

Supporting Information

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1. General Considerations

Chemicals and solvents were purchased from commercially available sources and used without any purification. Dry solvents were obtained from a solvent purification system using columns of Al_2O_3 under argon. ^1H , ^{13}C and ^{19}F NMR spectra were obtained with Bruker Avance-400 MHz NMR spectrometer with residual solvent peaks or tetramethylsilane as the internal reference. Multiplicities are described using the following abbreviations: s = singlet, bs = broad singlet, d = doublet, dd = doublet of doublet, t = triplet, m = multiplet. High-resolution mass spectra were obtained using a Thermo Q Exactive™ Plus via (ASAP-MS) by the mass spectrometry facility at the University of Wisconsin-Madison (funded by NIH grant: 1S10OD020022-1). Voltammetric experiments and bulk electrolysis reactions were performed using Nuvant Array PGStats (Serial number: APG15091801), from Nuvant System Inc (130 N. West Street Crown Point, Indiana 46307, USA). Compact fluorescent lamp (CFL, spiral, 20 W, 120 V, 60 Hz, 315mA, 1200 lumens, 6500 K) is purchased from Amazon, produced by Sunlite. The cell for bulk electrolysis was made by Tracy Owen Drier, scientific glassblower in Department of Chemistry at University of Wisconsin – Madison. The O-ring seal joint (7646-08 15 mm joints o-ring seal -116) was purchased from Ace Glass Inc. (1430 North West Blvd. Vineland, NJ, USA).

2. CV Studies

For all the voltammetric experiments, a glassy carbon disk electrode (2 mm diameter) and a platinum wire (1.0 cm, spiral wire) were used as working and counter electrodes, respectively. The working electrode potentials were measured versus Ag/AgNO_3 reference electrode (internal solution, 0.1 M Bu_4NClO_4 and 0.01 M AgNO_3 in CH_3CN). The redox potential of ferrocene/ferrocenium (Fc/Fc^+) was measured (same experimental conditions) and used to provide an internal reference. The potential values were then adjusted relative to Fc/Fc^+ , and electrochemical studies in organic solvents were recorded accordingly.

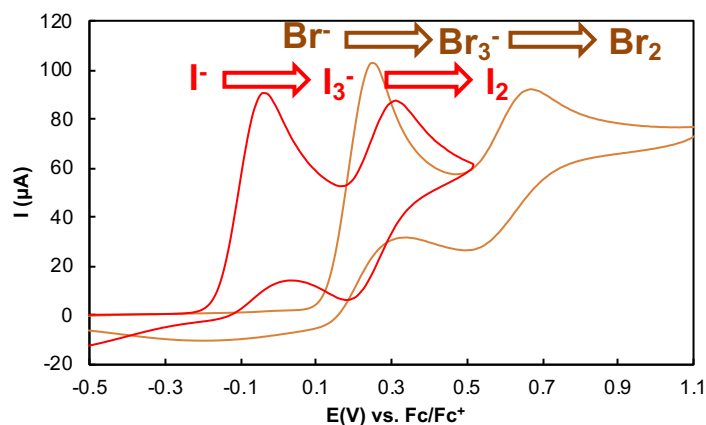


Figure S1. CVs of $n\text{Bu}_4\text{NI}$ (TBAI) and Et_4NBr (5 mM) in acetonitrile with KPF_6 (0.1 M) as supporting electrolyte, glassy carbon as working electrode ($\sim 7.0 \text{ mm}^2$) and a platinum wire (1.0 cm, spiral wire) as counter electrode, scan rate = 100 mV/s.

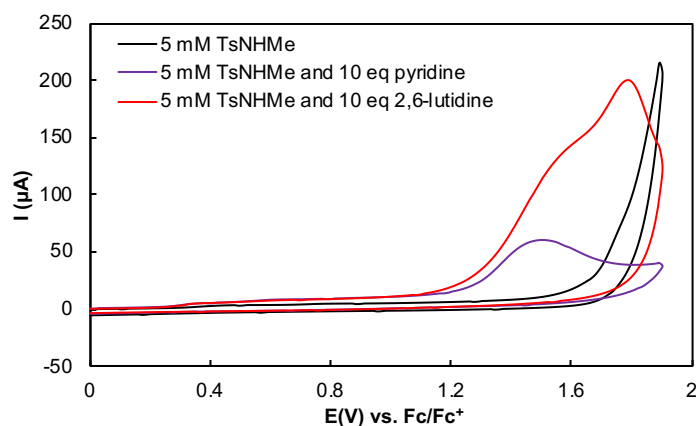
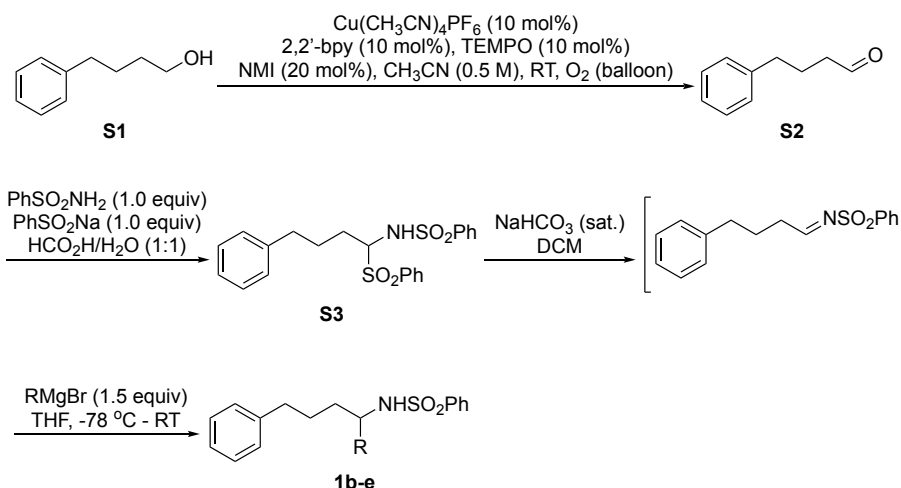


