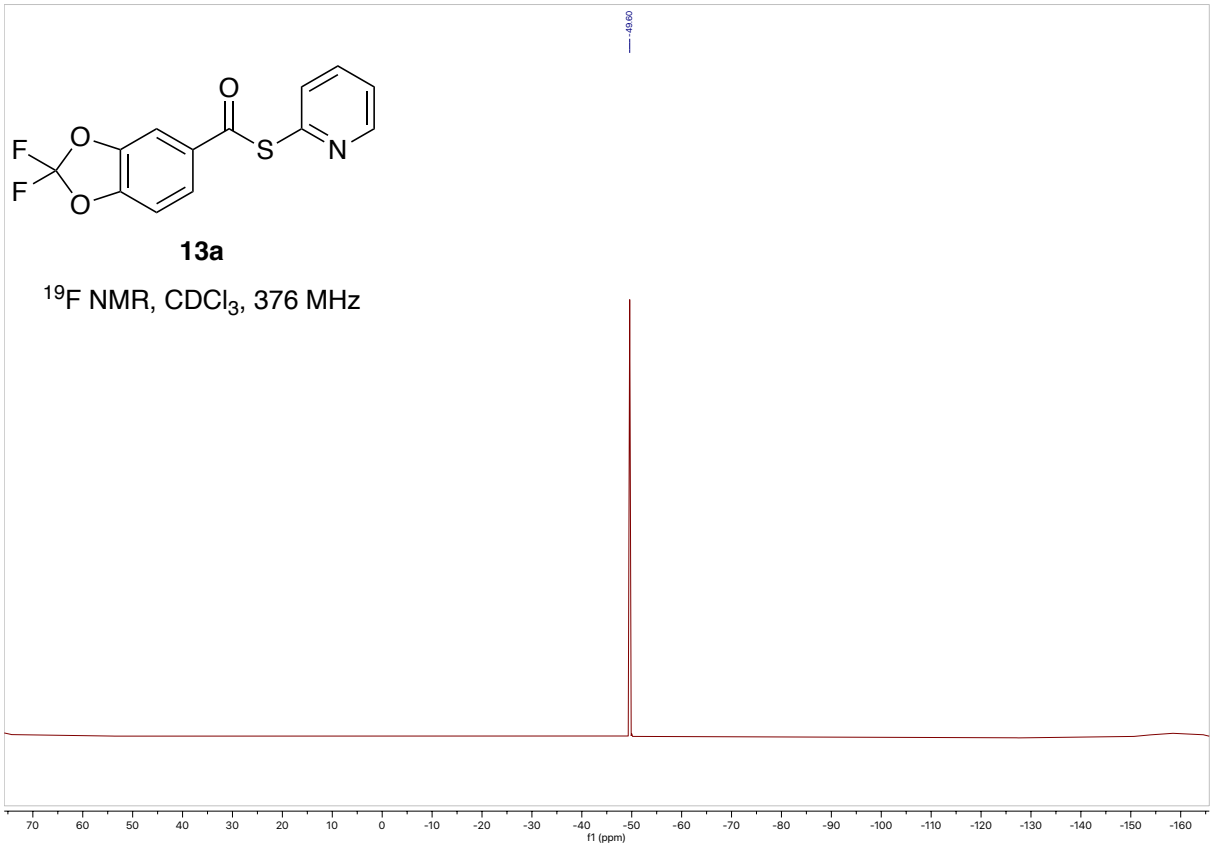


12a

^{13}C NMR, $CDCl_3$, 101 MHz

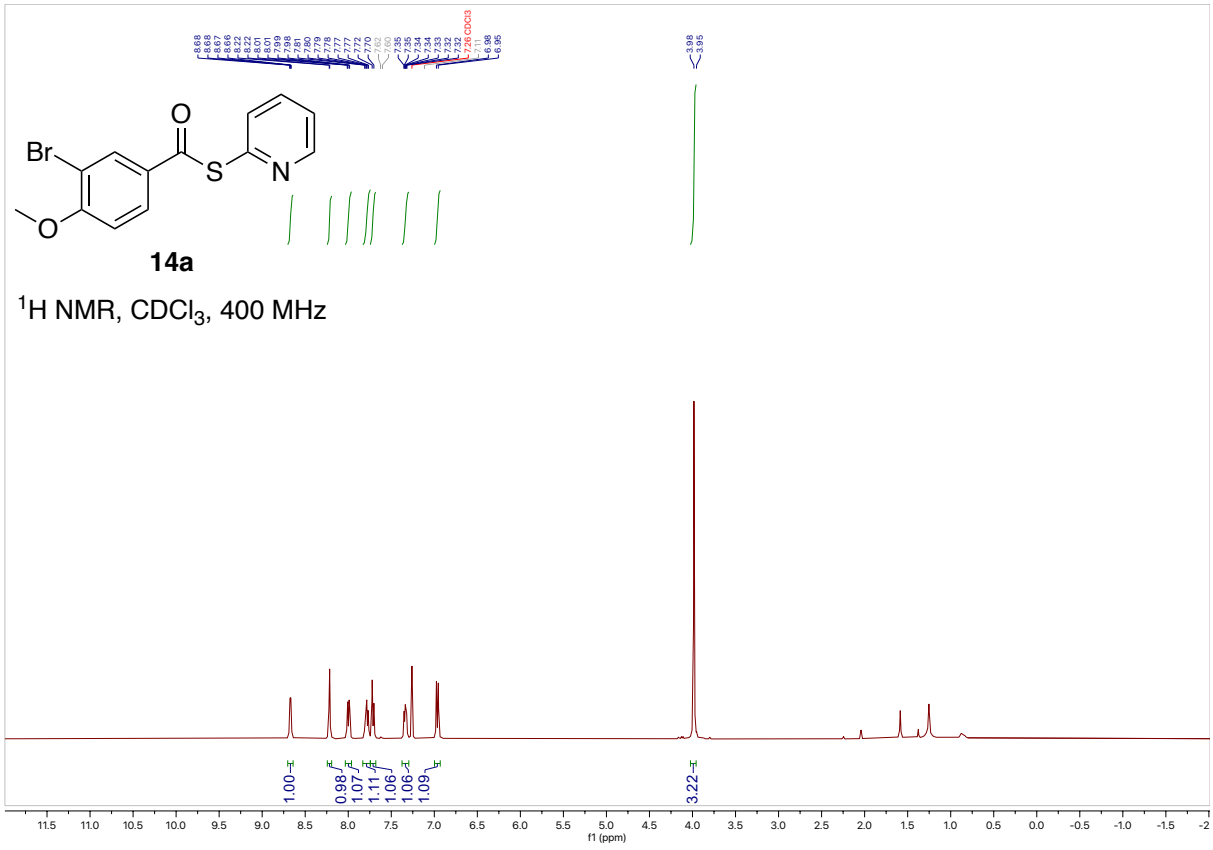

13a
^{1}H NMR, $CDCl_3$, 400 MHz
1.00
1.06
1.07
2.03
1.04
1.05
f1 (ppm)


13a
^{13}C NMR, $CDCl_3$, 101 MHz
A (t)
131.90
f1 (ppm)

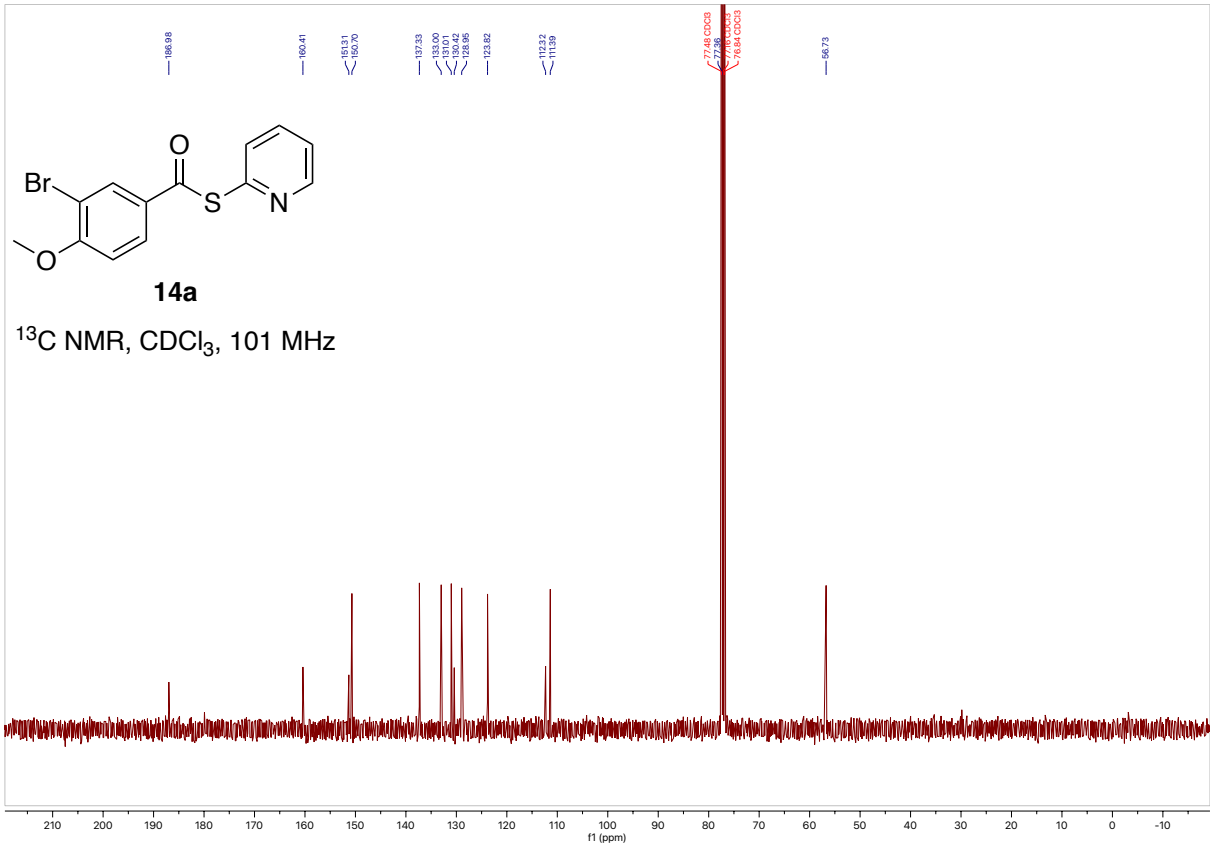


13a

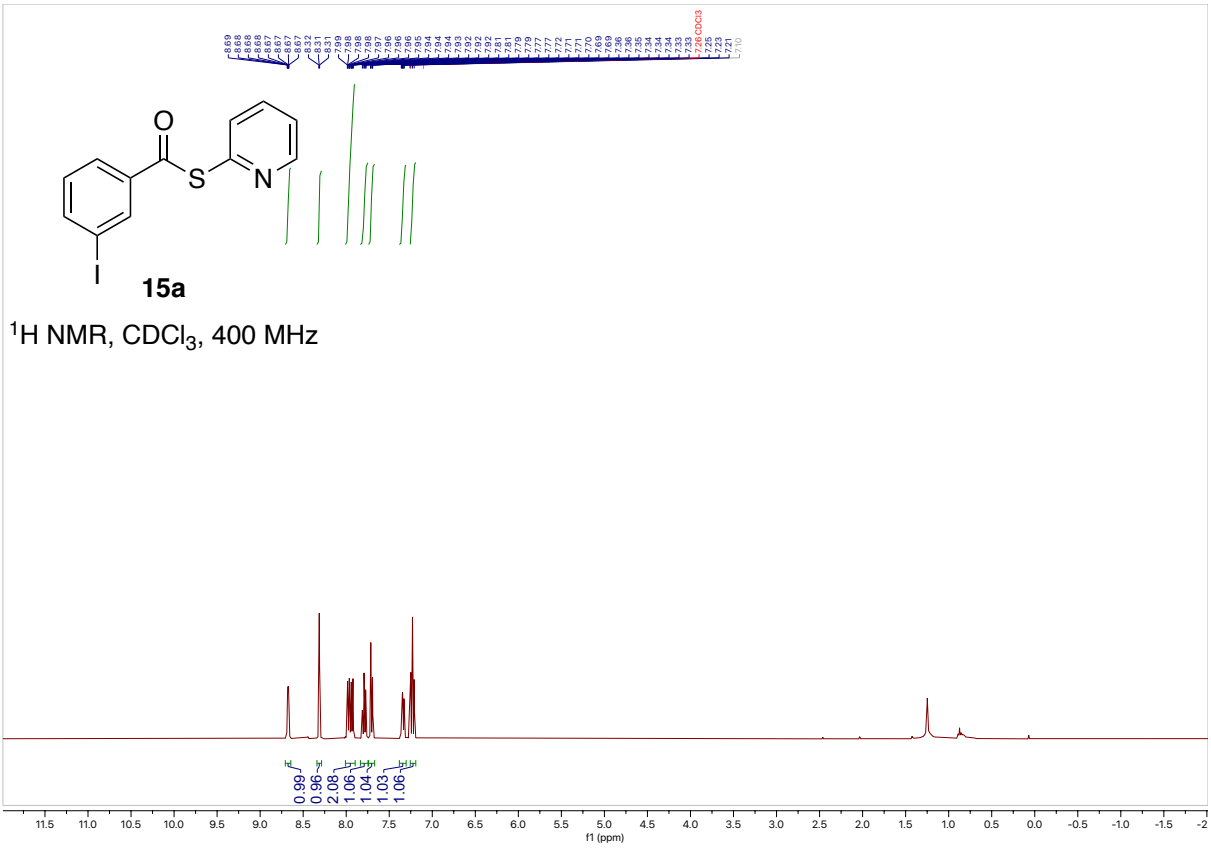
^{19}F NMR, $CDCl_3$, 376 MHz



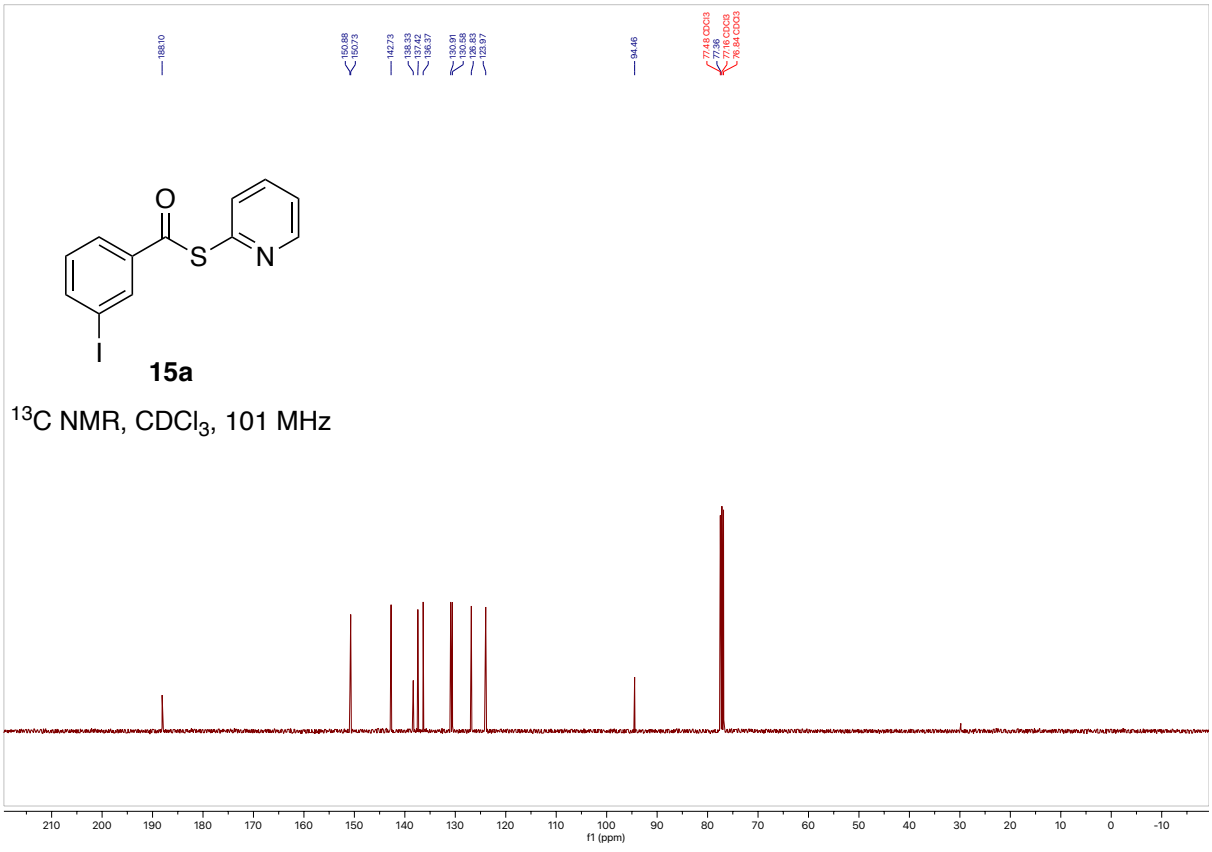
^{1}H NMR, $CDCl_3$, 400 MHz



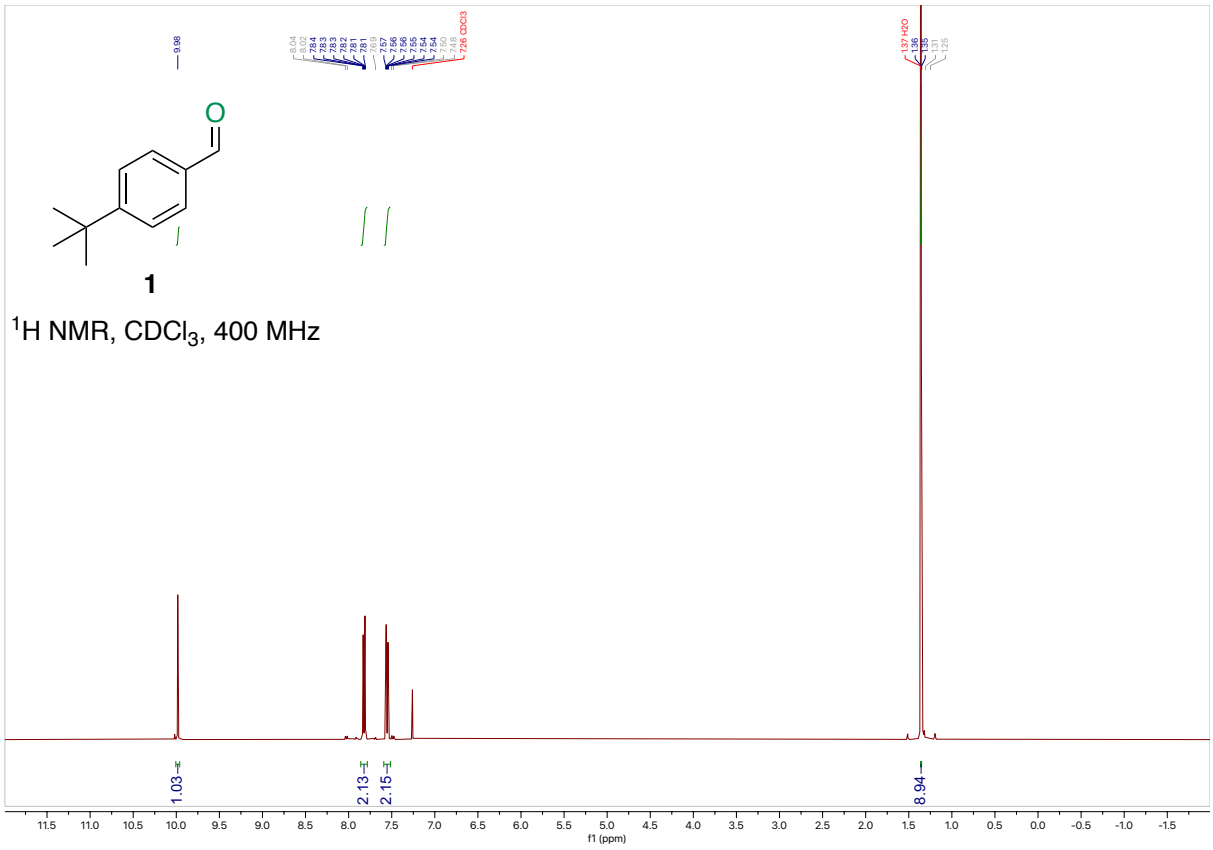
^{13}C NMR, $CDCl_3$, 101 MHz



^{1}H NMR, $CDCl_3$, 400 MHz

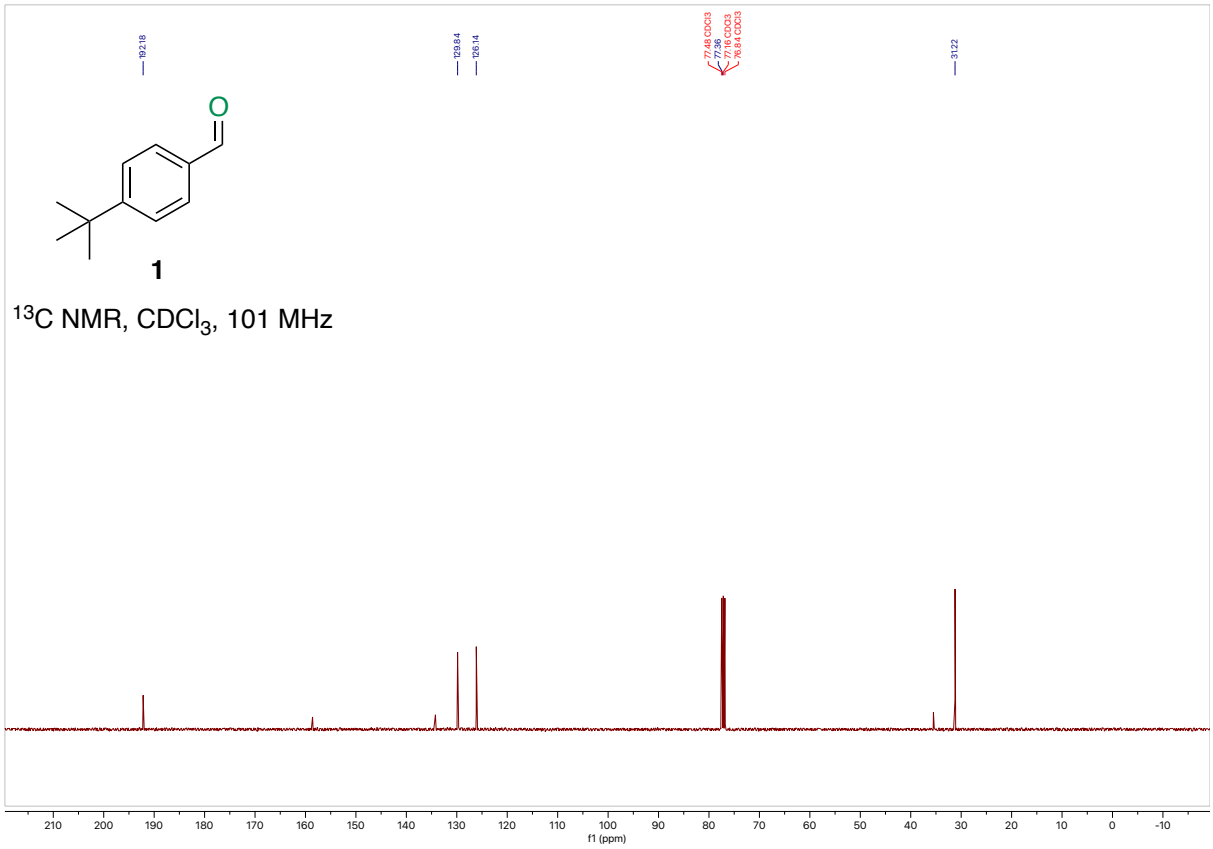


^{13}C NMR, $CDCl_3$, 101 MHz



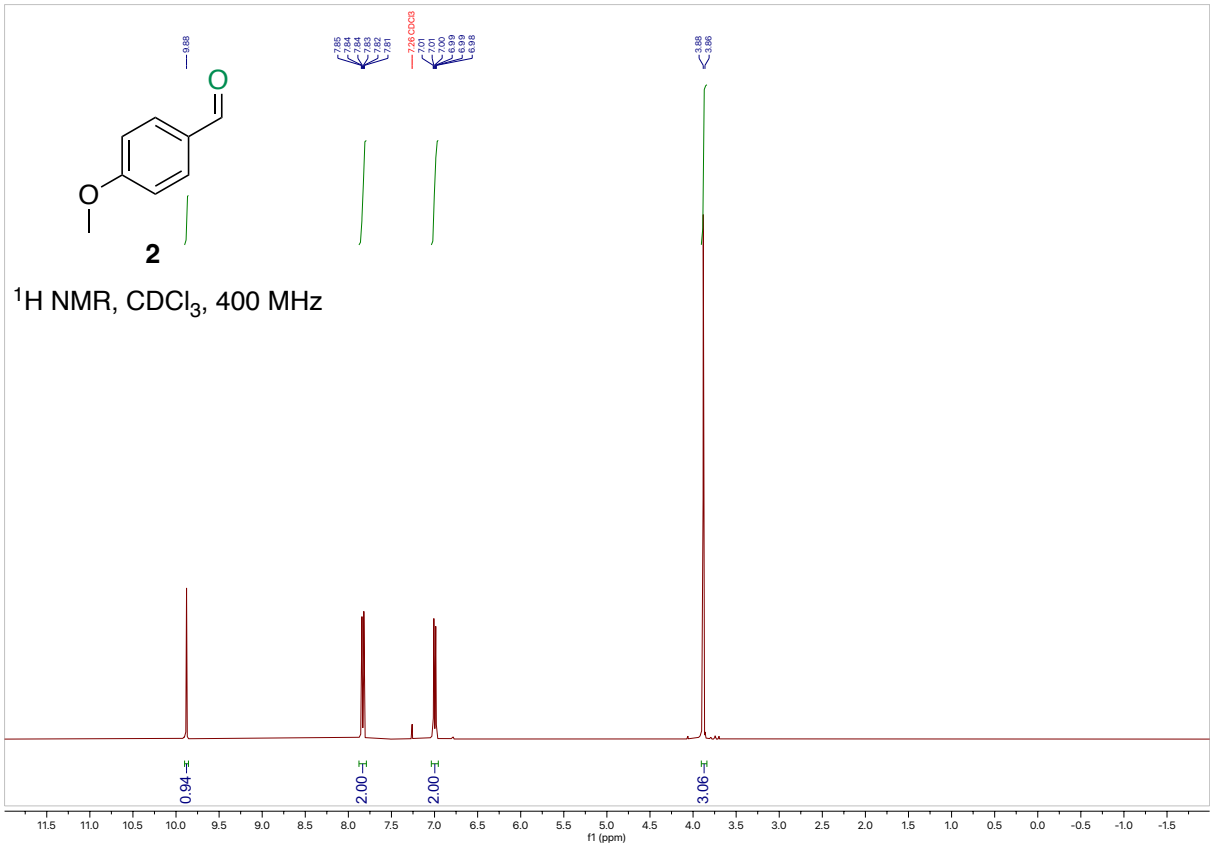
1

^{1}H NMR, $CDCl_3$, 400 MHz



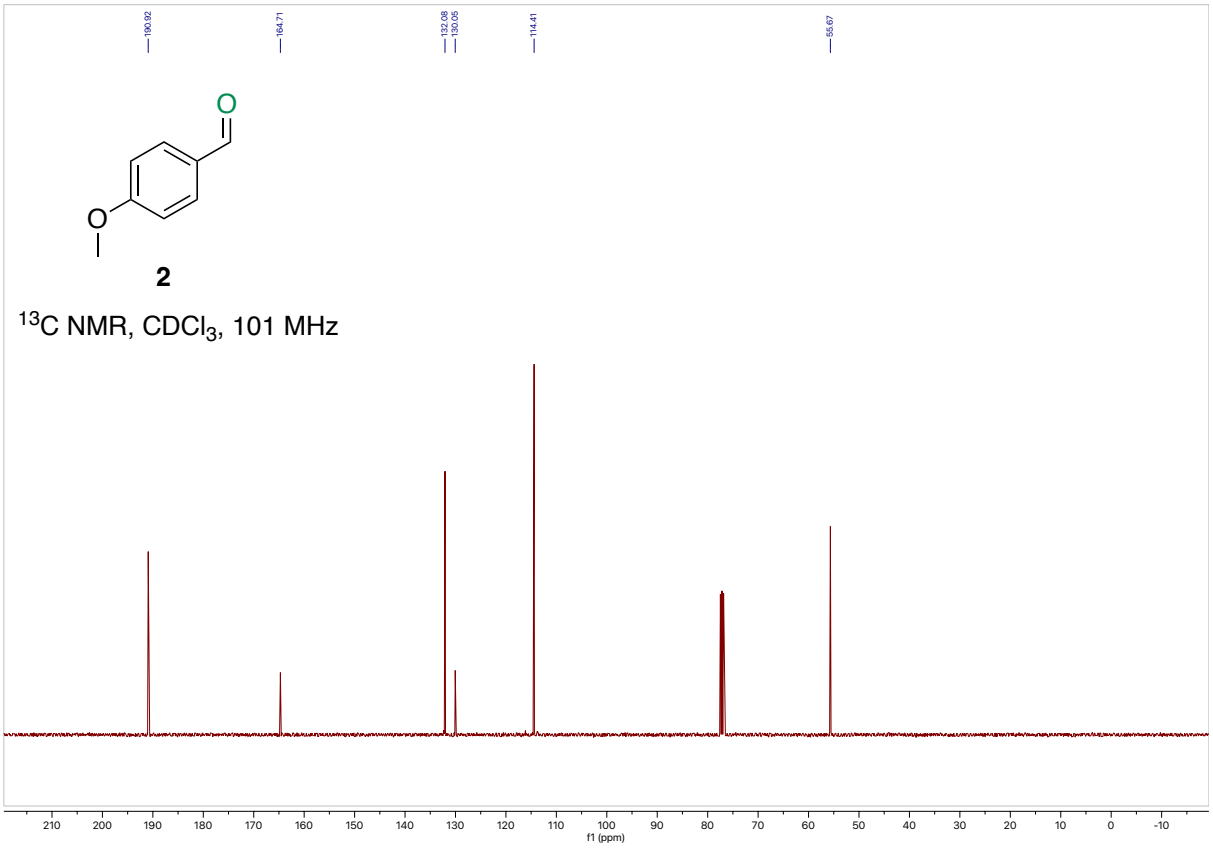
1

^{13}C NMR, $CDCl_3$, 101 MHz



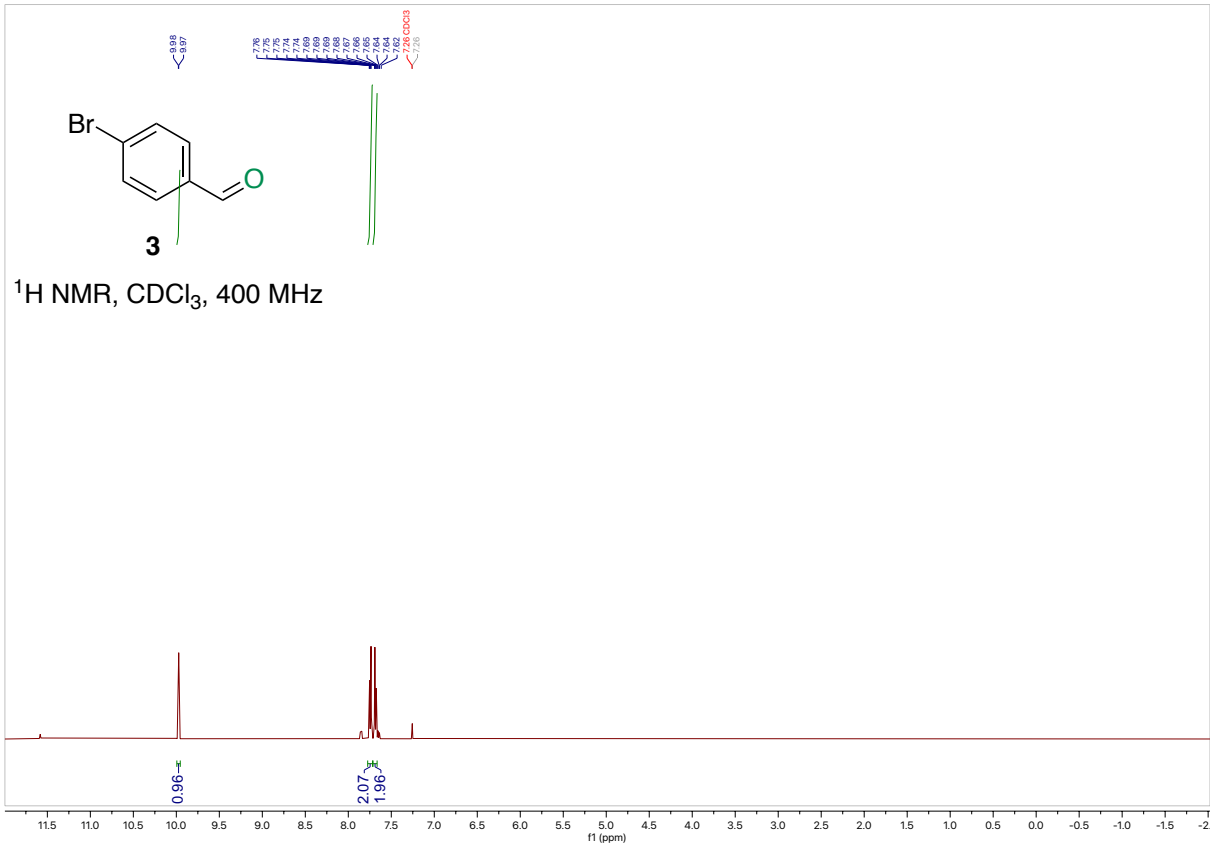
2

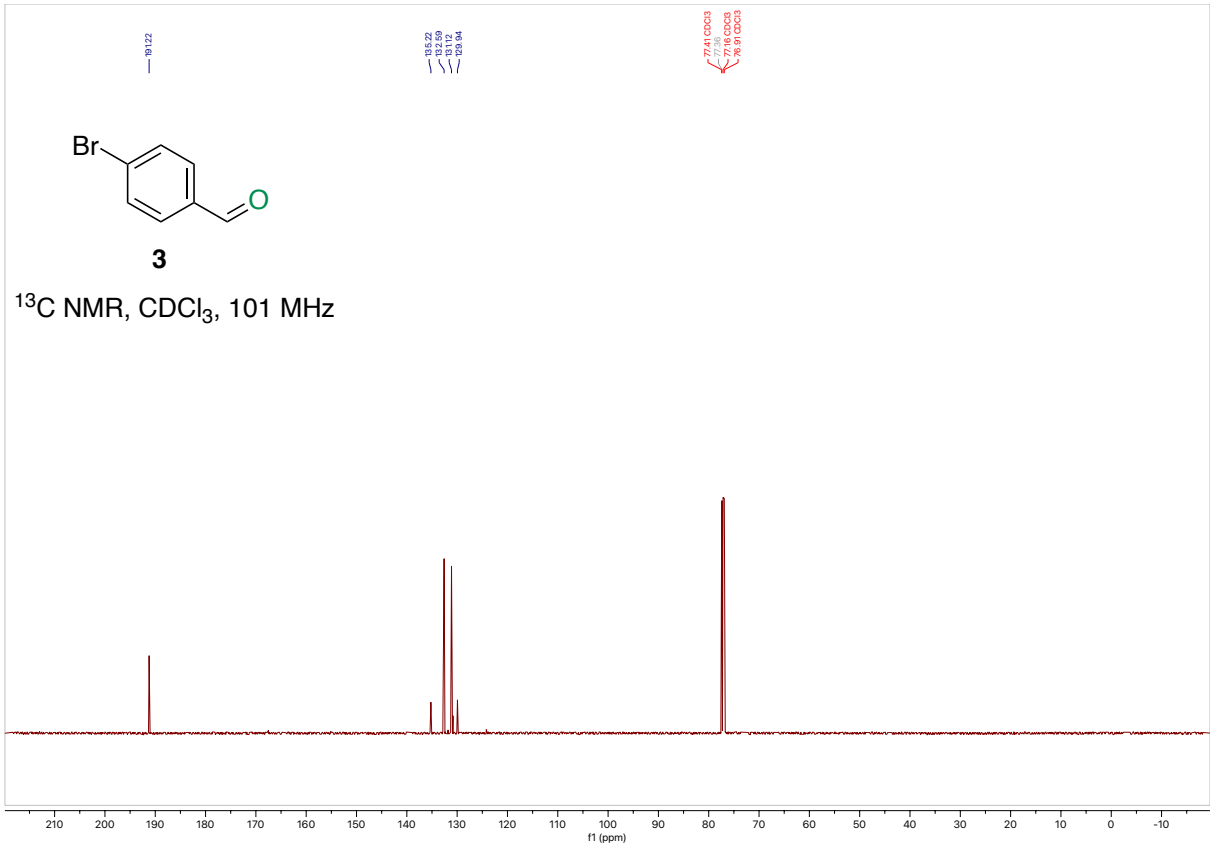
^{1}H NMR, $CDCl_3$, 400 MHz

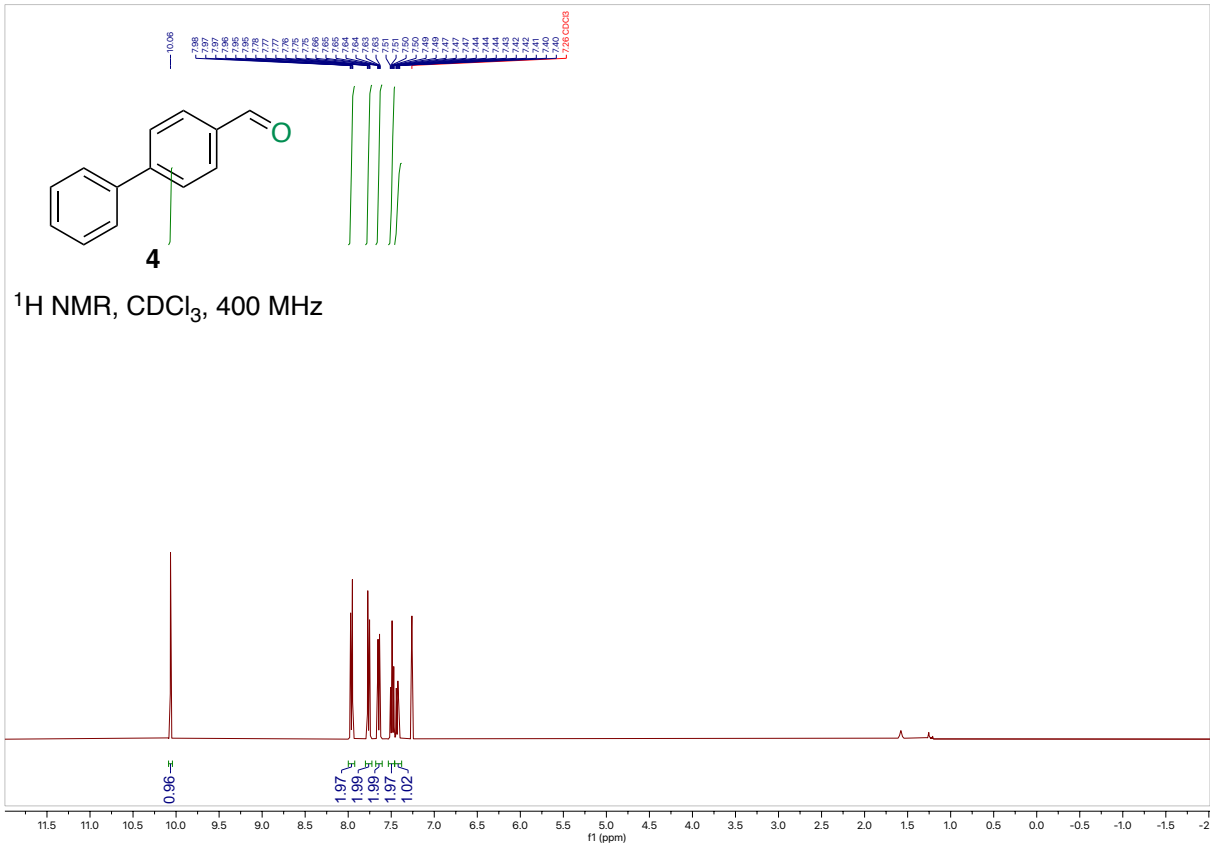


2

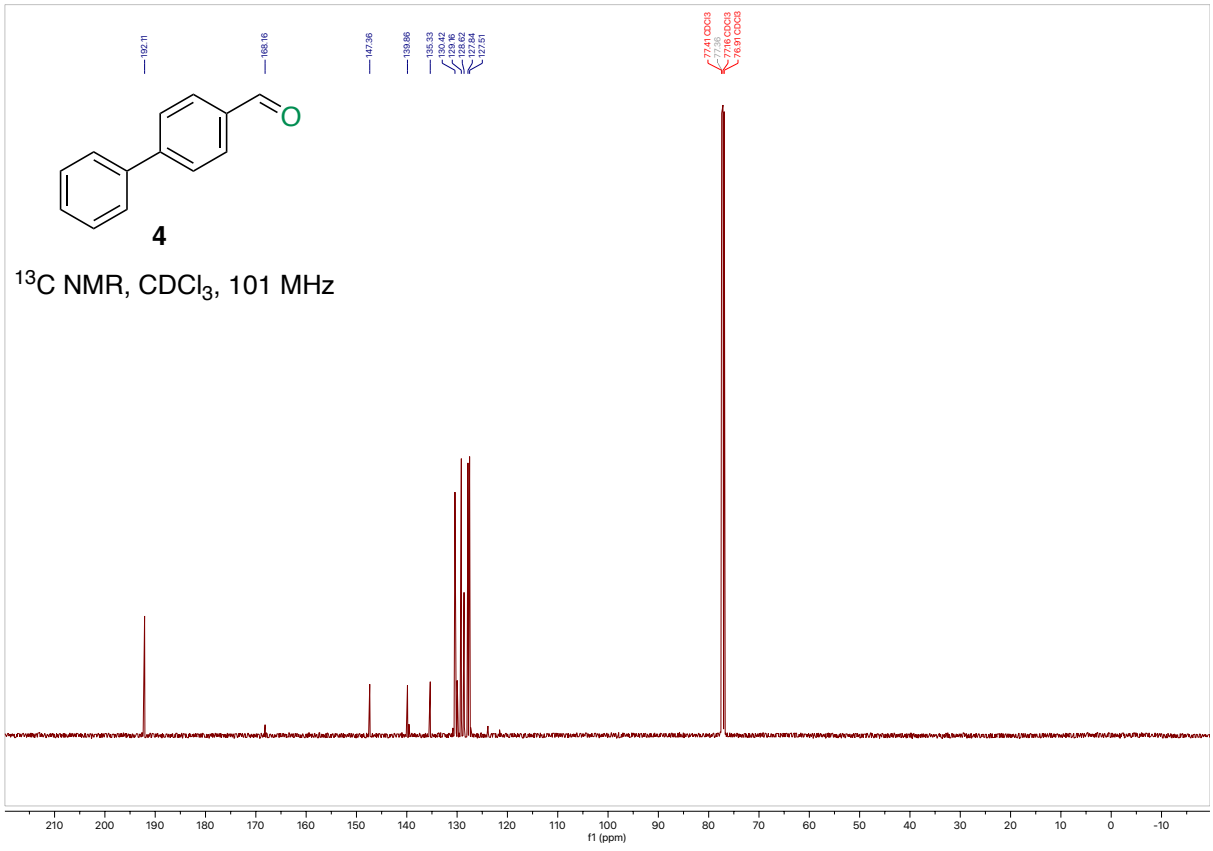
^{13}C NMR, $CDCl_3$, 101 MHz


Br
O
3
^{1}H NMR, $CDCl_3$, 400 MHz
f1 (ppm)

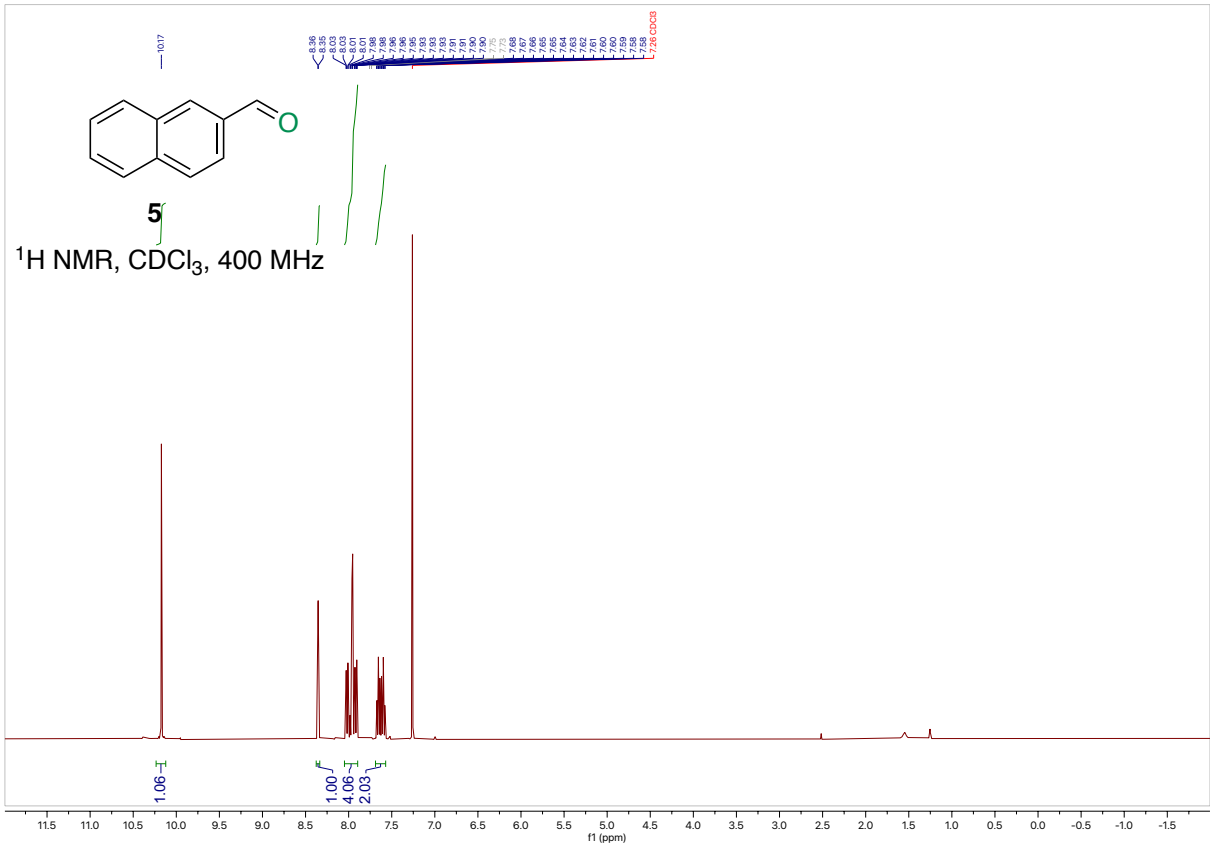

Br
O
3
^{13}C NMR, $CDCl_3$, 101 MHz
f1 (ppm)