Figure S2. CVs of TsNHMe (5 mM) in acetonitrile with and without base (10 equiv), with $n\text{Bu}_4\text{NBF}_4$ (0.1 M) as supporting electrolyte, glassy carbon as working electrode ($\sim 7.0 \text{ mm}^2$) and a platinum wire (1.0 cm, spiral wire) as counter electrode, scan rate = 100 mV/s.

3. Procedures for Substrate Synthesis

3.1 Substrate synthesis procedure 1



The first step was conducted following the reported procedure.^[1] To a solution of **S1** (50 mmol) in dry CH_3CN in a 1 L flask were added $[\text{Cu}(\text{MeCN})_4]\text{PF}_6$ (5 mmol, 10 mol%), 2,2'-dipyridyl (5 mmol, 10 mol%), TEMPO (5 mmol, 10 mol%) and N-methyl imidazole (NMI, 10 mmol, 20 mol%). The solution was purged with oxygen for 5 min and fitted with a septum and a balloon of O_2 . The brown reaction mixture was stirred rapidly and monitored by thin layer chromatography (TLC) until no starting

material remained (the reaction color changed to blue). Upon completion, 3 mL of concentrated sulfuric acid was added into the reaction mixture to promote the disproportionation of TEMPO and further stirred for 20 min, simplifying the purification (TLC revealed that TEMPO and **S2** had similar R_f). The reaction mixture was then diluted with 1:1 ethyl acetate:hexane (200 mL) and filtered through a plug of silica. The filtrate was then concentrated and purified by silica column chromatography to give 6 g **S2** as light yellow oil (81%).

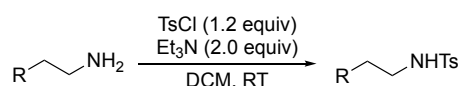
Steps 2-4 were conducted following the reported procedures.^[2]

Step 2: Aldehyde **S2** (40.0 mmol), benzenesulfonamide (40.0 mmol, 1.0 equiv) and sodium benzenesulfinate (40.0 mmol, 1.0 equiv) were stirred at room temperature in formic acid (70 mL) and water (70 mL) for 16 hrs. Precipitate was filtered and washed with water then hexane to give **S3** as a white solid, which was used directly for the next step.

Step 3: **S3** was dissolved in dichloromethane (100 mL) and stirred with a solution of aqueous saturated sodium bicarbonate (100 mL) for 1 hours. The organic layer was extracted and dried over sodium bicarbonate. Solvent was removed under reduced pressure to yield crude aldimine, which was used directly for the next step.

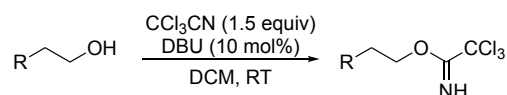
Step 4: Aryl-magnesium bromide (0.5 M in THF, 30 mL, 15 mmol) was added to a stirred solution of above crude aldimine (1/4 crude product of step 3) in THF (15 mL) at -78 °C for 2 hours. The solution was warmed to room temperature and stirred further at room temperature for 1 hour. The reaction was quenched with saturated ammonium chloride (30 mL) and extracted with ethyl acetate. The organic phases were combined, dried over sodium sulfate, and the solvent was evaporated under reduced pressure. The residue was purified via flash column chromatography and recrystallized from ethyl acetate and hexanes to yield sulfonamide **1b-e** (33%~52% yield after three steps).

3.2 Substrate synthesis procedure 2



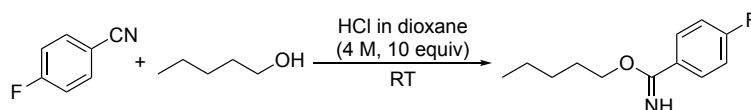
To the solution of amine (1.0 equiv) and tosyl chloride (1.2 equiv) in DCM was added Et₃N (2.0 equiv) dropwise at 0 °C cooling with ice bath. After that, the reaction mixture was warmed to room temperature and stirred overnight. Then the reaction mixture was washed with water (two times) and brine (once), and the aqueous solution was further extracted with DCM. The combined organic phases were dried over anhydrous MgSO₄, filtered, concentrated. The residue was purified via flash column chromatography to give the desired products **1a** and **1f-h**.

3.3 Substrate synthesis procedure 3



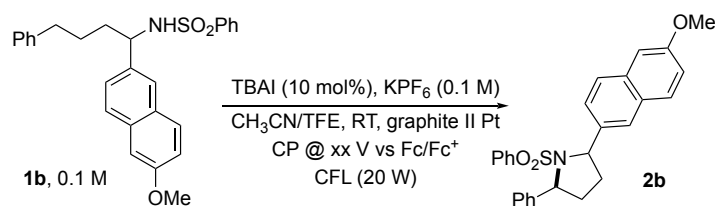
The reaction was conducted following the reported procedure.^[3] To a solution of alcohol (1.0 equiv, 0.1 M) in DCM was added trichloroacetonitrile (1.5 equiv) and DBU (0.1 equiv). The reaction was stirred at room temperature, and monitored by TLC until the alcohol was consumed. Upon completion, the mixture was concentrated and purified by silica gel chromatography (hexanes with 2% Et₃N to avoid imidate hydrolysis) to afford the desired products **3a-u**.

3.4 Substrate synthesis procedure 4



The reaction was conducted following the reported procedure.^[4] To a mixture of 1-pentyl alcohol (10 mmol, 1 equiv) and 4-fluorobenzonitrile (10 mmol, 1 equiv) in a well-sealed round-bottom flask was added 4 M HCl in dioxane (25 mL, 100 mmol, 10 equiv). The reaction mixture was stirred at room temperature for 24 hours. After that, it was concentrated and the residue was dissolved in dichloromethane, washed with sat. NaHCO₃ and brine. The organic layer was separated, dried with MgSO₄, filtered and concentrated in vacuo. The residue was purified by chromatography on silica gel (hexane/EtOAc = 10/1, with 2% Et₃N) to give the desired product **3v** in 51% yield as colorless oil.

4. Optimization of Reaction Conditions



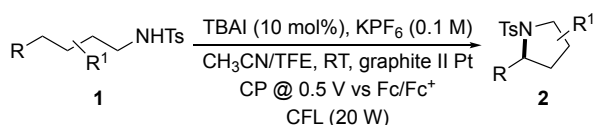
Entry	Potential/V vs Fc/Fc ⁺	TBAI	Yield/% ^a
1	0.3	10 mol%	4%
2	0.4	10 mol%	70%
3	0.5	10 mol%	75%
4	0.5	-	n.d.
5 ^b	0.5	10 mol%	19%
6 ^c	0.5	10 mol%	11%
7 ^d	0.5	10 mol%	72%
8 ^e	0.5	10 mol%	7%
9	0.5	5 mol%	18%
10 ^f	0.5	10 mol%	34%

Table S1. [a] The reaction was performed on a 0.5 mmol scale with CH₃CN/TFE (17:1) as co-solvent, ¹H NMR yield was shown with *m*-xylene as internal standard. [b] Without irradiation. [c] Without TFE. [d] With CH₃CN/TFE (34:1) as co-solvent. [e] With TBABF₄ as supporting electrolyte. [f] With CH₃CN/HFIP (12:1) as co-solvent. TFE = 2,2,2-Trifluoroethanol. HFIP = 1,1,1,3,3,3-Hexafluoro-2-propanol. n.d. = not detected.

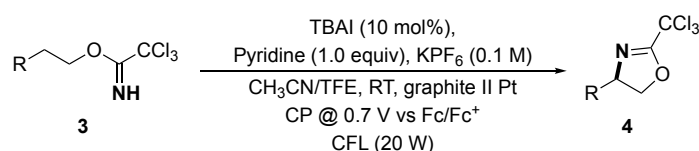
Entry	Potential/V vs Fc/Fc ⁺	Additive	TBAI	Yield/%
1	0.5	-	10 mol%	54%
2	0.3	-	10 mol%	47%
3	0.7	-	10 mol%	71%
4	0.7	pyridine (0.5 eq.)	10 mol%	78%
5	0.7	pyridine (1 eq.)	10 mol%	82%
6	0.7	pyridine (2 eq.)	10 mol%	74%
7	0.7	2,6-lutidine	10 mol%	62%
8 ^b	0.7	pyridine (1 eq.)	10 mol%	n.d.
9 ^c	0.7	pyridine (1 eq.)	10 mol%	75%
10 ^d	0.7	pyridine (1 eq.)	10 mol%	78%
11 ^e	0.7	pyridine (1 eq.)	10 mol%	36%
12 ^f	0.7	pyridine (1 eq.)	10 mol%	2%
13 ^g	0.7	pyridine (1 eq.)	10 mol%	3%
14	0.7	pyridine (1 eq.)	5 mol%	49%
15	0.7	pyridine (1 eq.)	2.5 mol%	23%
16	0.7	pyridine (1 eq.)	-	n.d.
17 ^h	0.2 - 1.0	pyridine (1 eq.)	10 mol%	72%
18 ⁱ	0.3 - 1.0	pyridine (1 eq.)	10 mol%	72%
19 ^j	0.7	pyridine (1 eq.)	10 mol%	79%

Table S2. [a] The reaction was performed on a 0.5 mmol scale with CH₃CN/TFE (17:1) as co-solvent, ¹H NMR yield was shown with *m*-xylene as internal standard. [b] Without TFE. [c] With CH₃CN/TFE (34:1) as co-solvent. [d] With CH₃CN/HFIP (12:1) as co-solvent. [e] With CH₃CN/MeOH (31:1) as co-solvent. [f] Without irradiation [g] Under ambient light conditions. [h] Constant current @ 5 mA for 2.5 F/mol. [i] Constant current @ 10 mA for 2.5 F/mol. [j] With RVC as working electrode (30 PPI, 0.635*1*2 cm³, ~ 24 cm²) instead of graphite rod, 4.8 h, initial current ~ 20 mA. TFE = 2,2,2-Trifluoroethanol. HFIP = 1,1,1,3,3,3-Hexafluoro-2-propanol. n.d. = not detected.