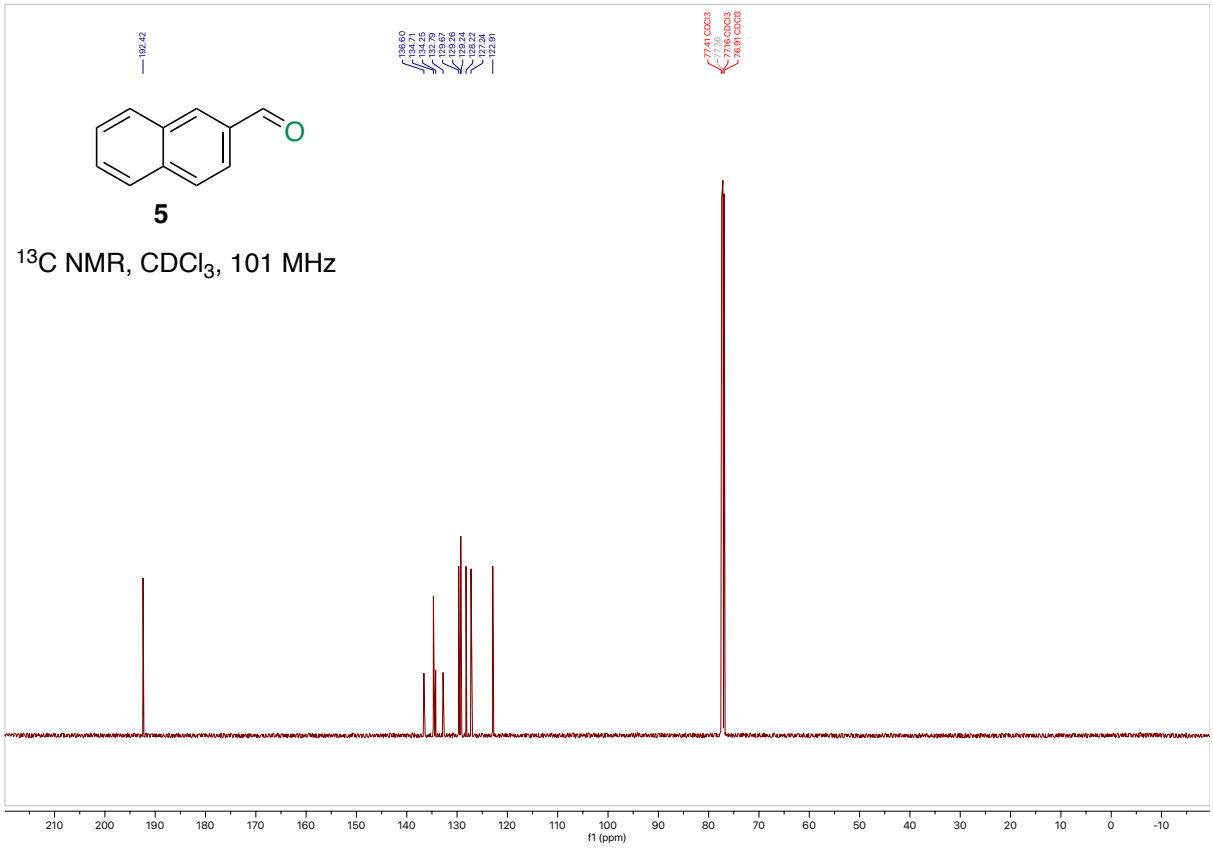


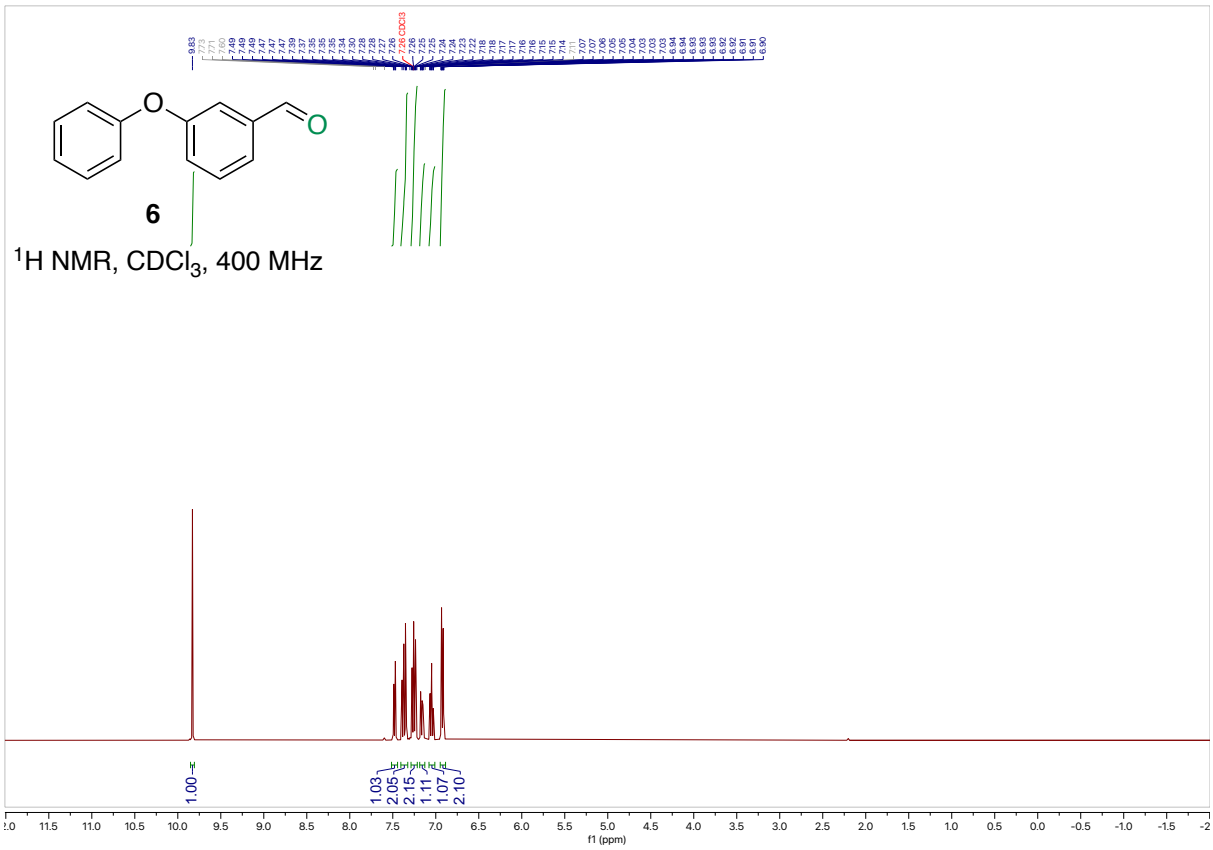
^{1}H NMR, $CDCl_3$, 400 MHz

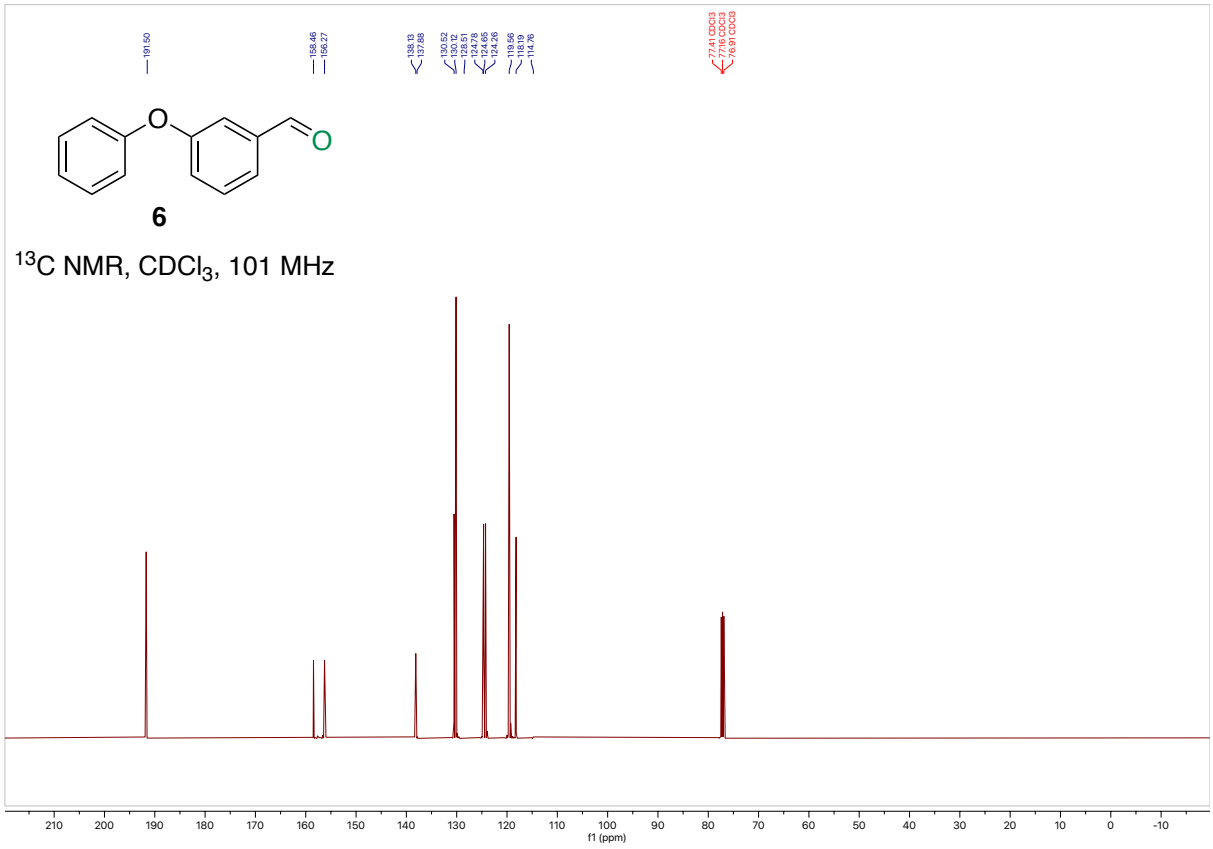


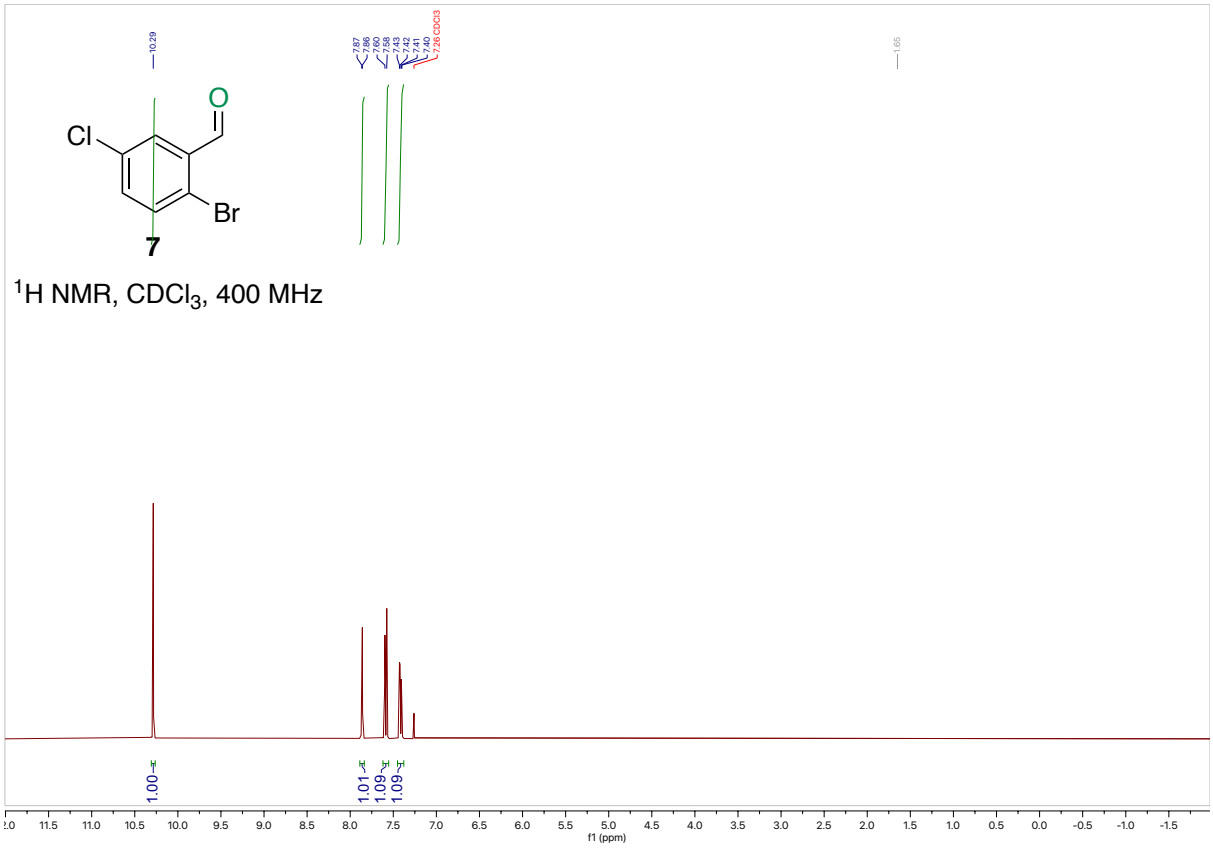
^{13}C NMR, $CDCl_3$, 101 MHz


10.17
8.36
8.35
8.03
8.01
7.98
7.96
7.95
7.93
7.91
7.90
7.68
7.67
7.66
7.65
7.64
7.62
7.61
7.60
7.59
7.58
7.26 CDCl3
O
5
1H NMR, CDCl3, 400 MHz
1.06
1.00
4.06
2.03
f1 (ppm)

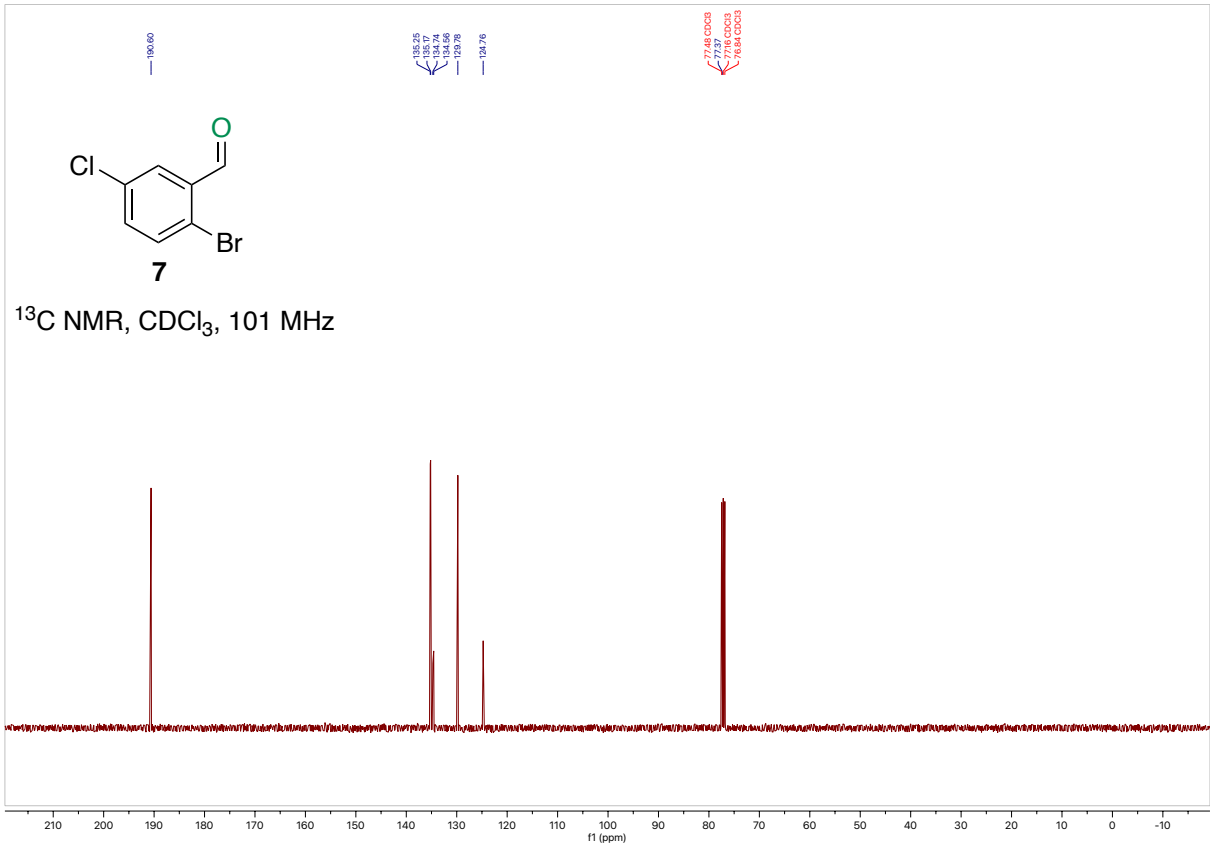

192.42
136.60
134.71
134.35
132.79
129.67
129.26
128.34
128.22
127.24
122.91
77.41 CDCl3
77.16
77.16 CDCl3
76.91 CDCl3
O
5
13C NMR, CDCl3, 101 MHz
f1 (ppm)


6
1H NMR, CDCl3, 400 MHz
f1 (ppm)

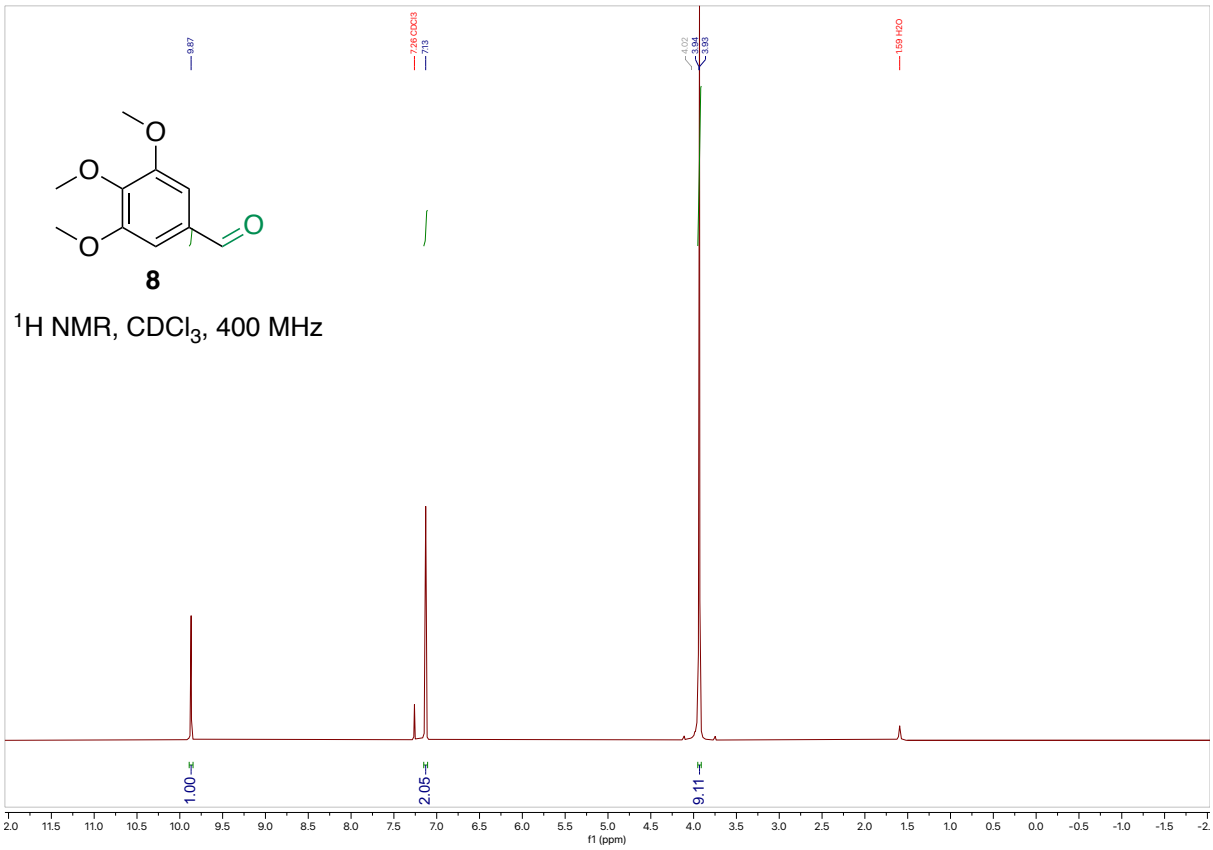

6
13C NMR, CDCl3, 101 MHz
f1 (ppm)



^{1}H NMR, $CDCl_3$, 400 MHz

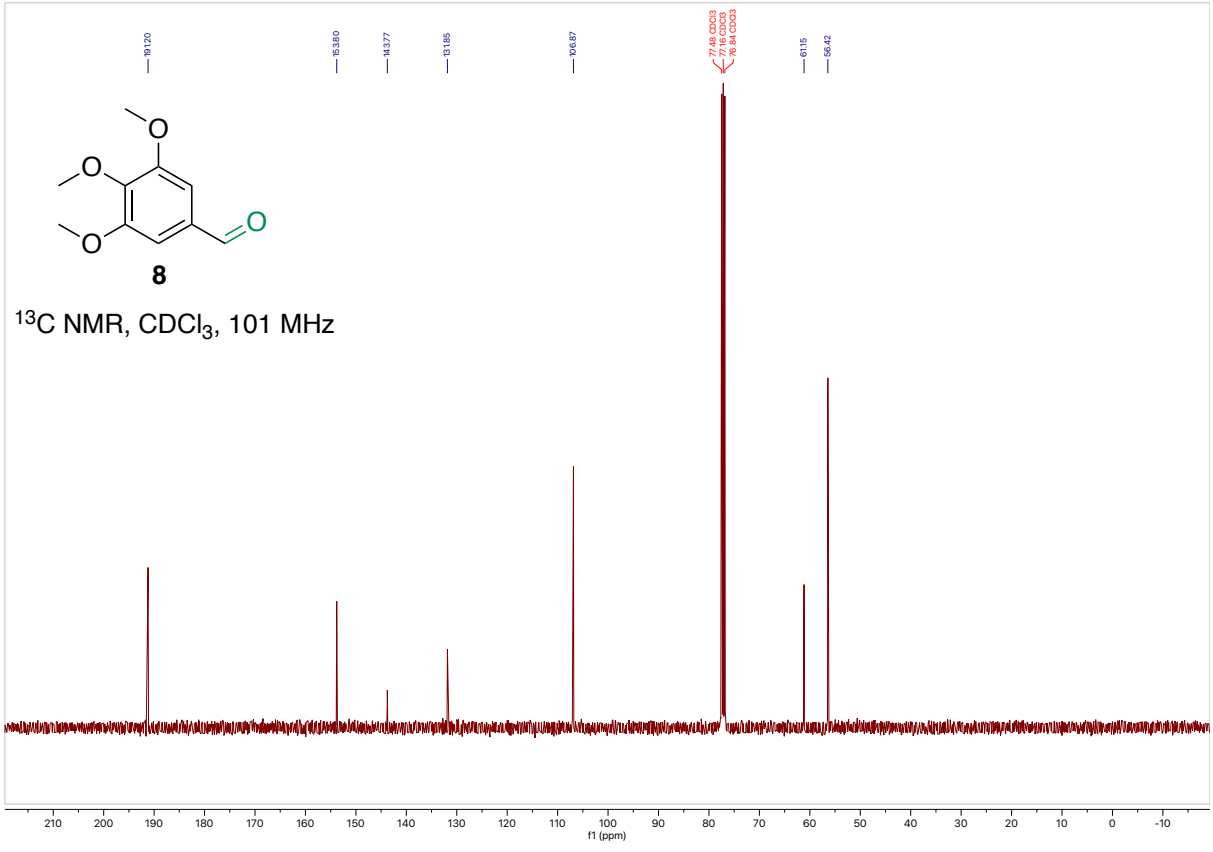


^{13}C NMR, $CDCl_3$, 101 MHz



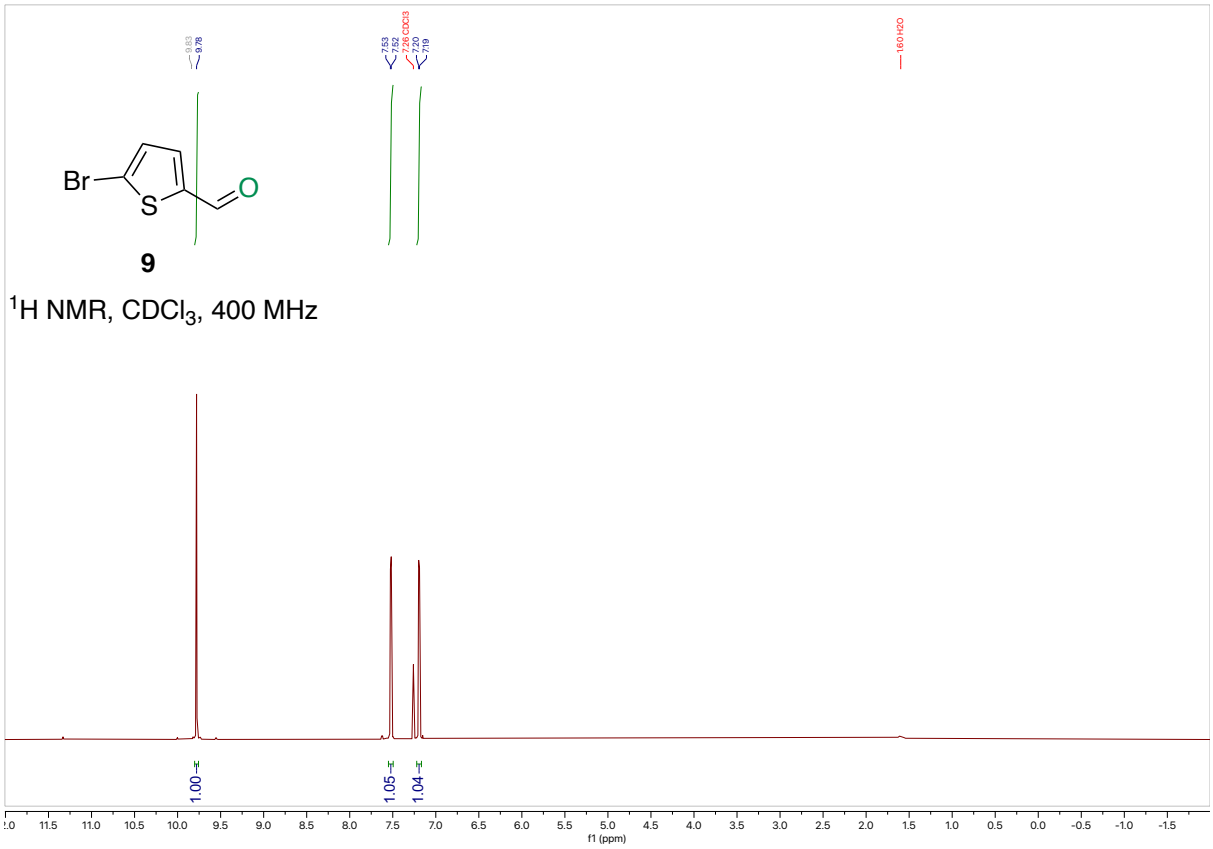
8

^{1}H NMR, $CDCl_3$, 400 MHz



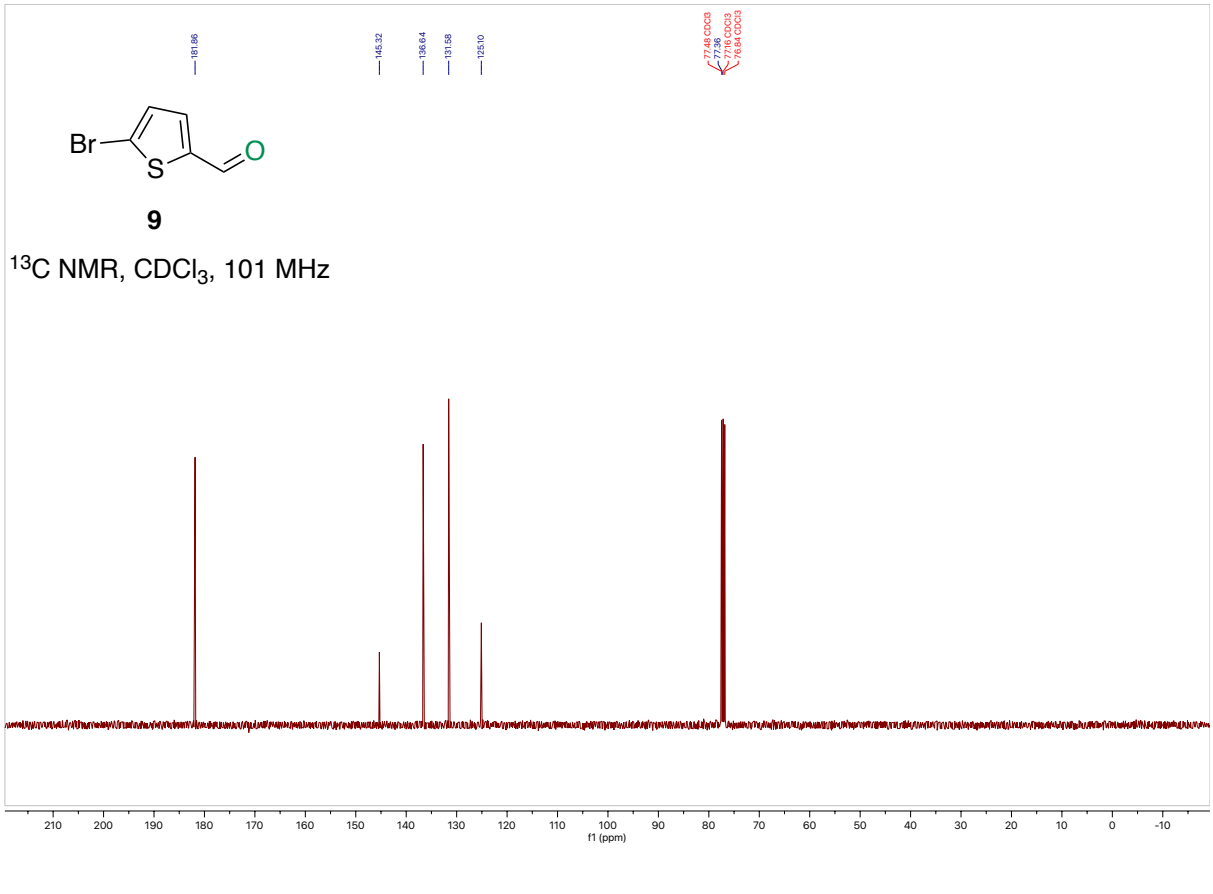
8

^{13}C NMR, $CDCl_3$, 101 MHz



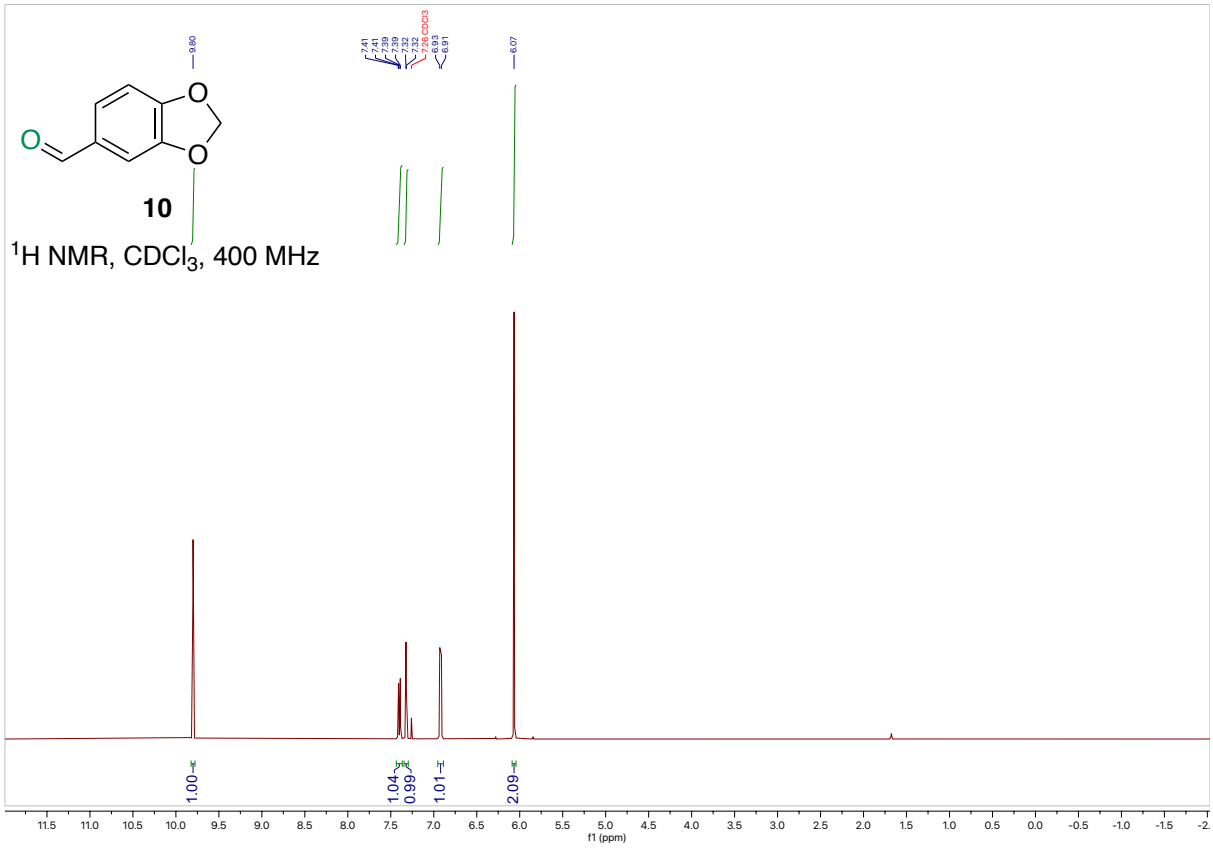
9

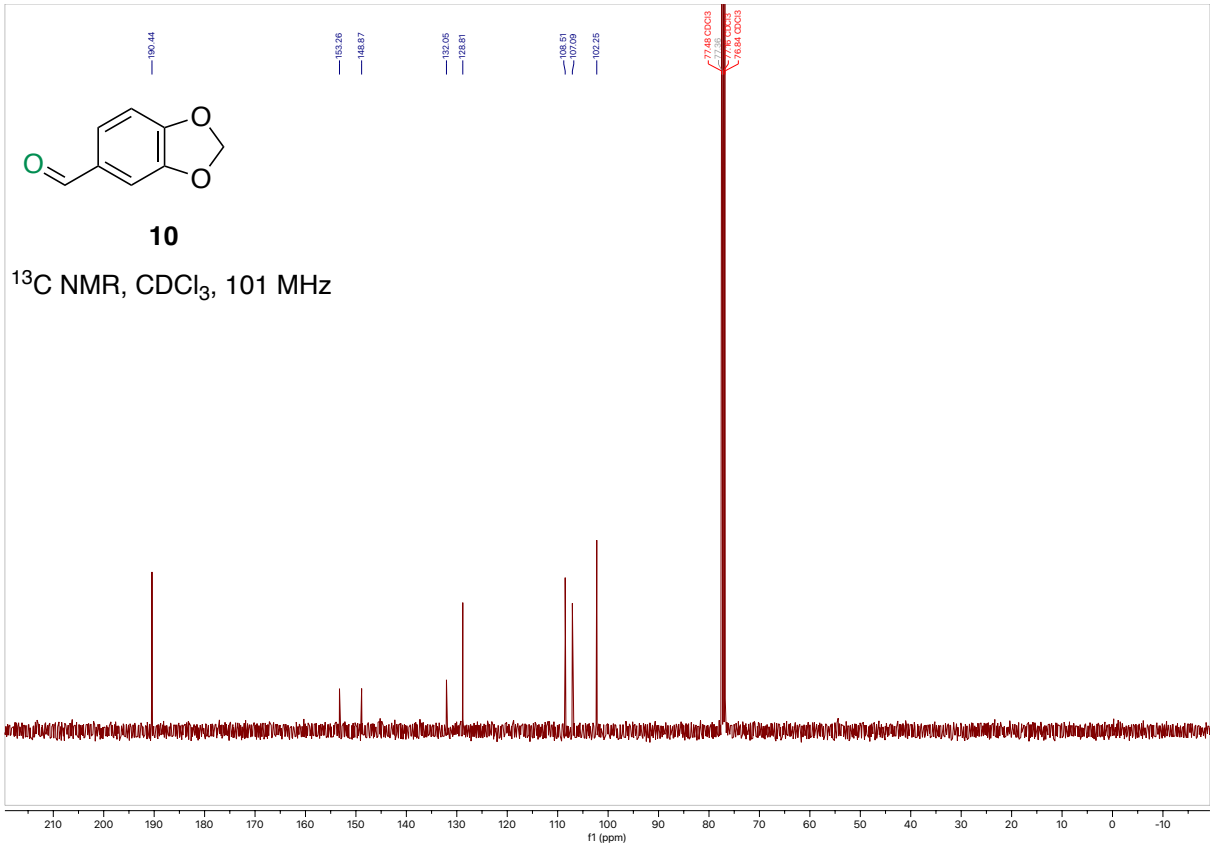
^{1}H NMR, $CDCl_3$, 400 MHz

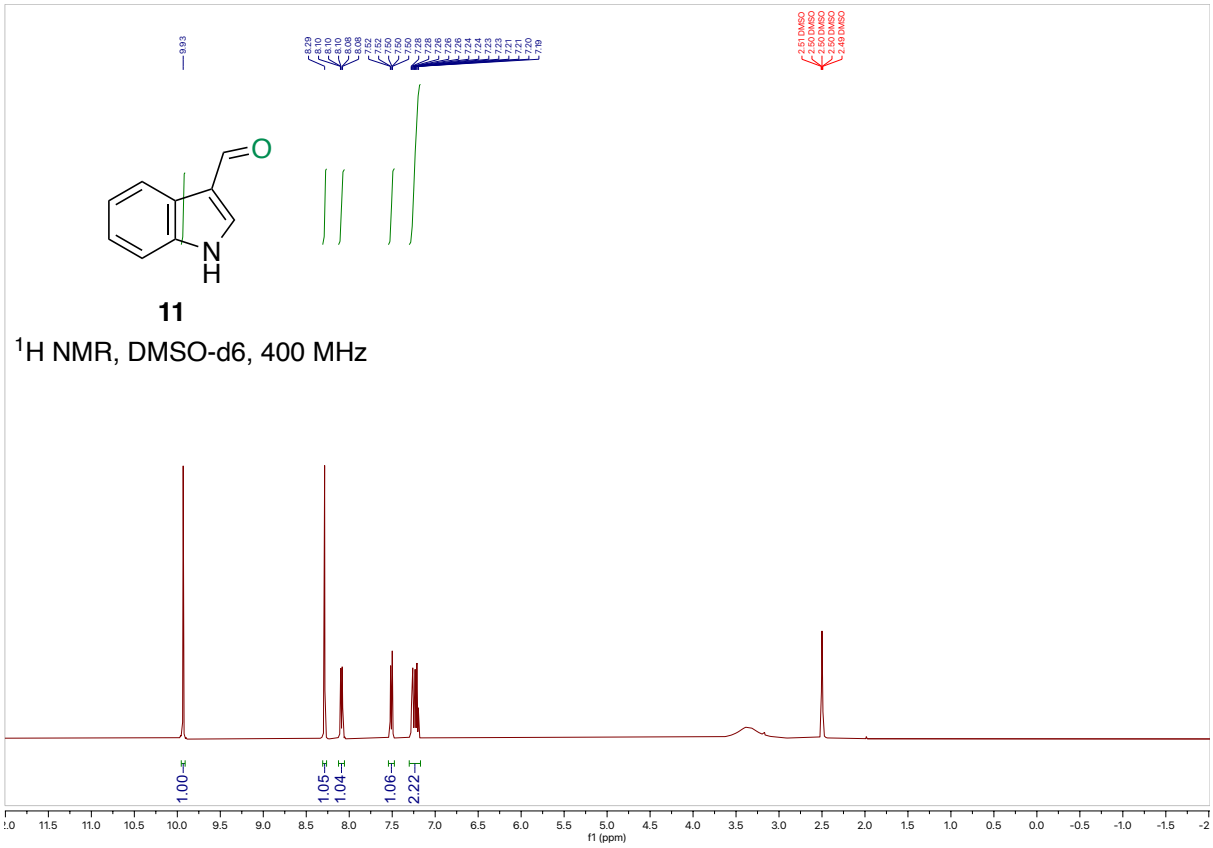


9

^{13}C NMR, $CDCl_3$, 101 MHz

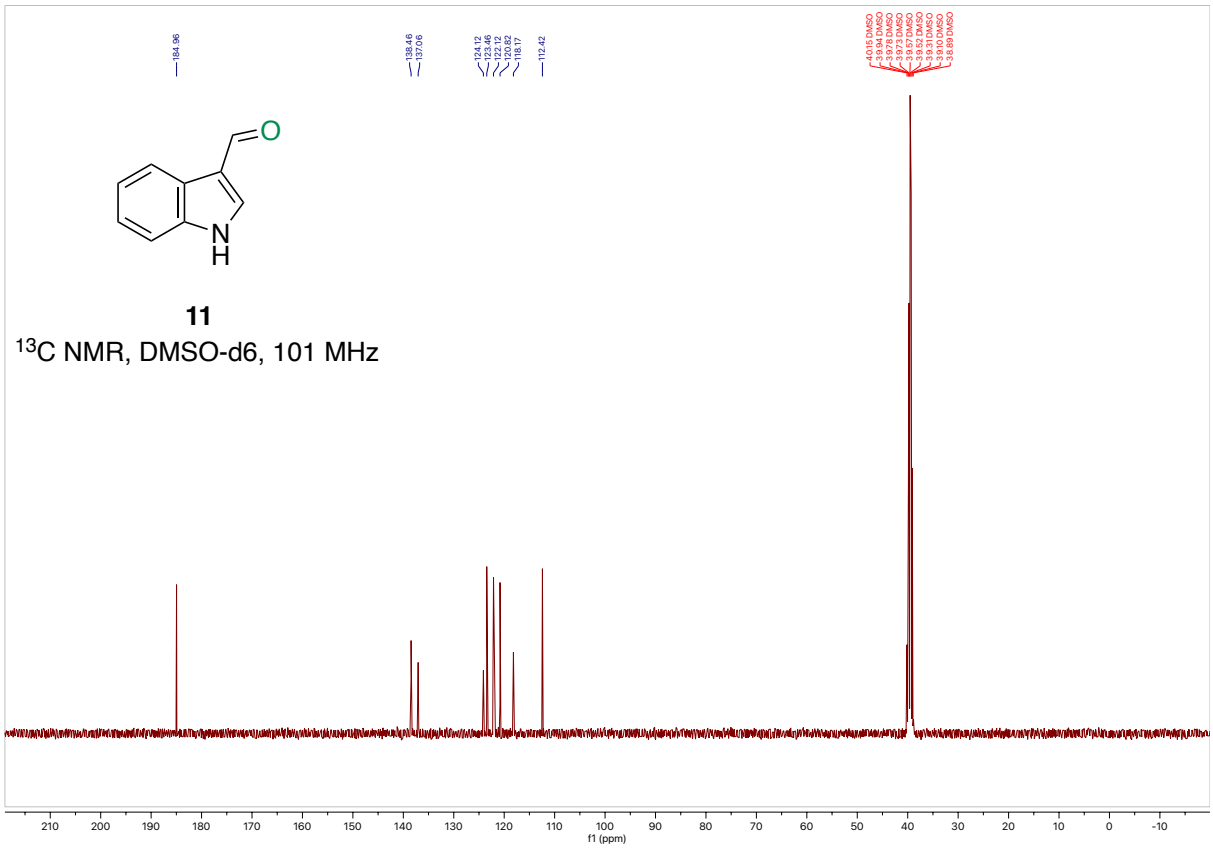

9.80
7.41
7.41
7.39
7.39
7.32
7.32
7.26 CDCl3
6.93
6.91
6.07
O
O
O
10
1H NMR, CDCl3, 400 MHz
1.00
1.04
0.99
1.01
2.09
f1 (ppm)


190.44
153.26
148.87
132.05
128.81
108.51
107.09
102.25
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
O
O
O
10
13C NMR, CDCl3, 101 MHz
f1 (ppm)