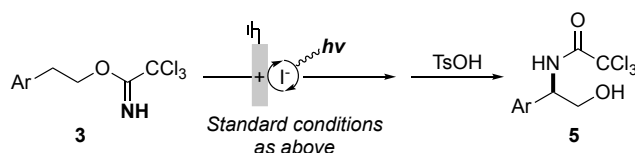
5. Procedures for Electrolysis



General procedure A for pyrrolidine synthesis: Reactions were carried out using an undivided cell equipped with a graphite anode (0.6 cm OD x 6 cm L, ~ 3 cm was immersed in the solution, apparent area ~ 6 cm²), a platinum wire cathode (1.0 cm, spiral wire) and a Ag/Ag⁺ reference electrode. A mixture of **1** (0.5 mmol), KPF₆ (0.5 mmol) as supporting electrolyte, TBAI (0.05 mmol), and TFE (8.0 equiv) in acetonitrile (5 mL) was purged with nitrogen for 20 min, and then electrolyzed at 0.5 V vs Fc/Fc⁺ with magnetic stirring. The mixture was irradiated with a CFL (20 W) during bulk electrolysis. The reaction was stopped automatically when the current decreased to 1 mA or manually after full consumption of substrates was detected by TLC. After that, solvent was removed under reduced pressure to leave a residue. The reaction was analyzed by crude ¹H NMR spectroscopy with *m*-xylene as internal standard, followed by chromatography on silica gel to afford the pure products **2**.



General procedure B for oxazoline synthesis: Reactions were carried out using an undivided cell equipped with a graphite anode (0.6 cm OD x 6 cm L, ~ 3 cm was immersed in the solution apparent area ~ 6 cm²), a platinum wire cathode (1.0 cm, spiral wire) and a Ag/Ag⁺ reference electrode. A mixture of **3** (0.5 mmol), KPF₆ (0.5 mmol) as supporting electrolyte, TBAI (0.05 mmol), pyridine (0.5 mmol), and TFE (8.0 equiv) in acetonitrile (5 mL) was purged with nitrogen for 20 min, and then electrolyzed at 0.7 V vs Fc/Fc⁺ with magnetic stirring. The mixture was irradiated with CFL (20 W) during bulk electrolysis. The reaction was stopped automatically when the current decreased to 0.5 mA or manually after full consumption of substrates was detected by TLC. After that, solvent was removed under reduced pressure to leave a residue. The reaction was analyzed by crude ¹H NMR spectroscopy with *m*-xylene as internal standard, followed by chromatography on silica gel to afford the pure products **4**.



General procedure C for amino alcohol synthesis: The first step was the same as **general procedure B**, after which the reaction mixture was evaporated under reduced pressure to remove the volatiles. The residue was dissolved in CH₃CN (3 mL), and then *p*-toluenesulfonic acid monohydrate (5.0 equiv), was added and the resulting mixture was stirred at room temperature for 1 h. Then, the

solvent was removed under reduced pressure to leave a residue, which was purified by chromatography on silica gel to afford the pure products **5**.

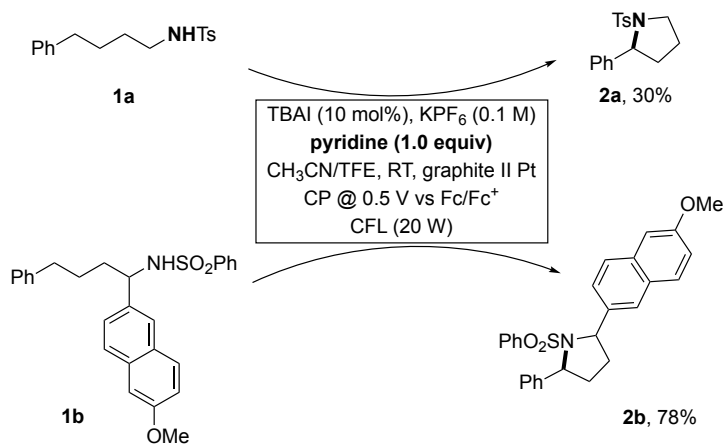
Note 1: The oxazoline products can undergo partial decomposition during column chromatography on silica gel, so Et₃N was added to the eluent.

Note 2: The outer layer of the carbon electrode should be removed with a razor blade or sand paper if it has been used previously.

Note 3: Good yield could be obtained under constant current conditions (Table **S2**, entries 17-18), so a simpler two-electrode set-up should also work.