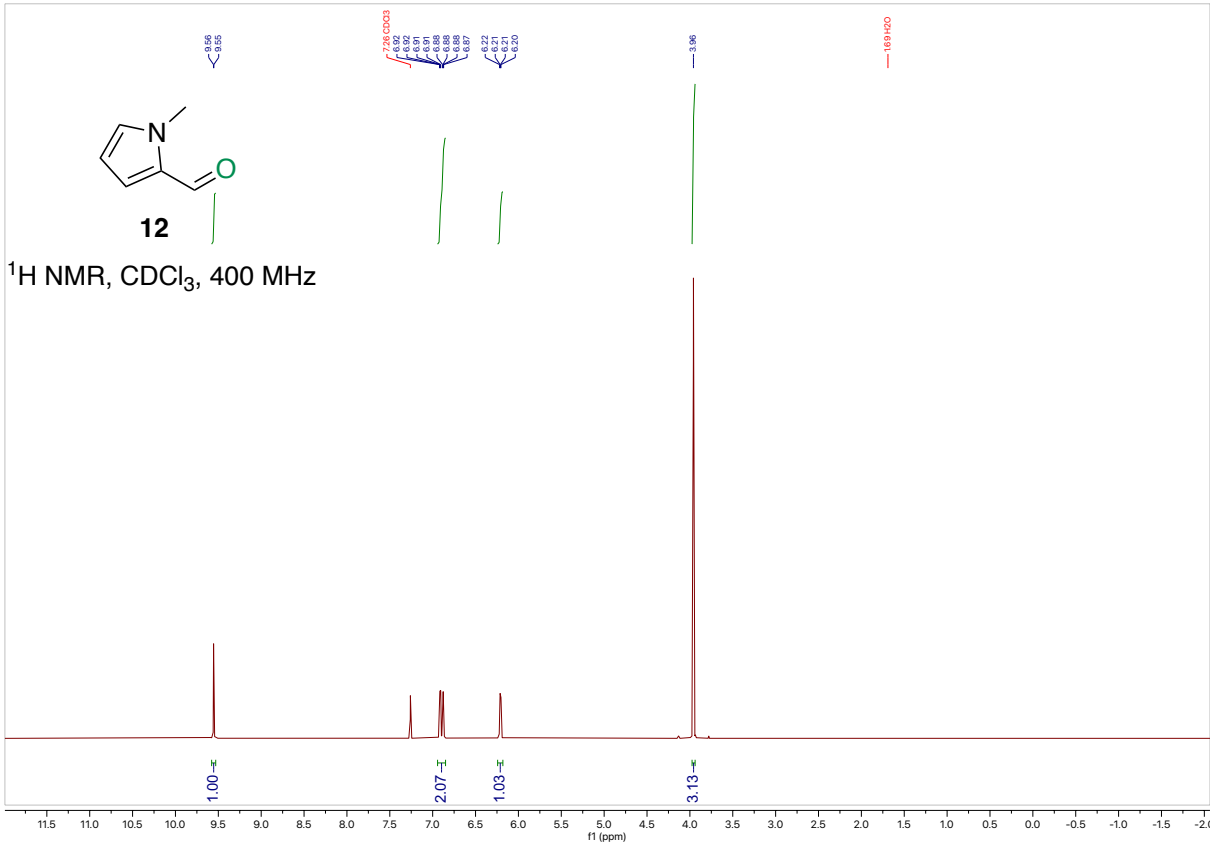
11

^{1}H NMR, DMSO-d6, 400 MHz

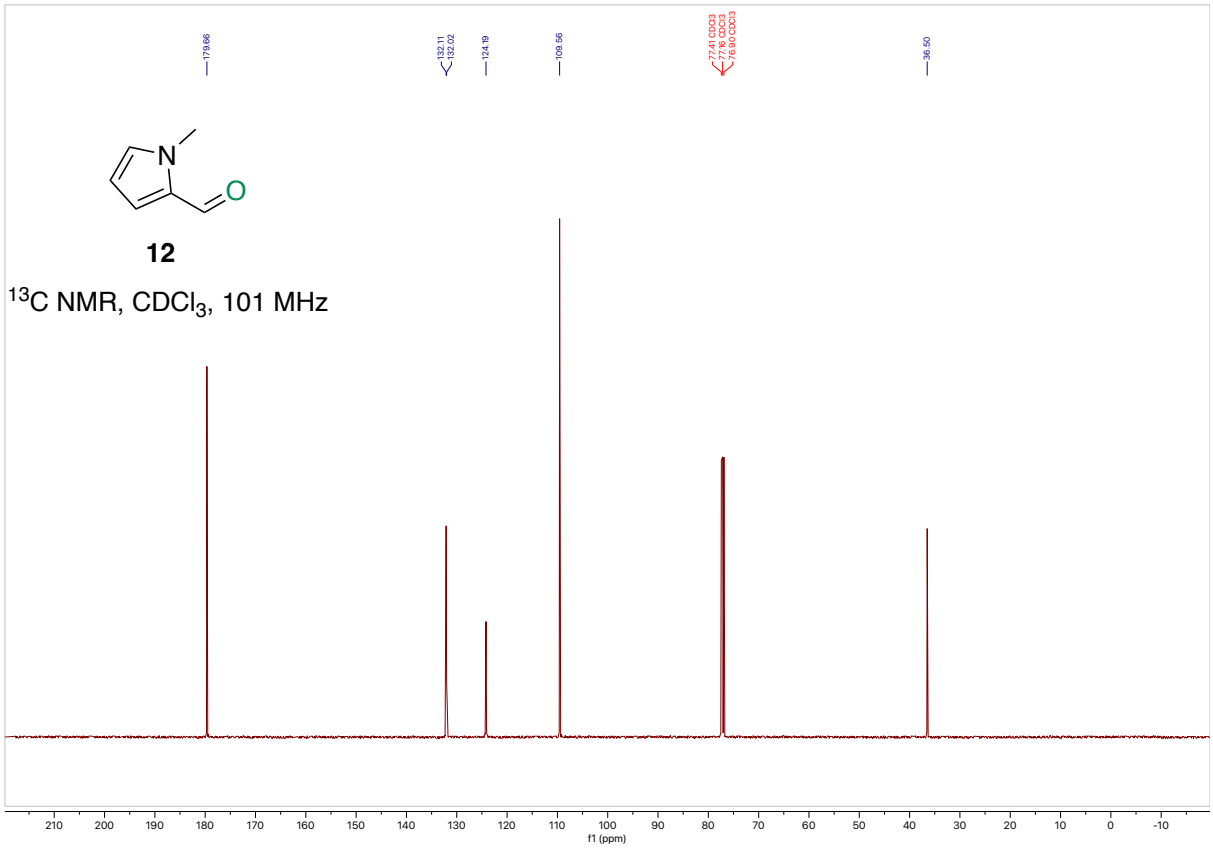


11

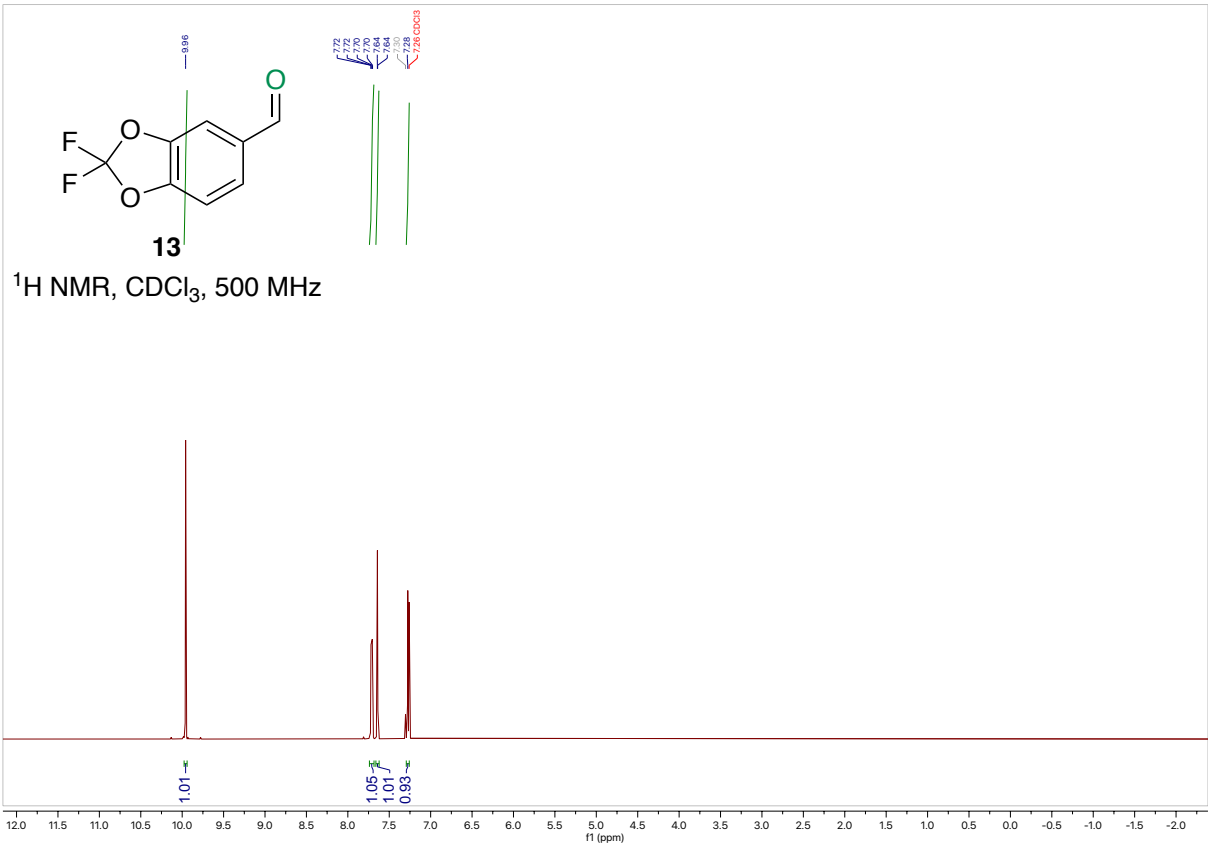
^{13}C NMR, DMSO-d6, 101 MHz



^{1}H NMR, $CDCl_3$, 400 MHz

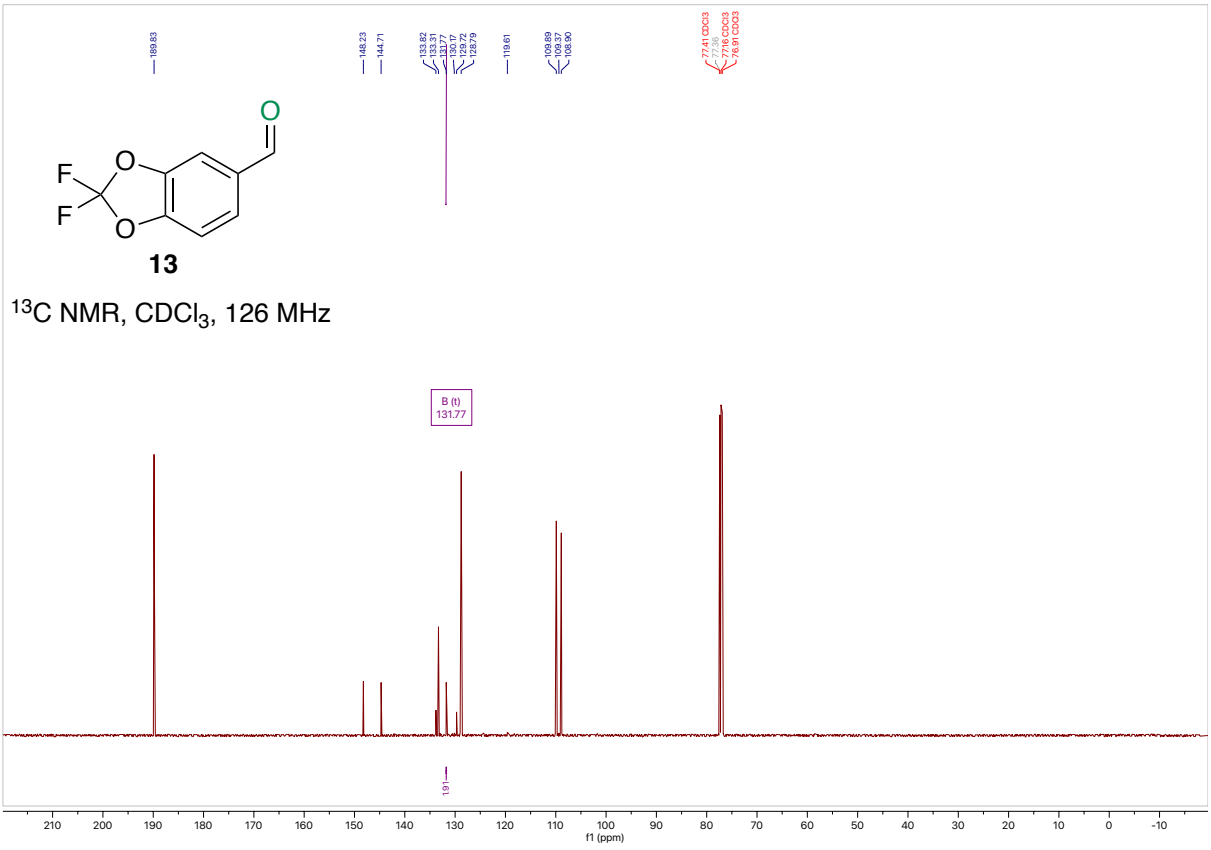


^{13}C NMR, $CDCl_3$, 101 MHz



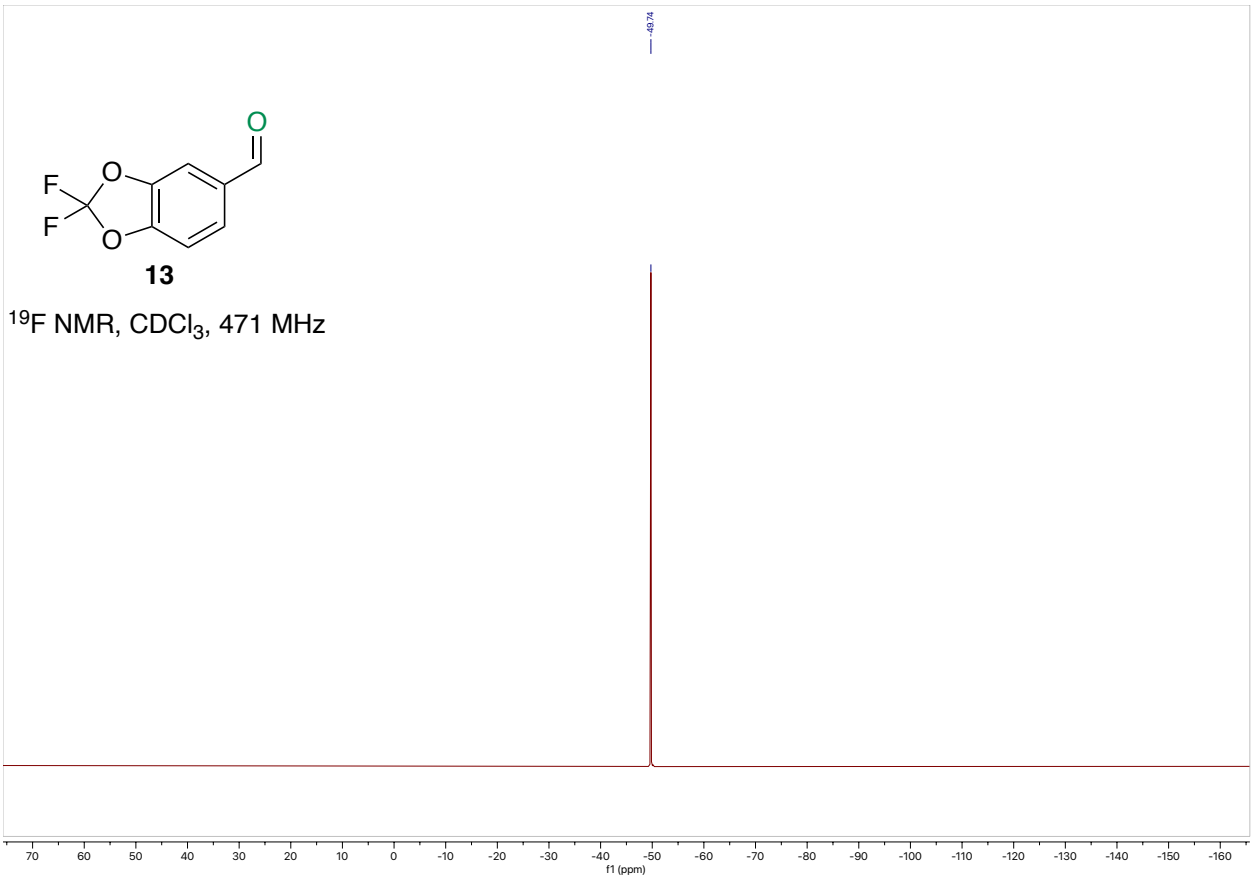
13

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



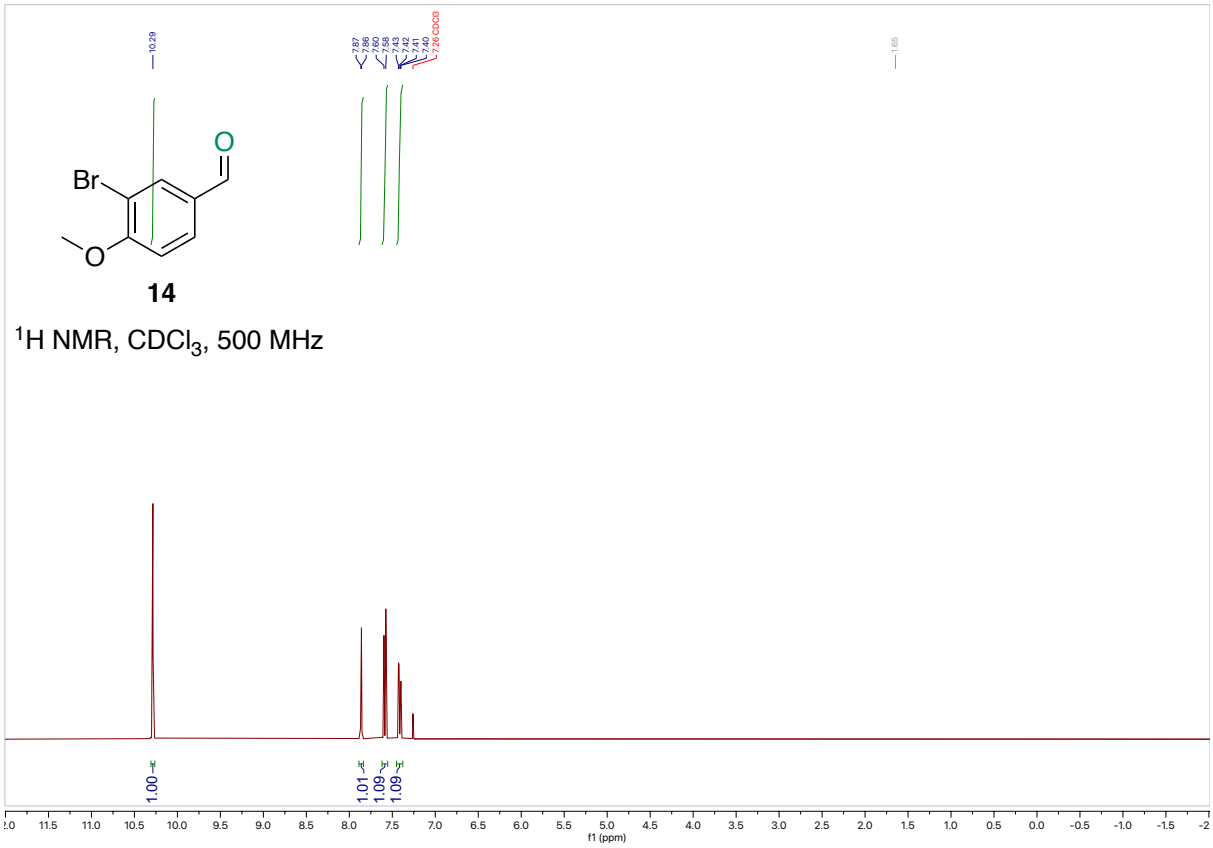
13

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



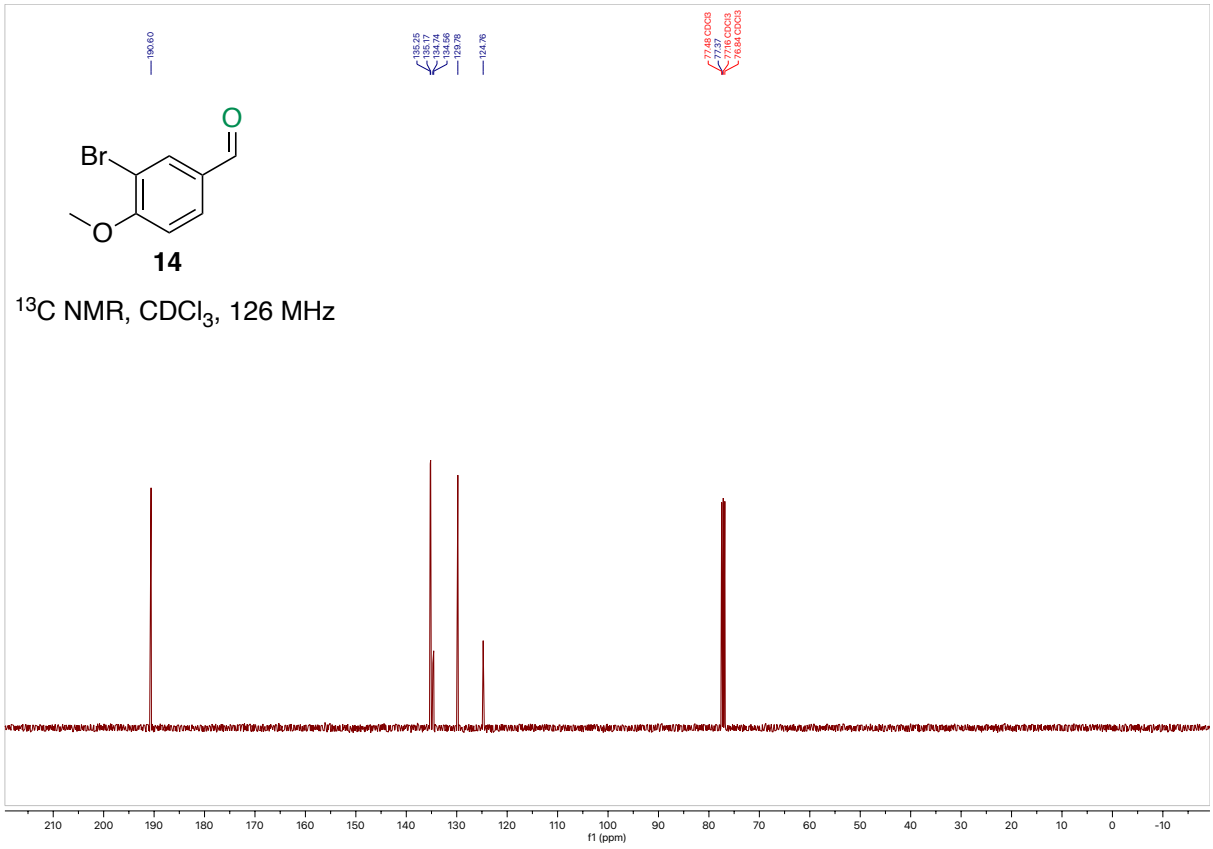
13

^{19}F NMR, $CDCl_3$, 471 MHz



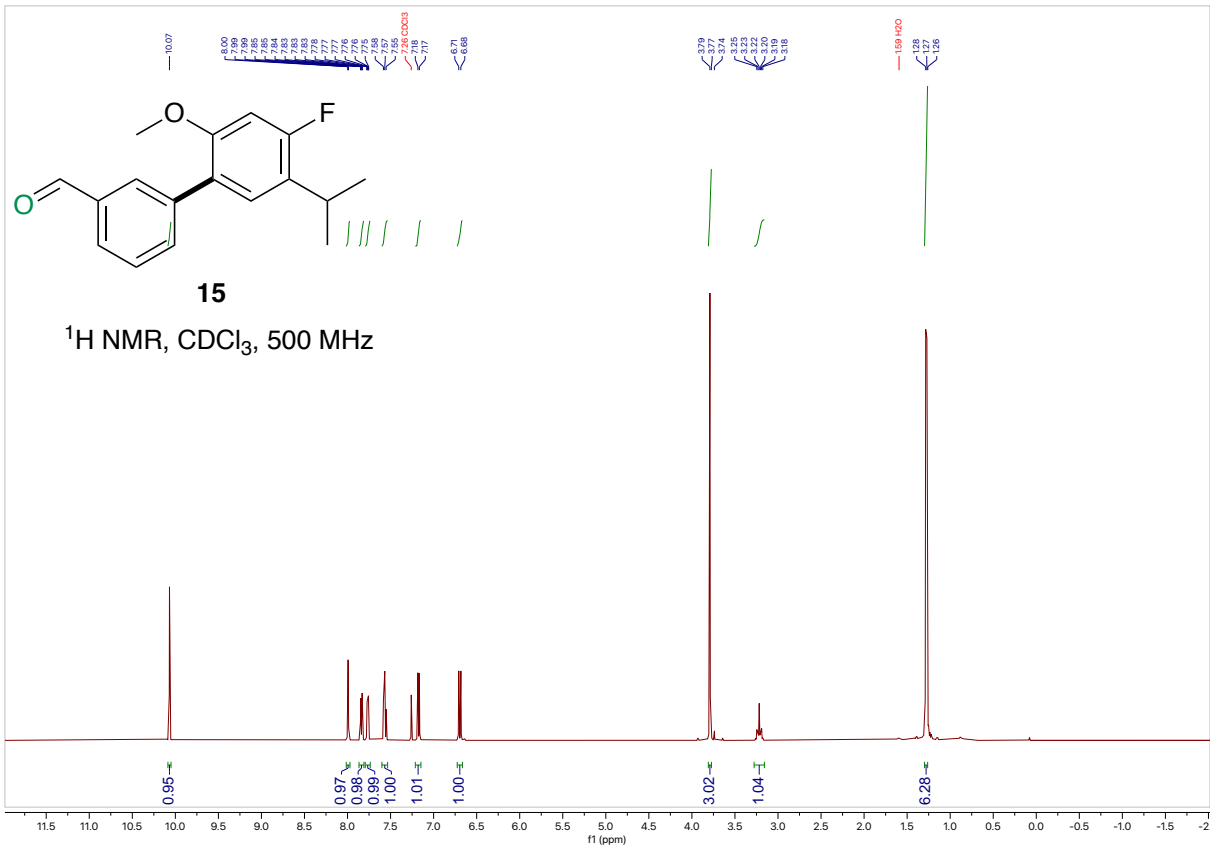
14

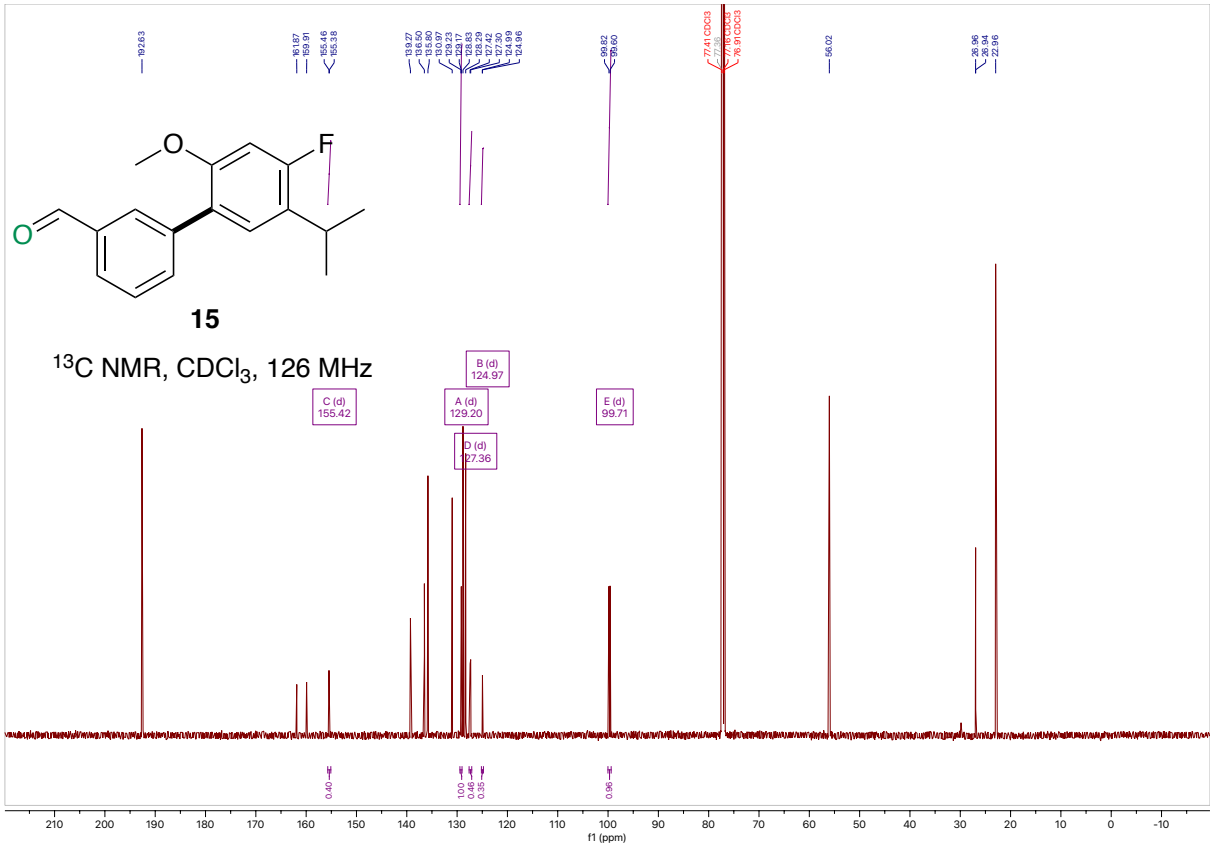
^{1}H NMR, $CDCl_3$, 500 MHz

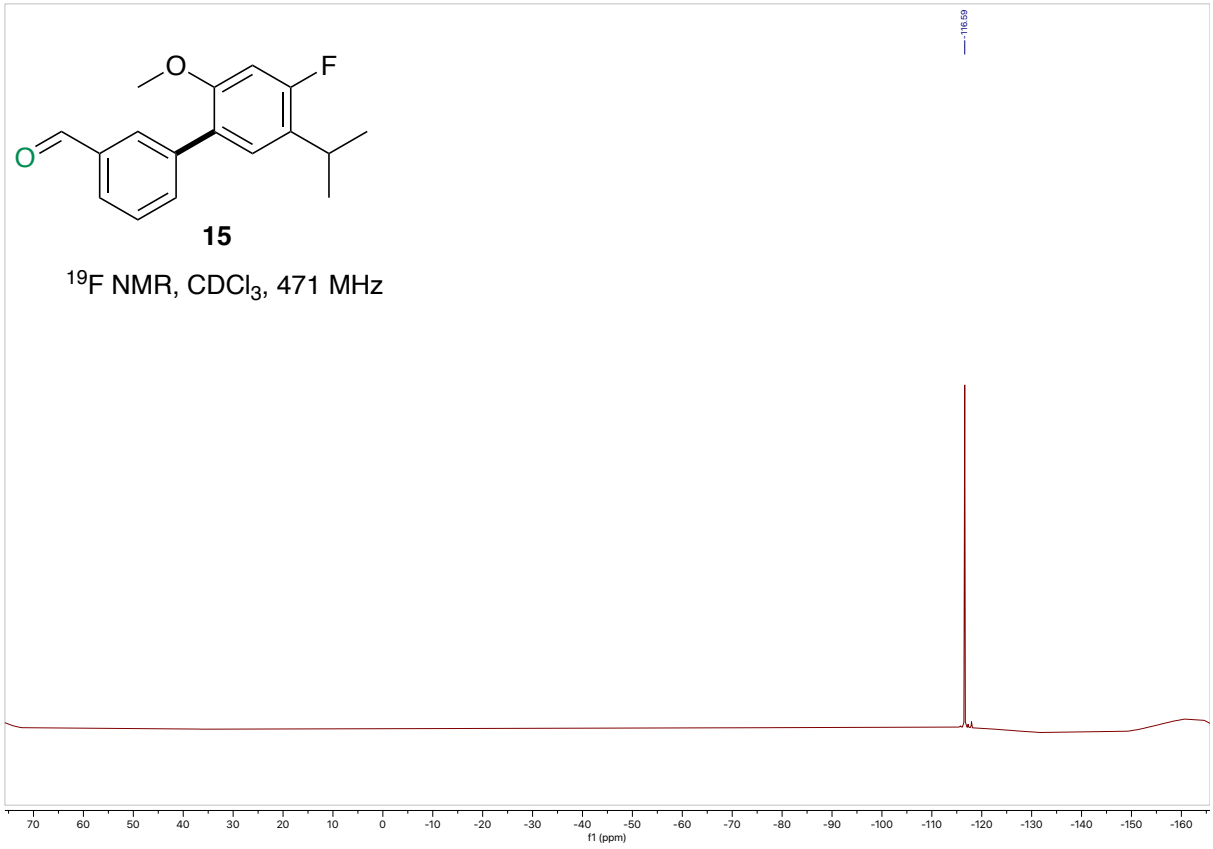


14

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

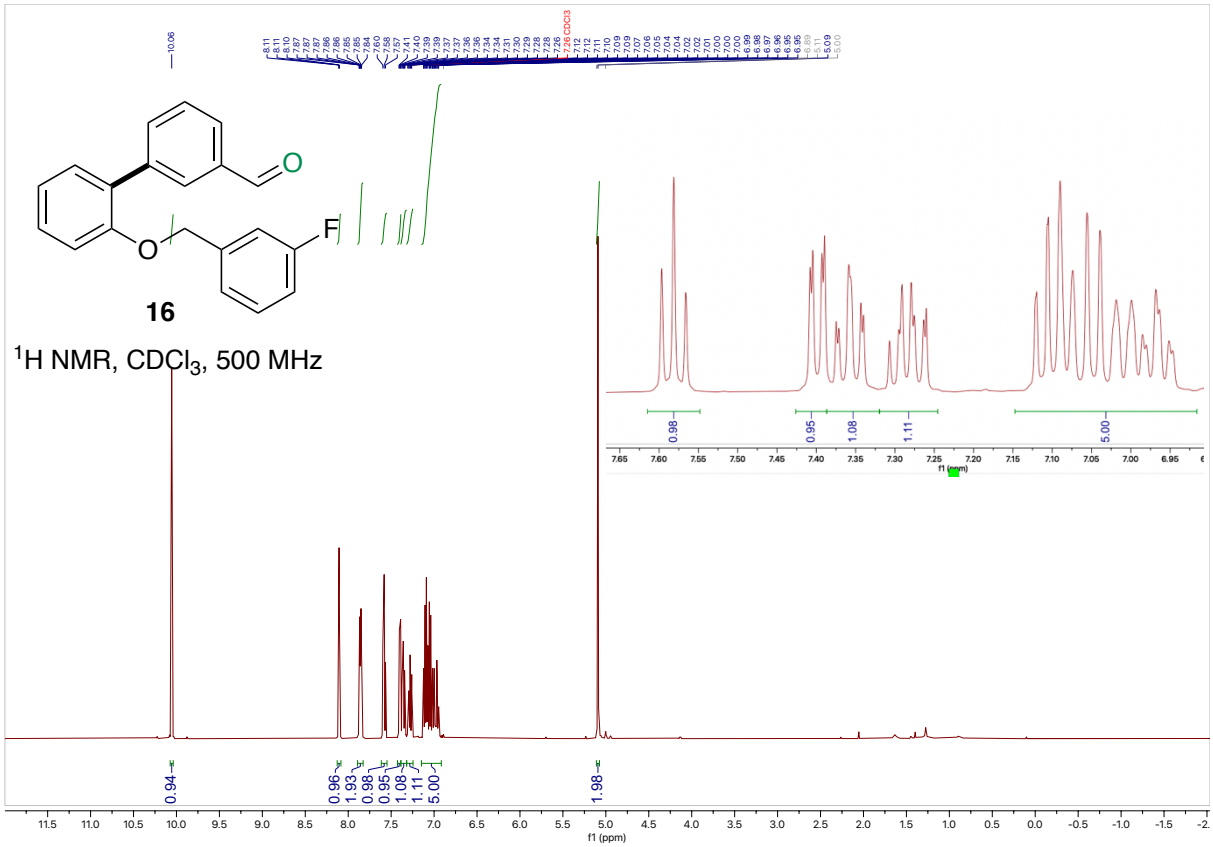

O
F
O
15
1H NMR, CDCl3, 500 MHz
f1 (ppm)

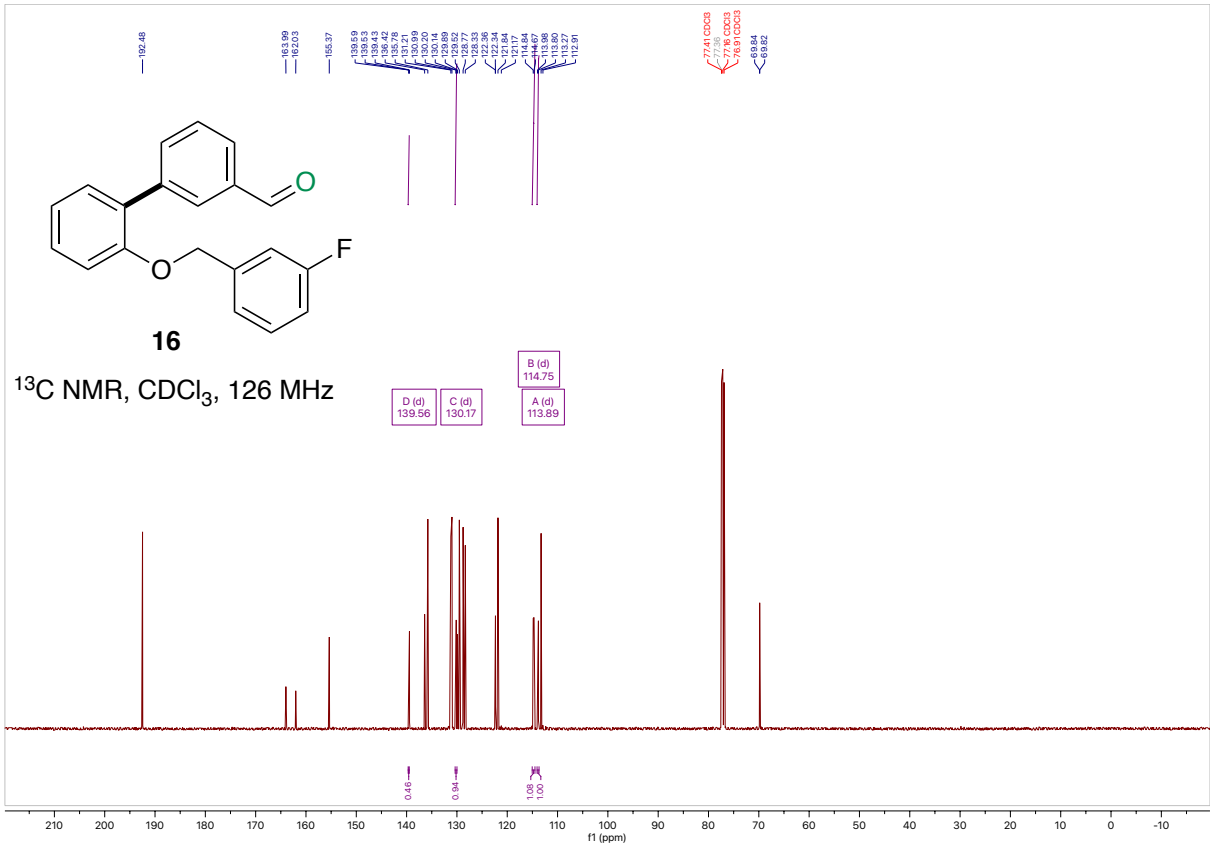

O
F
O
15
13C NMR, CDCl3, 126 MHz
B (d) 124.97
C (d) 155.42
A (d) 129.20
D (d) 127.36
E (d) 99.71
f1 (ppm)

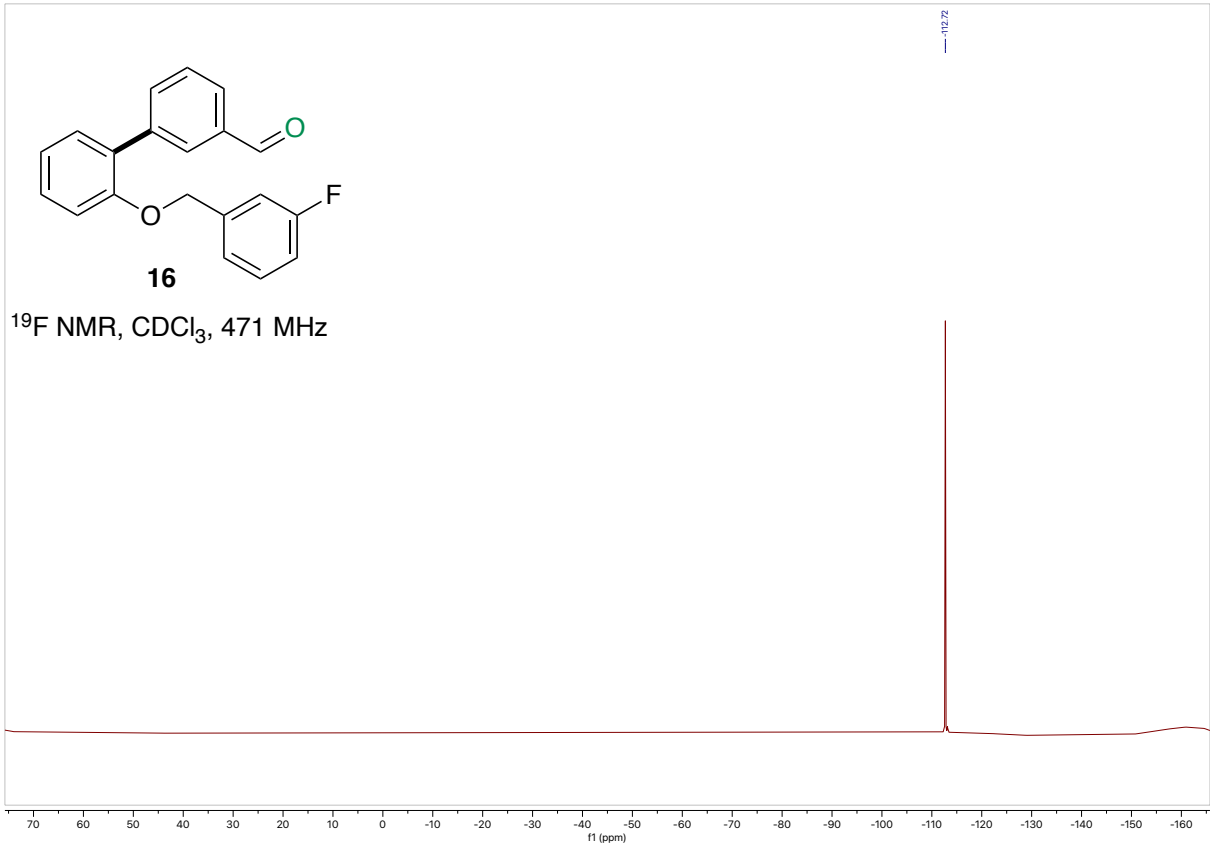


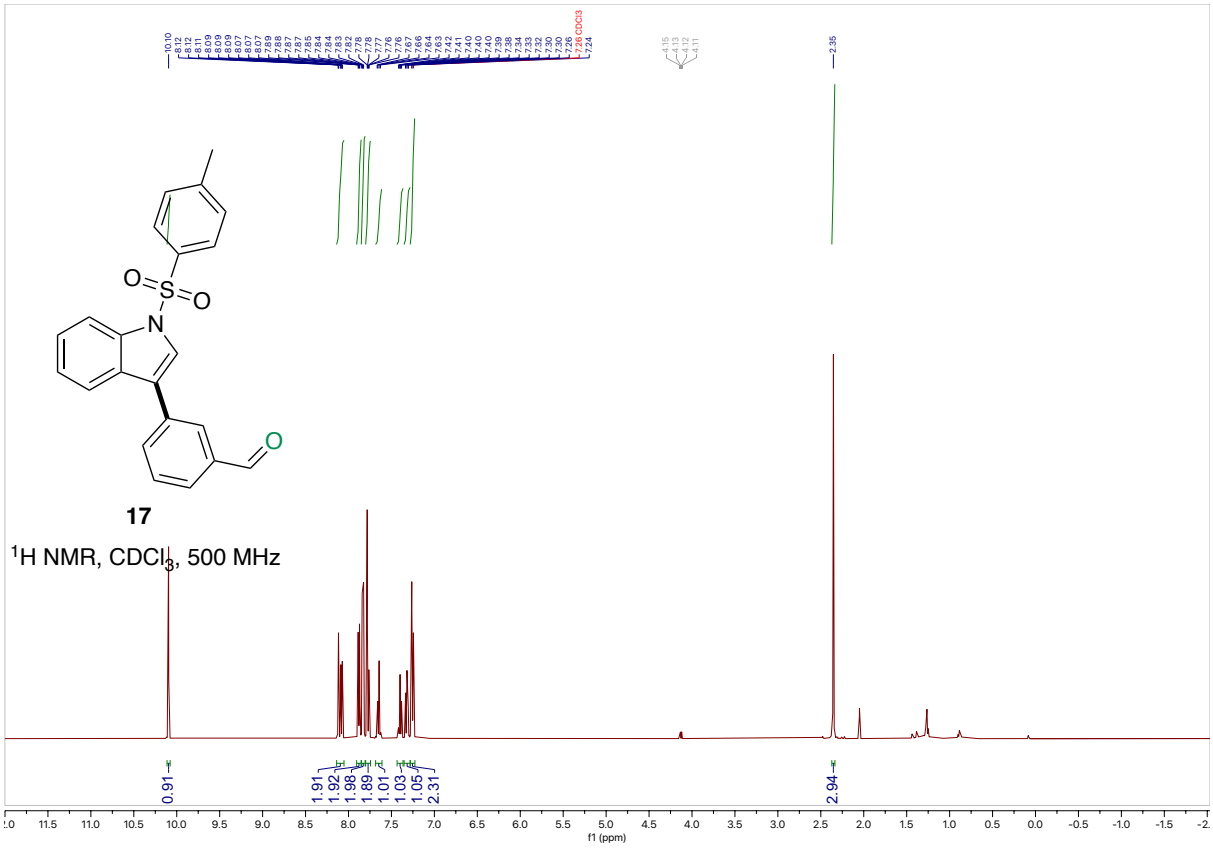
15

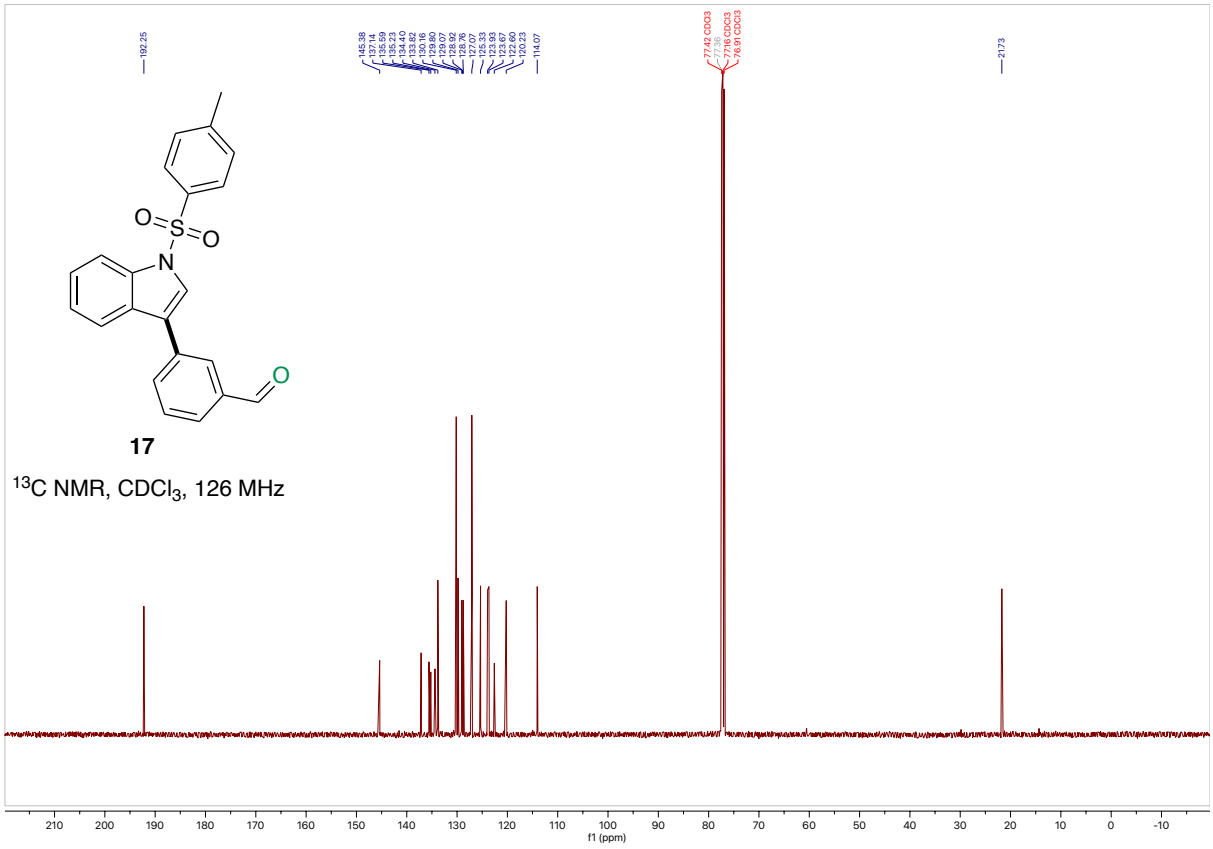
^{19}F NMR, $CDCl_3$, 471 MHz

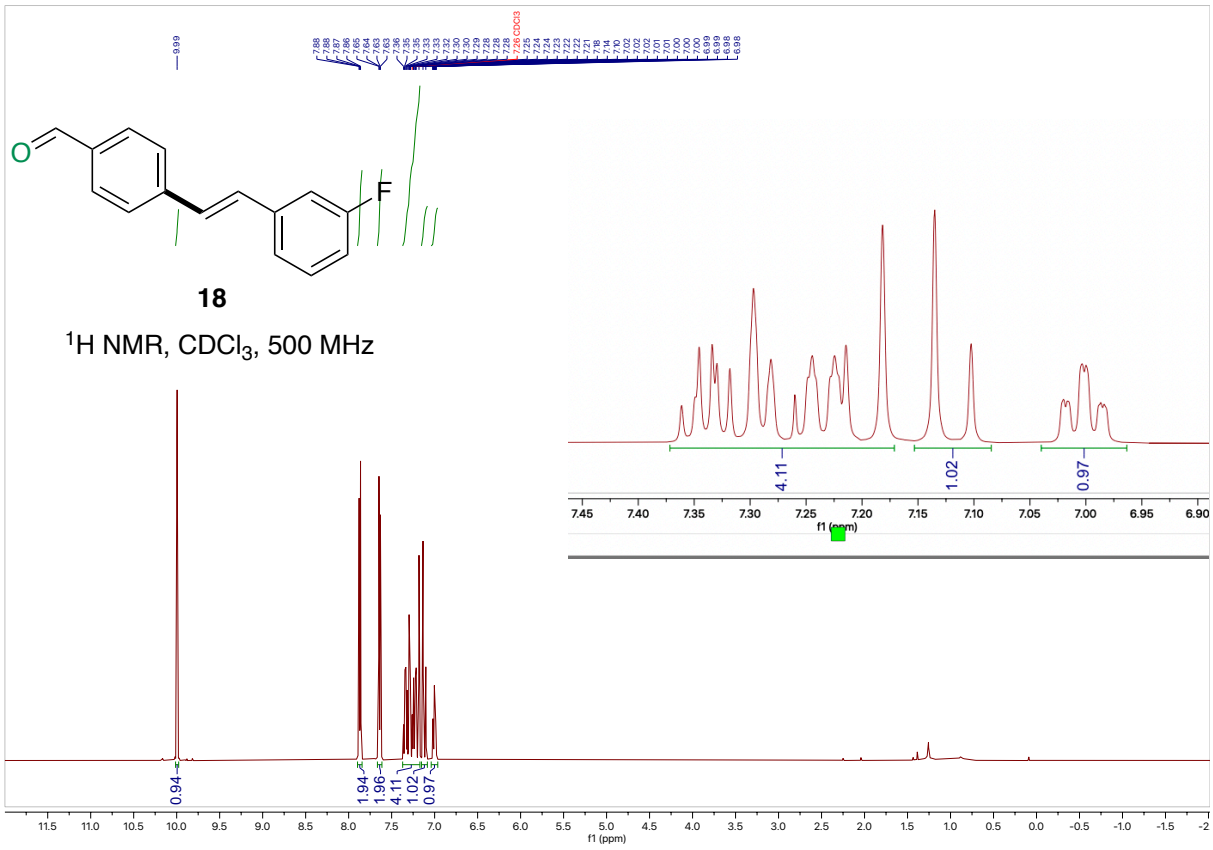

16
^{1}H NMR, $CDCl_3$, 500 MHz
f1 (ppm)

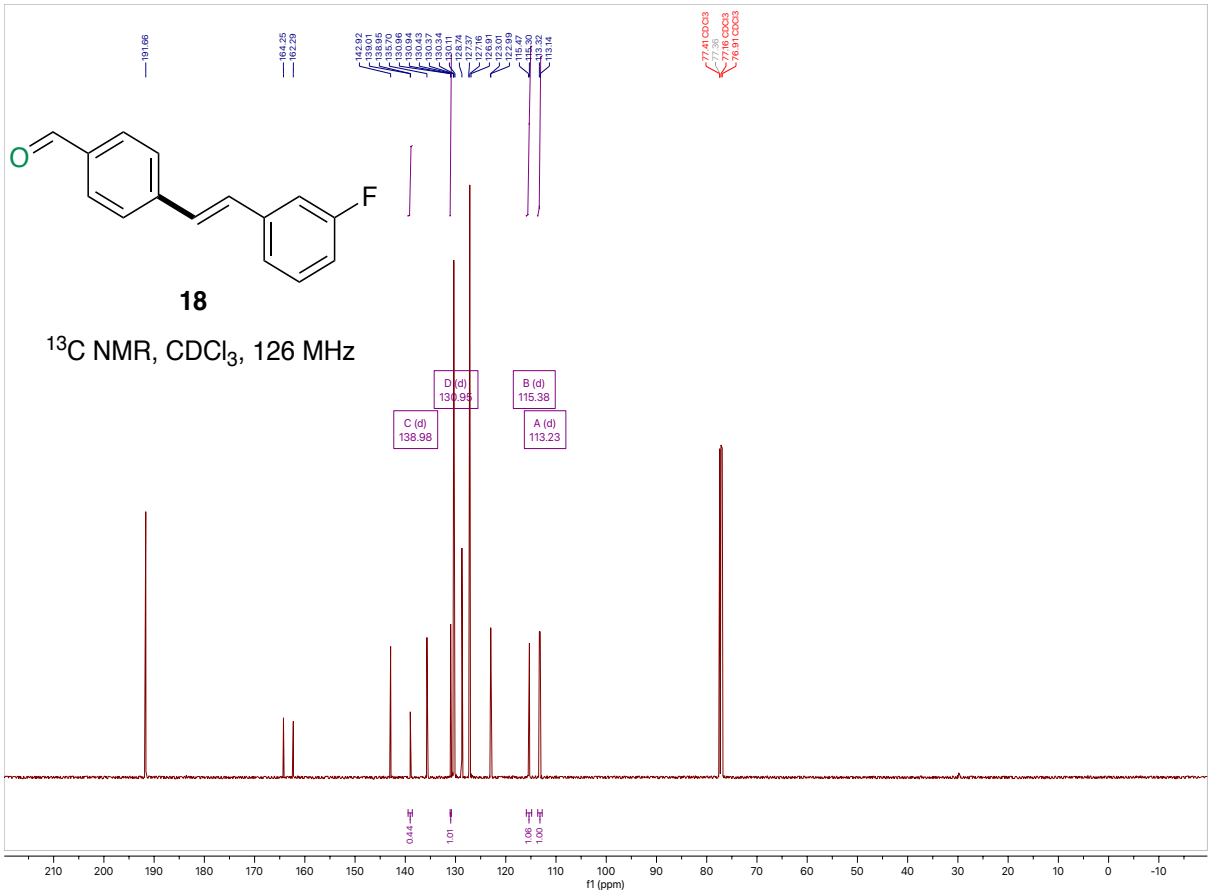

16
^{13}C NMR, $CDCl_3$, 126 MHz
D (d) 139.56
C (d) 130.17
B (d) 114.75
A (d) 113.89
f1 (ppm)

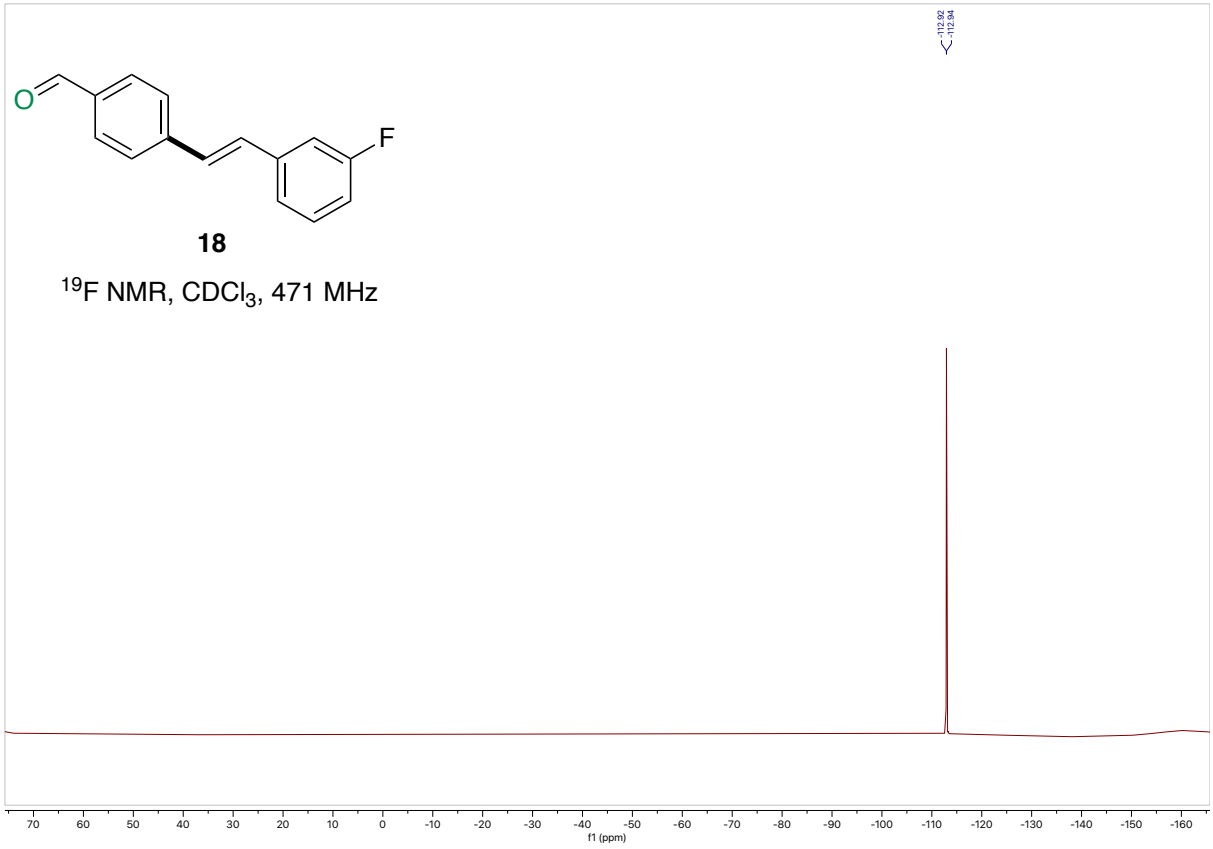

-112.72
O
F
O
16
^{19}F NMR, $CDCl_3$, 471 MHz
70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160
f1 (ppm)


17
^{1}H NMR, $CDCl_3$, 500 MHz
f1 (ppm)


17
^{13}C NMR, $CDCl_3$, 126 MHz
f1 (ppm)

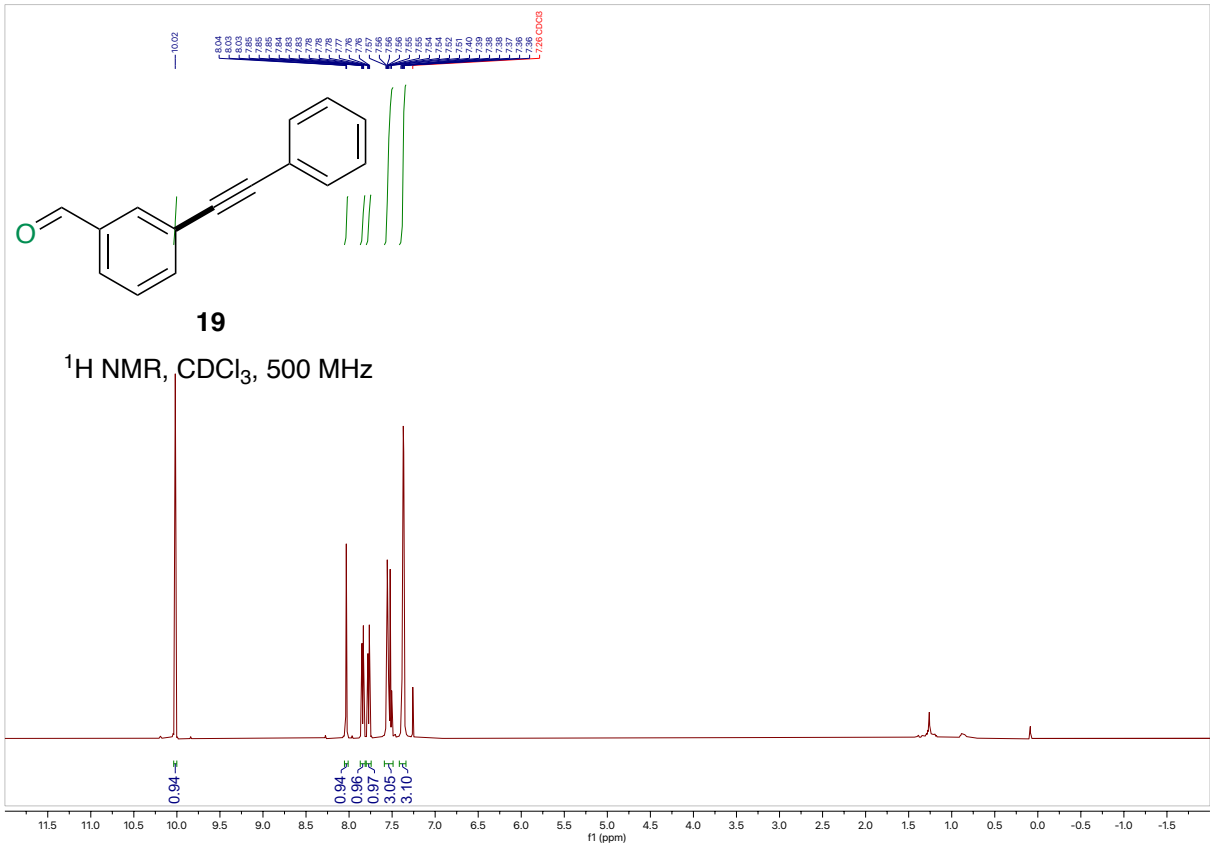

O
F
18
1H NMR, CDCl3, 500 MHz
4.11
1.02
0.97
f1 (ppm)


O
F
18
13C NMR, CDCl3, 126 MHz
D (d) 130.95
B (d) 115.38
C (d) 138.98
A (d) 113.23
f1 (ppm)



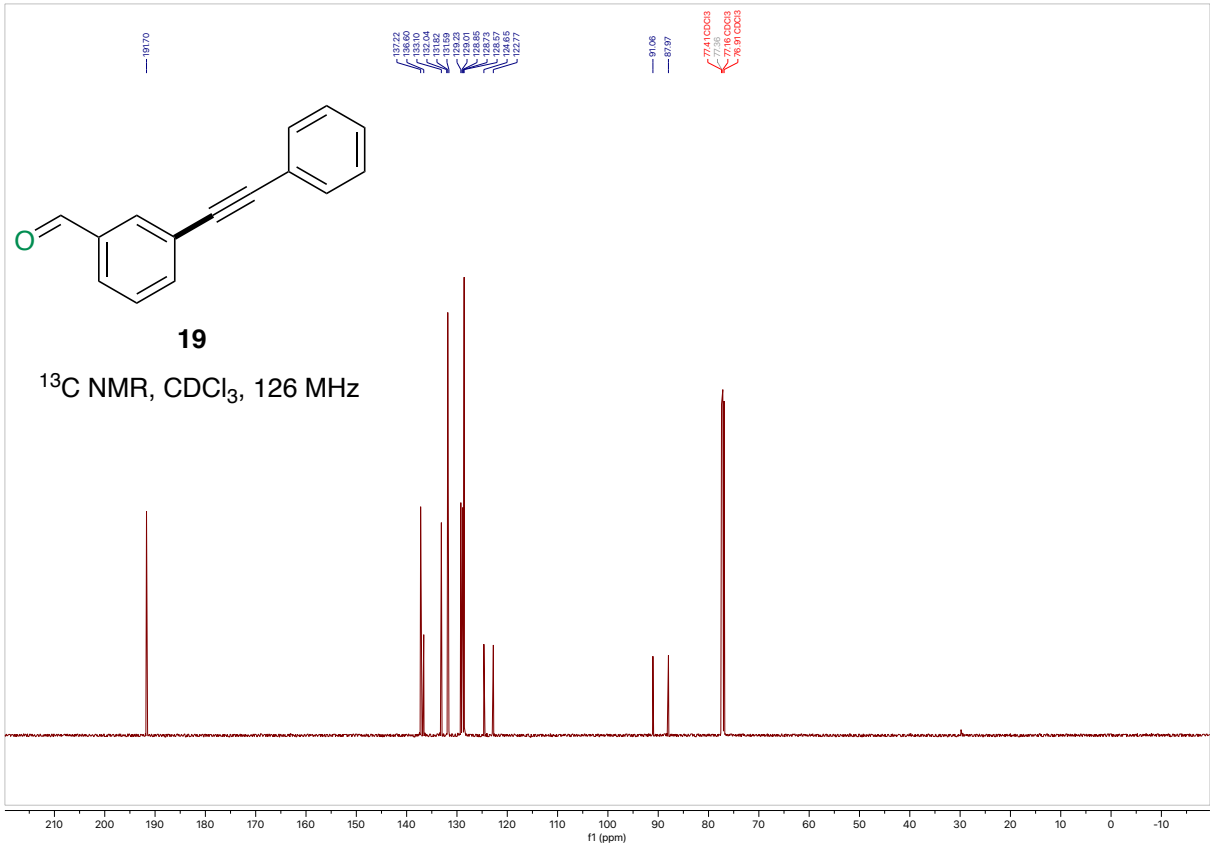
18

^{19}F NMR, $CDCl_3$, 471 MHz



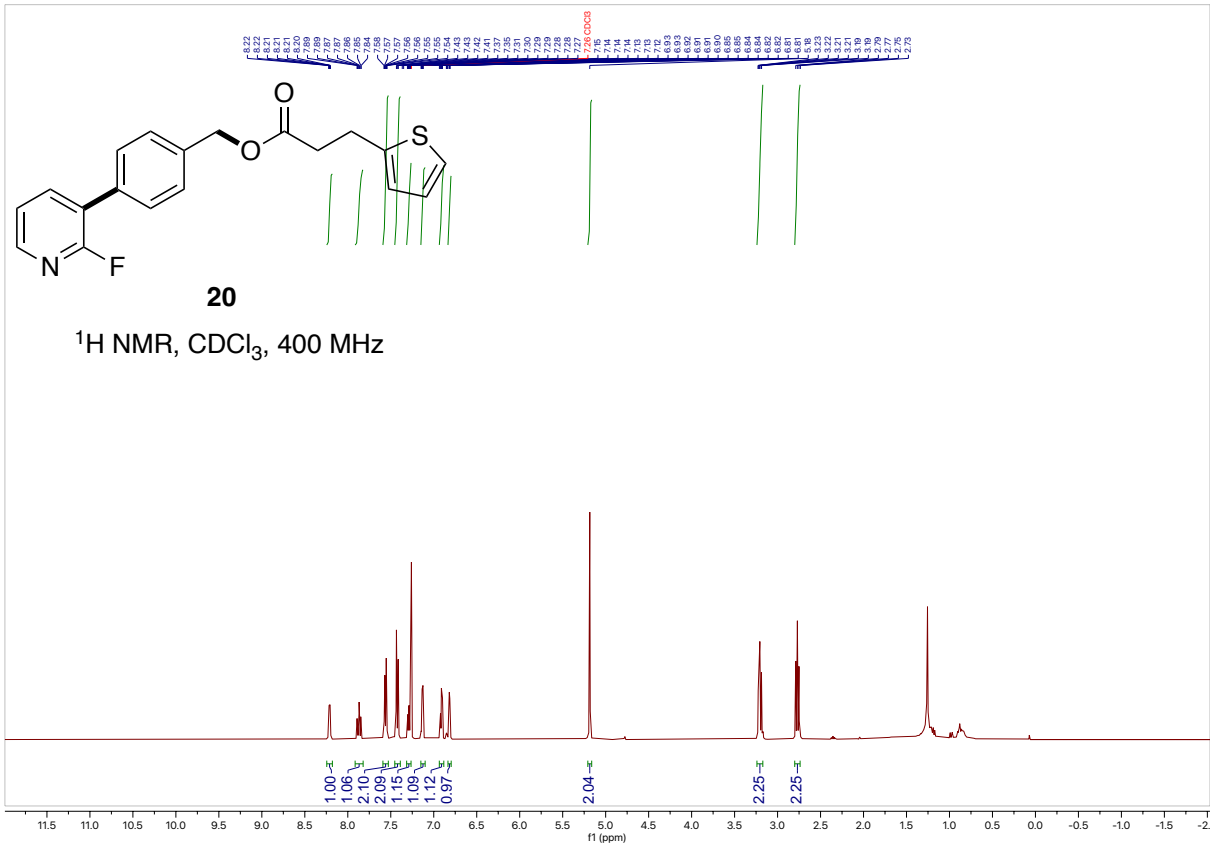
19

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



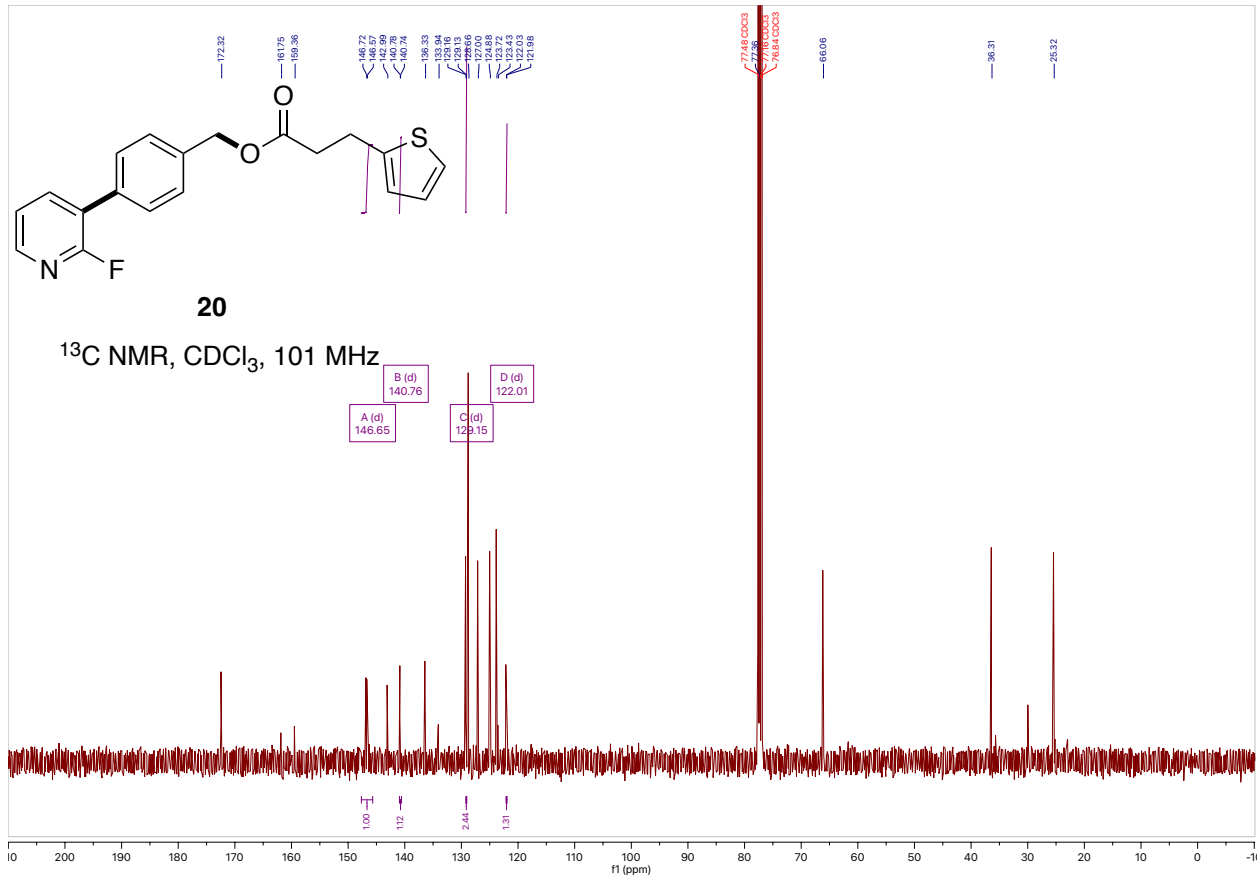
19

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



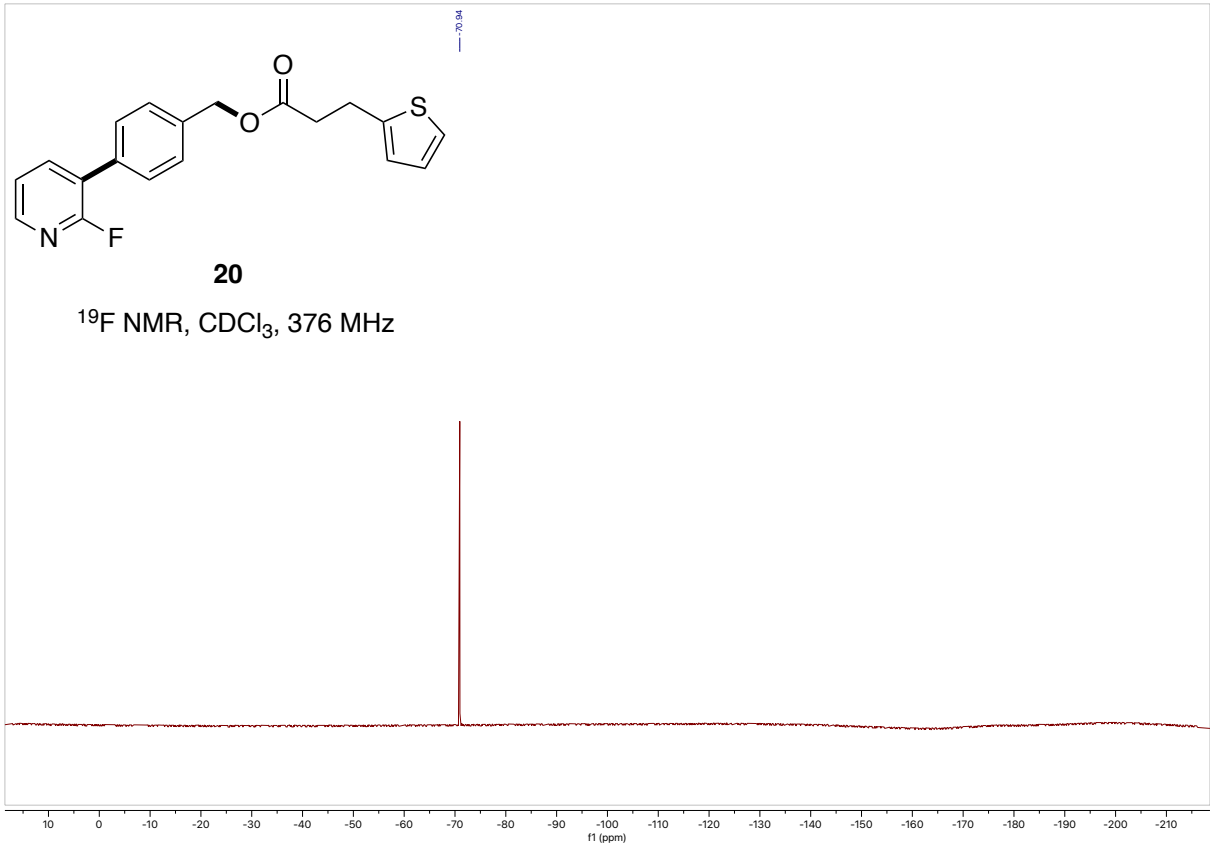
20

^{1}H NMR, $CDCl_3$, 400 MHz



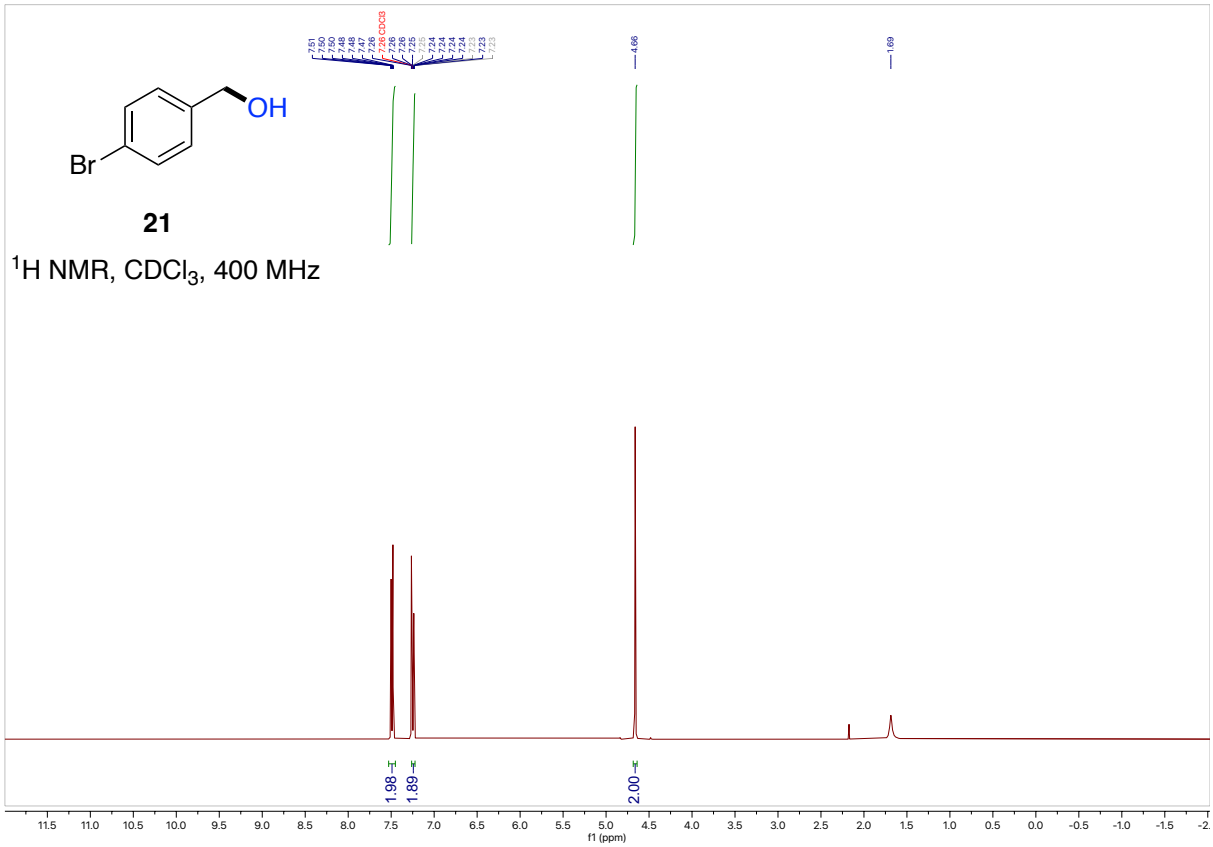
20

^{13}C NMR, $CDCl_3$, 101 MHz



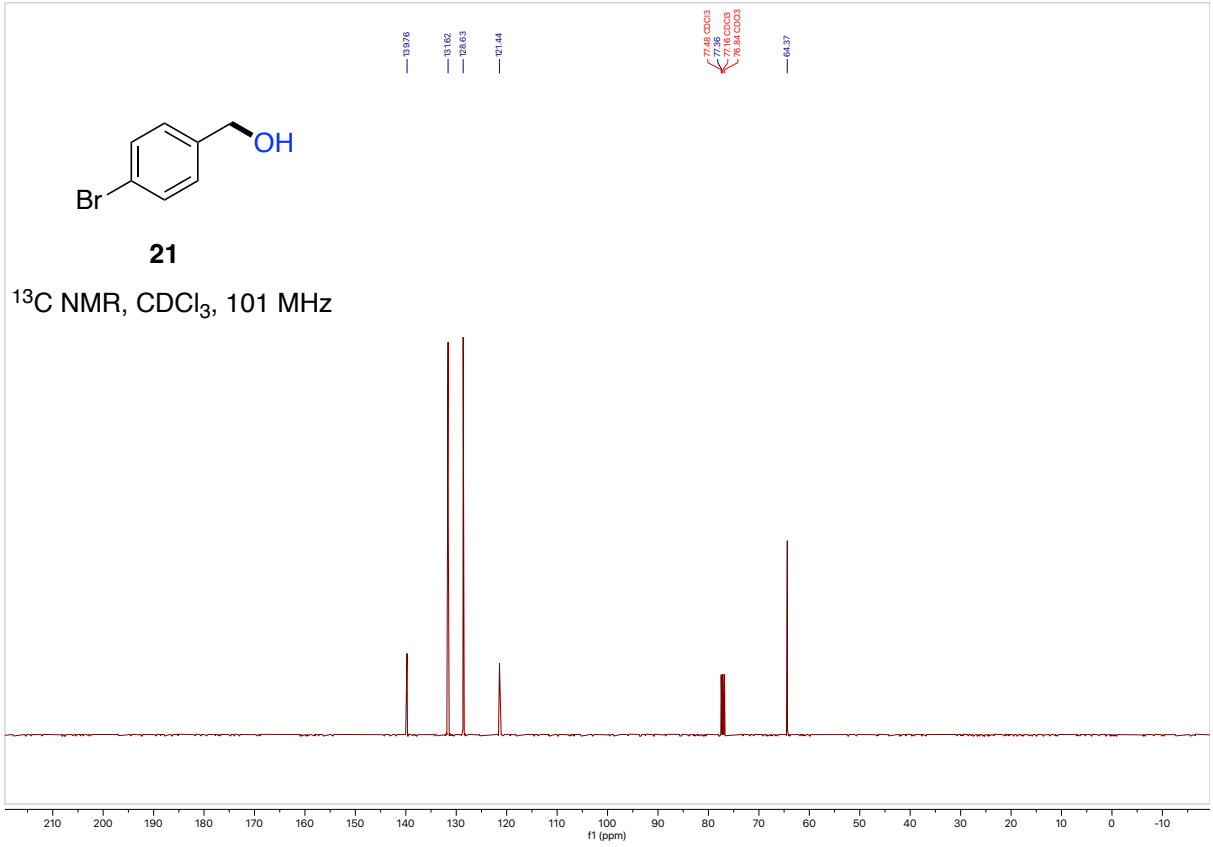
20

^{19}F NMR, $CDCl_3$, 376 MHz



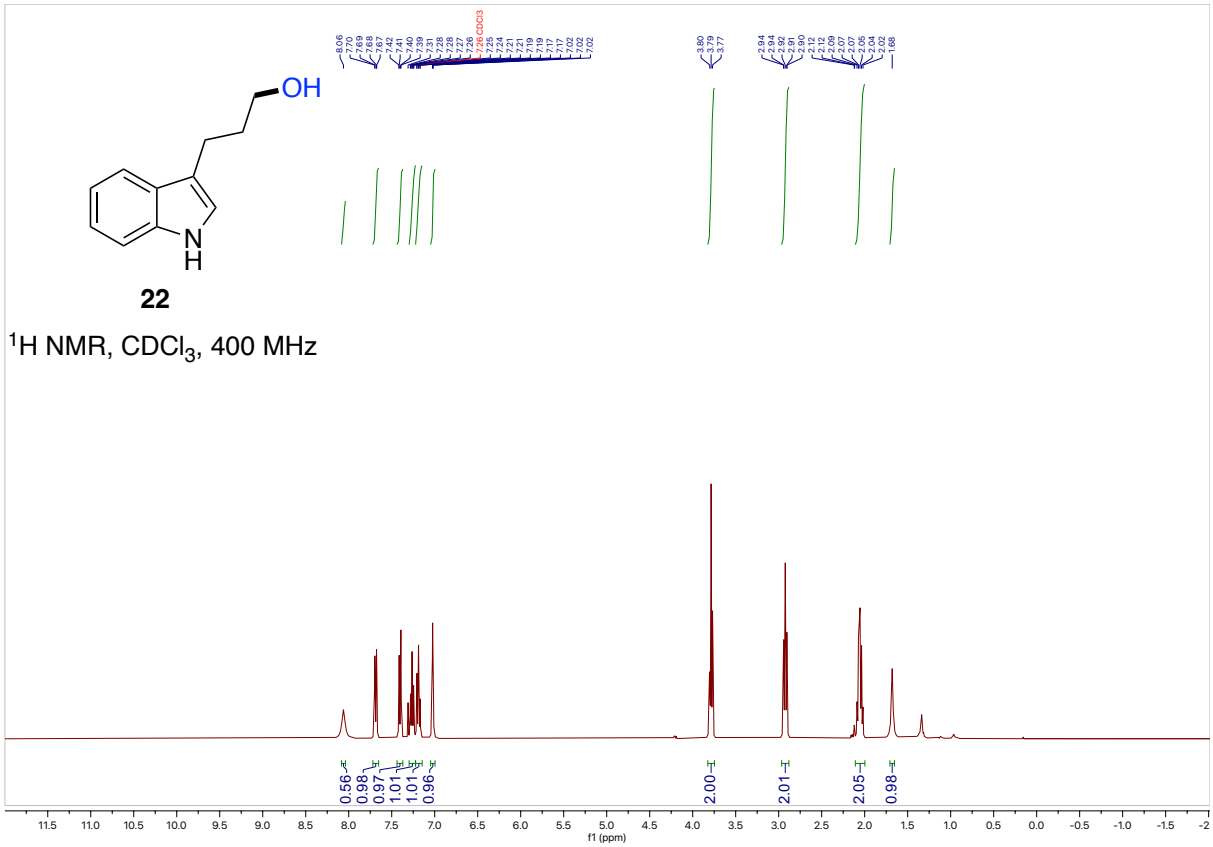
21

^{1}H NMR, $CDCl_3$, 400 MHz



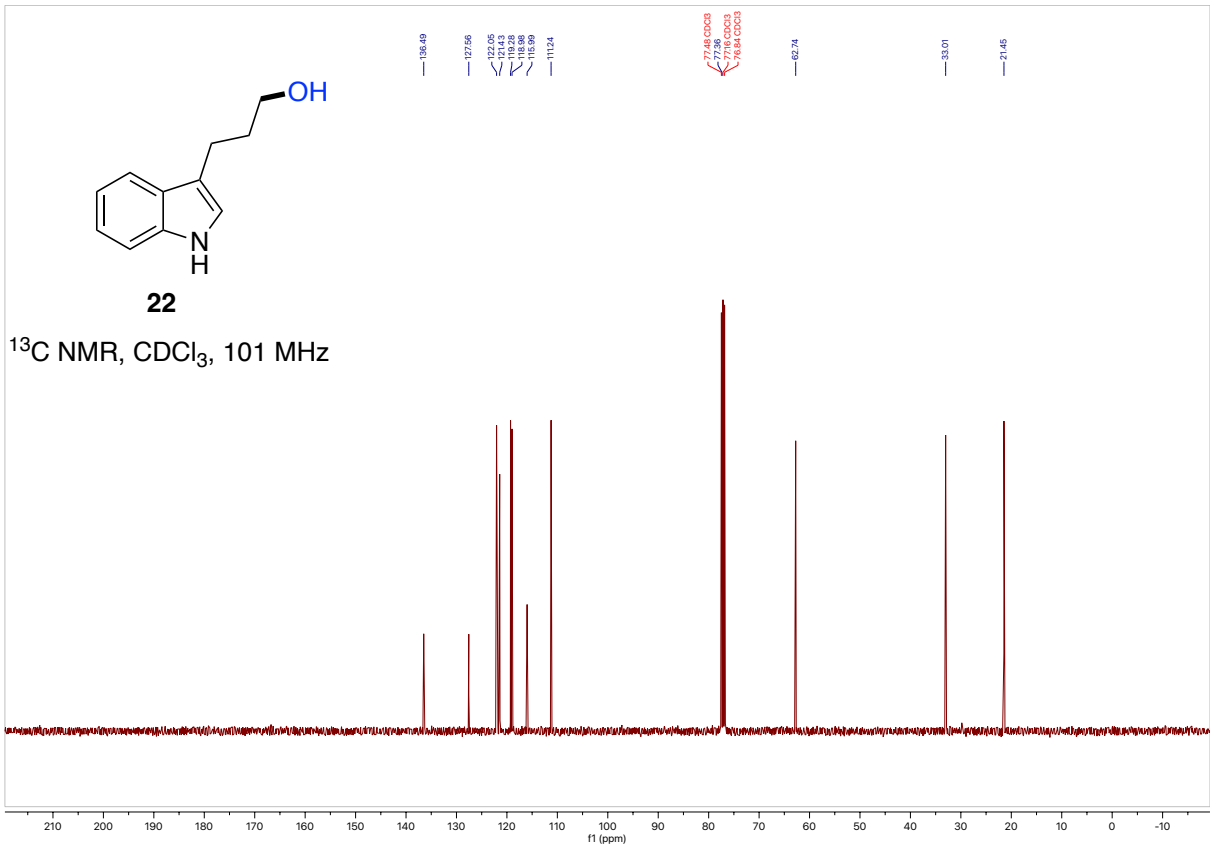
21

^{13}C NMR, $CDCl_3$, 101 MHz



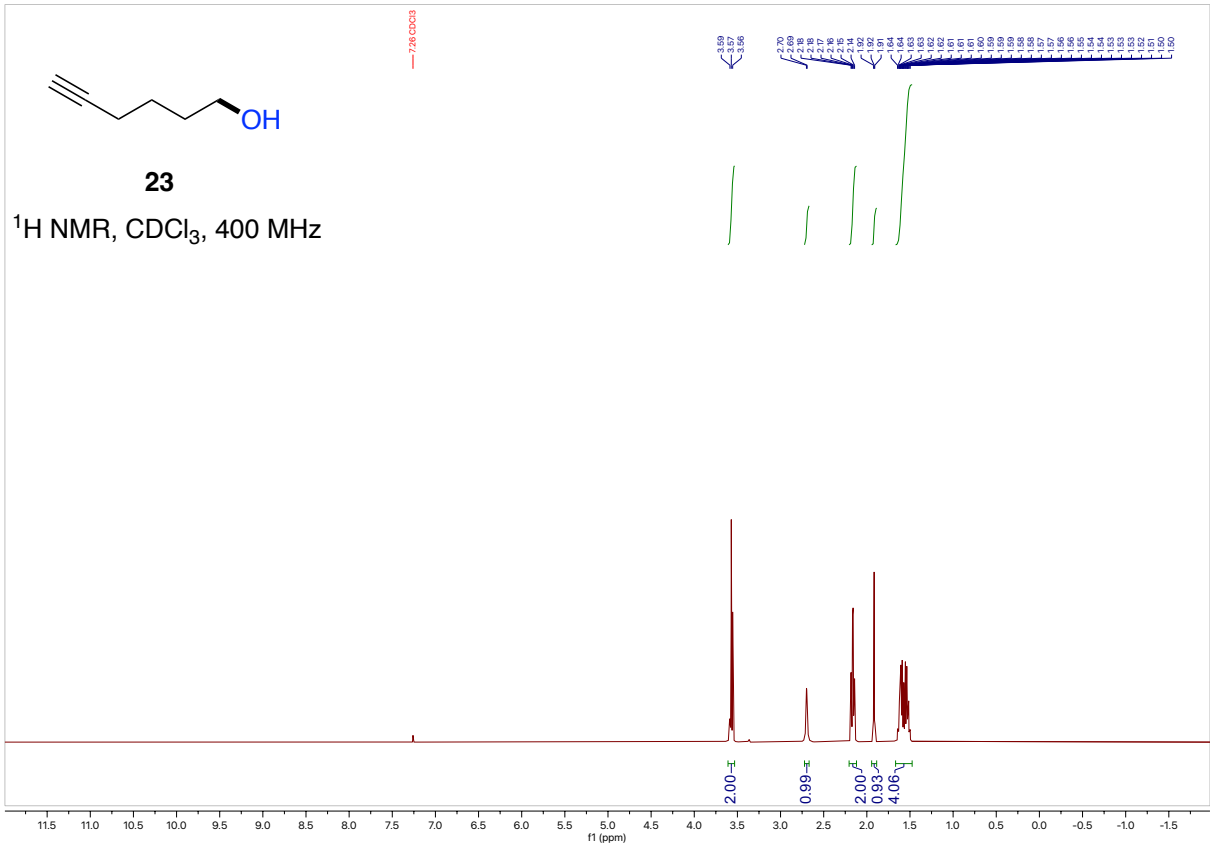
22

^{1}H NMR, $CDCl_3$, 400 MHz



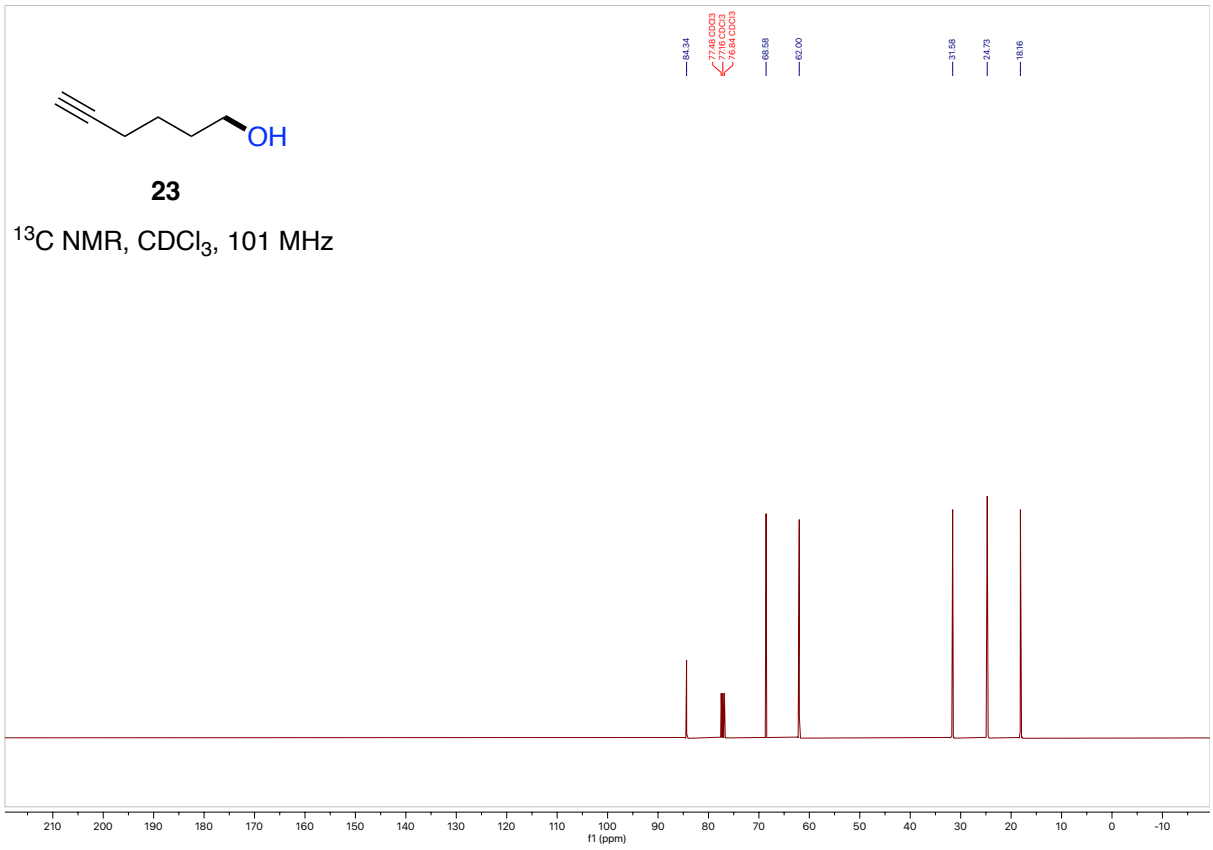
22

^{13}C NMR, $CDCl_3$, 101 MHz



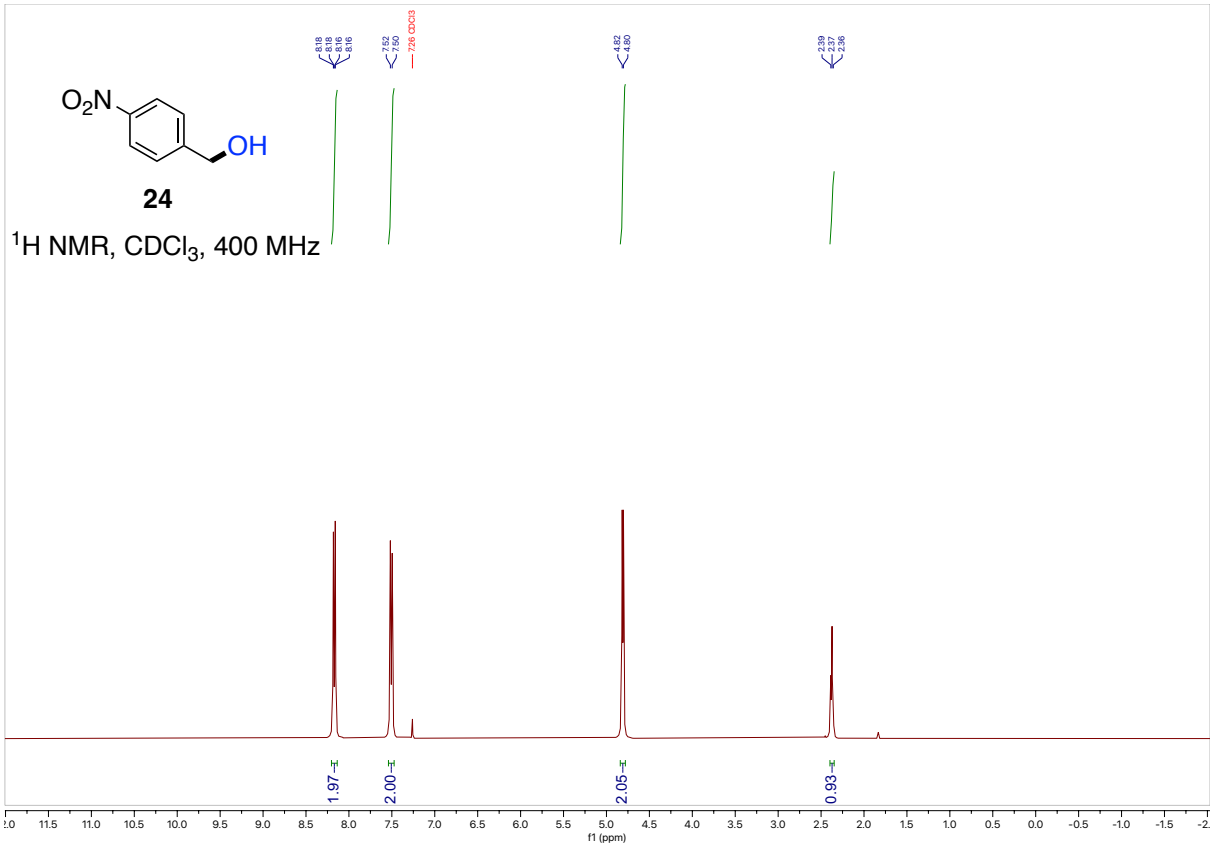
23

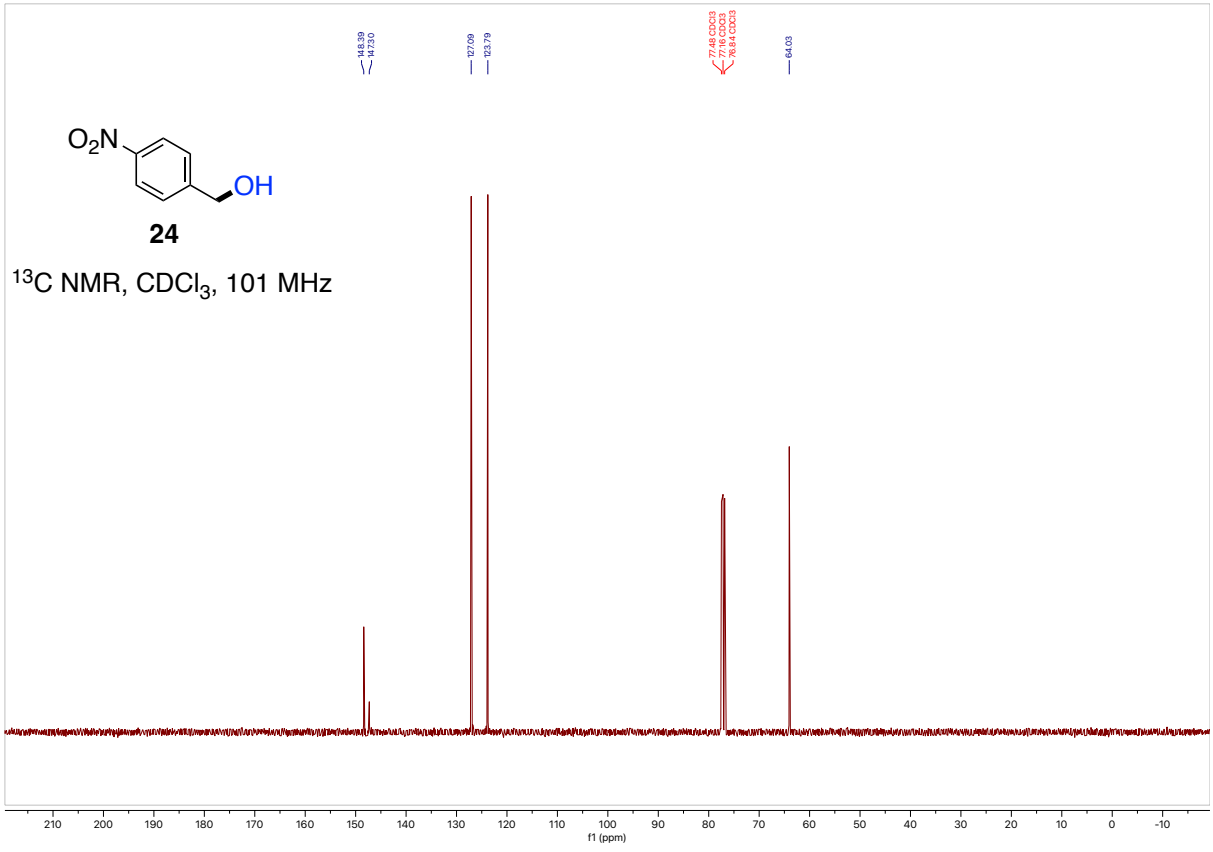
^{1}H NMR, $CDCl_3$, 400 MHz

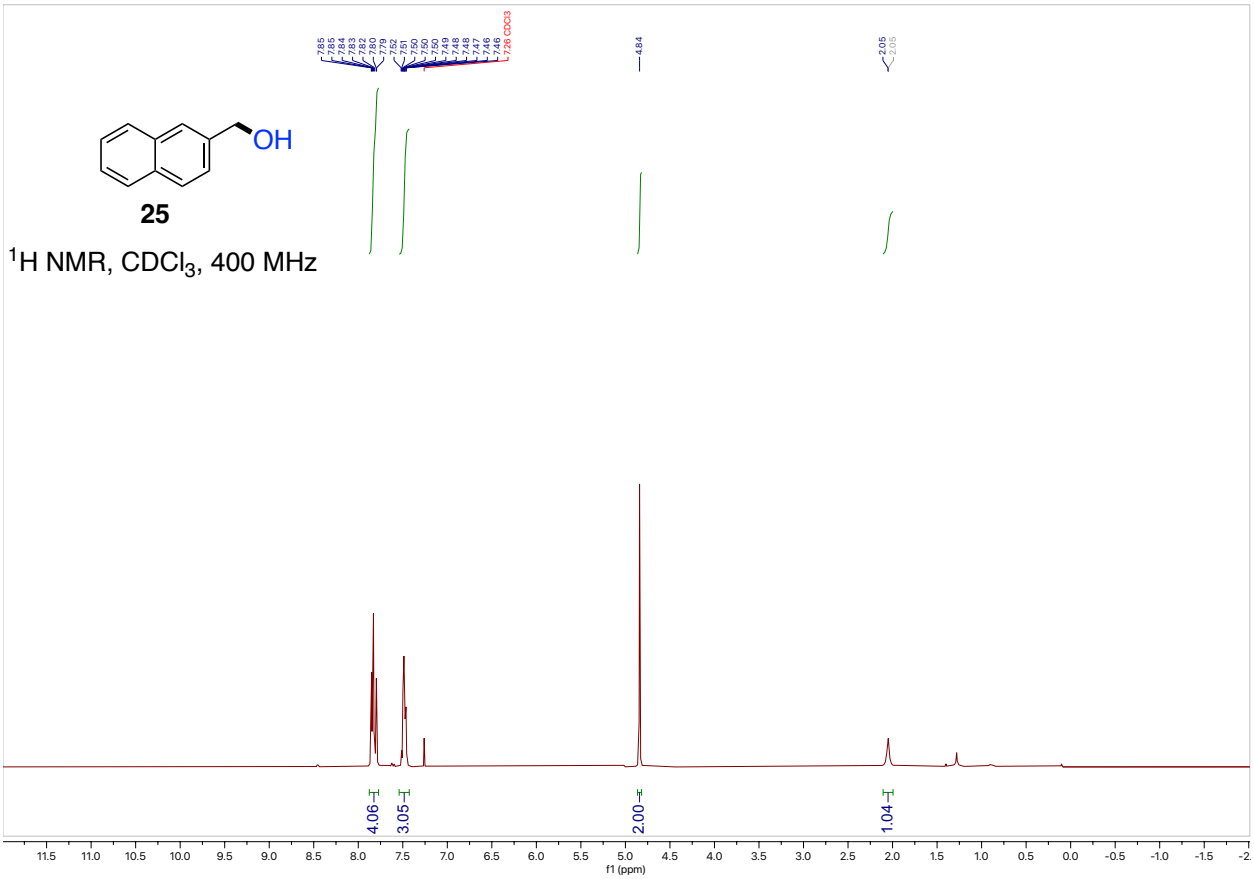


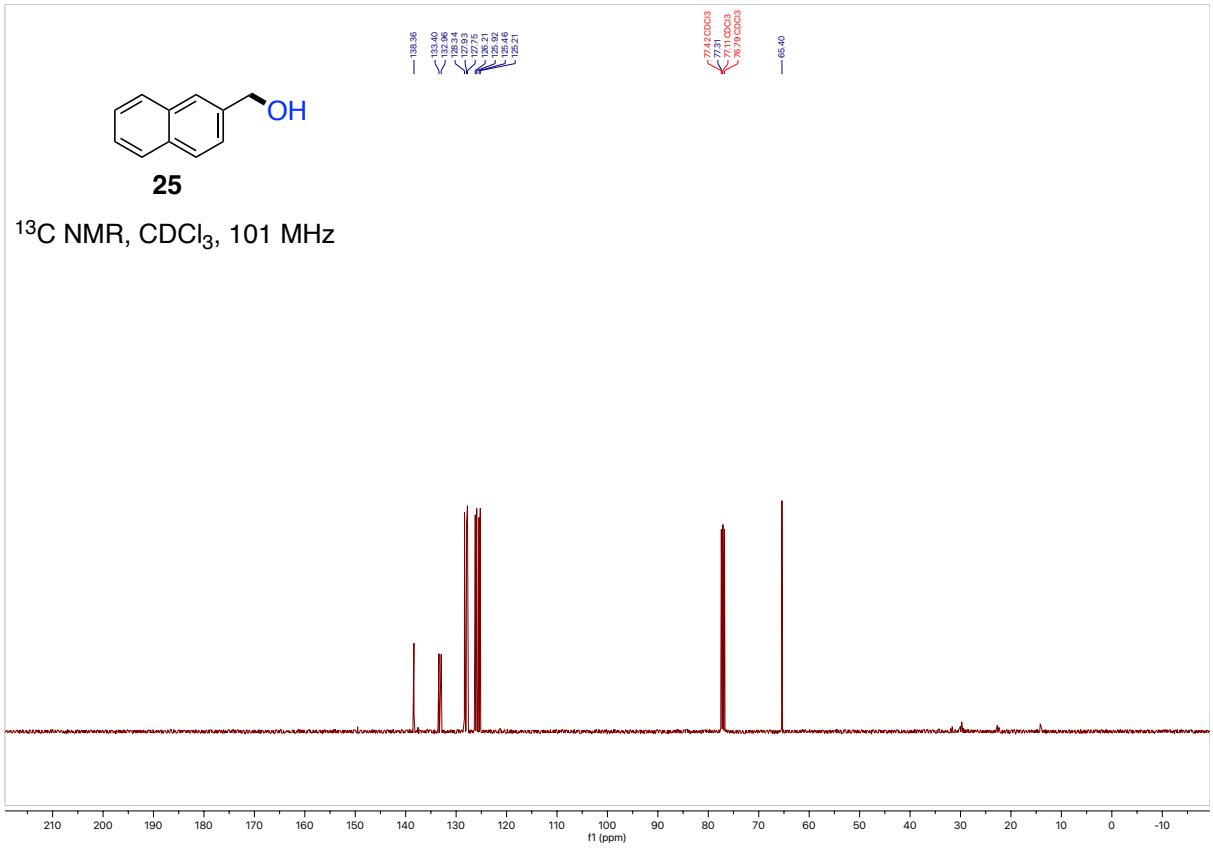
23

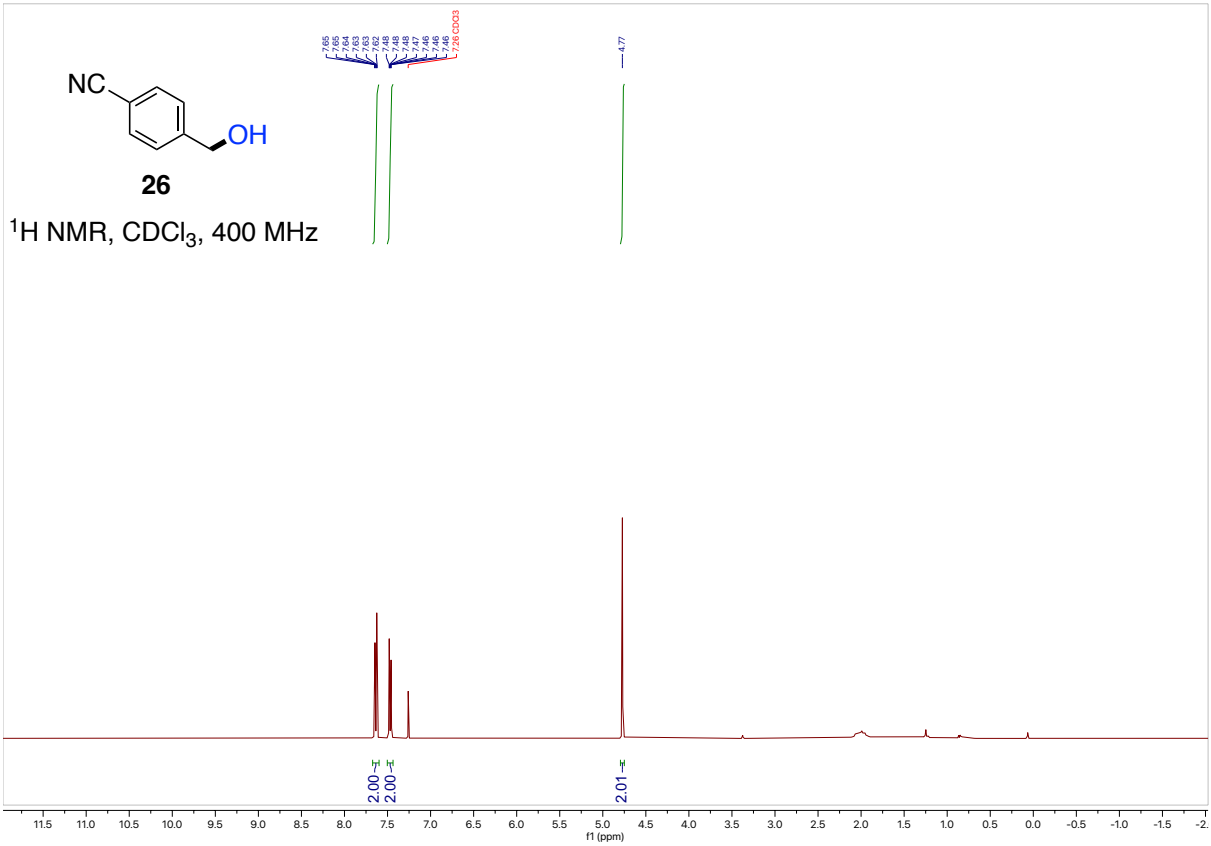
^{13}C NMR, $CDCl_3$, 101 MHz

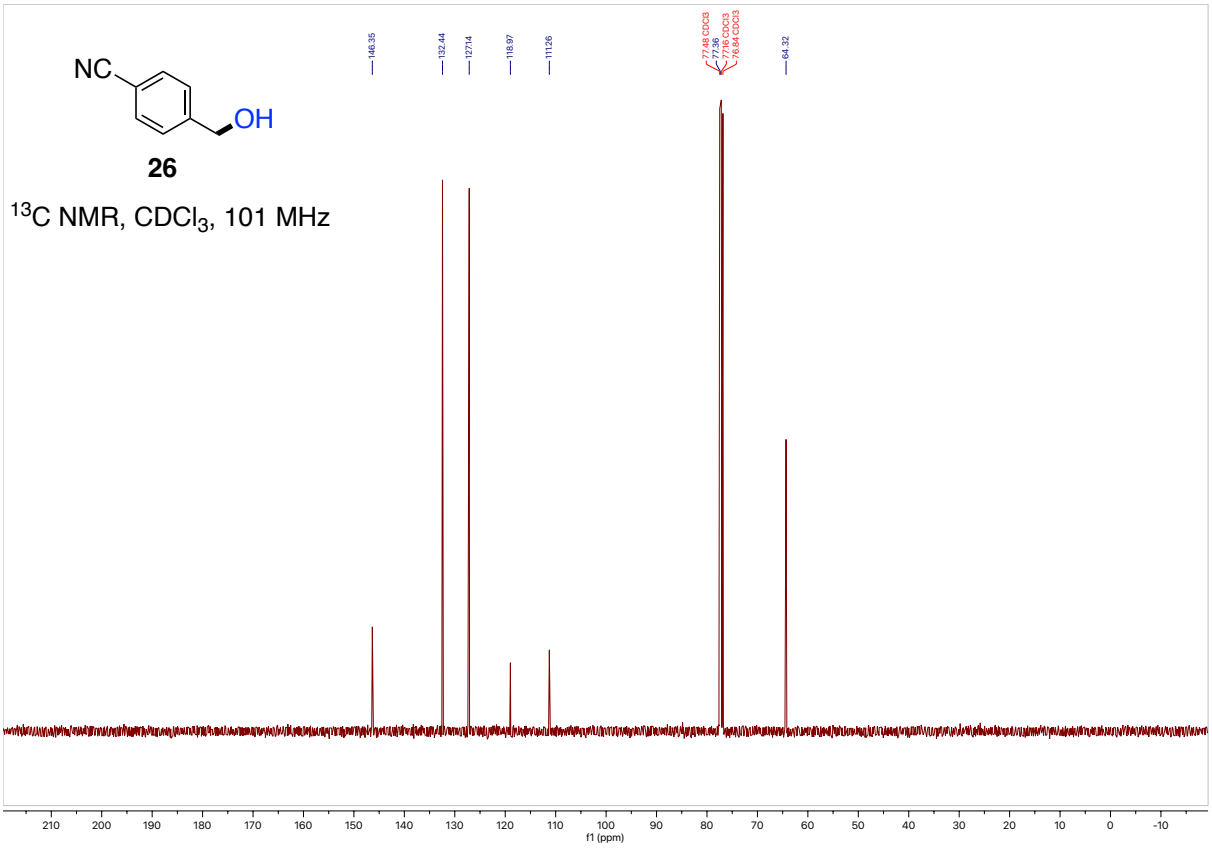

O2N
OH
24
1H NMR, CDCl3, 400 MHz
8.18
8.18
8.16
8.16
7.52
7.50
7.26 CDCl3
4.82
4.80
2.39
2.37
2.36
1.97
2.00
2.05
0.93
f1 (ppm)