Note 4: As shown in Table **S2**, inclusion of pyridine contributed to the better yield for the reactions of imidates. There are two possibilities to explain this phenomenon: 1) pyridine acts as base to facilitate the *N*-iodo intermediate formation; 2) given the favorable interaction between iodine and pyridine derivatives,^[5] pyridine might act as iodine activator rather than a simple base since a stronger base (CF₃CH₂O⁻) is furnished at the cathodic side.

Note 5: We noted that the inclusion of pyridine did not give any obvious improvement, and may even lead to inferior yield, for the reaction of sulfonamide substrates (see below). For example, comparable yield was obtained for **2b**, while lower yield and mass balance was observed in the reaction of **1a**, possibly a result of pyridine-promoted beta-elimination of HI.



Note 6: The reaction was run under constant potential, so that current (density) would decrease with the consumption of substrate. A typical plot of current vs time is shown below.

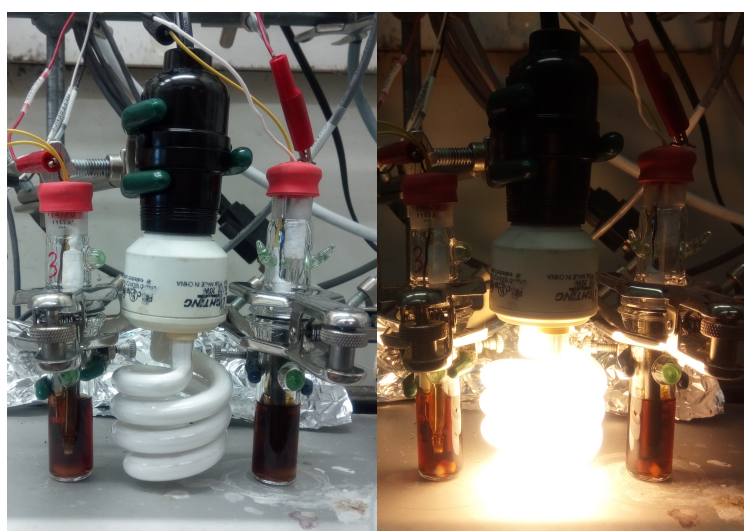
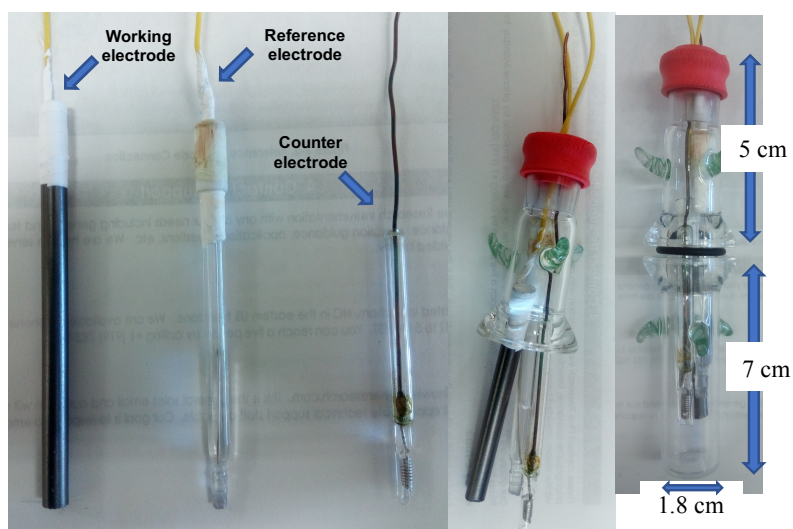
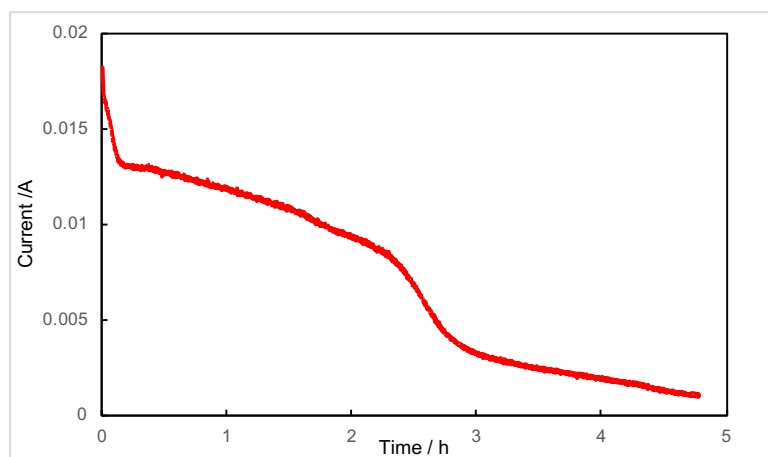


Figure S3. Reaction set-up (undivided cell).