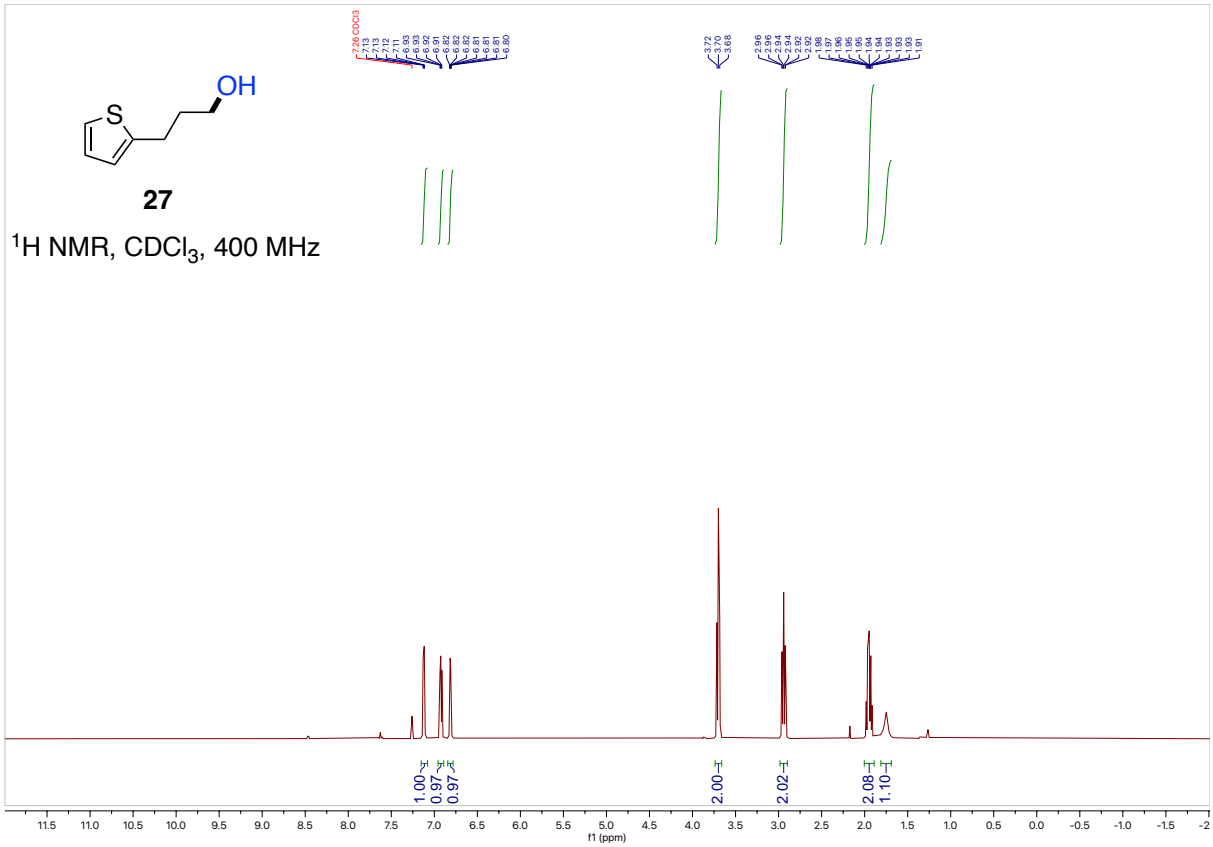

O2N
OH
24
13C NMR, CDCl3, 101 MHz
148.39
147.30
127.09
123.79
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
64.03
f1 (ppm)

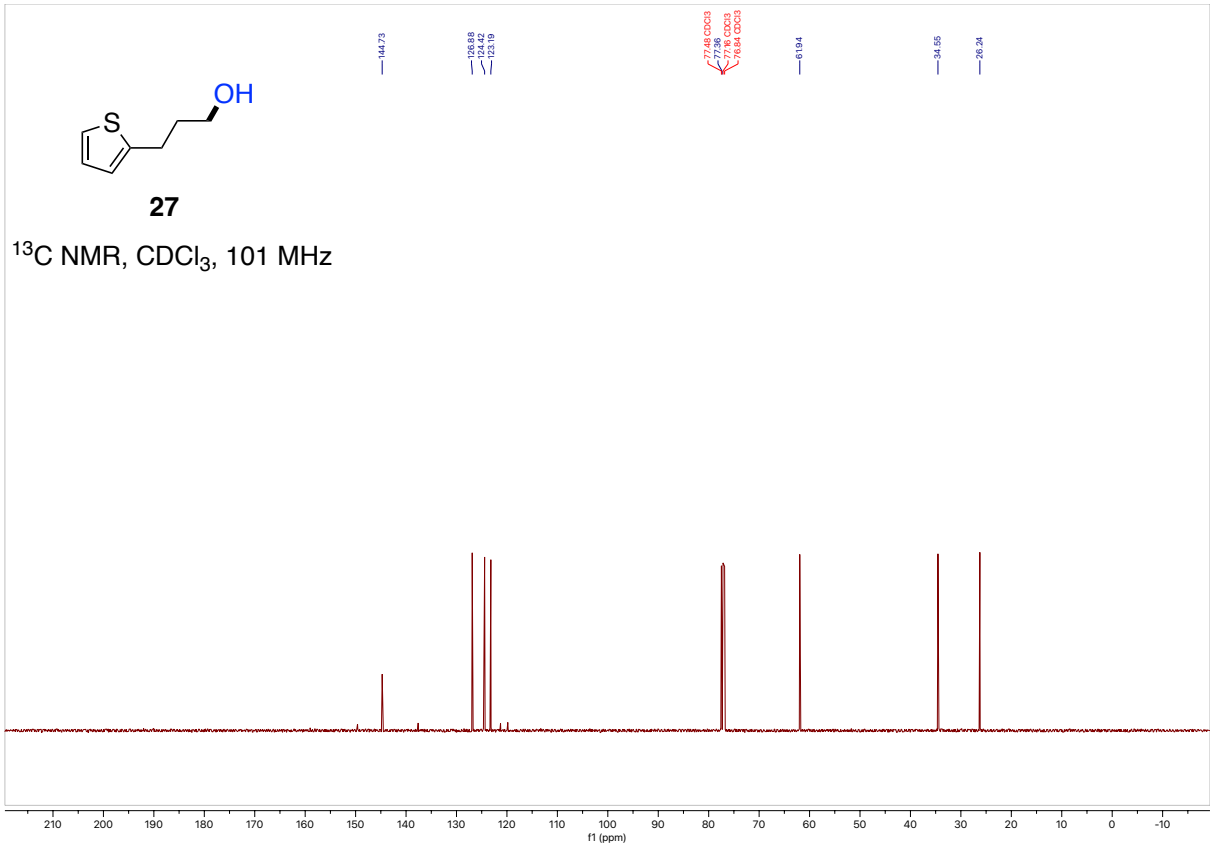

OH
25
1H NMR, CDCl3, 400 MHz
7.85
7.85
7.84
7.84
7.83
7.82
7.80
7.79
7.52
7.51
7.50
7.50
7.50
7.49
7.48
7.47
7.46
7.46
7.26 CDCl3
4.84
2.05
2.05
4.06
3.05
2.00
1.04
f1 (ppm)

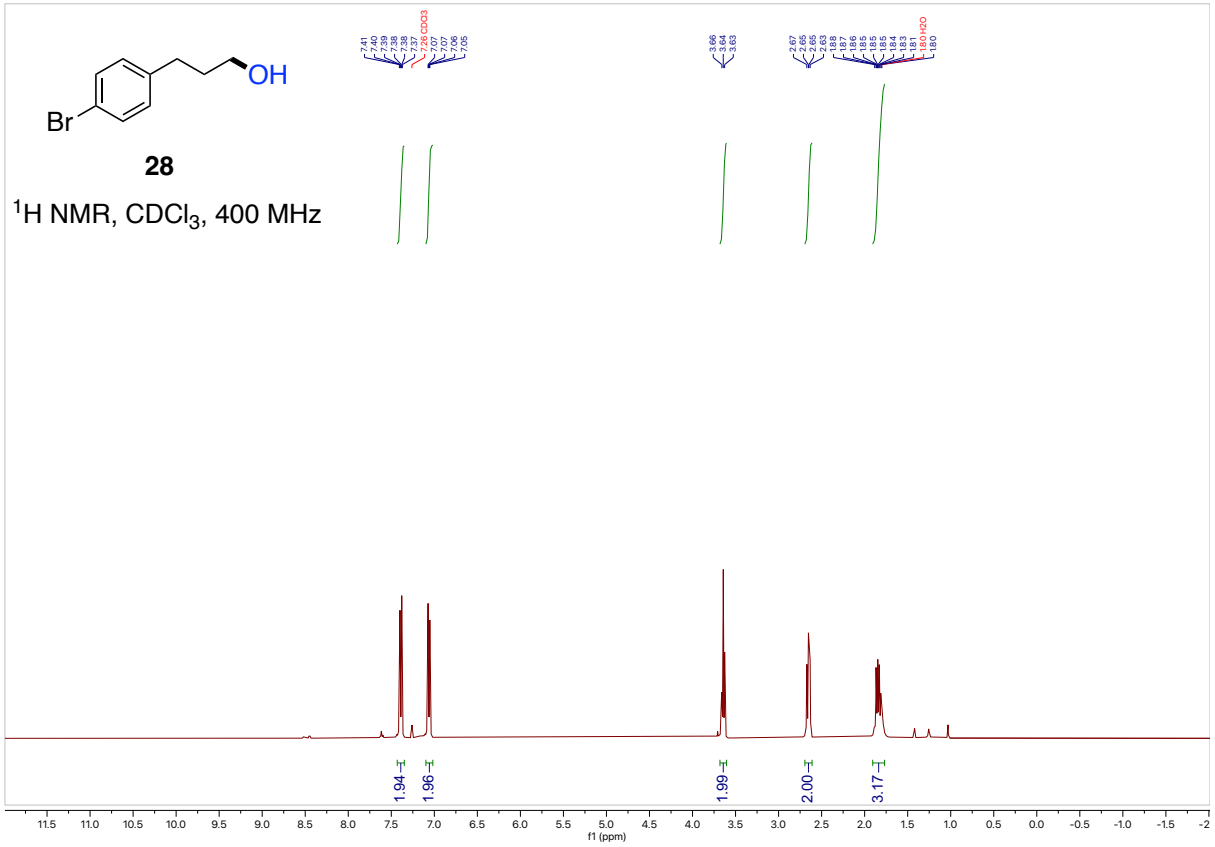

OH
25
13C NMR, CDCl3, 101 MHz
138.36
133.40
132.96
128.34
127.93
127.75
126.21
125.92
125.46
125.21
77.42 CDCl3
77.31
77.11 CDCl3
76.79 CDCl3
65.40
f1 (ppm)

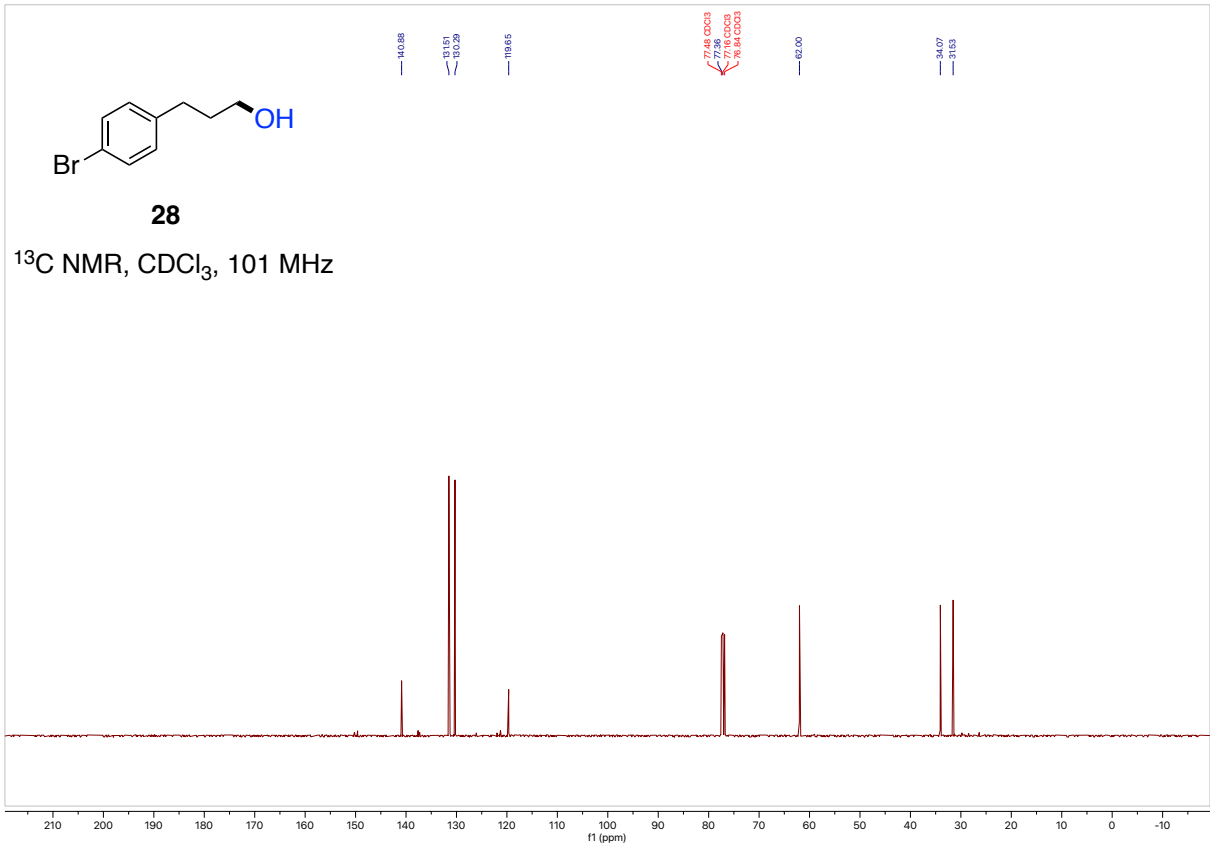

NC
OH
26
1H NMR, CDCl3, 400 MHz
7.65
7.65
7.64
7.63
7.63
7.62
7.48
7.48
7.48
7.47
7.46
7.46
7.46
7.26 CDCl3
4.77
2.00
2.00
2.01
f1 (ppm)

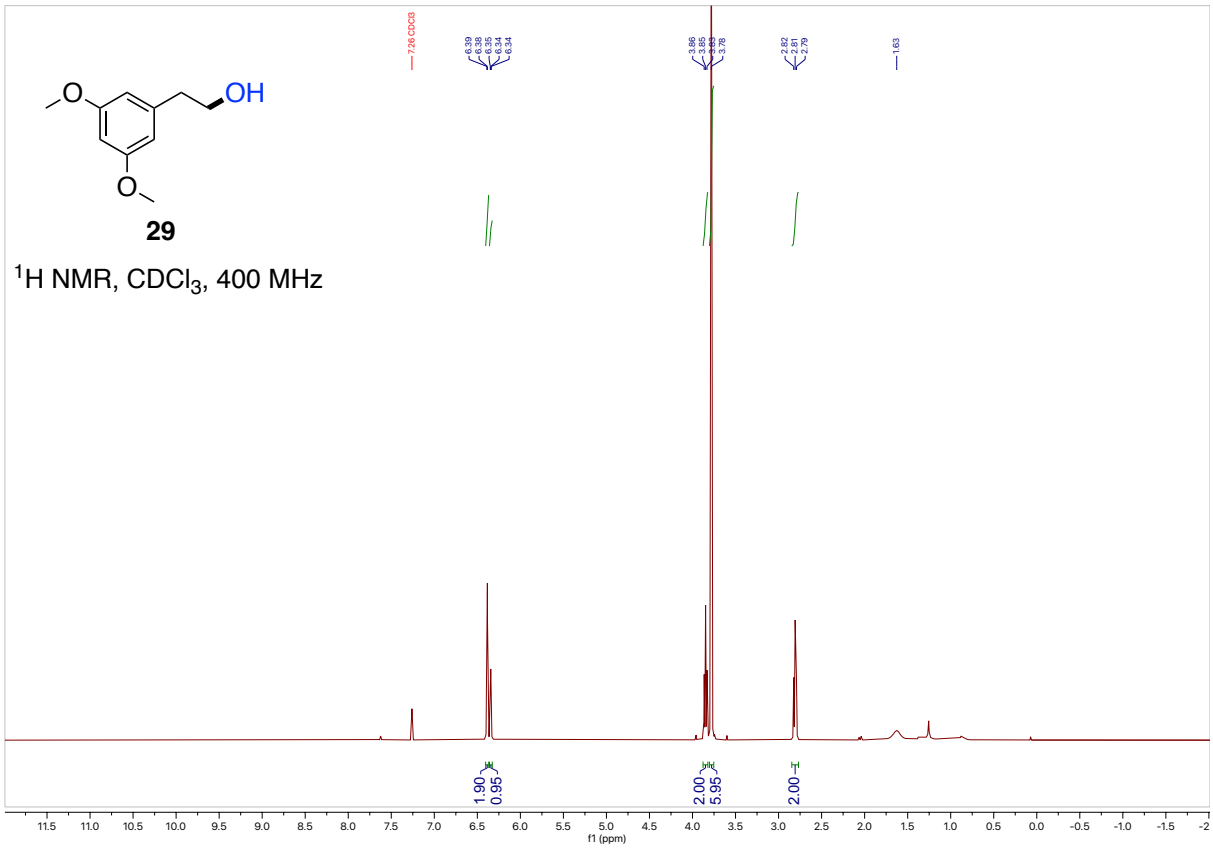

NC
OH
26
13C NMR, CDCl3, 101 MHz
146.35
132.44
127.14
118.97
111.26
77.48 CDCl3
77.16
77.16 CDCl3
76.84 CDCl3
64.32
f1 (ppm)

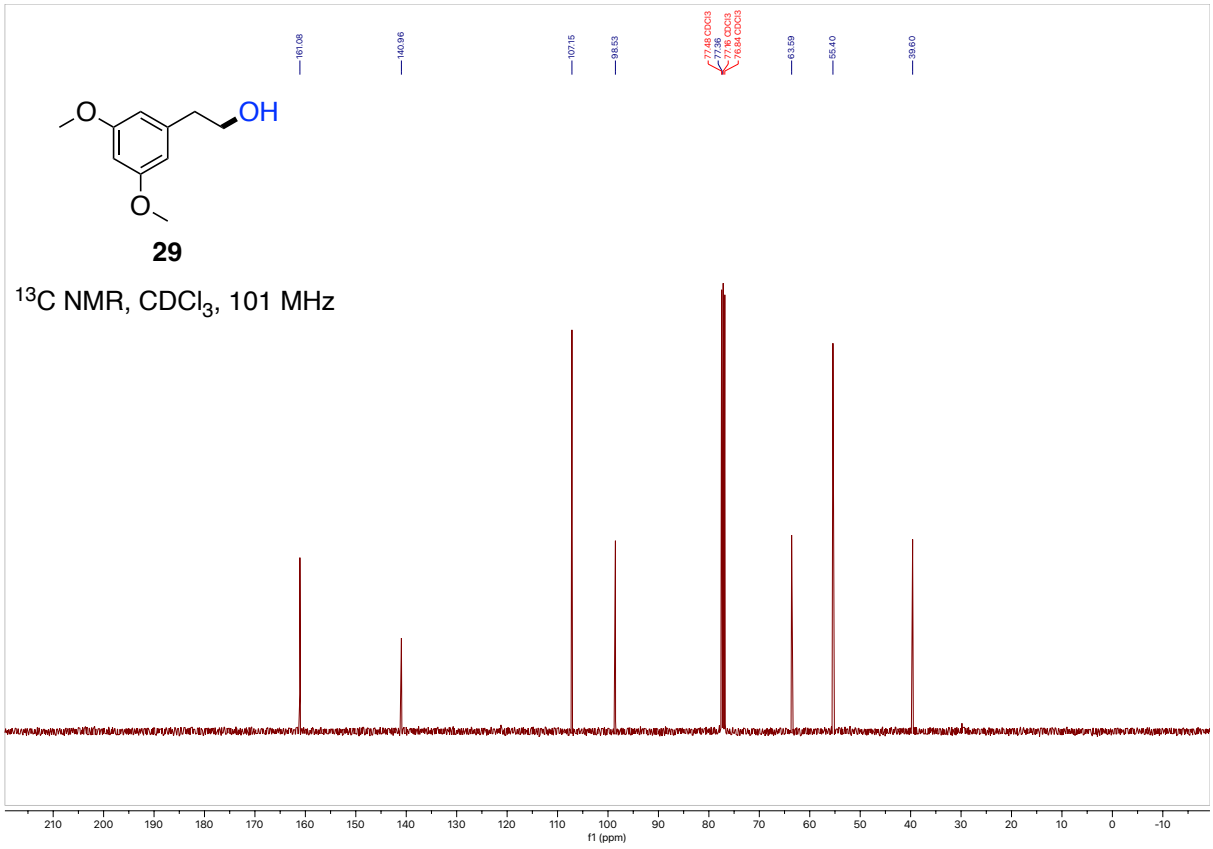

OH
S
27
1H NMR, CDCl3, 400 MHz
f1 (ppm)

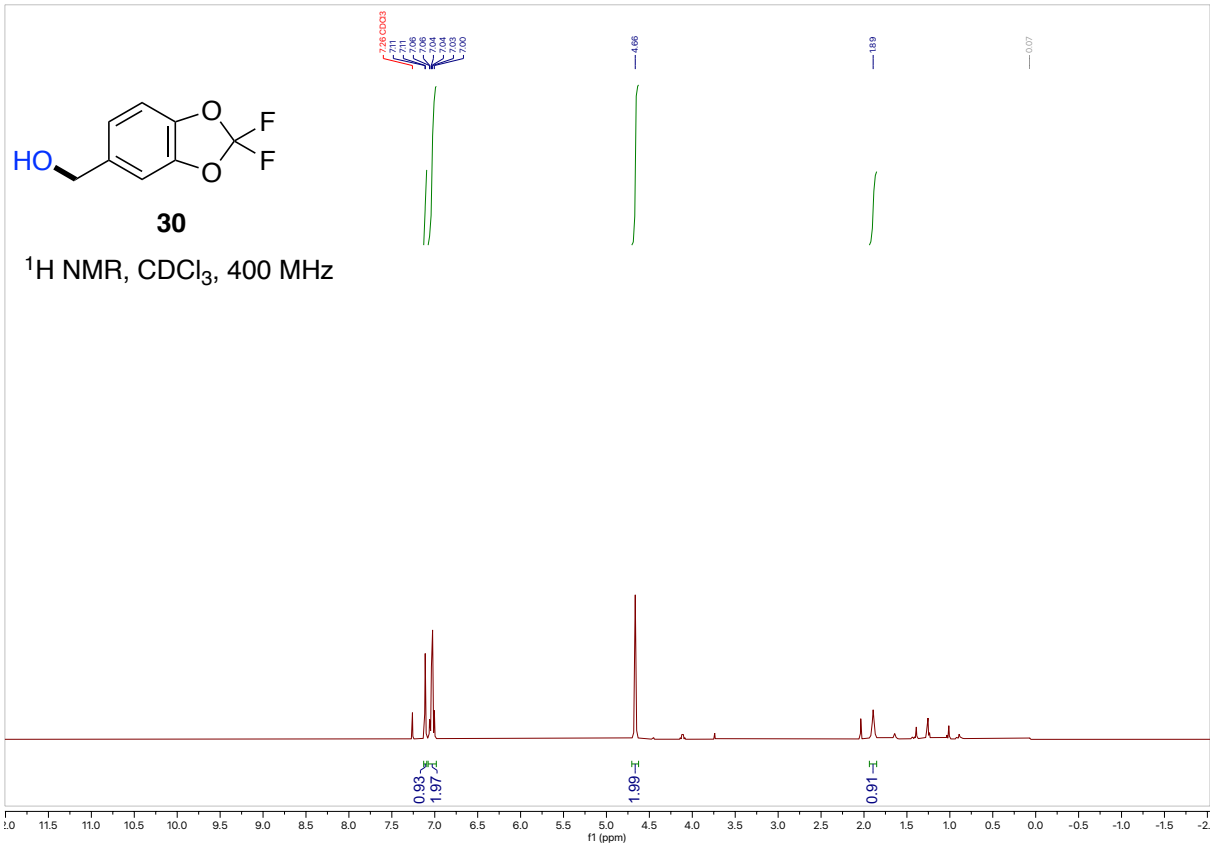

OH
S
27
13C NMR, CDCl3, 101 MHz
f1 (ppm)


OH
Br
28
1H NMR, CDCl3, 400 MHz
1.94
1.96
1.99
2.00
3.17
f1 (ppm)


OH
Br
28
13C NMR, CDCl3, 101 MHz
f1 (ppm)

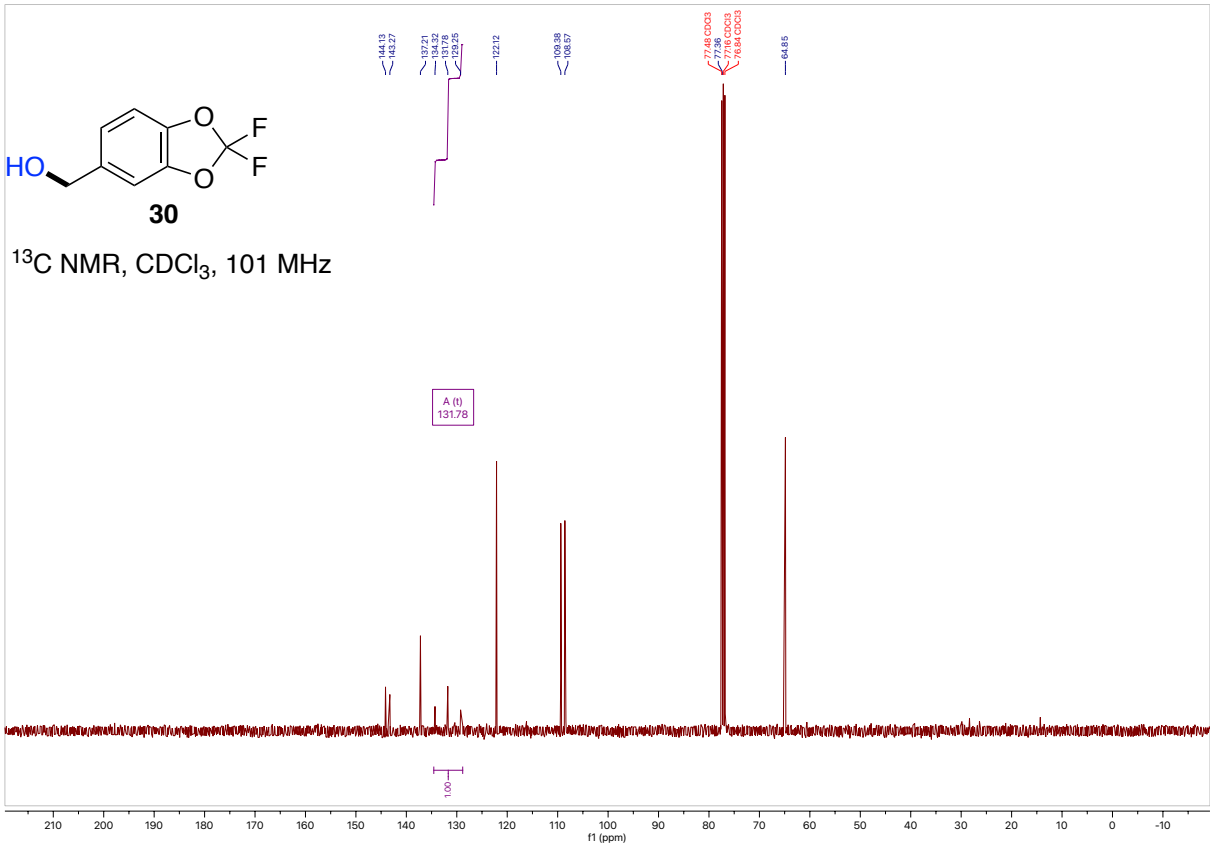

OH
O
O
29
^{1}H NMR, $CDCl_3$, 400 MHz
7.26 CDCl3
6.39
6.38
6.35
6.34
6.34
3.86
3.85
3.83
3.78
2.82
2.81
2.79
1.63
1.90
0.95
2.00
5.95
2.00
f1 (ppm)


OH
O
O
29
^{13}C NMR, $CDCl_3$, 101 MHz
161.08
140.96
107.15
98.53
77.48 CDCl3
77.26
77.05 CDCl3
76.84 CDCl3
63.59
55.40
39.60
f1 (ppm)



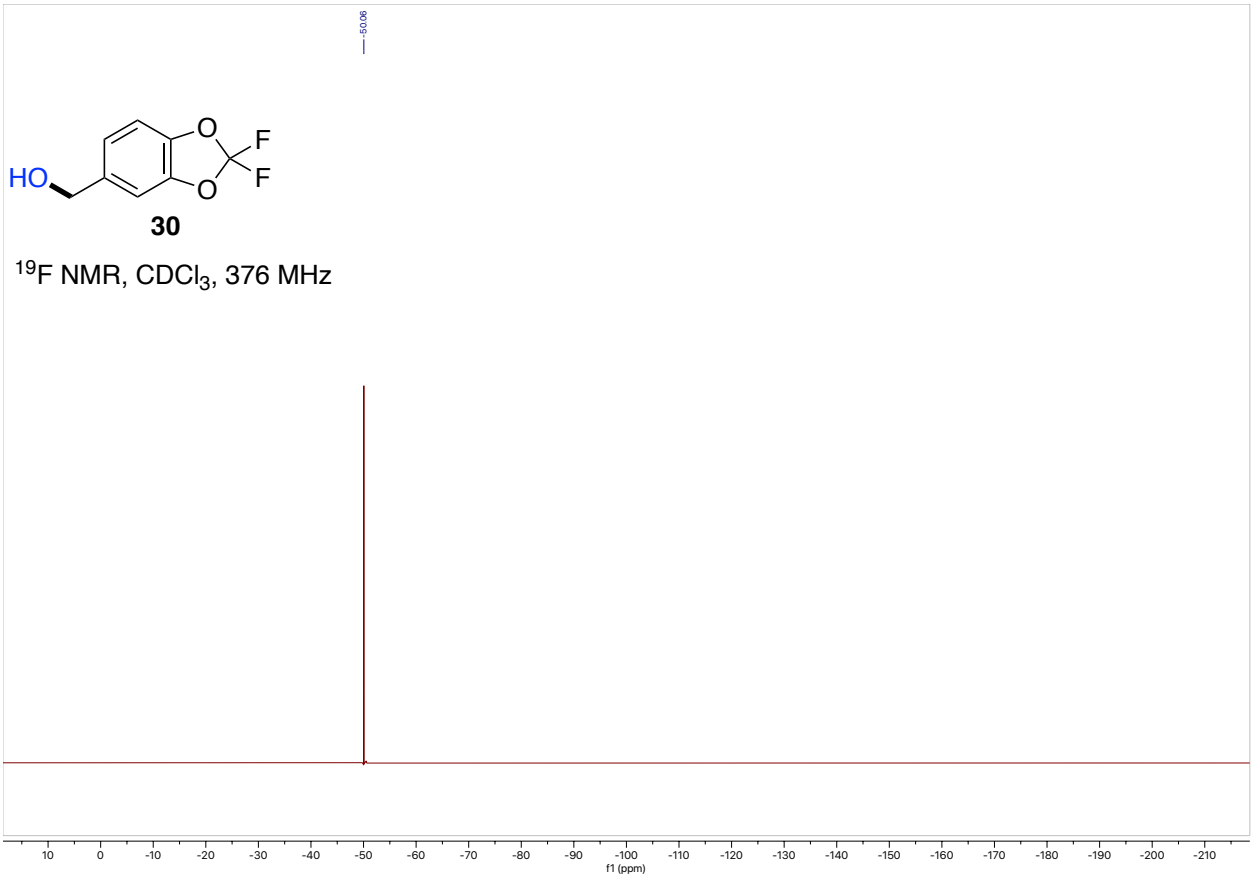
30

^{1}H NMR, $CDCl_3$, 400 MHz



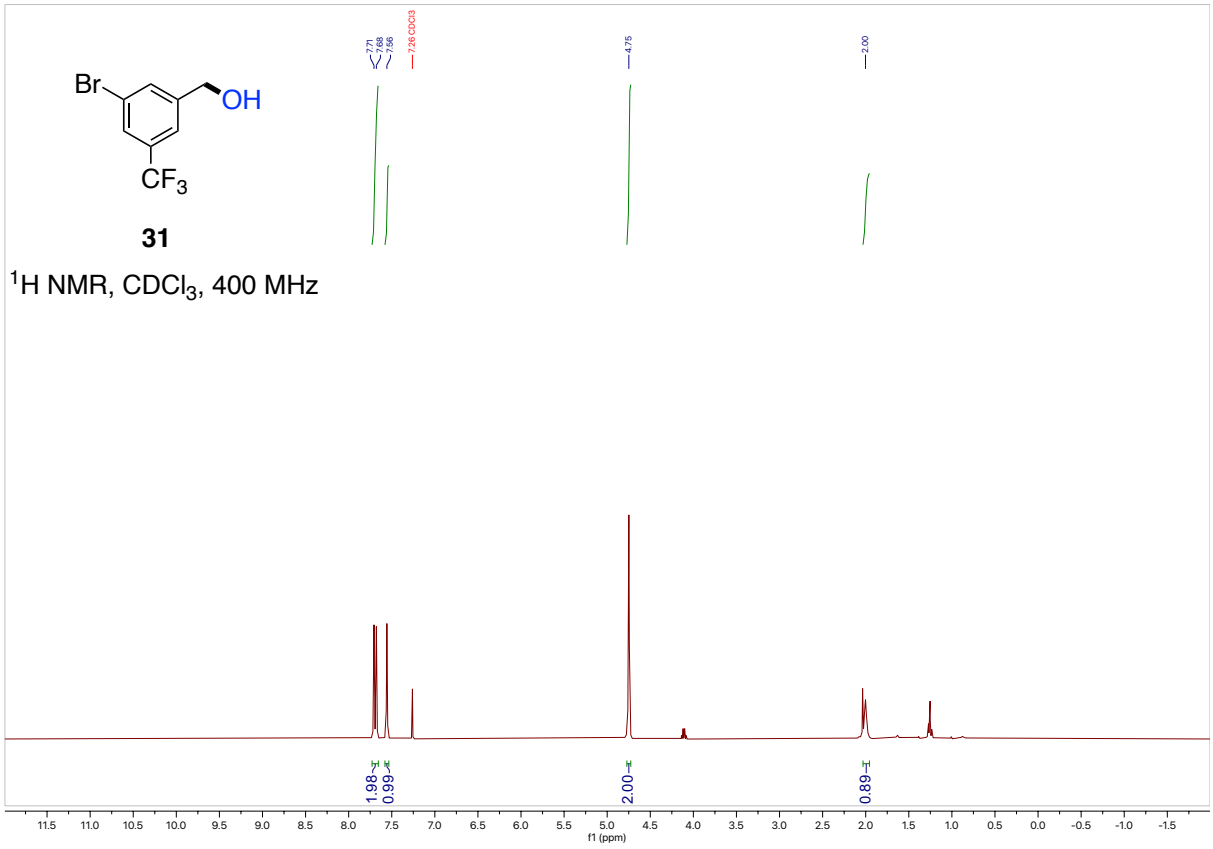
30

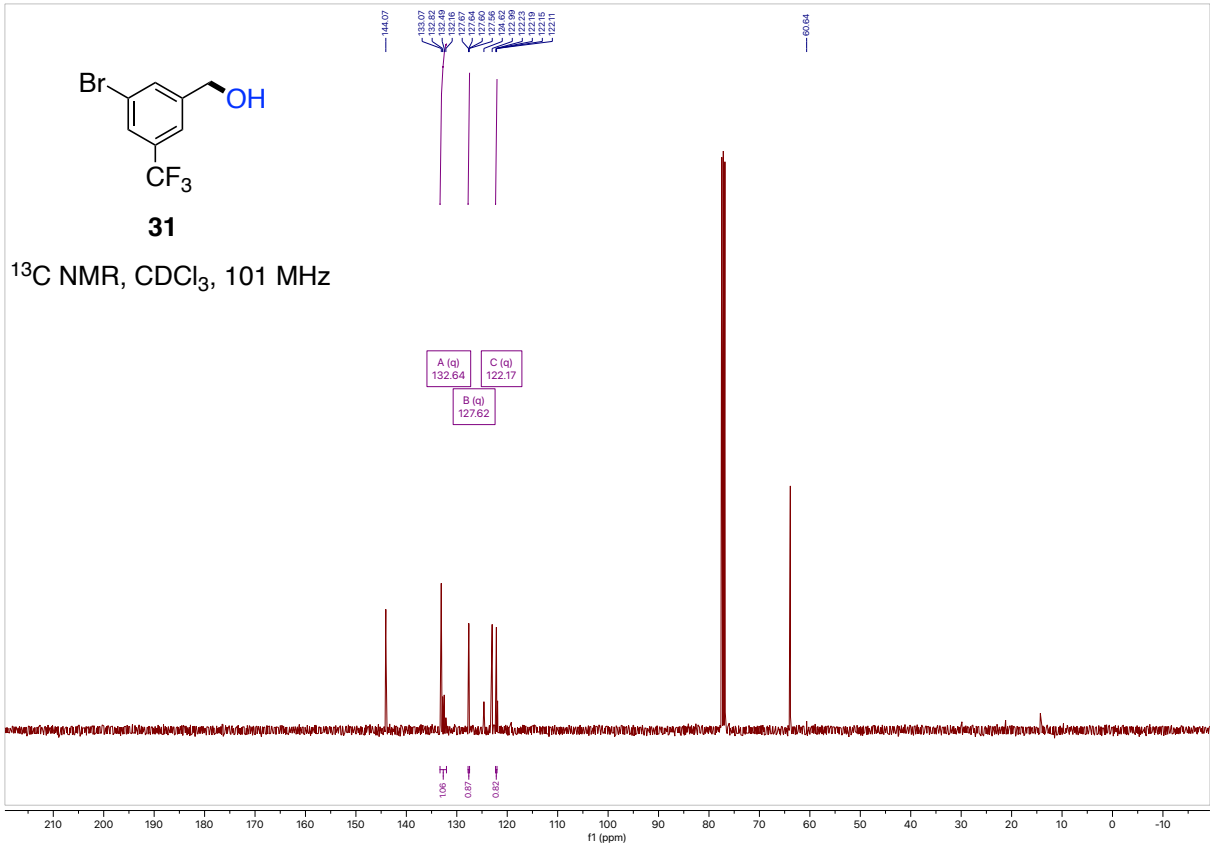
^{13}C NMR, $CDCl_3$, 101 MHz

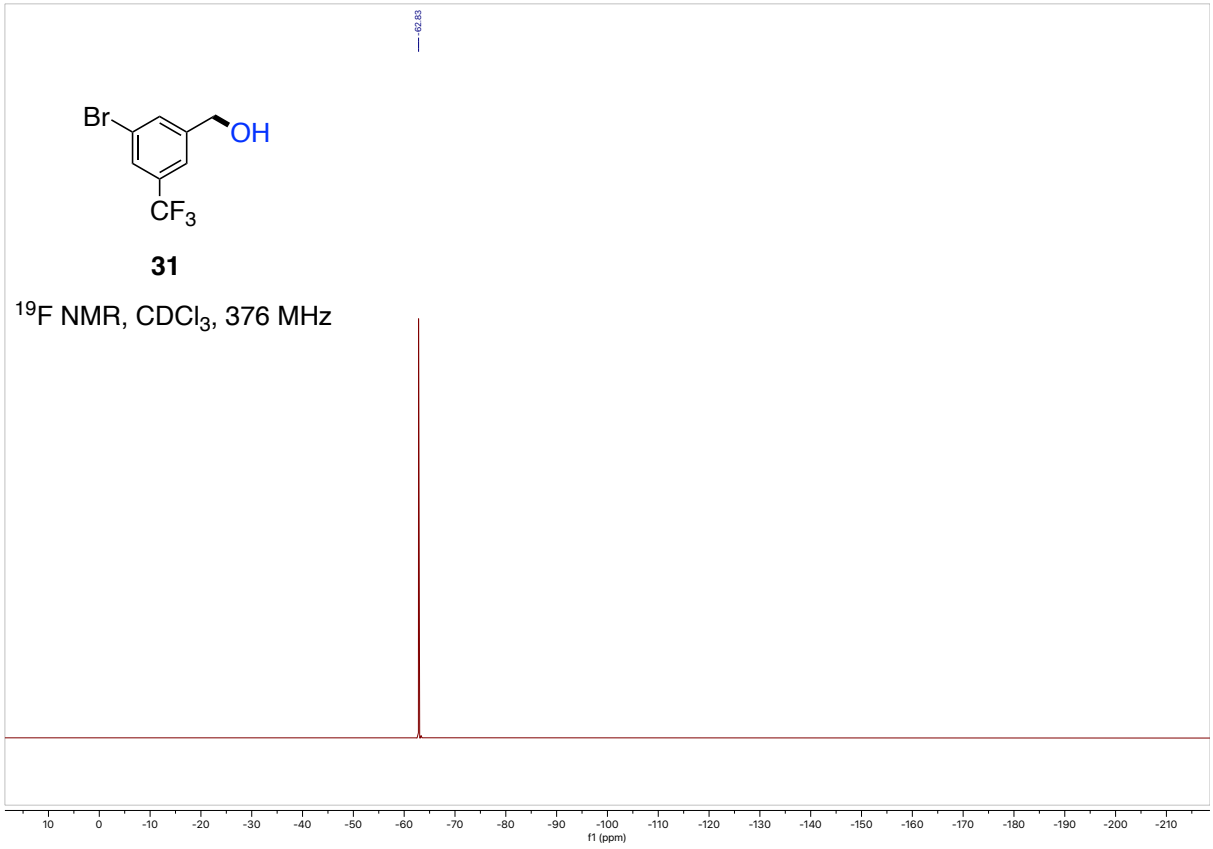


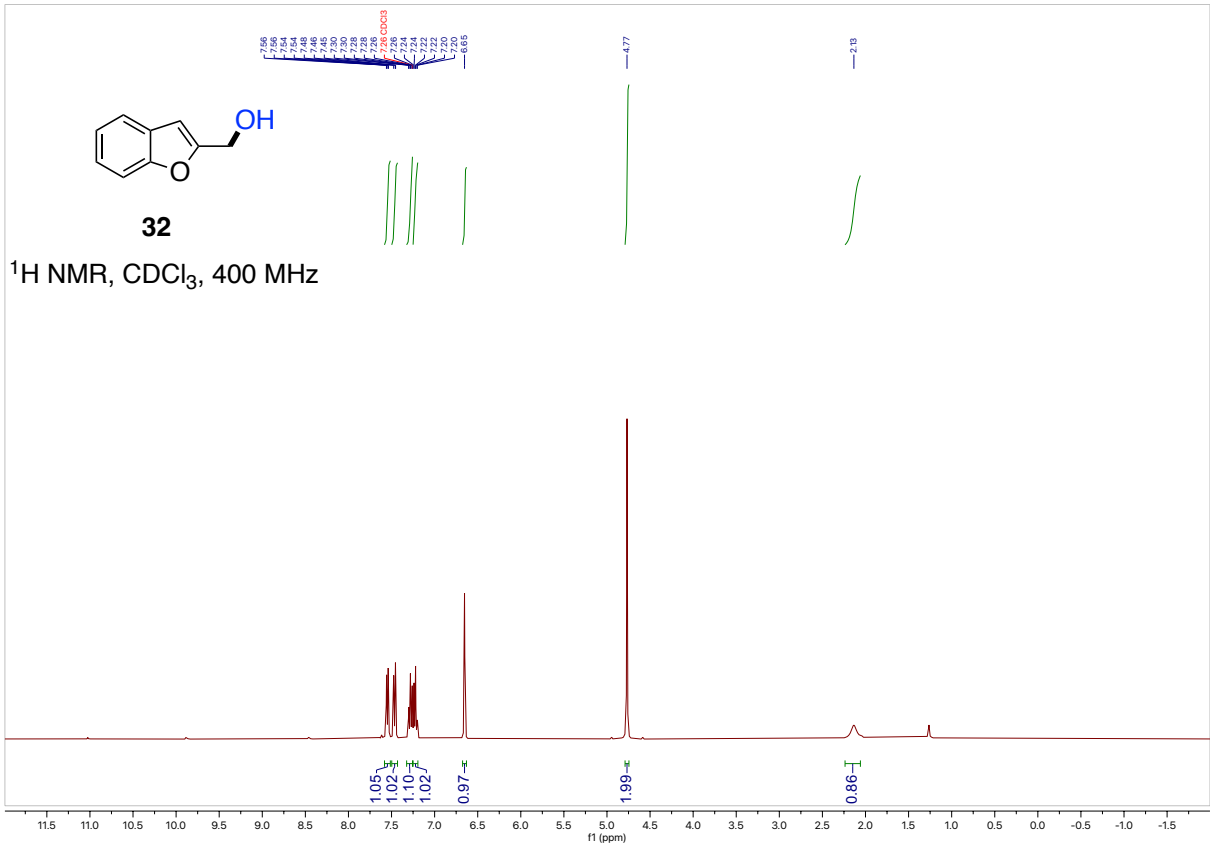
30

^{19}F NMR, $CDCl_3$, 376 MHz

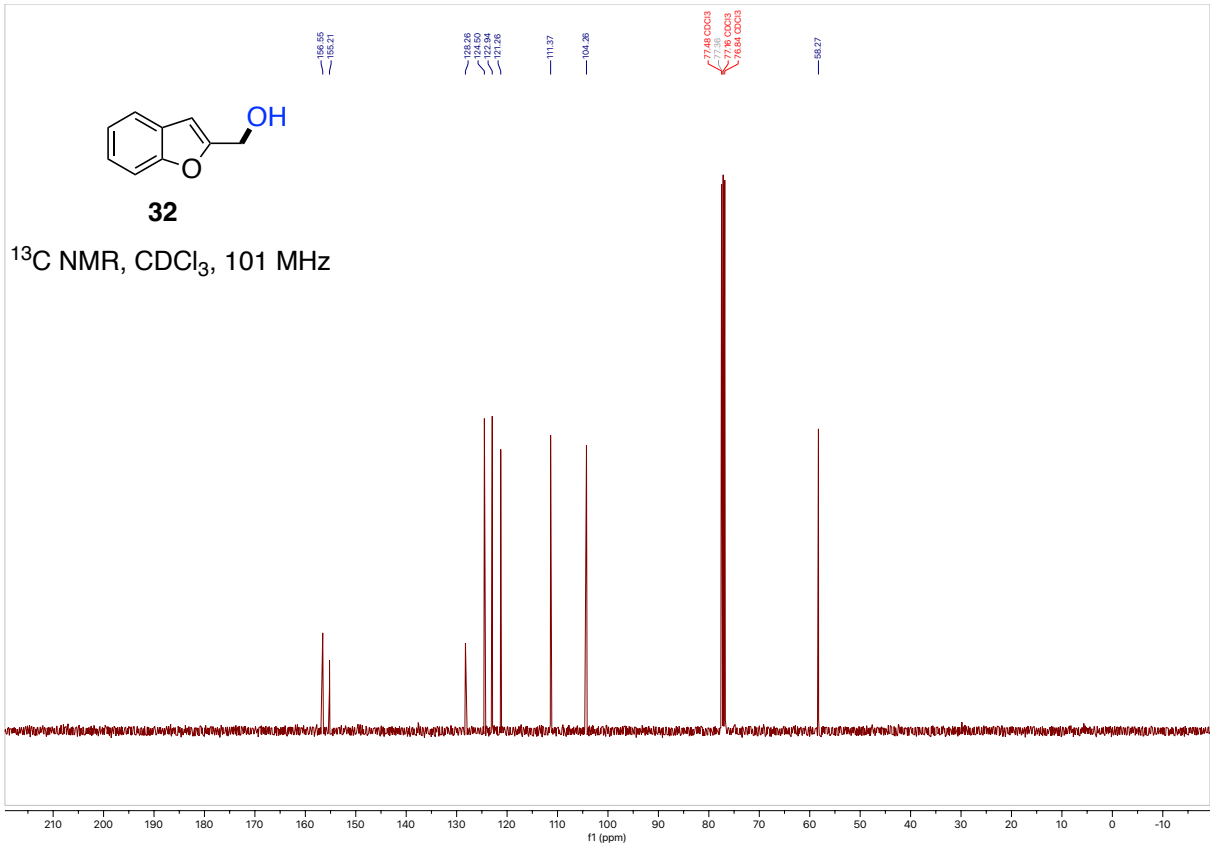

Br
OH
CF3
31
1H NMR, CDCl3, 400 MHz
7.71
7.68
7.56
7.26 CDCl3
4.75
2.00
1.98
0.99
2.00
0.89
f1 (ppm)


Br
OH
CF3
31
13C NMR, CDCl3, 101 MHz
144.07
133.07
132.82
132.49
132.16
127.62
127.64
127.60
127.56
124.62
122.99
122.23
122.19
122.15
122.11
60.64
A (q)
132.64
C (q)
122.17
B (q)
127.62
1.06
0.87
0.82
f1 (ppm)

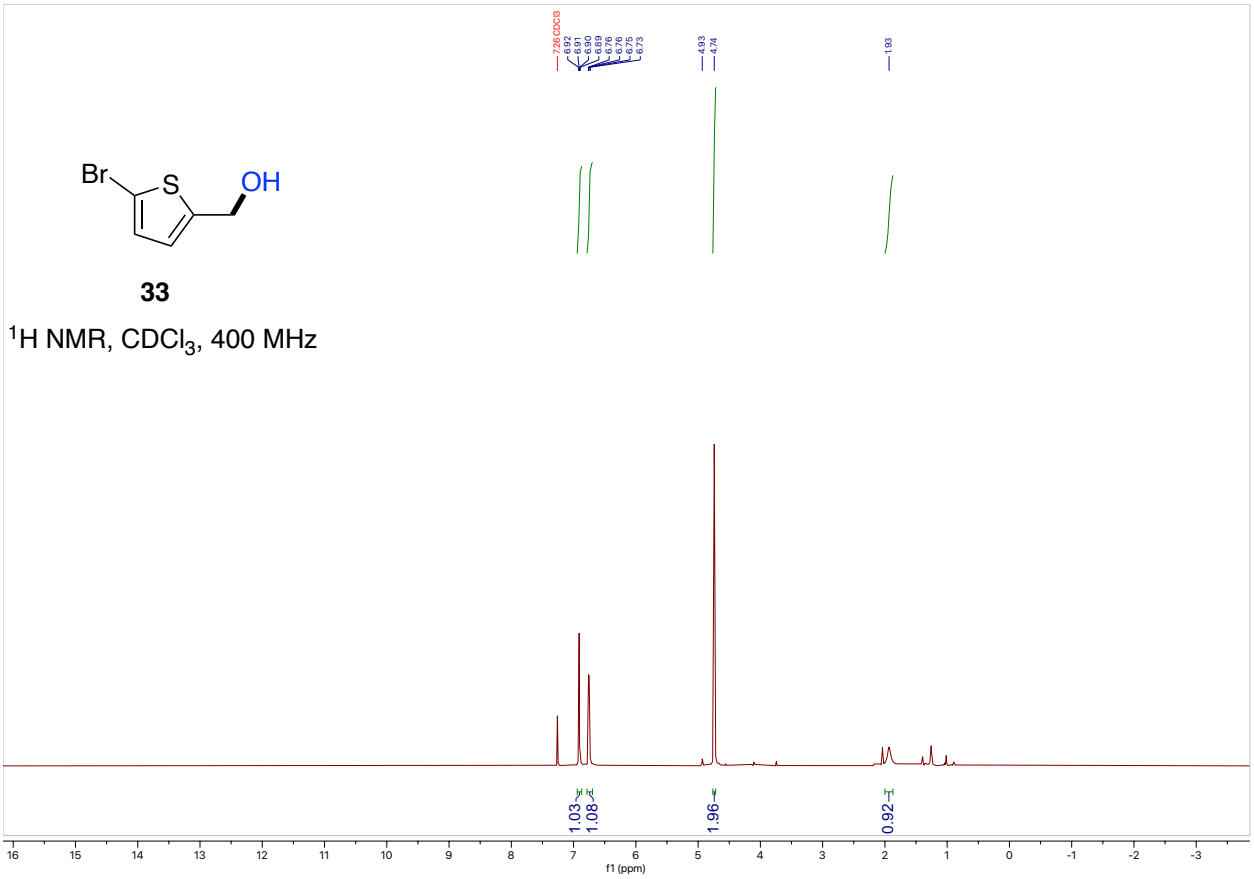

-62.83
Br
OH
CF3
31
19F NMR, CDCl3, 376 MHz
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)



^{1}H NMR, $CDCl_3$, 400 MHz

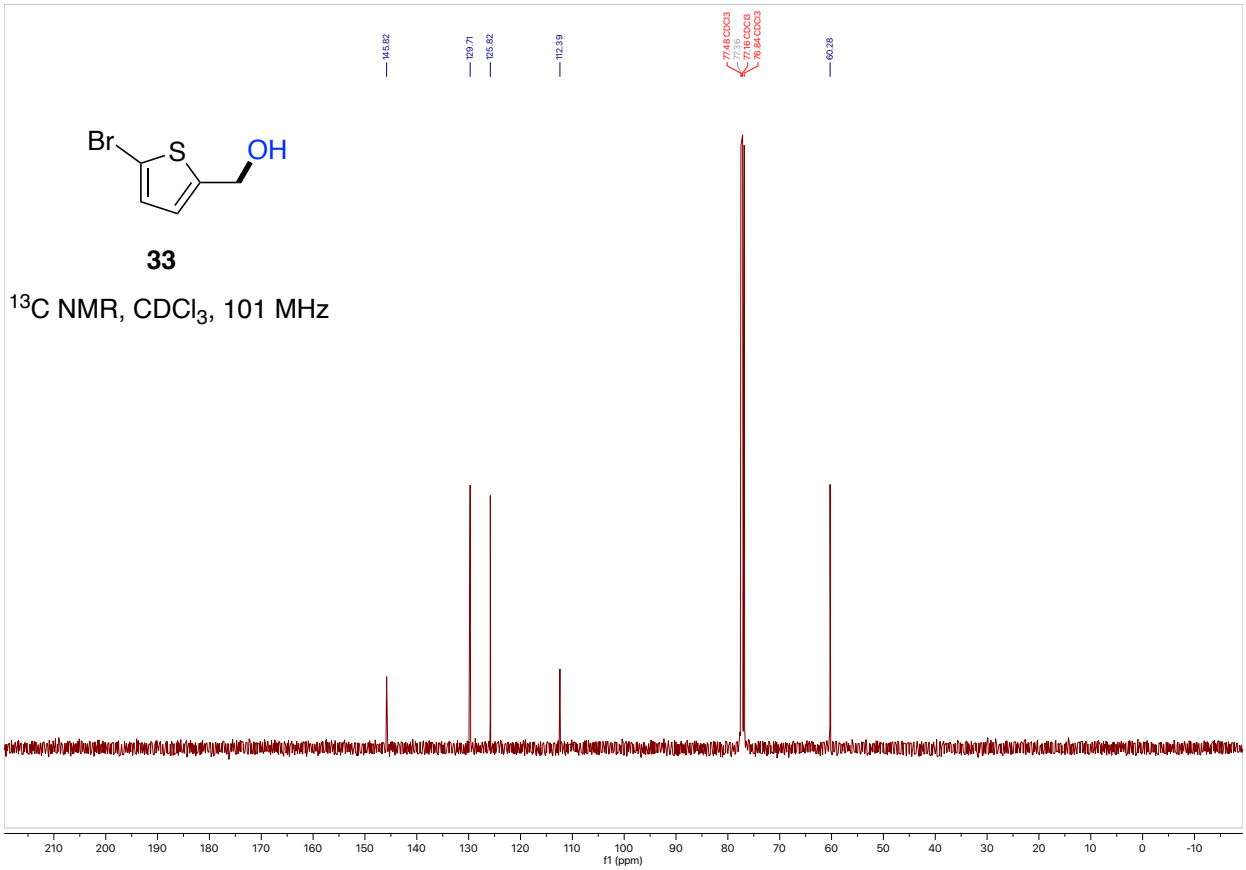


^{13}C NMR, $CDCl_3$, 101 MHz



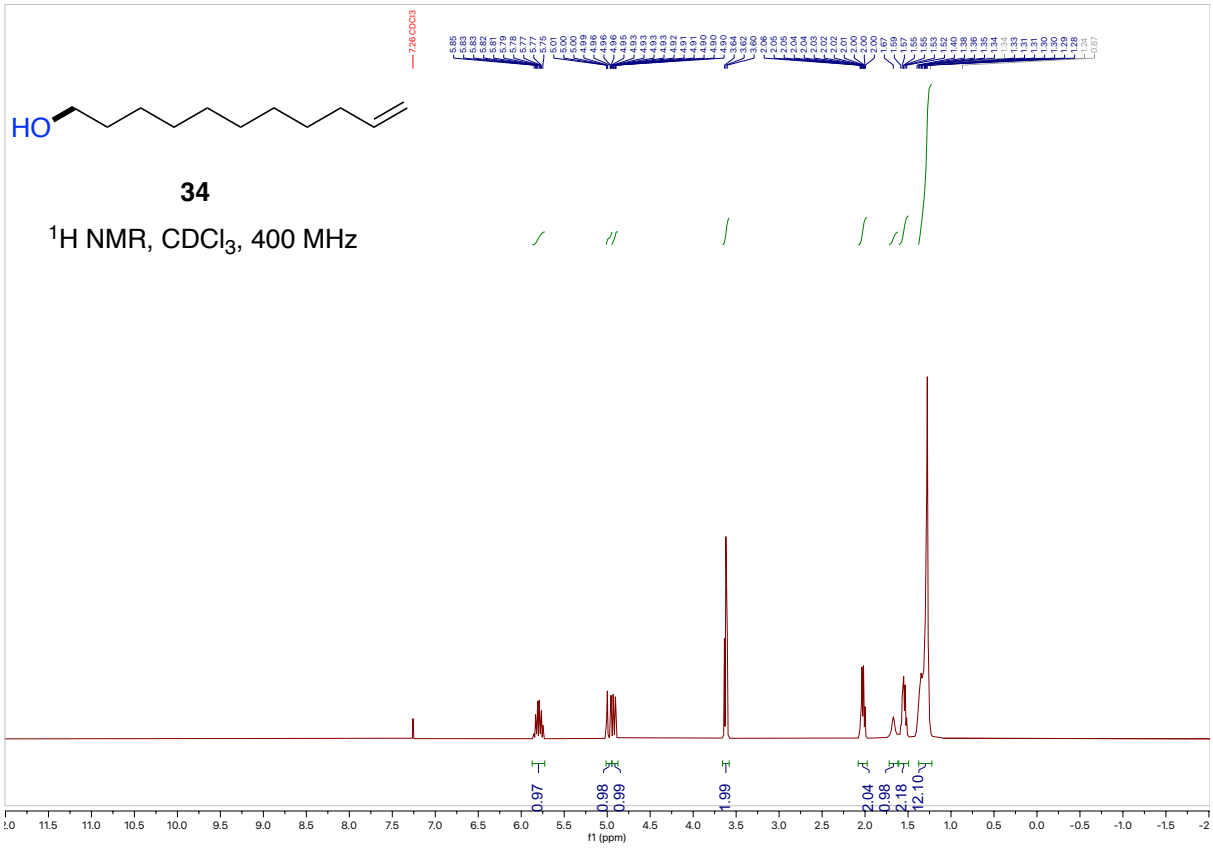
33

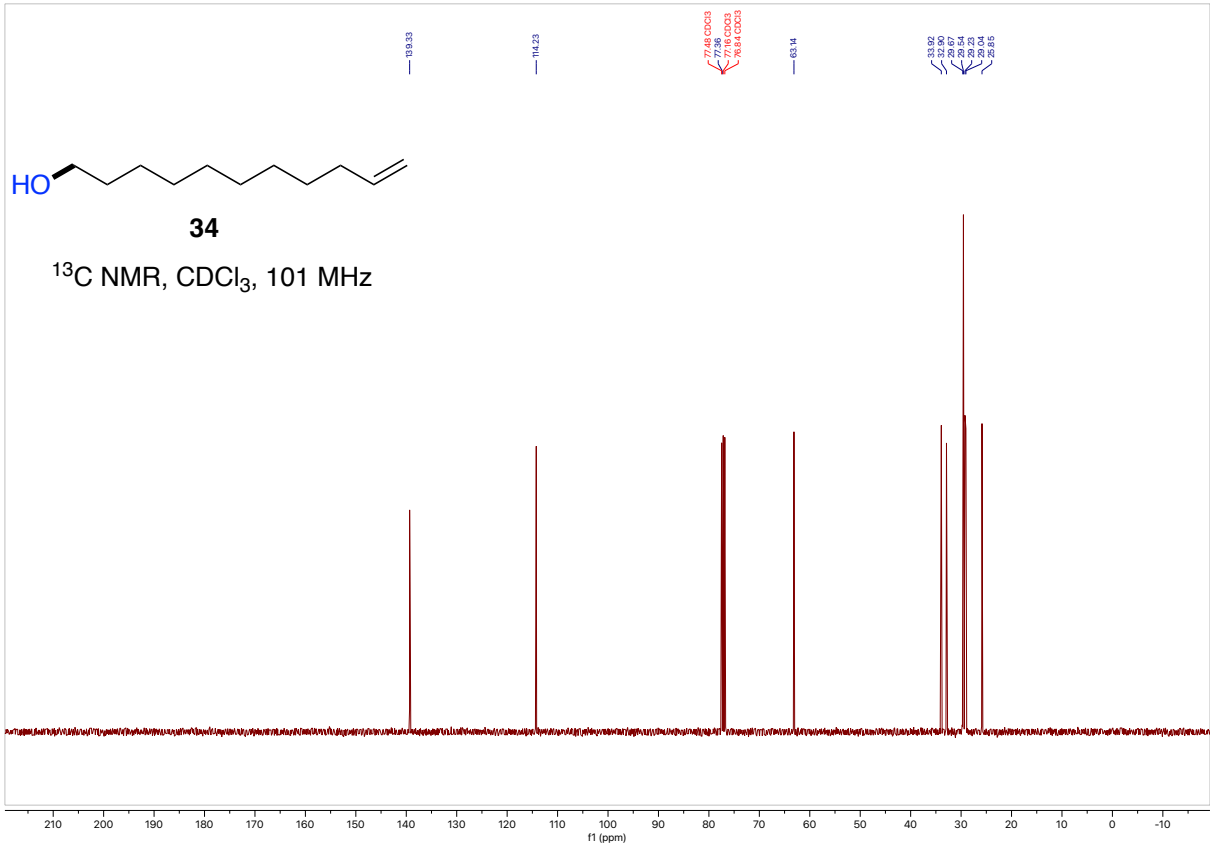
^{1}H NMR, $CDCl_3$, 400 MHz

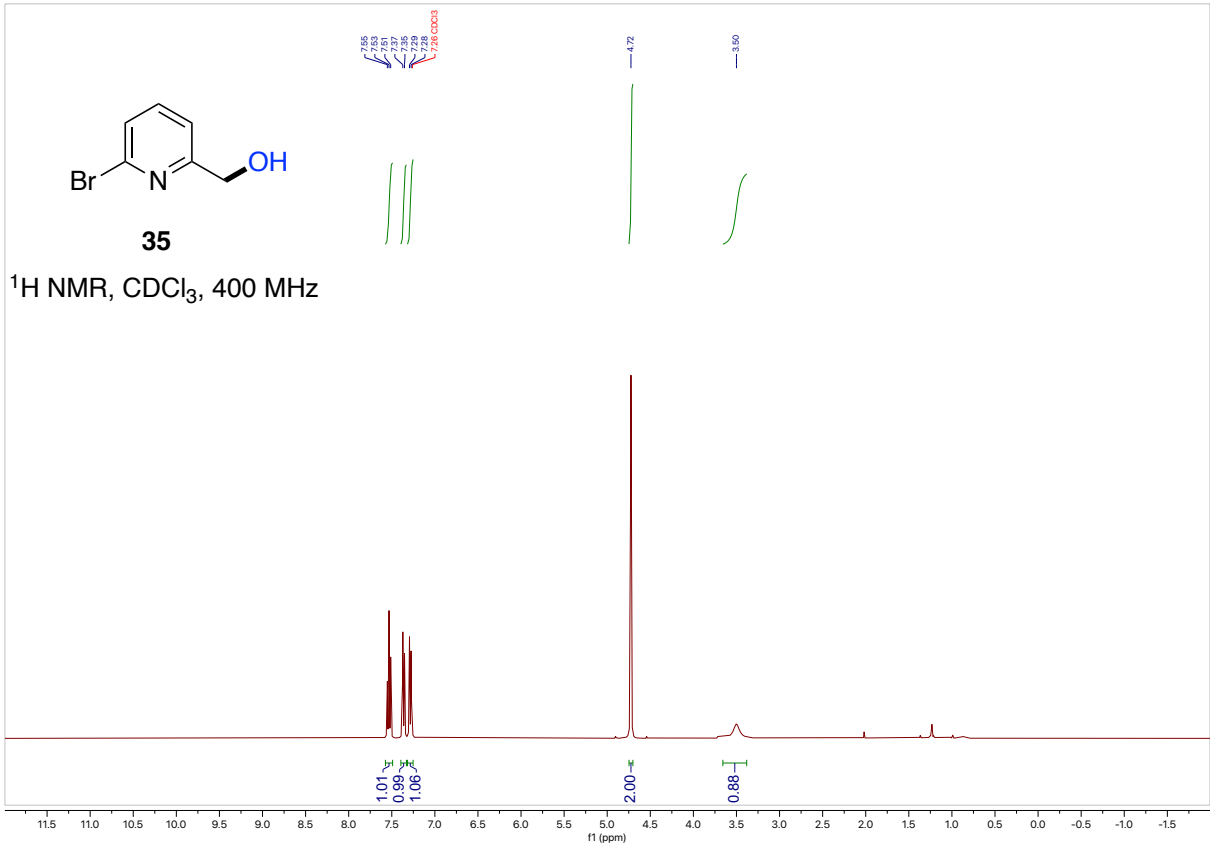


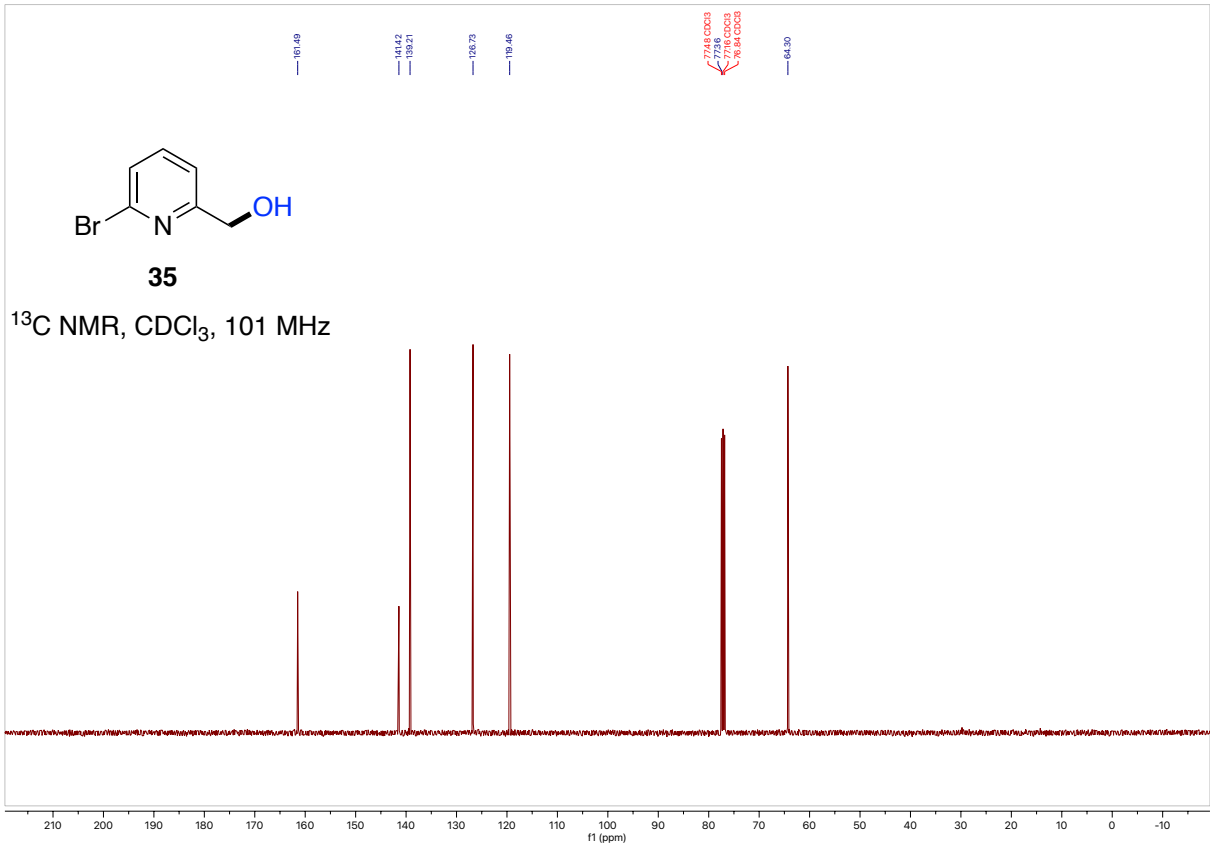
33

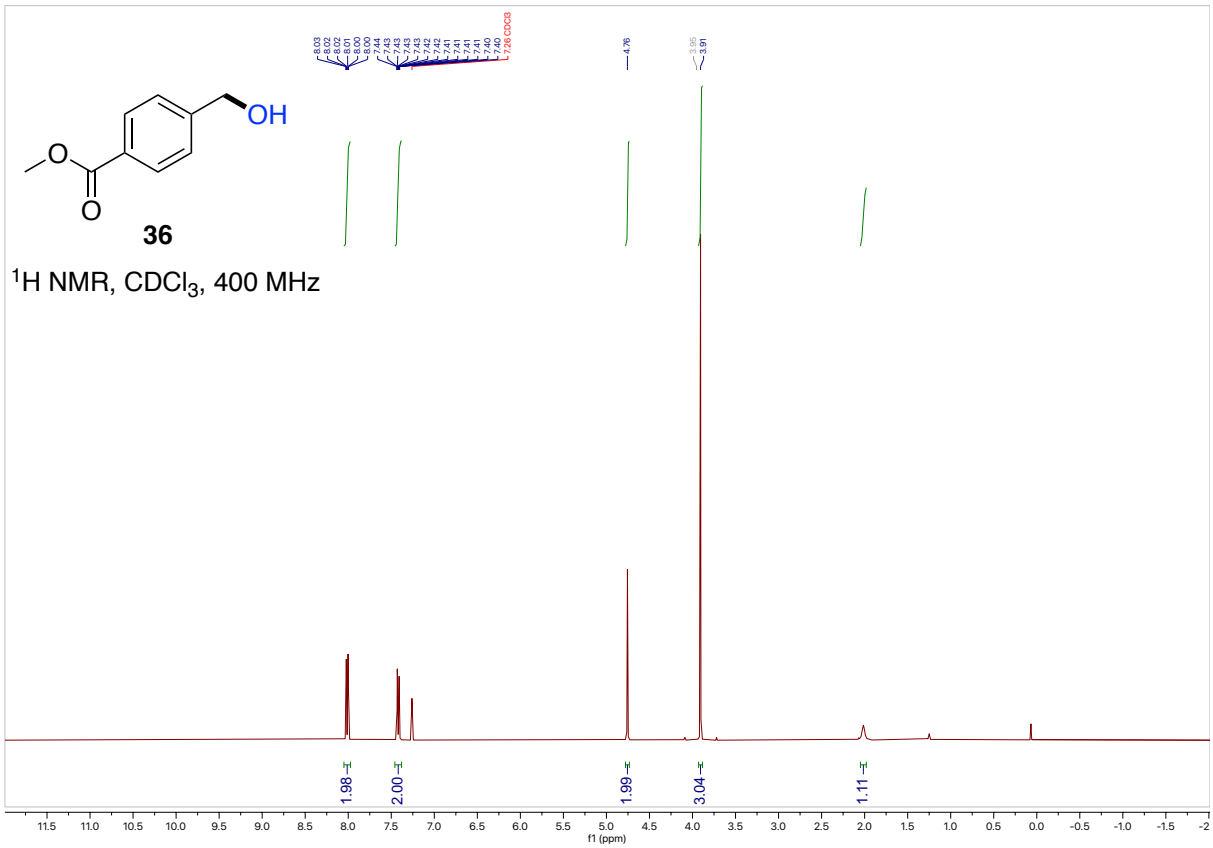
^{13}C NMR, $CDCl_3$, 101 MHz

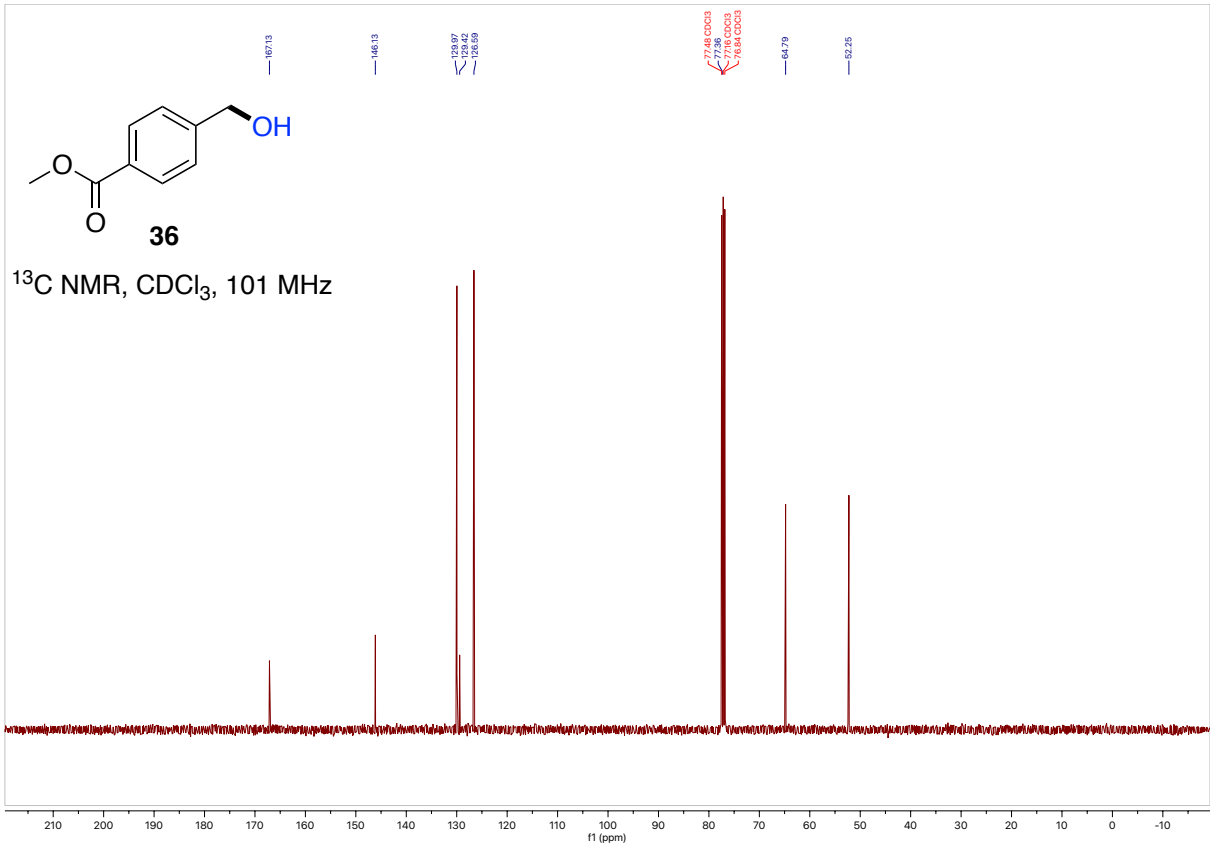

HO
34
^{1}H NMR, $CDCl_3$, 400 MHz
f1 (ppm)

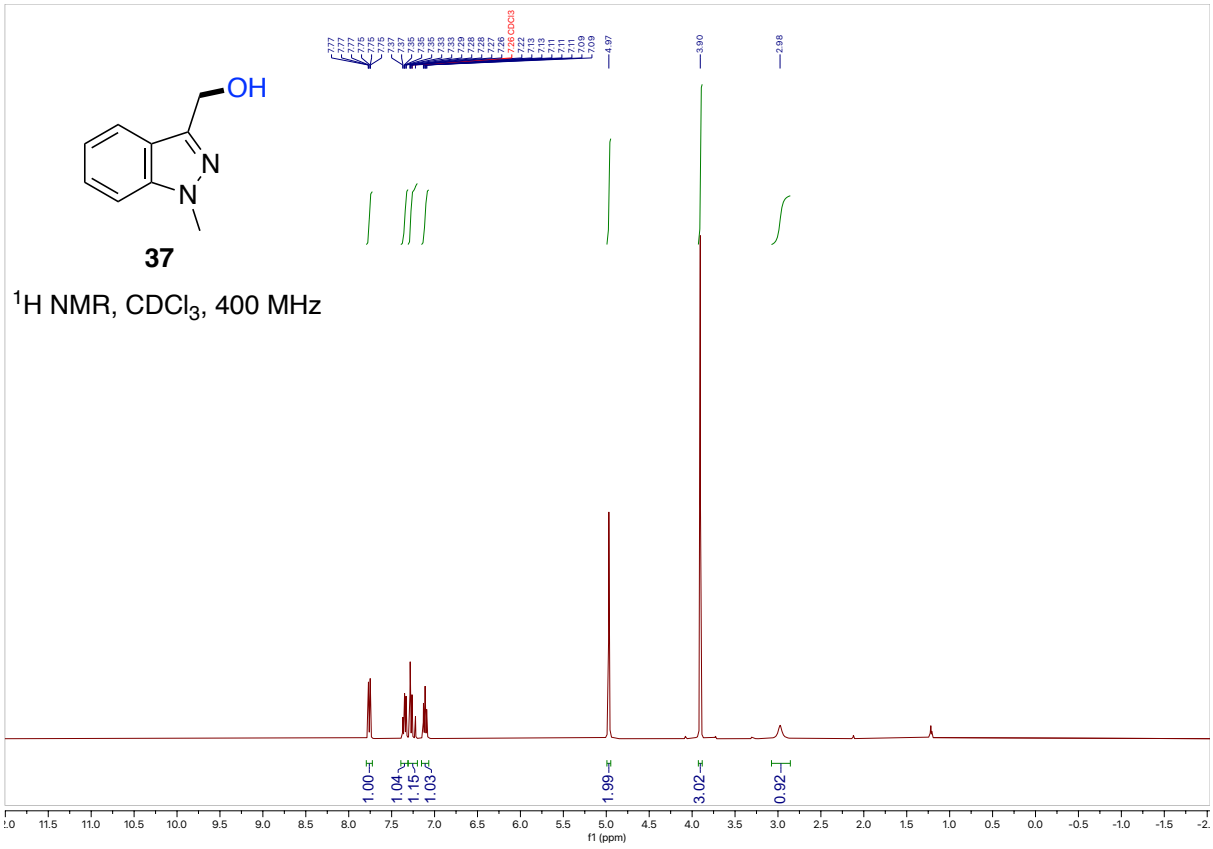

HO
34
^{13}C NMR, $CDCl_3$, 101 MHz
f1 (ppm)


Br
N
OH
35
1H NMR, CDCl3, 400 MHz
7.55
7.53
7.51
7.37
7.35
7.29
7.28
7.26 CDCl3
4.72
3.50
1.01
0.99
1.06
2.00
0.88
f1 (ppm)


Br
N
OH
35
13C NMR, CDCl3, 101 MHz
161.49
141.42
139.21
126.73
119.46
77.48 CDCl3
77.16
76.84 CDCl3
64.30
f1 (ppm)

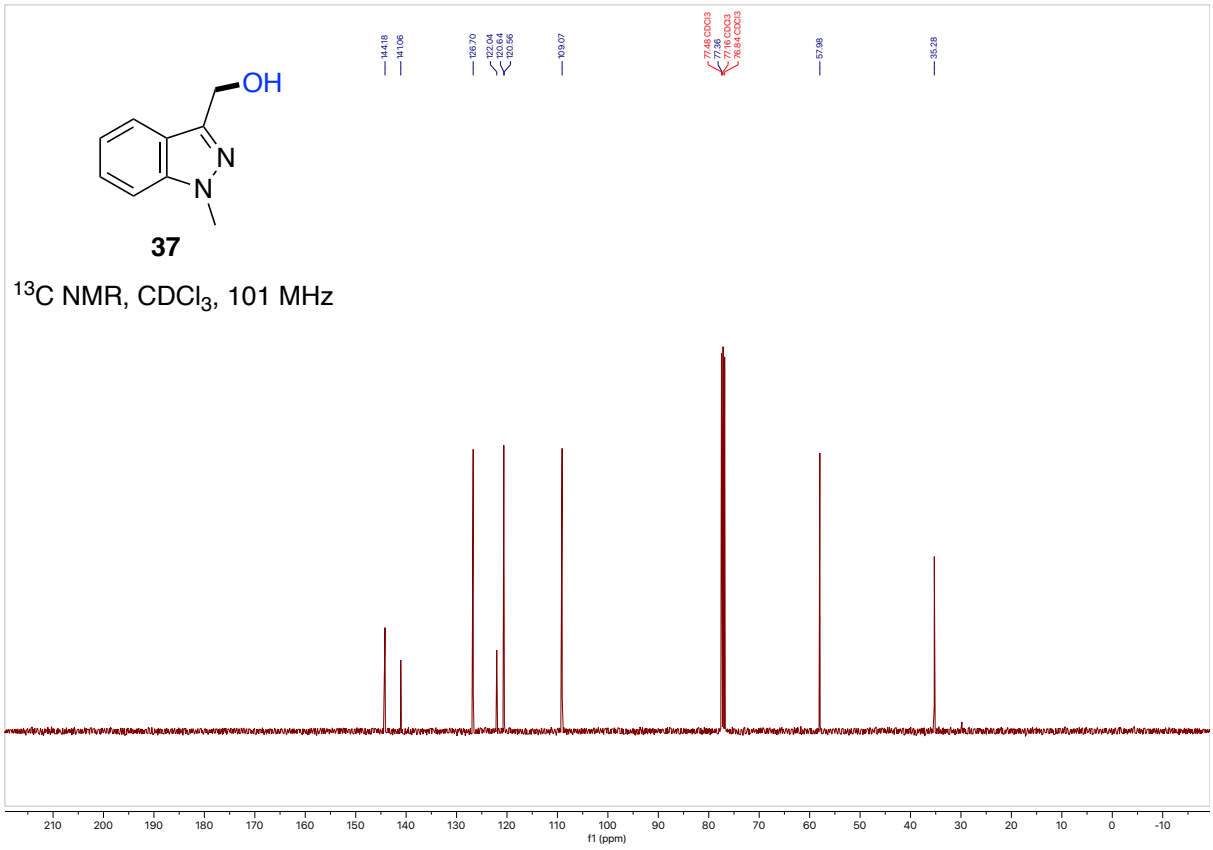

OH
O
O
36
1H NMR, CDCl3, 400 MHz
8.03
8.02
8.02
8.01
8.00
8.00
7.44
7.43
7.43
7.43
7.43
7.42
7.42
7.41
7.41
7.41
7.40
7.40
7.26 CDCl3
4.76
3.95
3.91
1.98
2.00
1.99
3.04
1.11
f1 (ppm)


OH
O
O
36
13C NMR, CDCl3, 101 MHz
167.13
146.13
129.97
128.42
126.59
77.48 CDCl3
77.36
77.16 CDCl3
76.84 CDCl3
64.79
52.25
f1 (ppm)



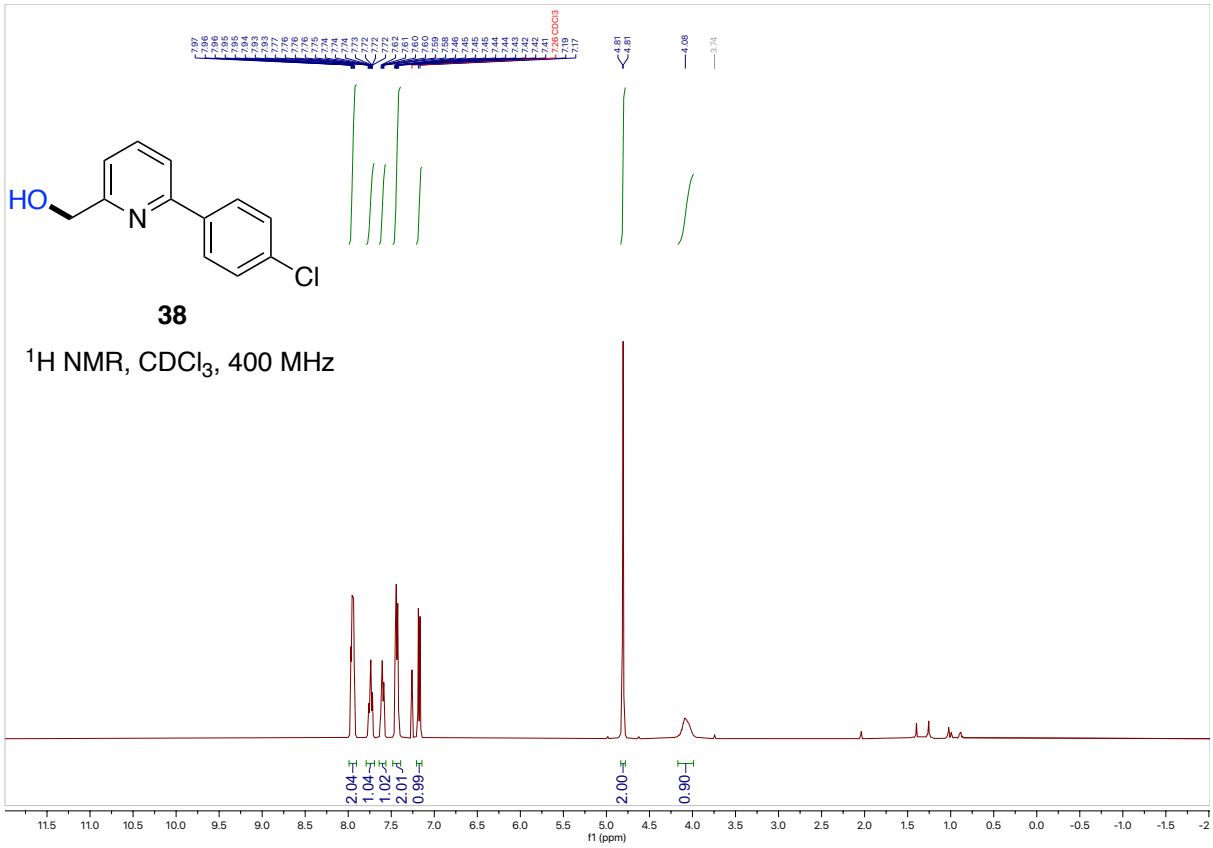
37

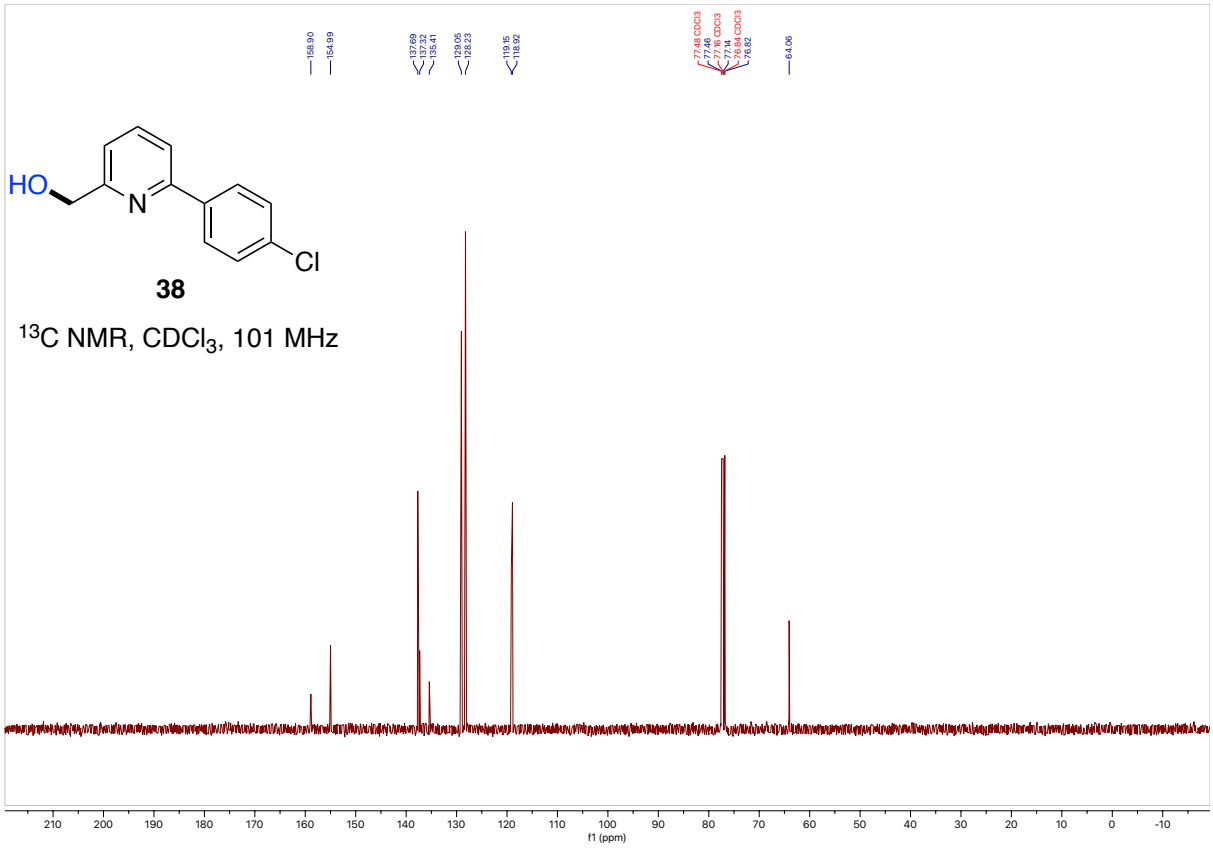
^{1}H NMR, $CDCl_3$, 400 MHz

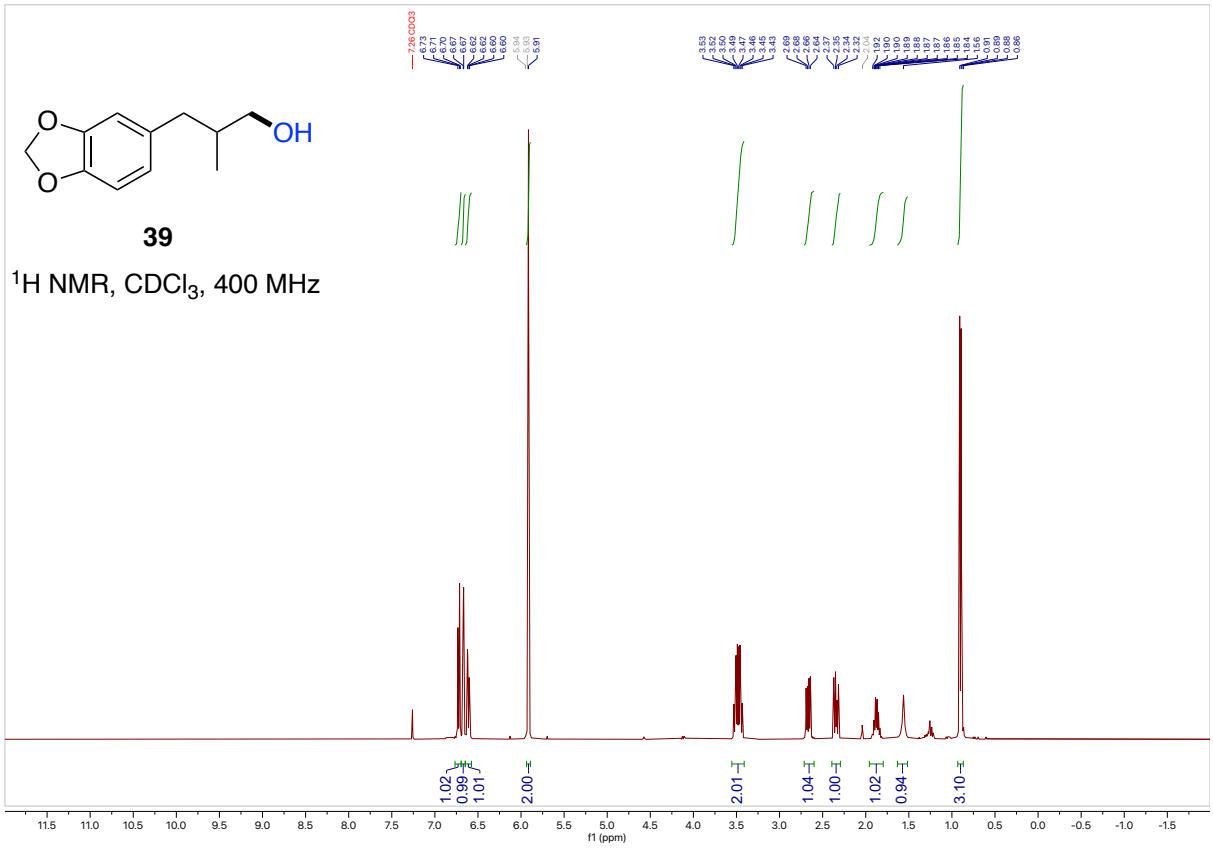


37

^{13}C NMR, $CDCl_3$, 101 MHz

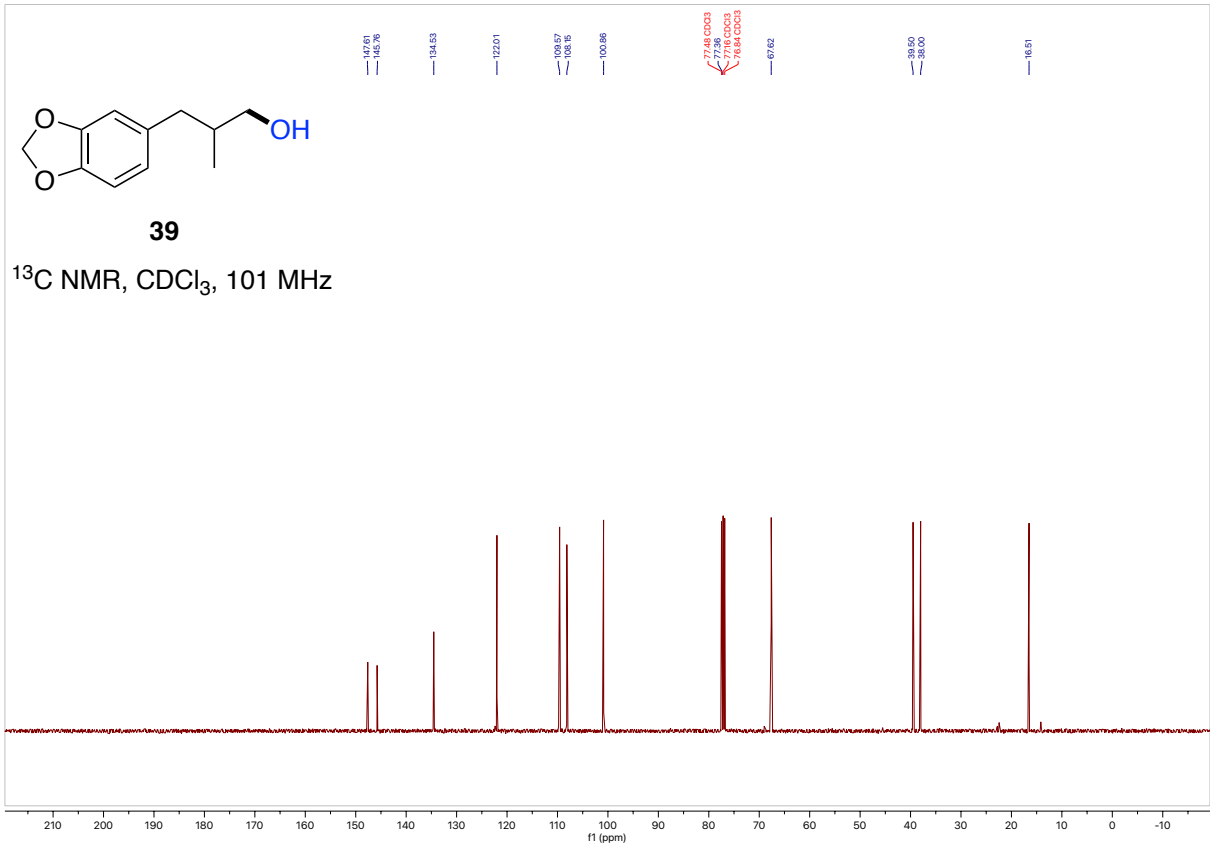

HO
N
Cl
38
^{1}H NMR, $CDCl_3$, 400 MHz
f1 (ppm)


HO
N
Cl
38
^{13}C NMR, $CDCl_3$, 101 MHz
f1 (ppm)