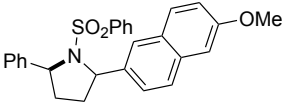
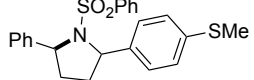
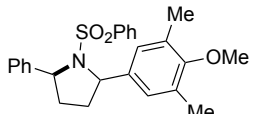
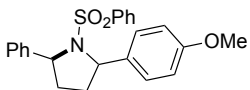

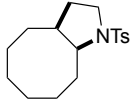
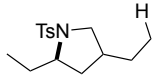
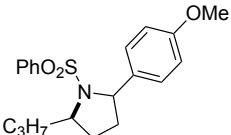
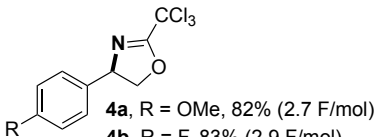
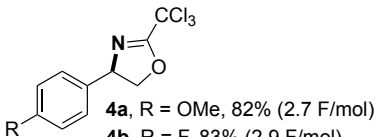
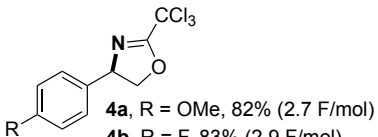
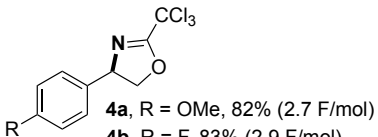
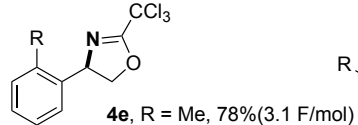
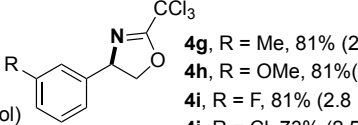
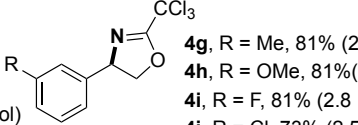
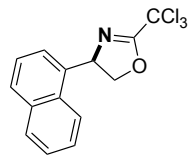
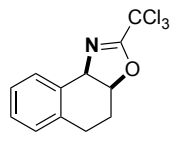
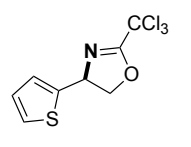
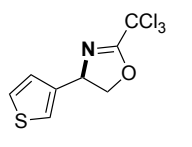
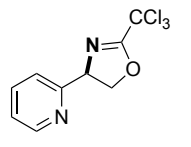
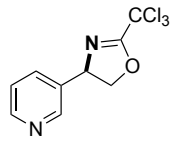
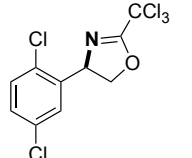
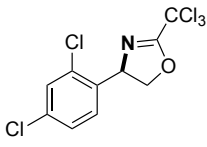
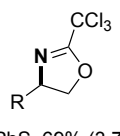
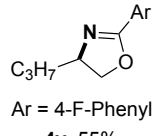
1H NMR yield (Consumed charge)				
				
2a , 72% (5.7 F/mol)	2b , 75% (4.1 F/mol)	2c , 67% (2.9 F/mol)	2d , 81% (3.1 F/mol)	
				
2e , 85% (8.4 F/mol)	2f , 59% (3.6 F/mol)	2g , 43% (2.8 F/mol)	2h , 74% (8.6 F/mol)	2i , 61% (8.1 F/mol)
				
4a , R = OMe, 82% (2.7 F/mol)	4b , R = F, 83% (2.9 F/mol)	4c , R = H, 92% (2.6 F/mol)	4d , R = Cl, 69% (2.5 F/mol)	4e , R = Me, 78% (3.1 F/mol)
				
				4f , R = Cl, 87% (2.7 F/mol)
				
				4g , R = Me, 81% (2.7 F/mol)
				4h , R = OMe, 81% (2.5 F/mol)
				4i , R = F, 81% (2.8 F/mol)
				4j , R = Cl, 73% (2.5 F/mol)
				4k , R = CF ₃ , 63% (1.9 F/mol)
				
4l , 89% (2.7 F/mol)	4m , 78% (2.1 F/mol)	4n , 68% (2.8 F/mol)	4o , 90% (2.6 F/mol)	4p , 89% (2.7 F/mol)
				
4q , 83% (2.4 F/mol)	4r , 78% (2.5 F/mol)	4s , 76% (2.7 F/mol)	4t , R = PhS, 69% (3.7 F/mol)	4v , 55% (3.8 F/mol)
			4u , R = MeO, 74% (2.9 F/mol)	Ar = 4-F-Phenyl

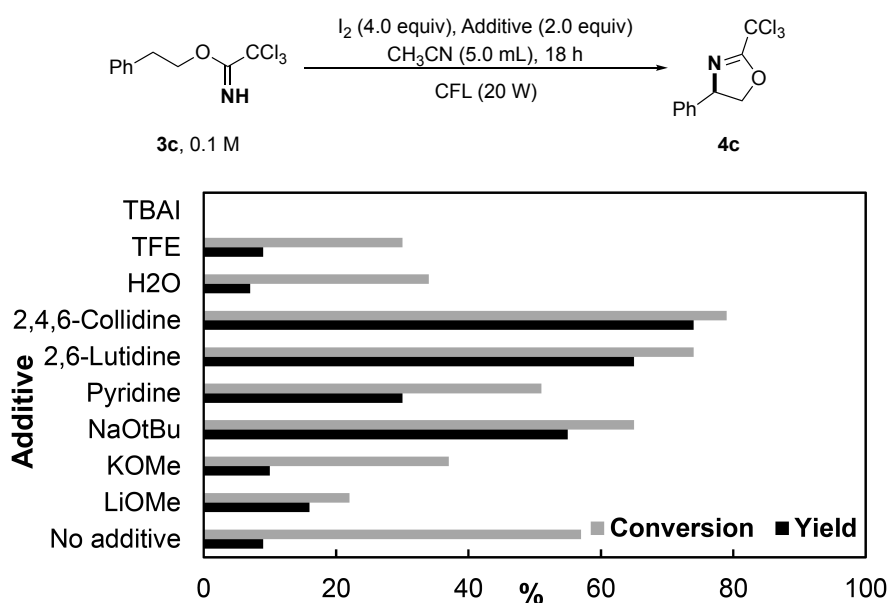
Table S3. Comparison of ¹H NMR yields and consumed charge. Note: Isolated yields are provided in Table 3 of the manuscript and in Section 9.2 below.