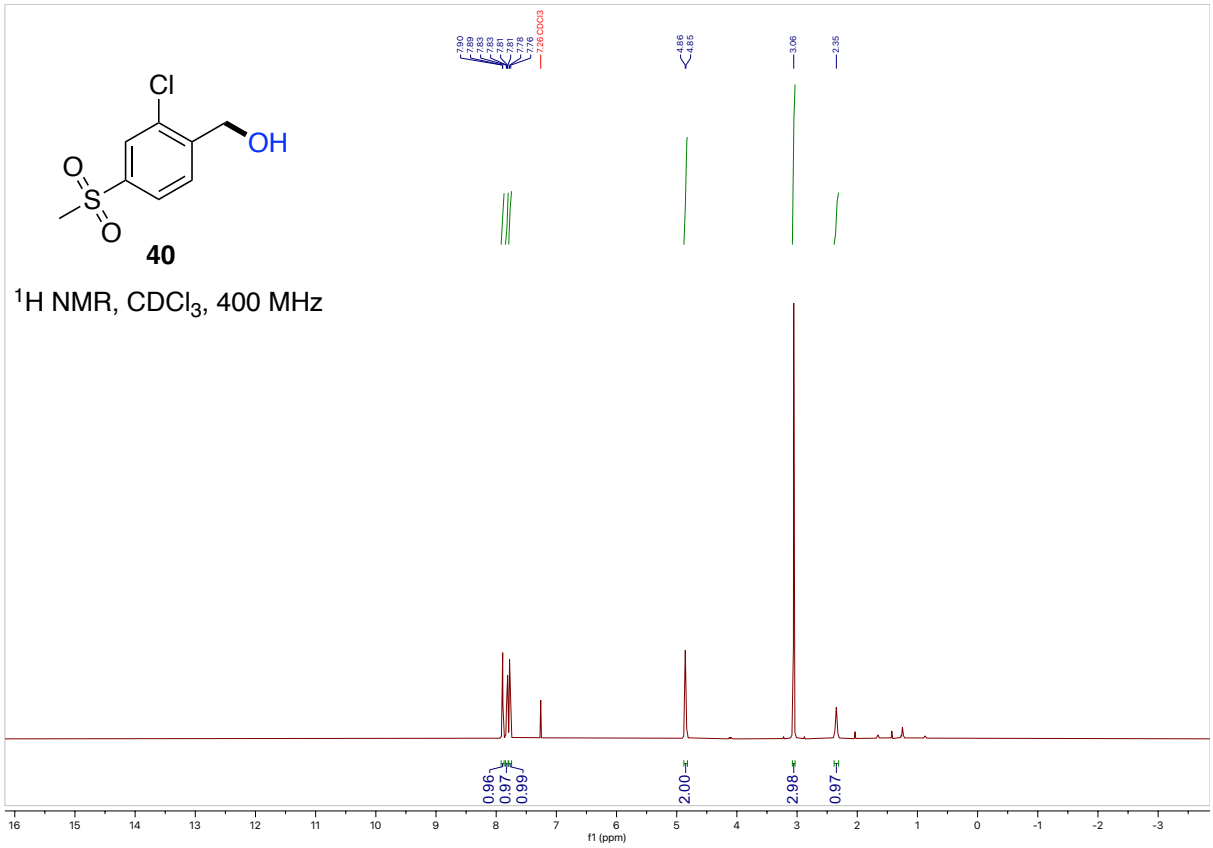
39

^{1}H NMR, $CDCl_3$, 400 MHz

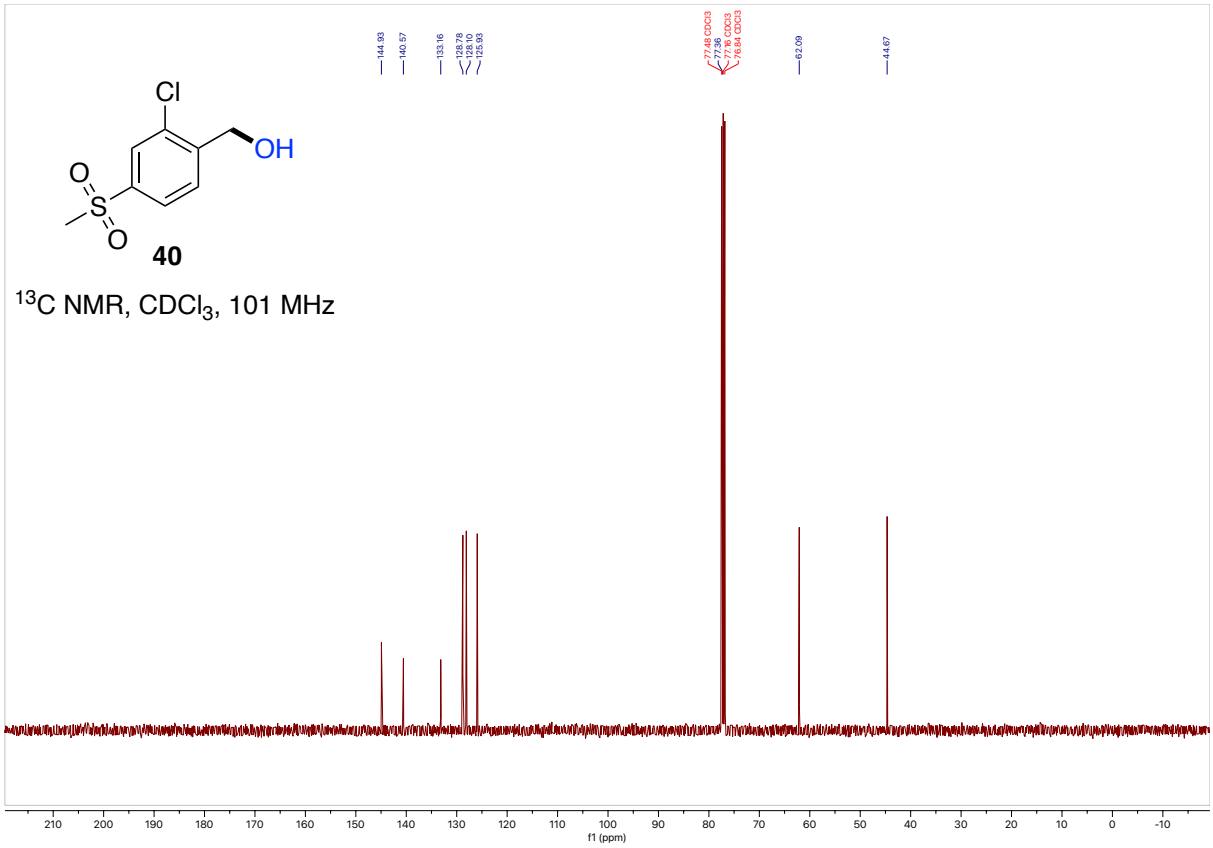


39

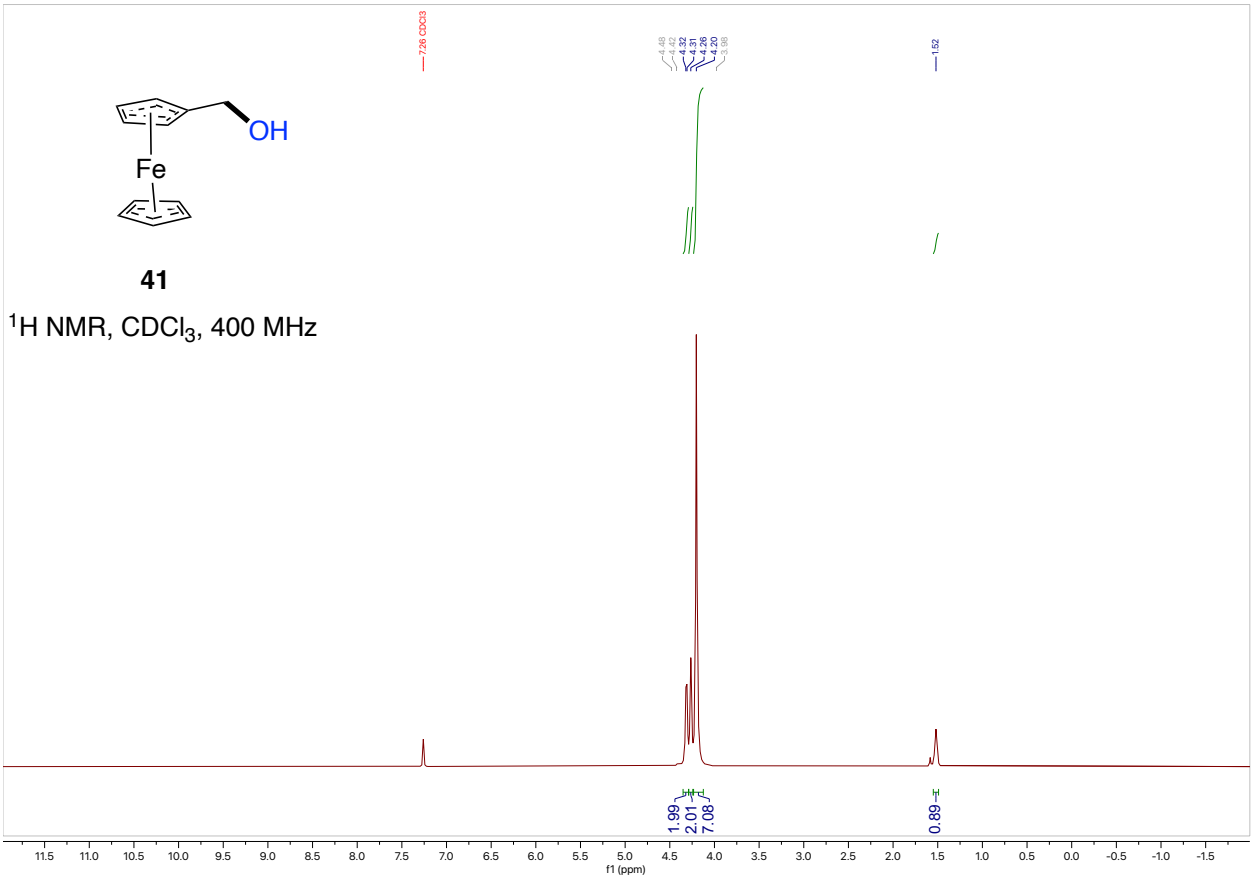
^{13}C NMR, $CDCl_3$, 101 MHz



^{1}H NMR, $CDCl_3$, 400 MHz

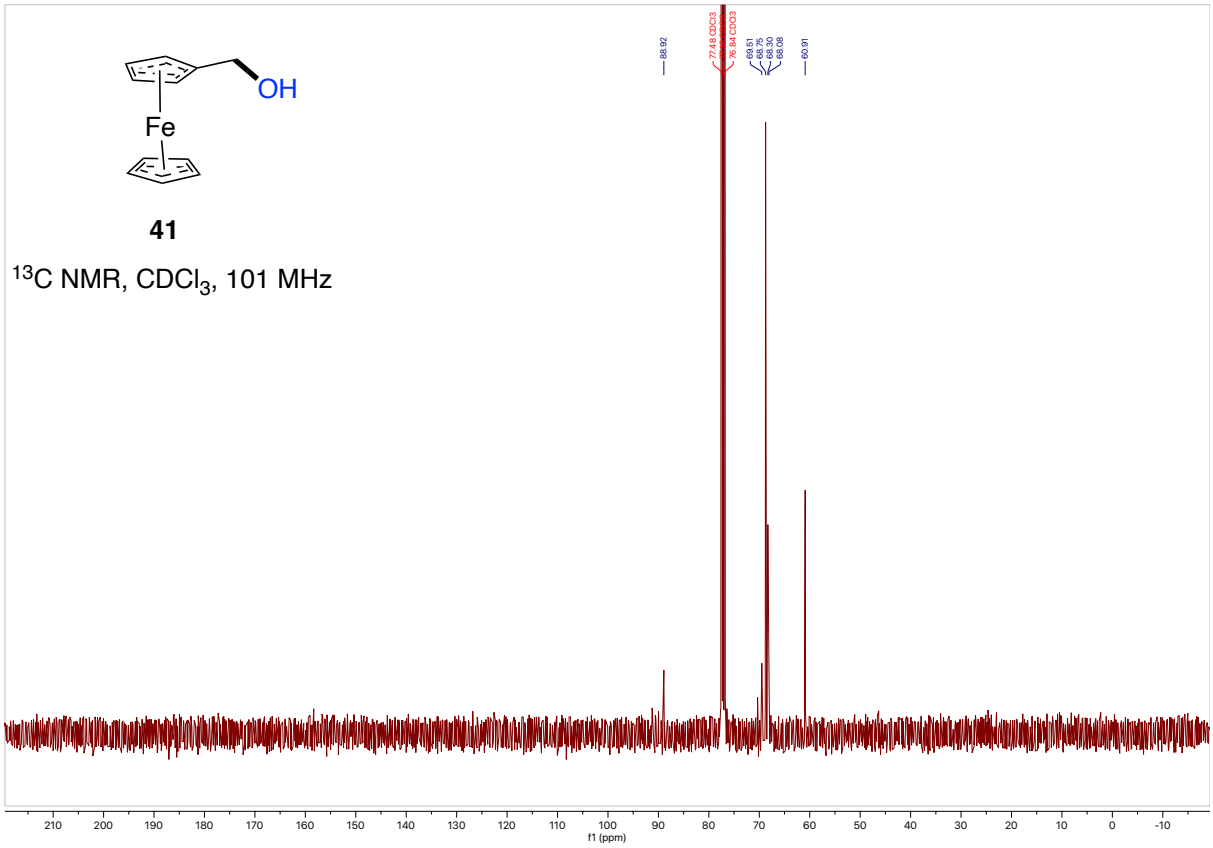


^{13}C NMR, $CDCl_3$, 101 MHz



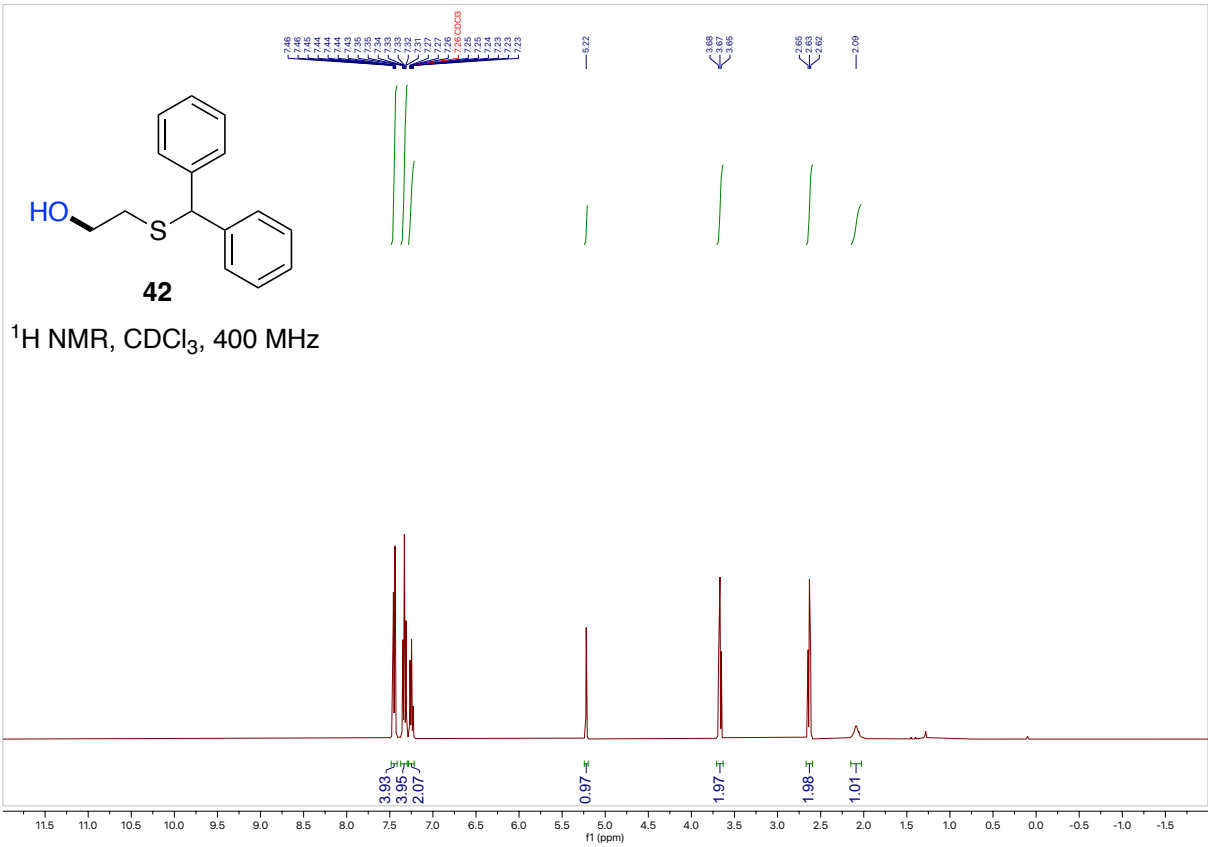
41

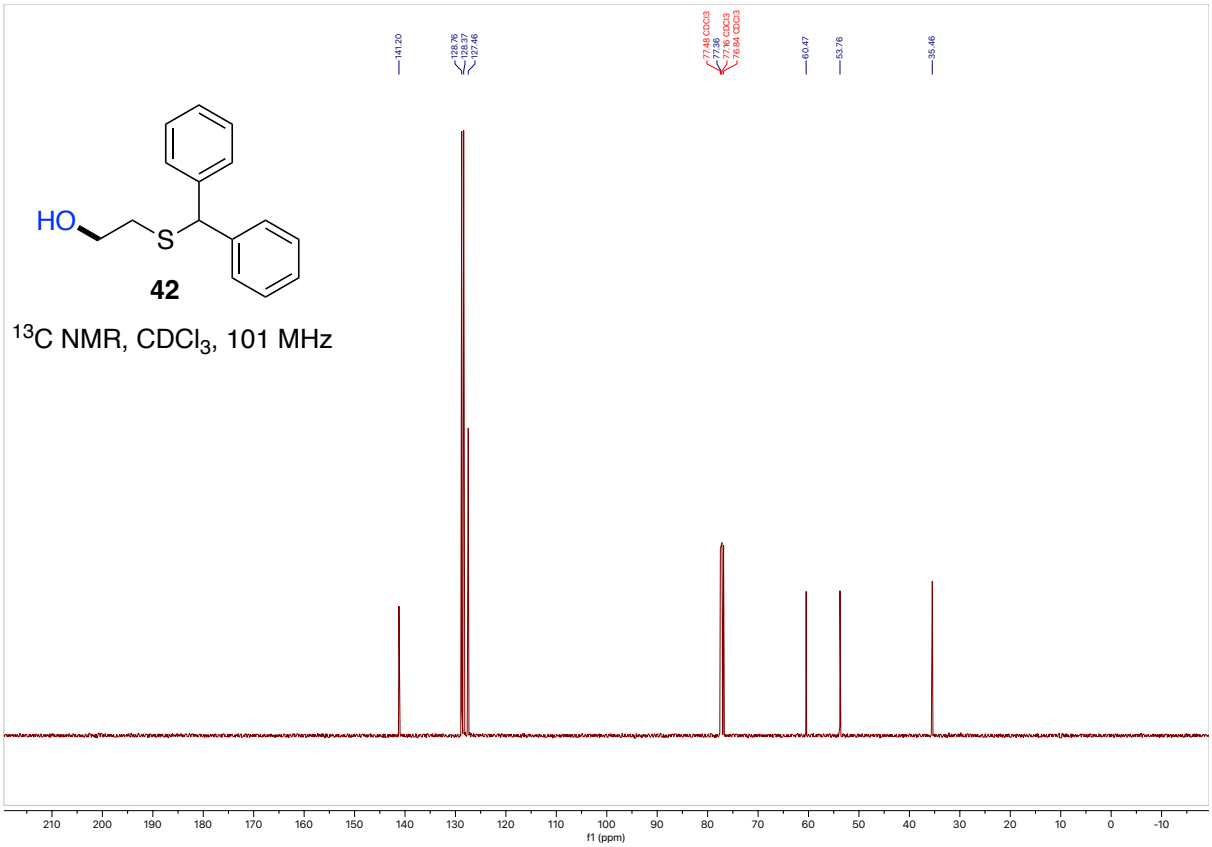
^{1}H NMR, $CDCl_3$, 400 MHz

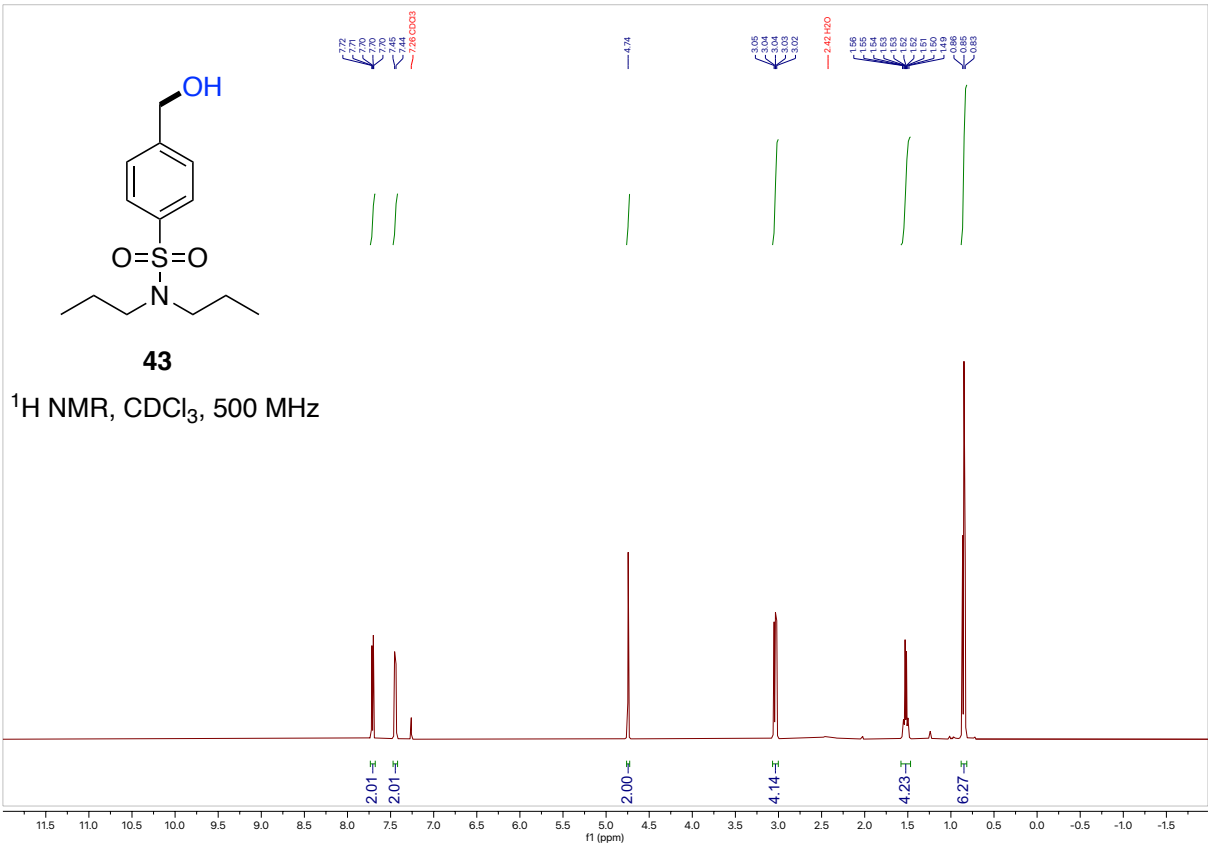


41

^{13}C NMR, $CDCl_3$, 101 MHz

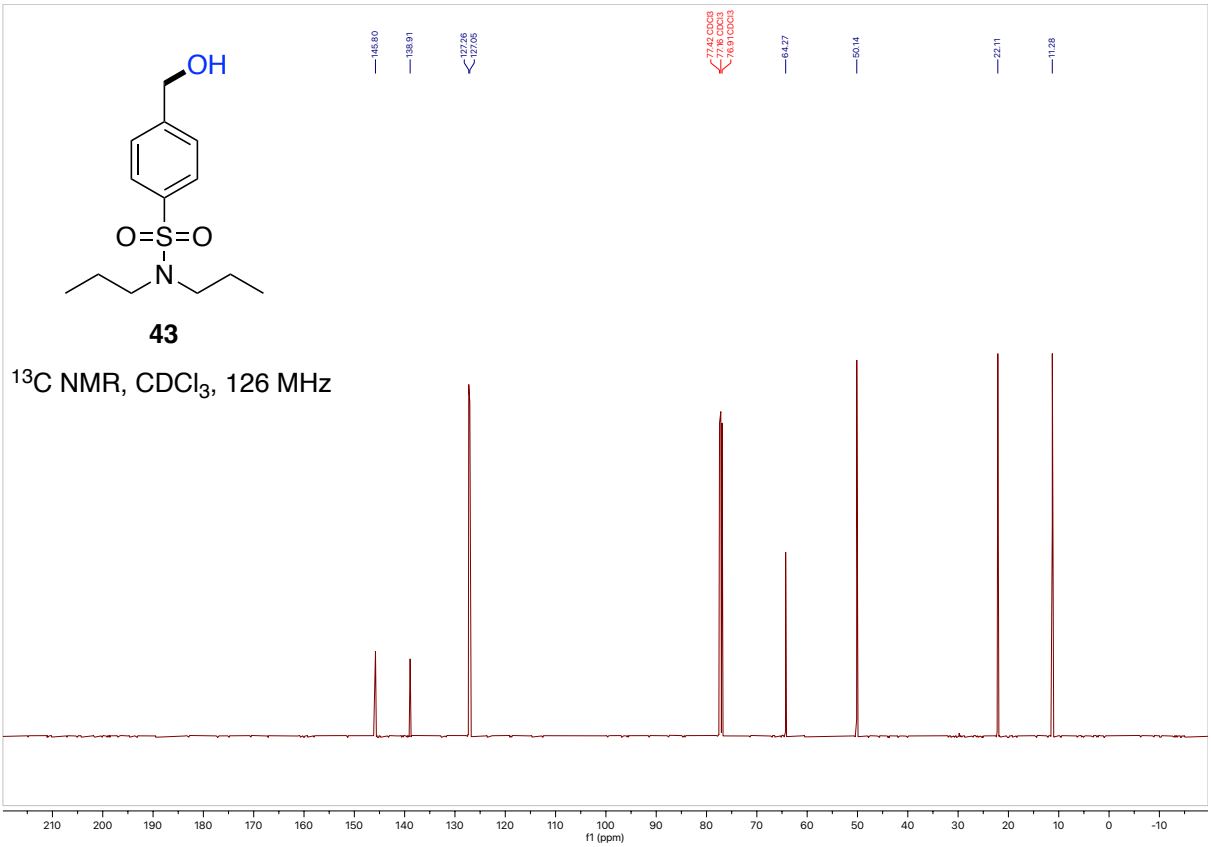

HO
S
42
^{1}H NMR, $CDCl_3$, 400 MHz


HO
S
42
^{13}C NMR, $CDCl_3$, 101 MHz



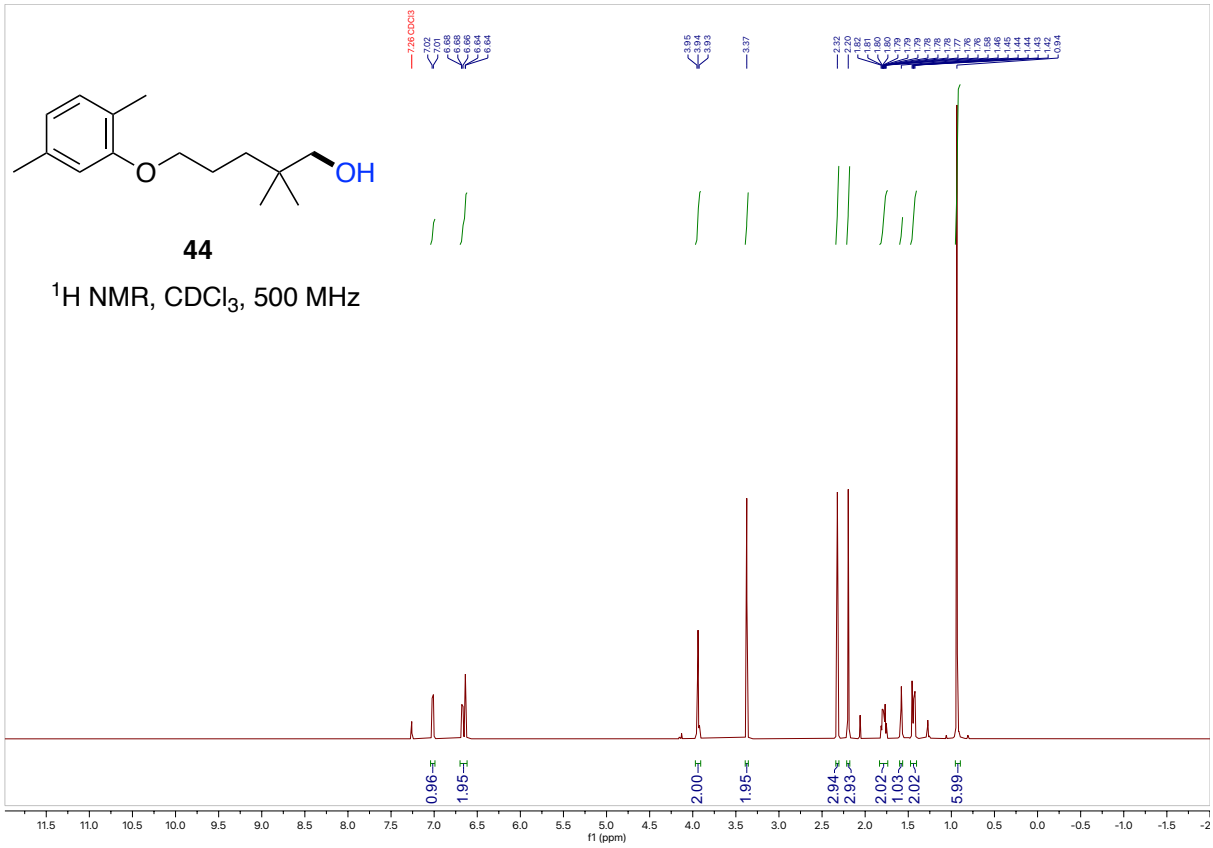
43

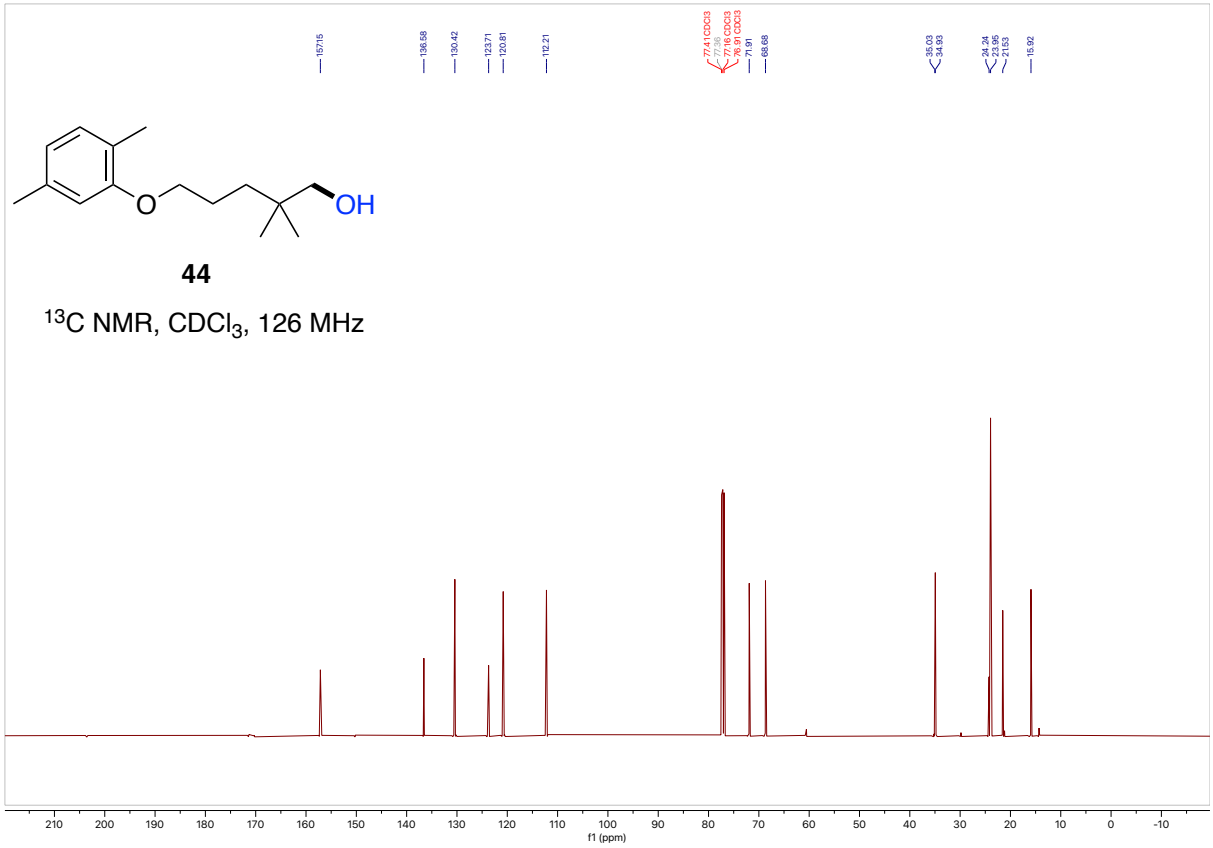
^{1}H NMR, $CDCl_3$, 500 MHz

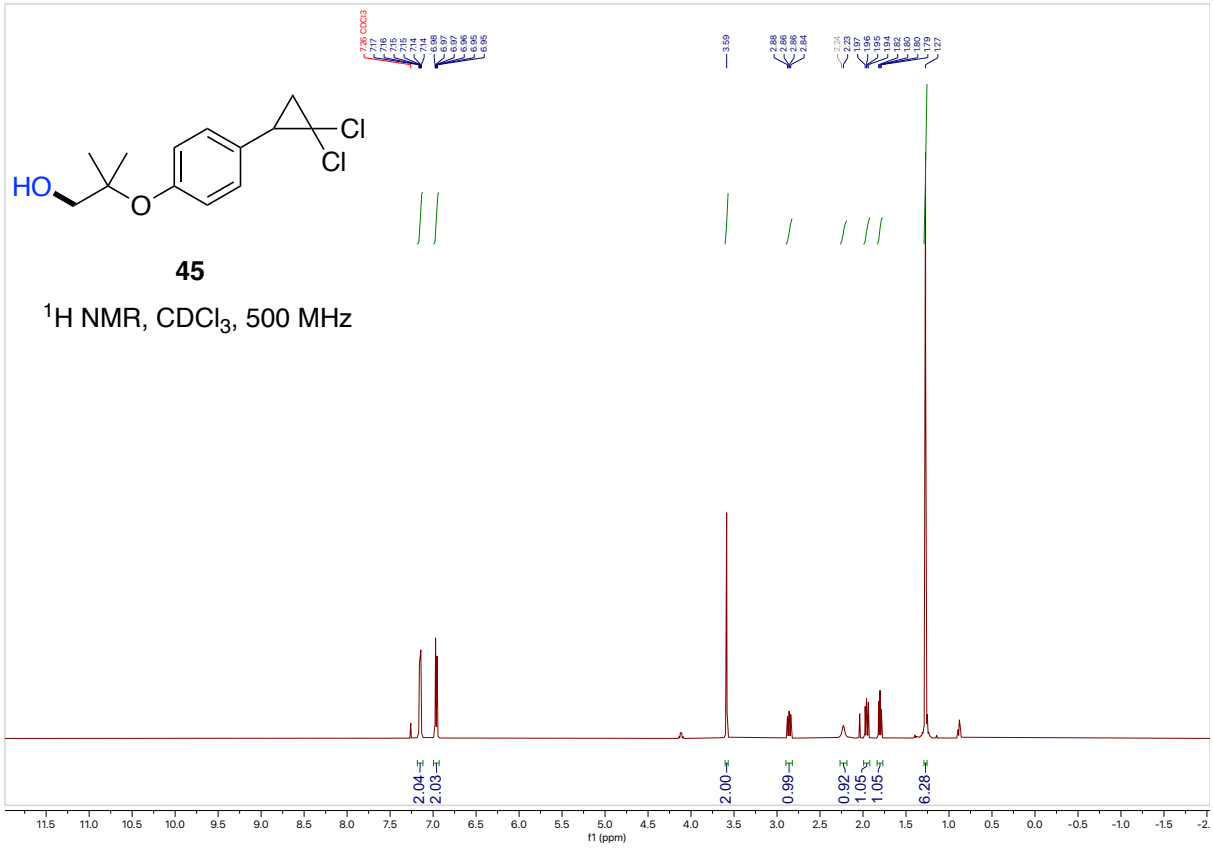


43

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

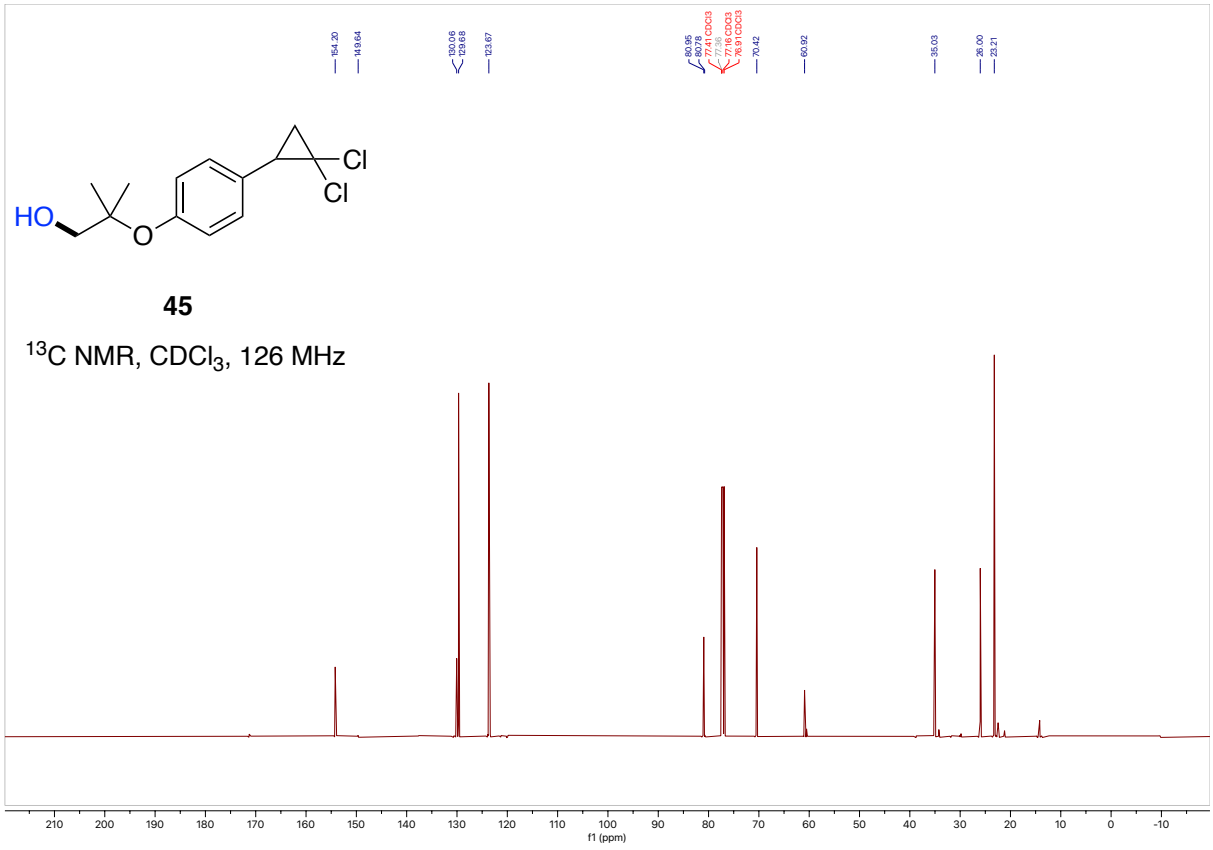

OH
O
44
1H NMR, CDCl3, 500 MHz
f1 (ppm)


OH
O
44
13C NMR, CDCl3, 126 MHz
f1 (ppm)



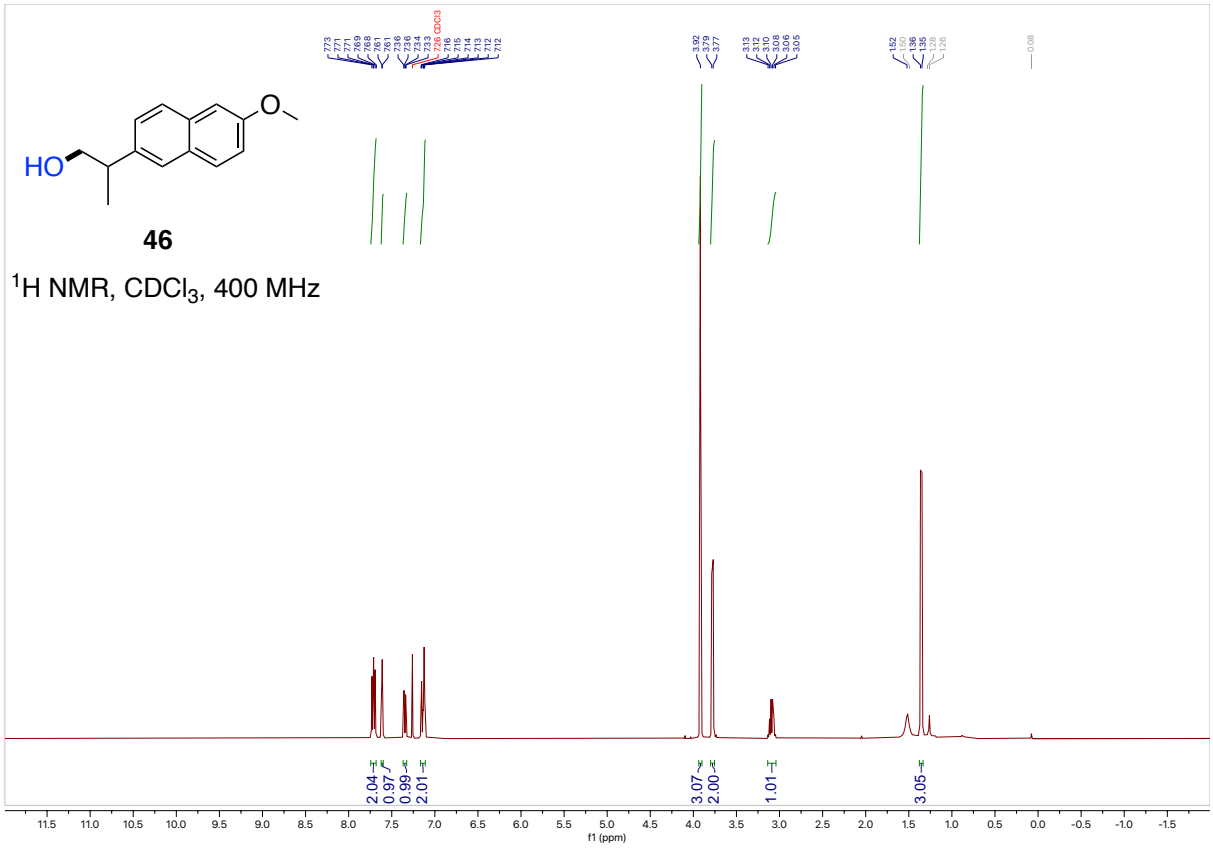
45

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

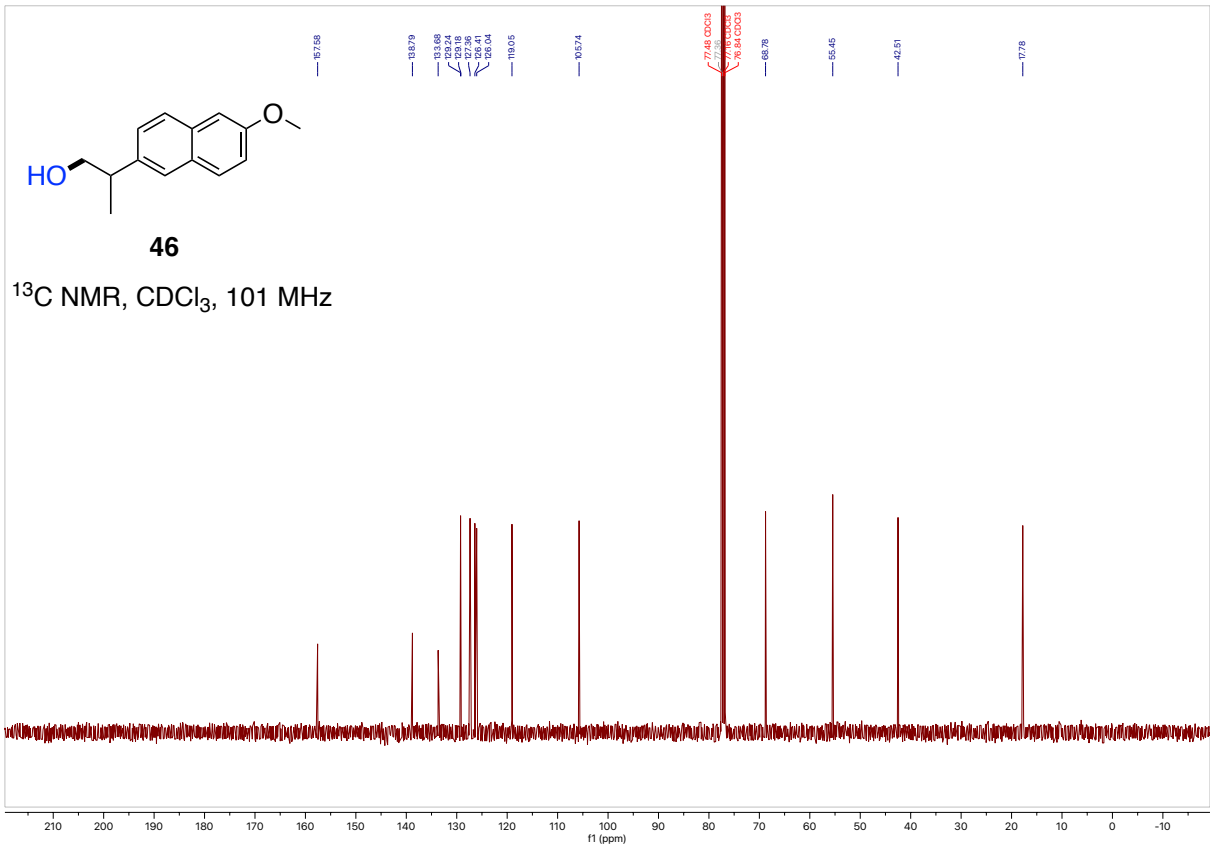


45

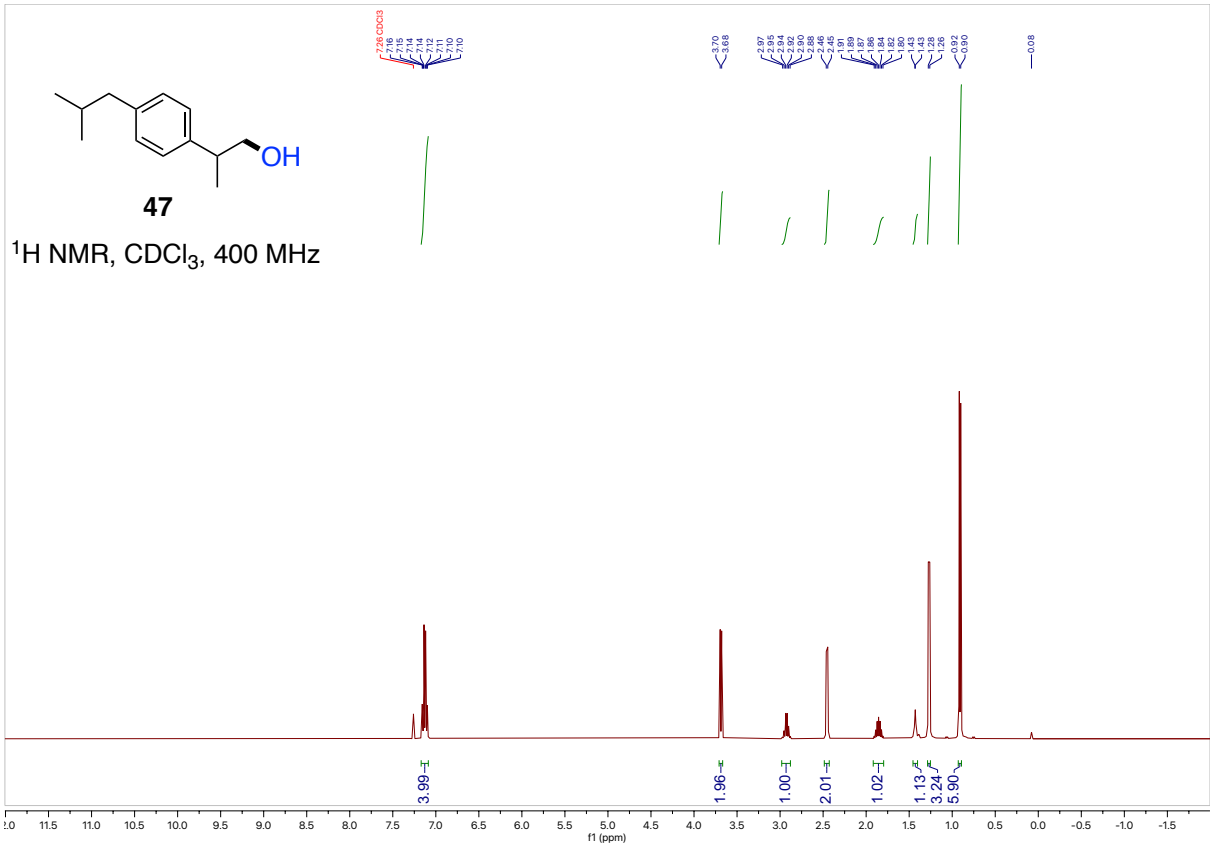
^{13}C NMR, $CDCl_3$, 126 MHz

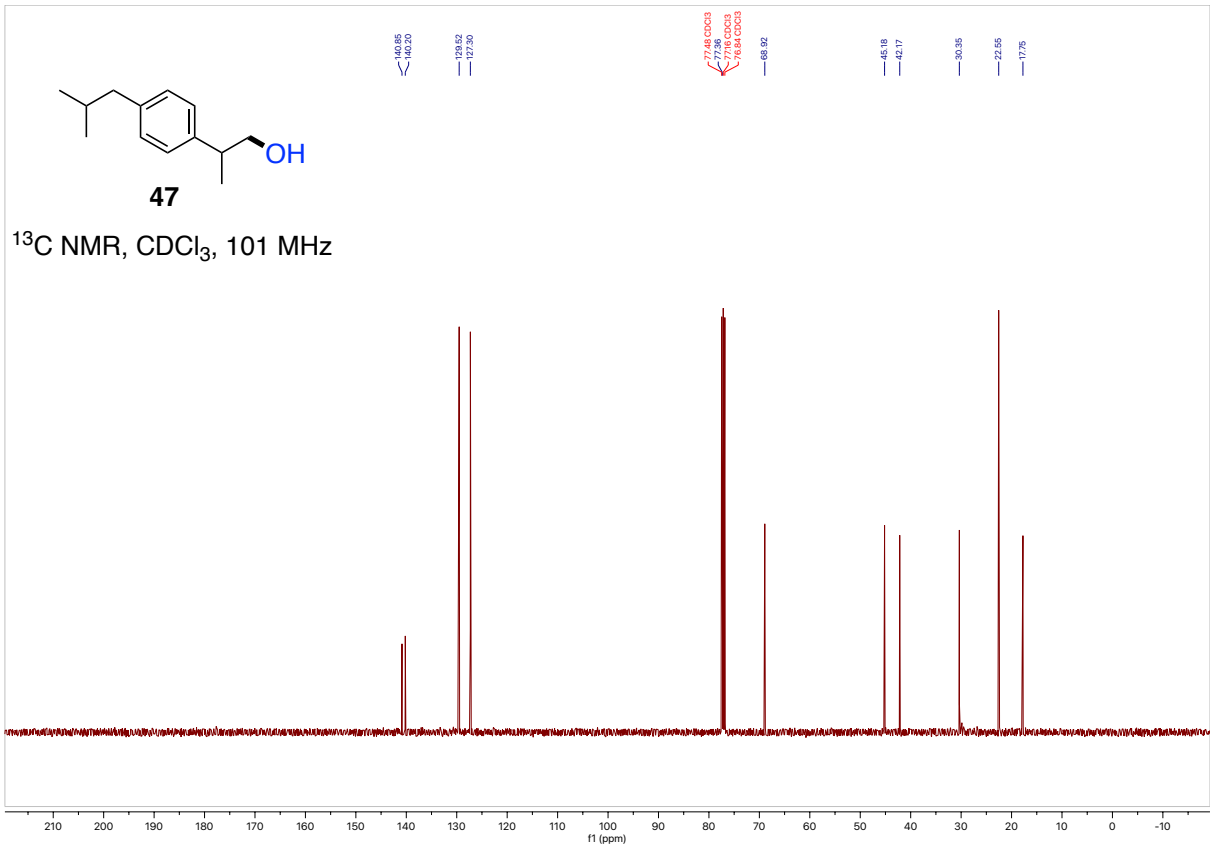


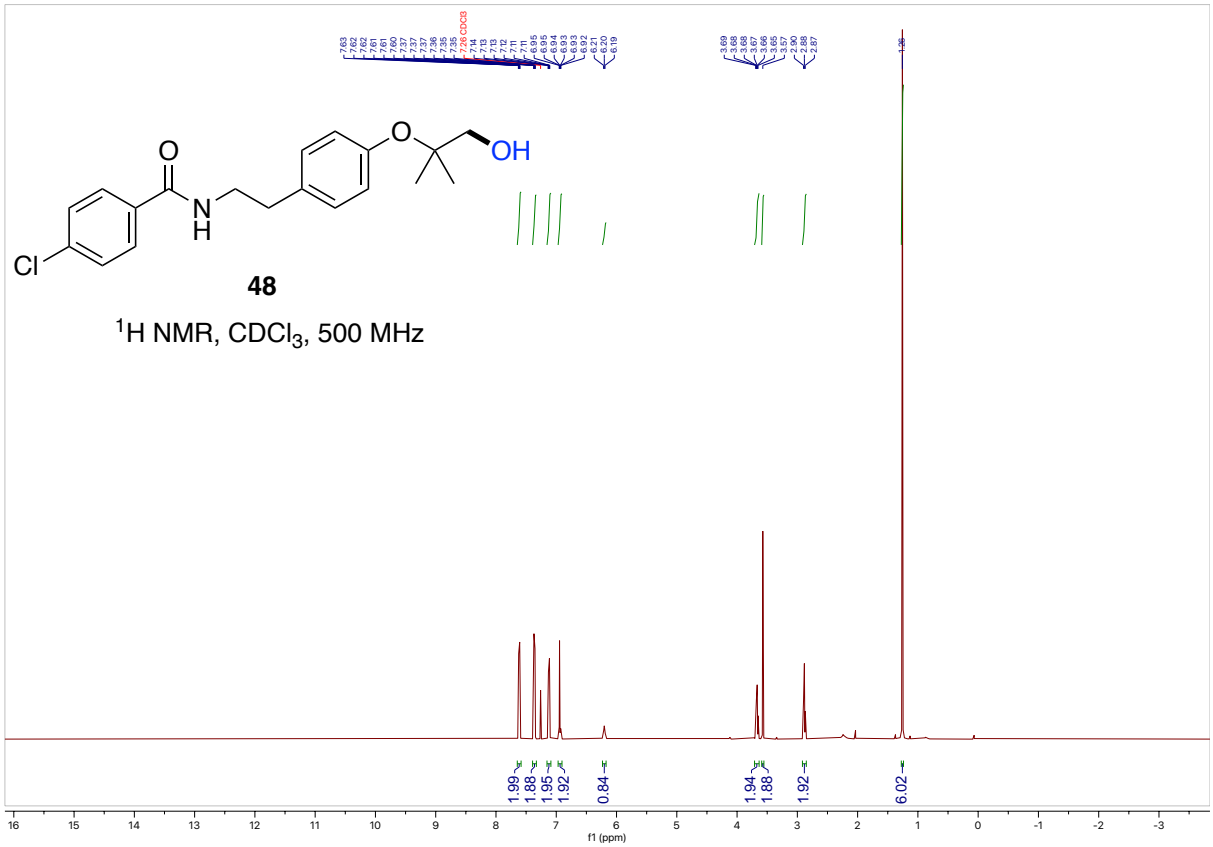
^{1}H NMR, $CDCl_3$, 400 MHz



^{13}C NMR, $CDCl_3$, 101 MHz

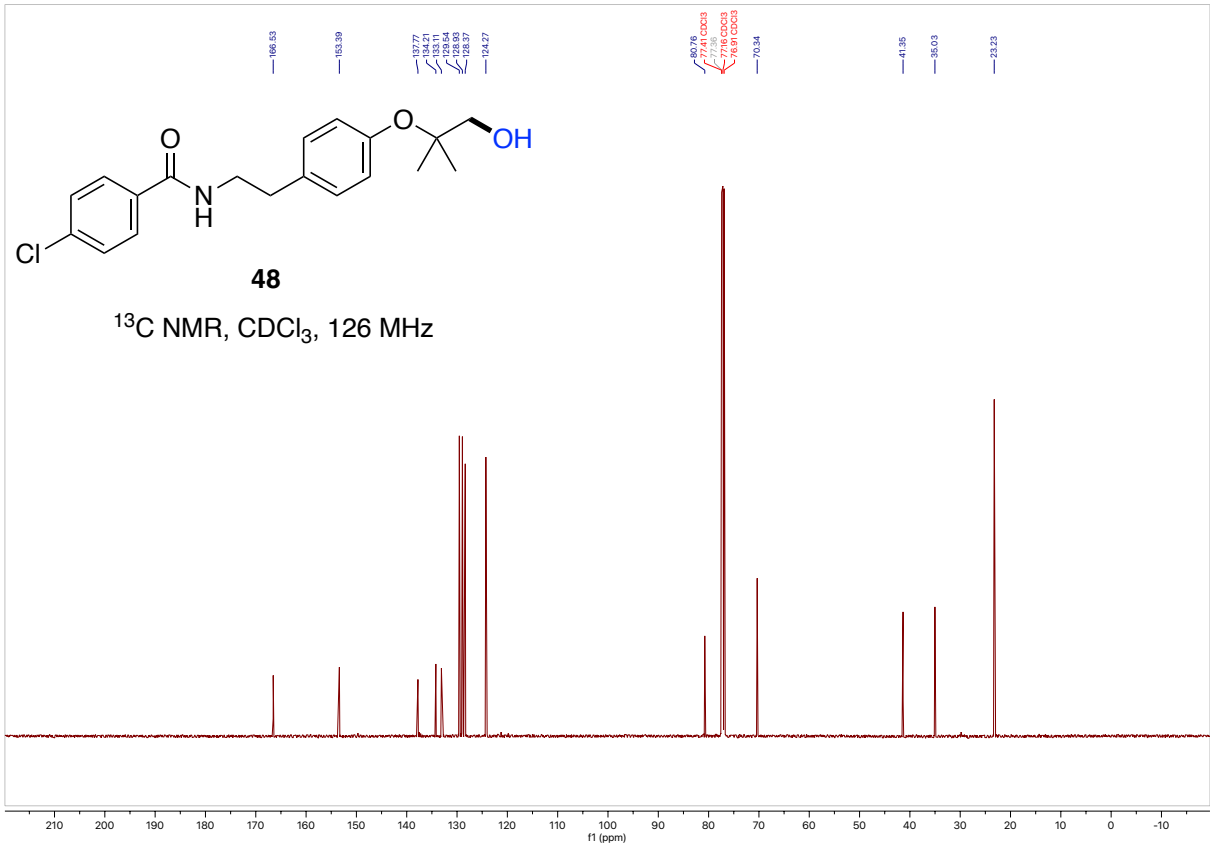

OH
47
^{1}H NMR, $CDCl_3$, 400 MHz
f1 (ppm)


OH
47
^{13}C NMR, $CDCl_3$, 101 MHz
f1 (ppm)



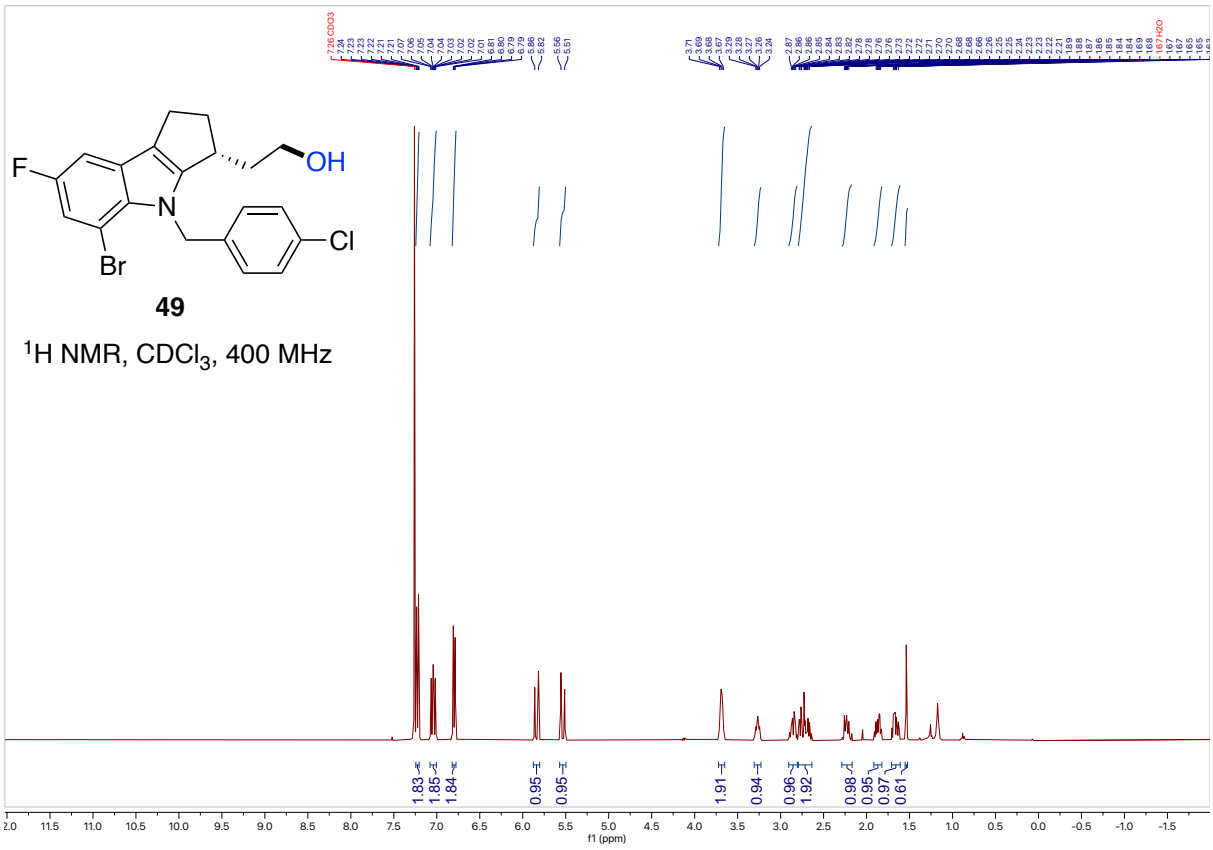
48

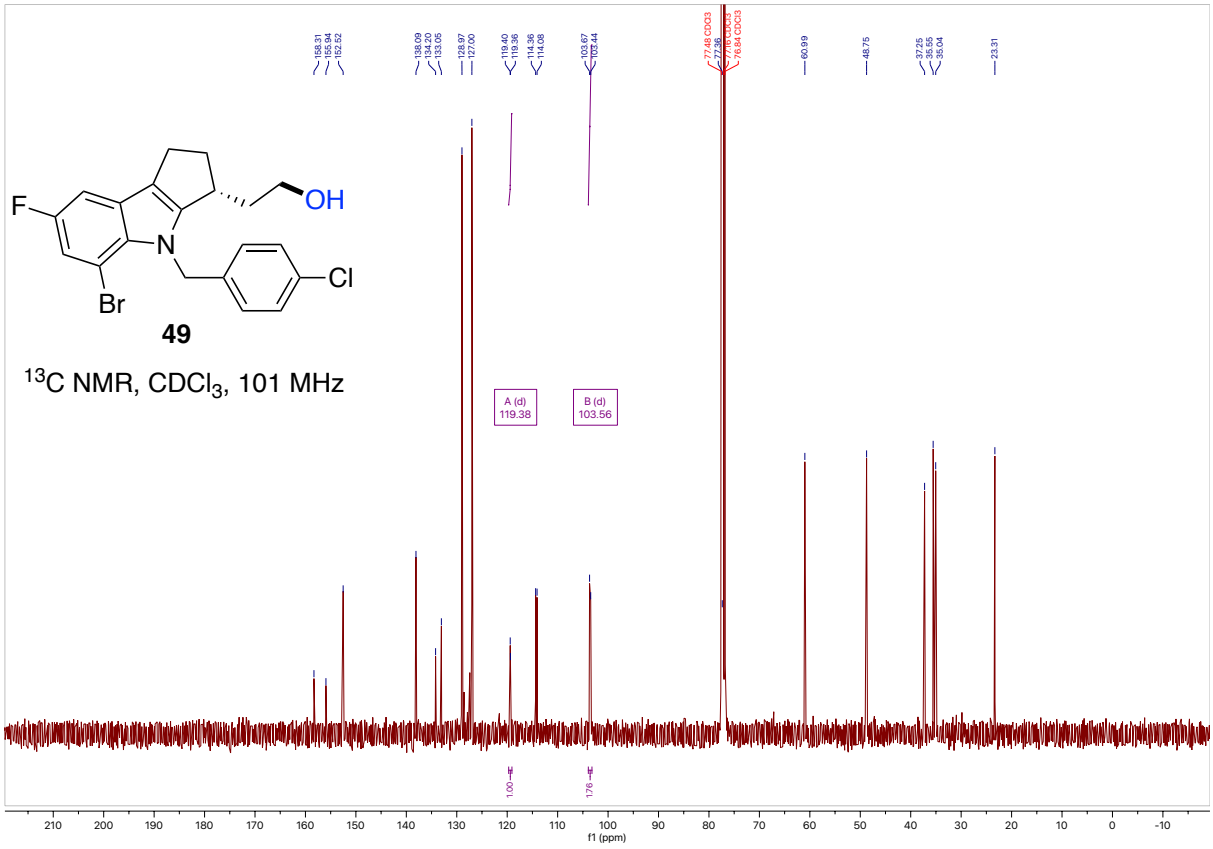
^{1}H NMR, $CDCl_3$, 500 MHz

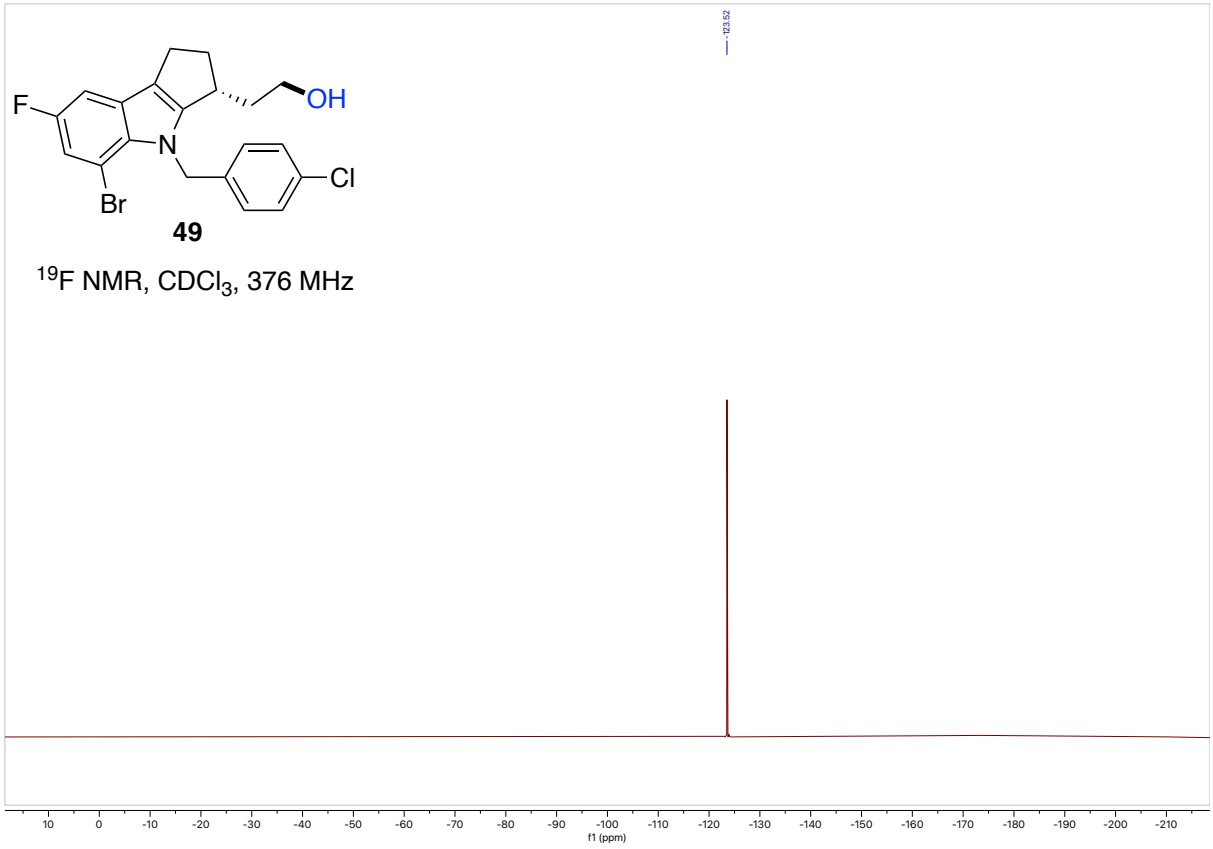


48

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

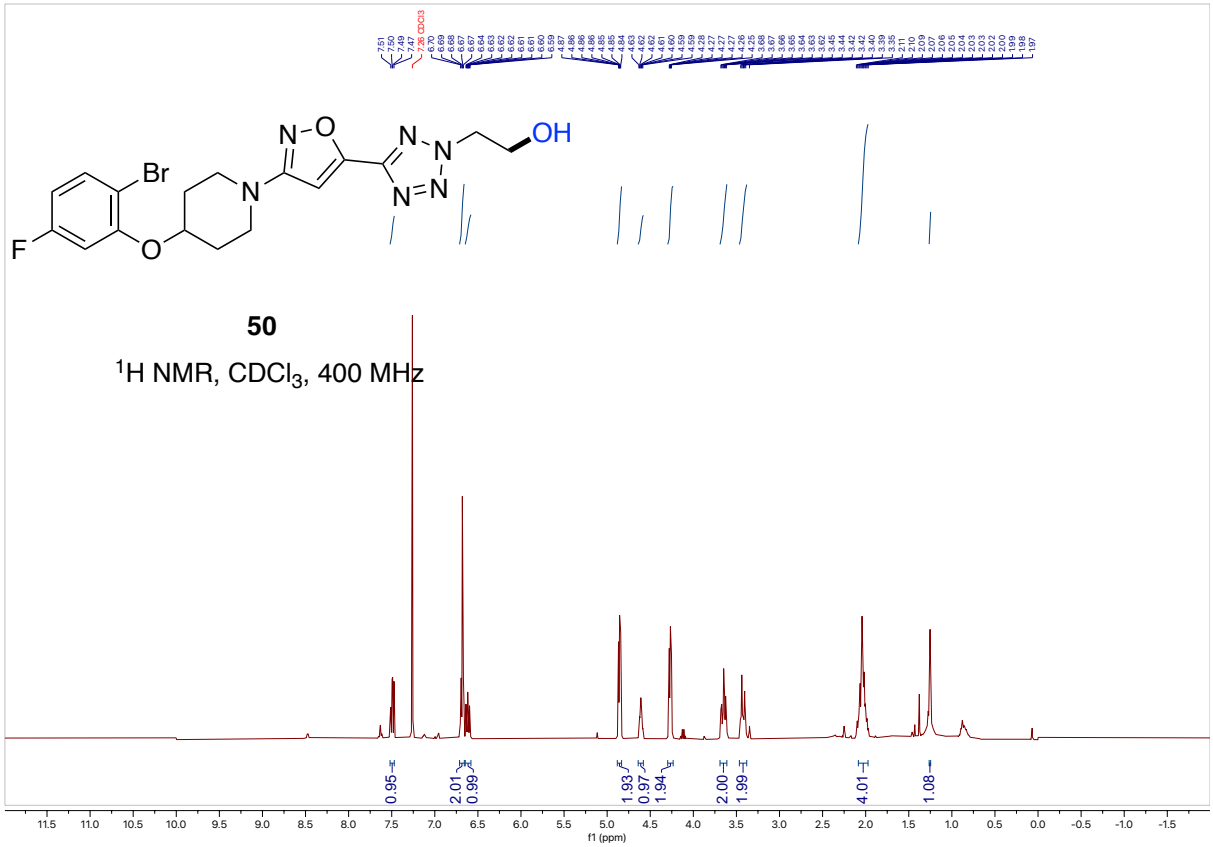

F
OH
N
Cl
Br
49
1H NMR, CDCl3, 400 MHz
f1 (ppm)


F
OH
N
Cl
Br
49
13C NMR, CDCl3, 101 MHz
A (d)
119.38
B (d)
103.56
f1 (ppm)



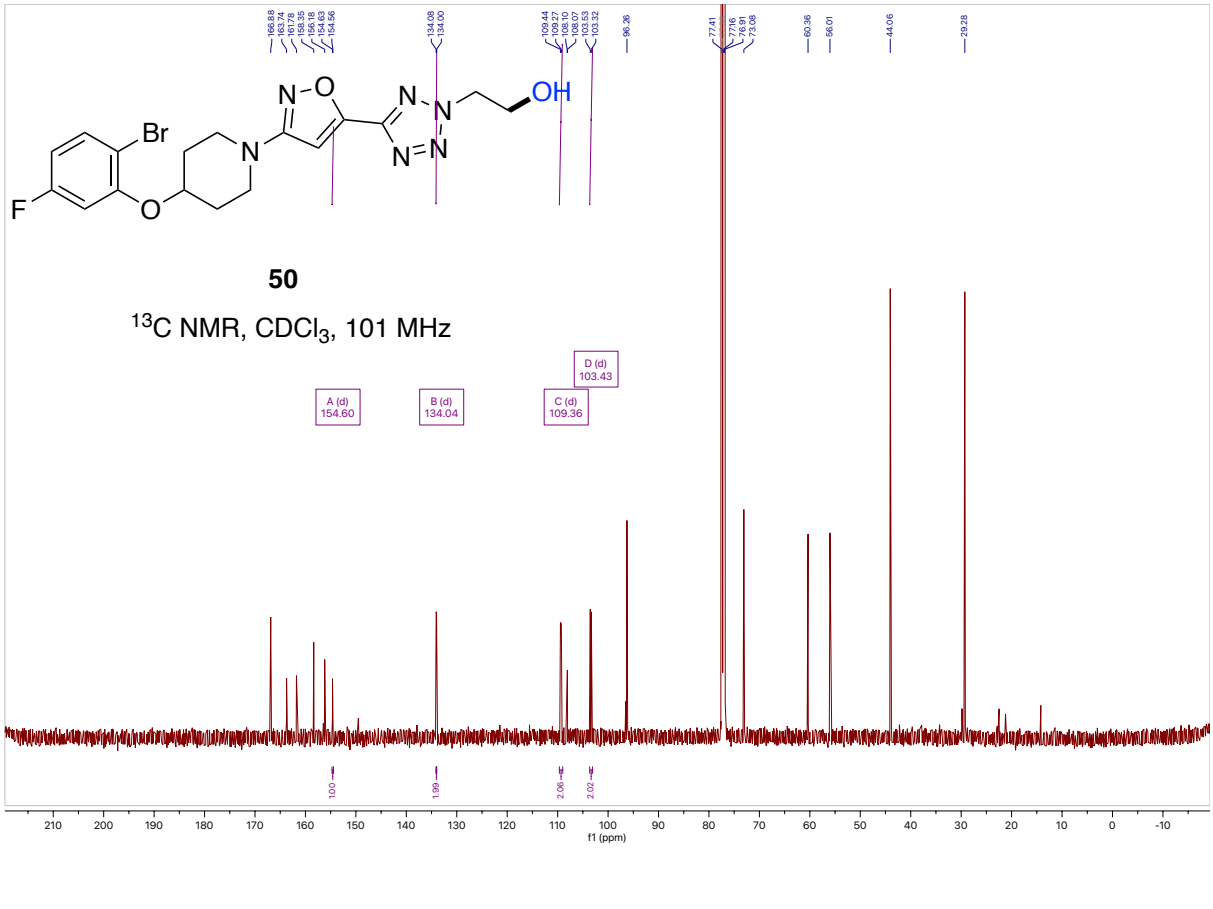
49

^{19}F NMR, $CDCl_3$, 376 MHz



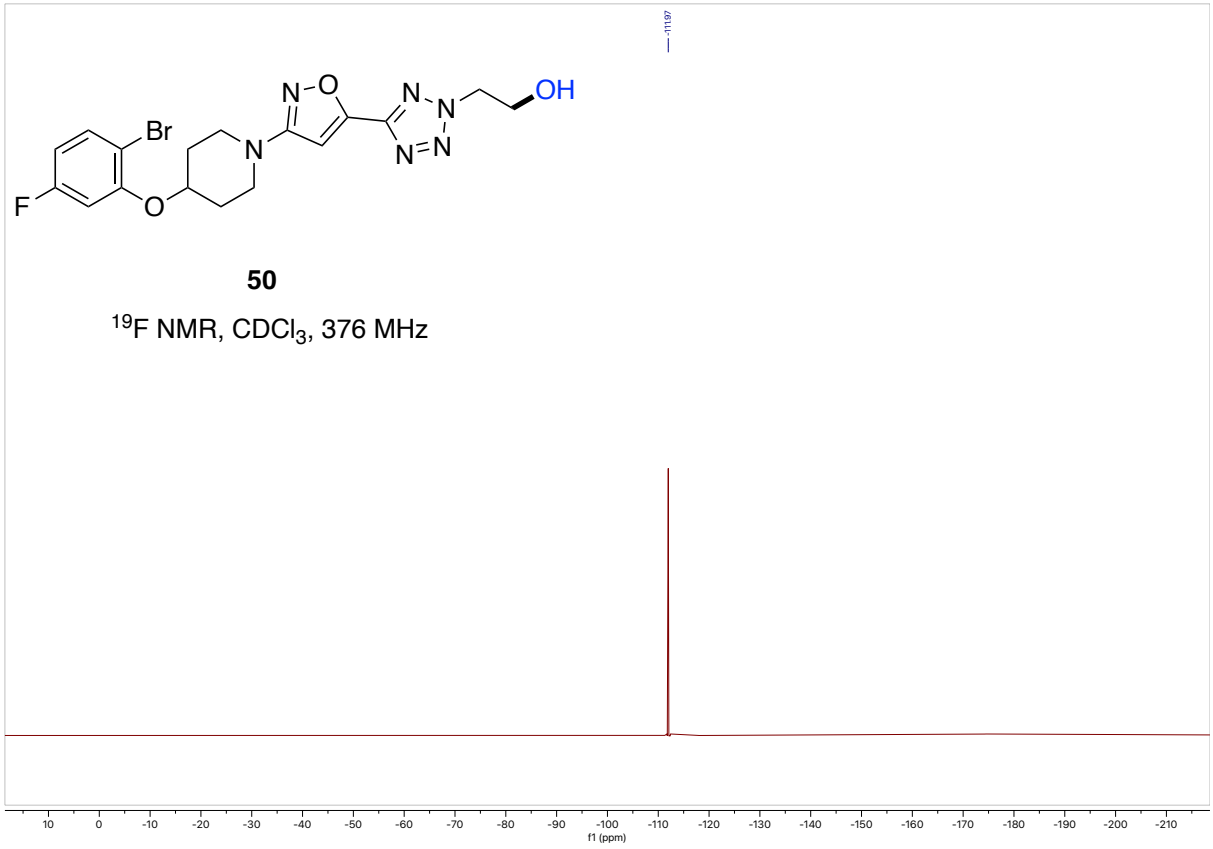
50

^{1}H NMR, $CDCl_3$, 400 MHz



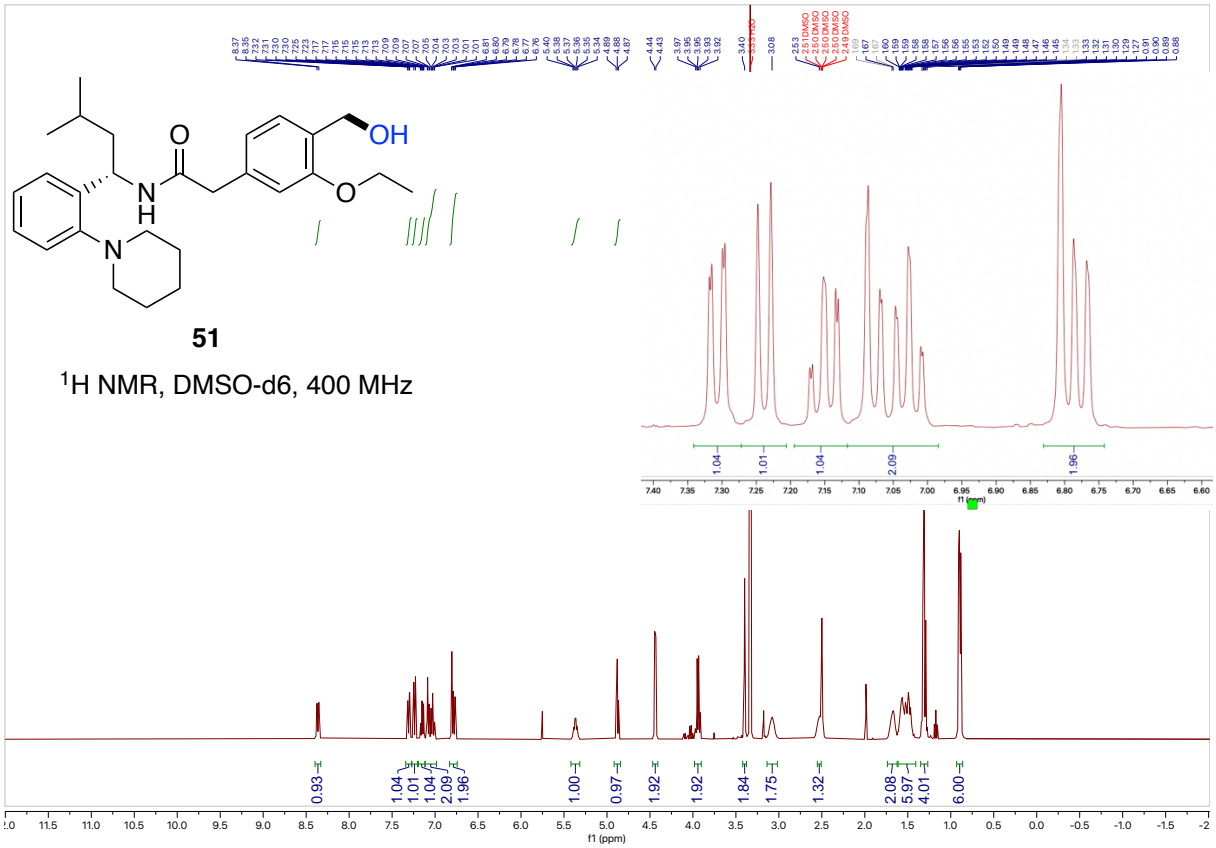
50

^{13}C NMR, $CDCl_3$, 101 MHz



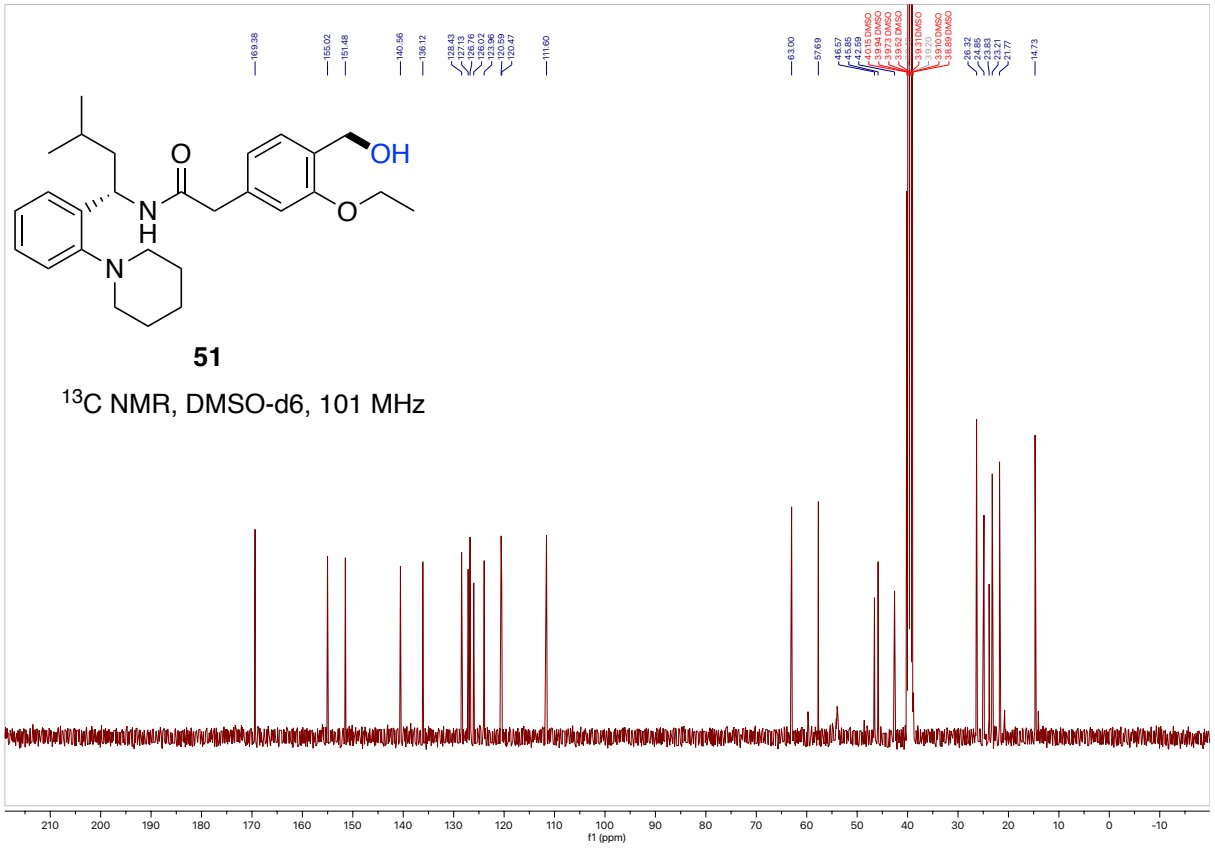
50

^{19}F NMR, $CDCl_3$, 376 MHz



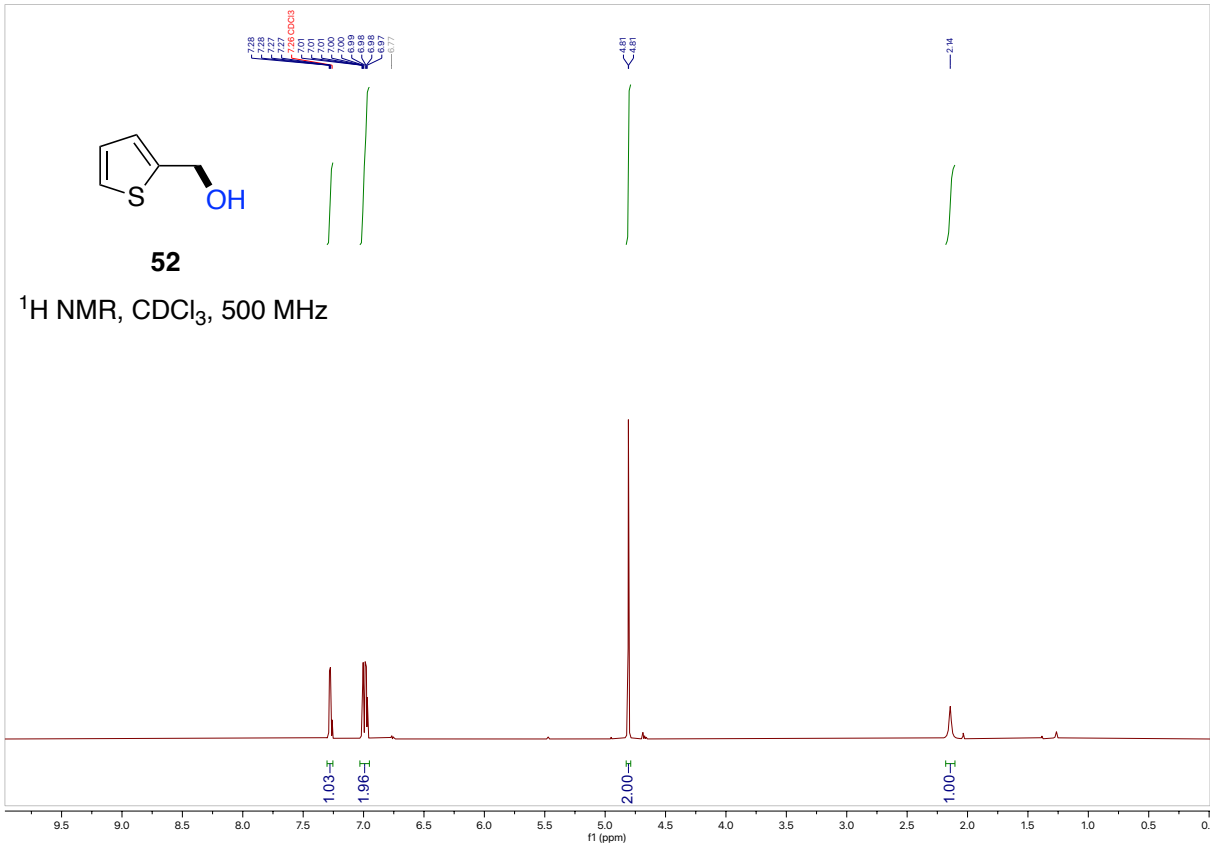
51

^{1}H NMR, DMSO-d6, 400 MHz



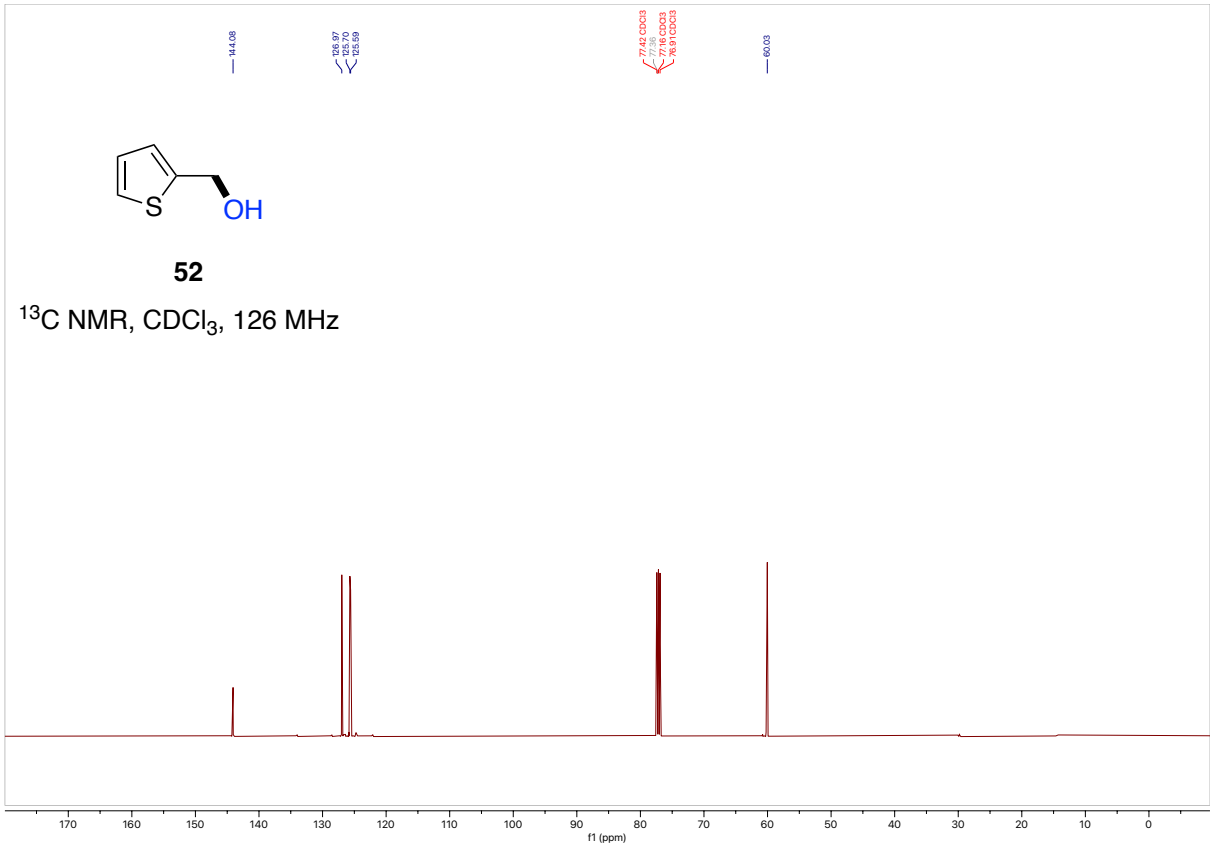
51

^{13}C NMR, DMSO-d6, 101 MHz



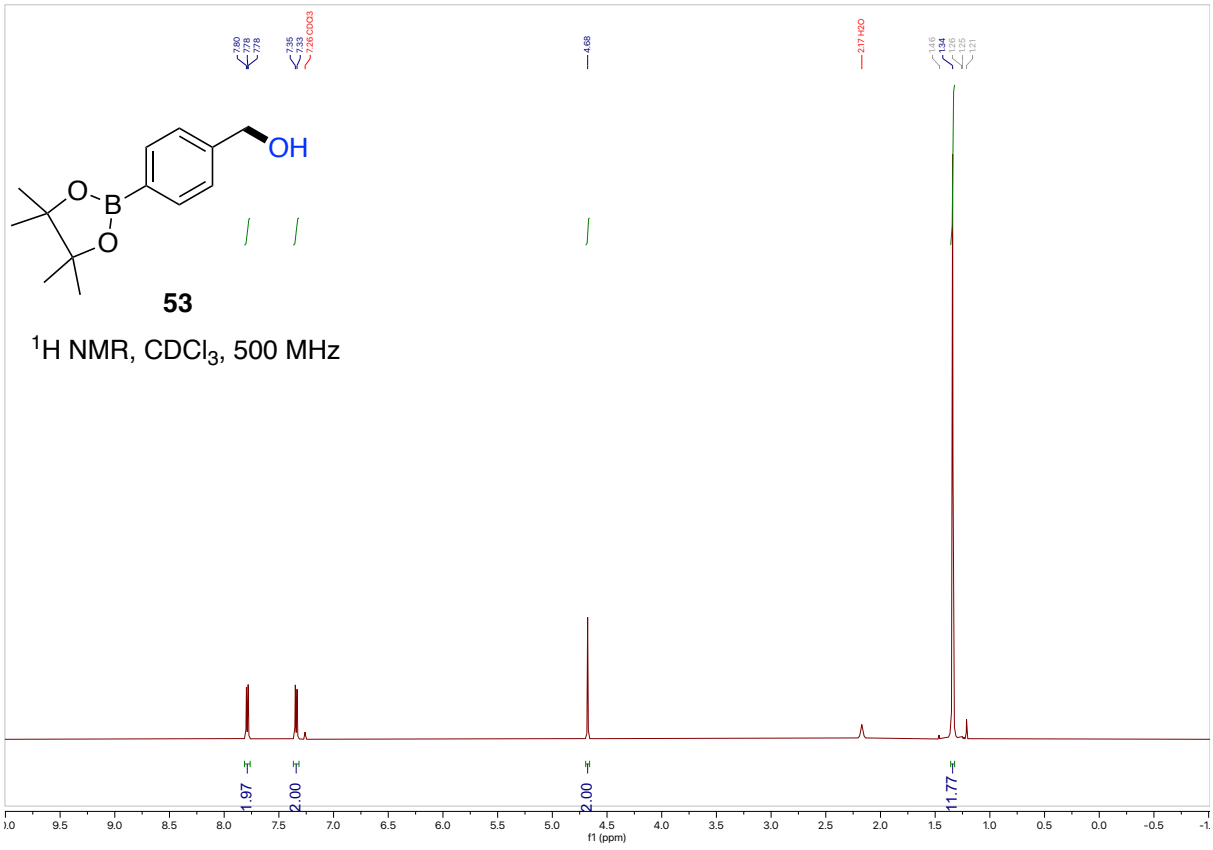
52

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



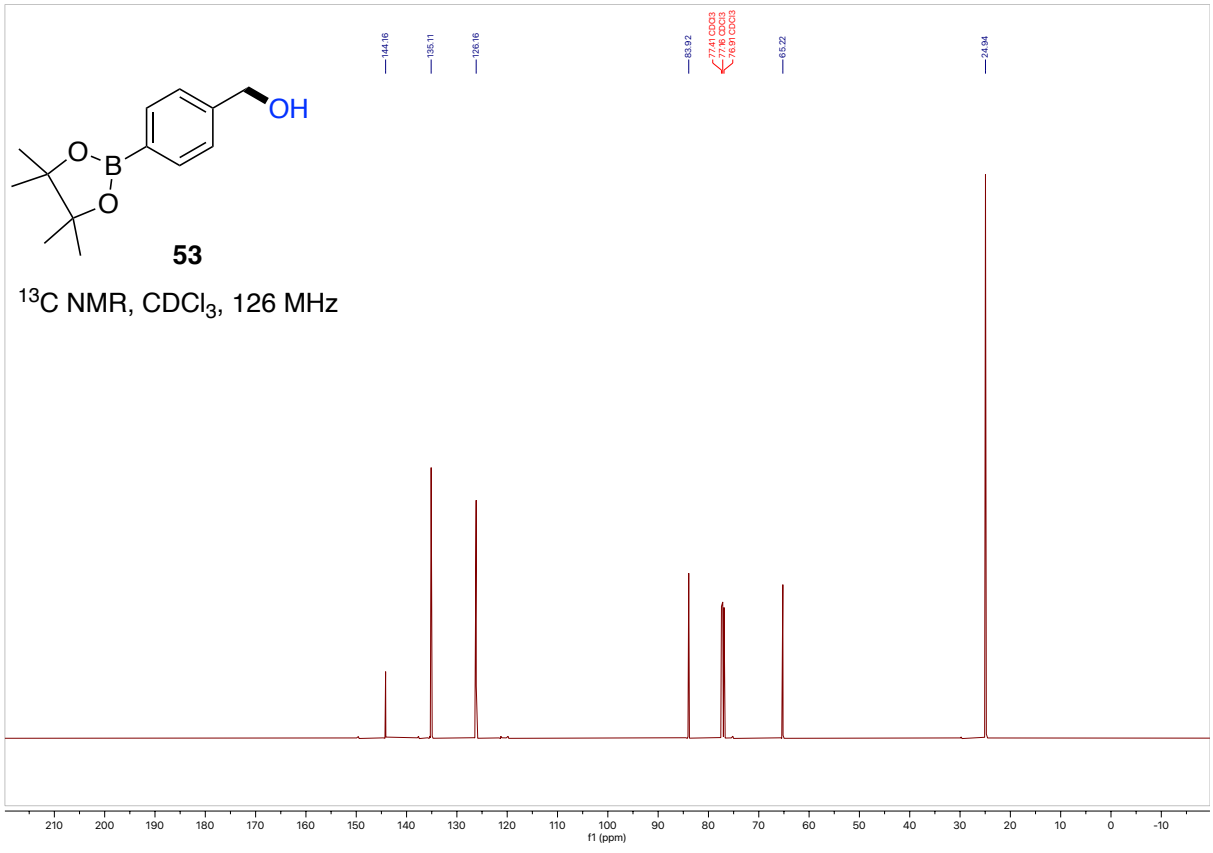
52

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



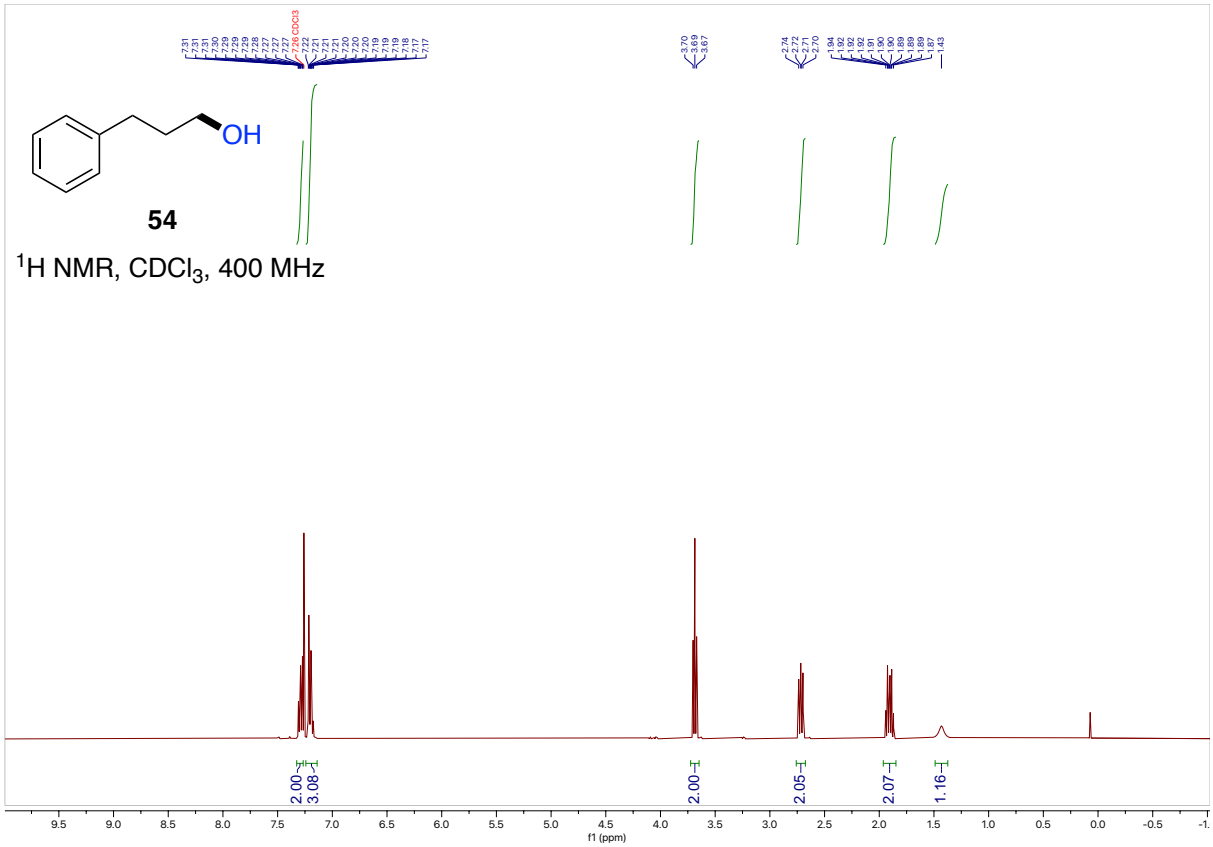
53

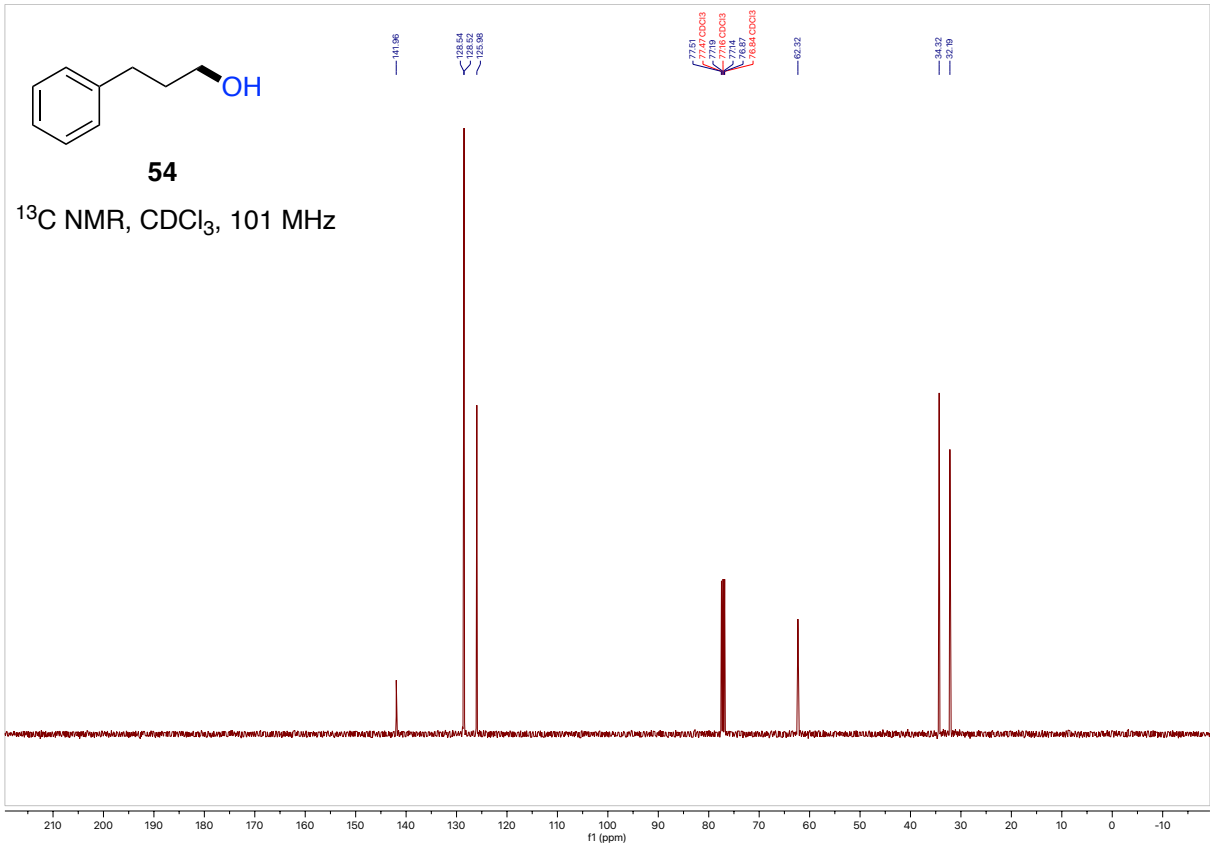
^{1}H NMR, $CDCl_3$, 500 MHz



53

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


OH
54
1H NMR, CDCl3, 400 MHz
2.00
3.08
2.00
2.05
2.07
1.16
f1 (ppm)


OH
54
13C NMR, CDCl3, 101 MHz
141.96
128.54
128.52
125.98
77.51
77.17 CDCl3
77.19
77.16 CDCl3
77.14
76.82
76.84 CDCl3
62.32
34.32
32.19
f1 (ppm)