6. Experiments with Stoichiometric Iodine as Oxidant

To a glass vial (6 mL) were added substrate **1a** or **3c** (0.5 mmol), I₂ (2.0 mmol, 4.0 equiv), additive/base (1.0 mmol, 2.0 equiv) and CH₃CN (5.0 mL). The resulting mixture was stirred with irradiation by CFL (20 W) at room temperature. After 18h, the reaction was detected by ¹H NMR with *m*-xylene as internal standard and the results were shown below (Table S4).

<chem>Ph-CH2-CH2-CH2-NHTs</chem> 1a , 0.1 M		I_2 (4.0 equiv), Base (2.0 equiv) CH_3CN (5 mL) CFL (20 W)		<chem>Ph-CH2-CH2-CH2-N(Ts)C1CCCC1</chem> 2a		
Base:	wo base	KOMe	LiOMe	Pyridine	2,6-Lutidine	2,4,6-Collidine
Yield: (conv.)	n.d. (< 3%)	16% (22%)	12% (16%)	10% (10%)	30% (42%)	21% (37%)

Table S4. Reactions of **1a** with iodine as oxidant. The reaction was performed on a 0.5 mmol scale, 1H NMR yield was shown with *m*-xylene as internal standard, conversion of **1a** shown in parenthesis.



Scheme S1. Reactions of **3c** with iodine as oxidant. The reaction was performed on a 0.5 mmol scale, yield and conversion were determined by 1H NMR with *m*-xylene as internal standard.

7. Intermolecular Additive Screening

These reactions were conducted following **General Procedure A**. A mixture of **1a** (0.5 mmol), KPF₆ (0.5 mmol) as supporting electrolyte, TBAI (0.05 mmol), additive (0.5 mmol, 1.0 equiv) and TFE (8.0 equiv) in acetonitrile (5 mL) was purged with nitrogen for 20 min, and then electrolyzed at 0.5 V vs Fc/Fc⁺ with magnetic stirring. The mixture was irradiated with CFL (20 W) during bulk electrolysis. The reaction was stopped automatically when the current decreased to 1 mA or manually after full consumption of **1a** was detected by TLC. After that, *m*-xylene (0.5 mmol) was added into the reaction mixture, an aliquot (~ 0.2 mL) was removed and diluted with CD₃CN (0.4 mL), which was then analyzed by crude ¹H NMR spectroscopy.

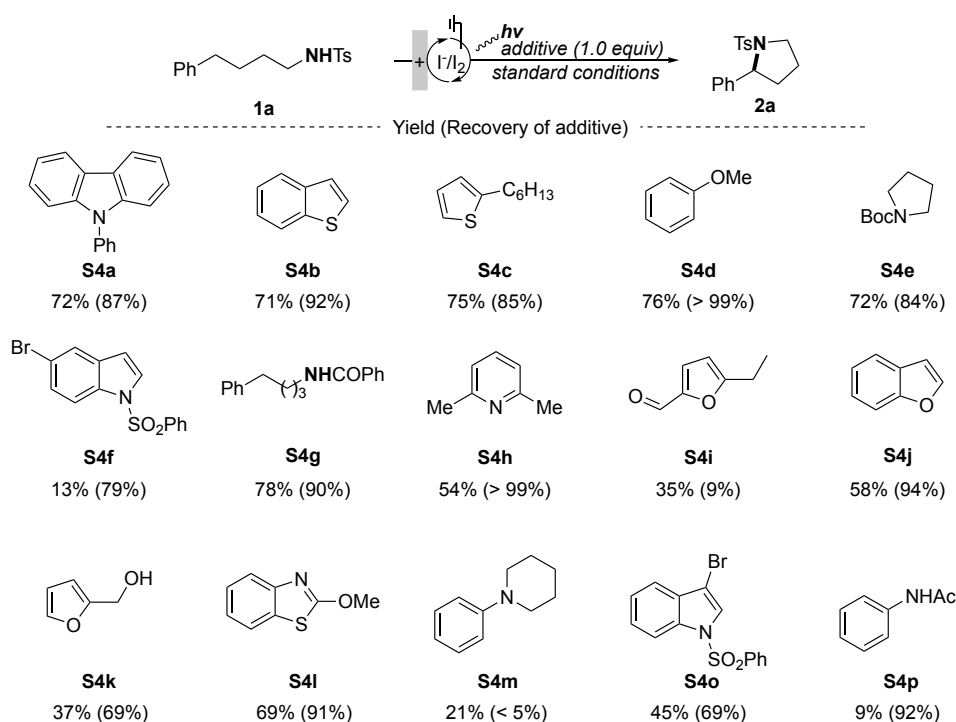


Table S5. Intermolecular additive screen.

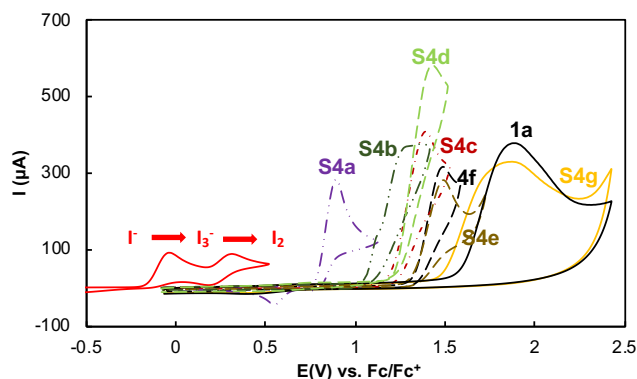
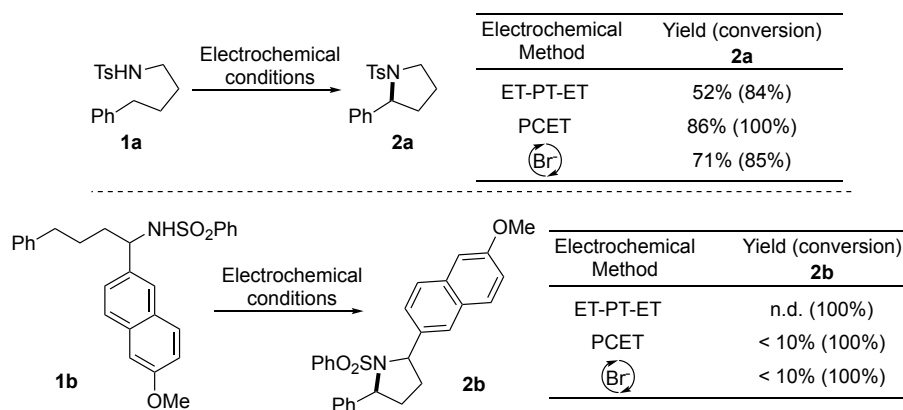


Figure S4. CVs of TBAI, **1a** and **S4a-S4f** (5 mM) in acetonitrile with KPF₆ (0.1 M) as supporting electrolyte, scan rate = 100 mV/s.

8. Control Experiments under Reported Conditions



Scheme S2. Comparison of different strategies.

8.1 Halide-mediated electrochemical dehydrogenative amination^[6]

A solution of **1a** or **1b** (0.4 mmol), NaOMe (10.8 mg, 0.2 mmol), and KBr (23.8 mg, 0.2 mmol) in methanol (6 mL) was placed in a vial (10 mL) equipped with two platinum electrodes and a magnetic stirbar. Anodic oxidation was carried out while being heated externally at 65°C under conditions of constant current (100 mA). After 3 hrs (28 F/mol), the reaction was detected by ¹H NMR with *m*-xylene as internal standard. The results shown in Scheme S2 demonstrated that the bromide-mediated system worked well for the simple substrate **1a**, while poor yield and mass balance were observed with **1b**, containing electron-rich aromatic ring. Meanwhile, this system is not applicable to imidate in that the substrate will undergo decomposition under these conditions (Figure S5). All these results indicated the limitation in Shono's conditions.

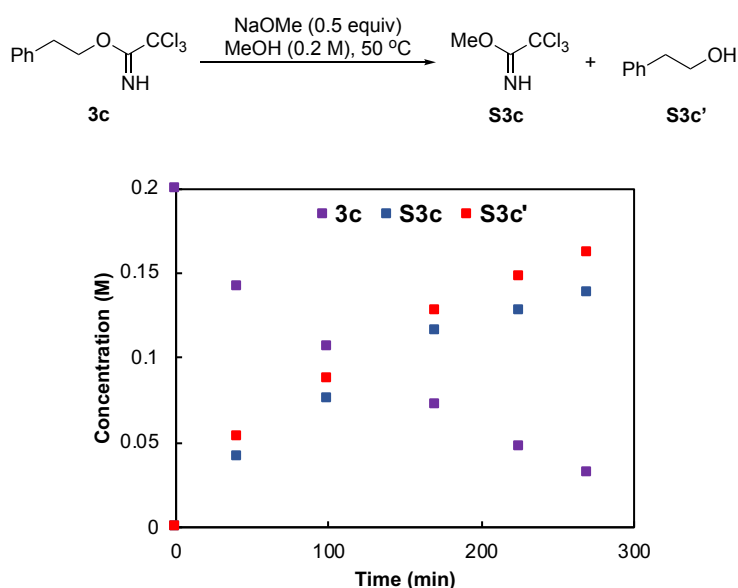


Figure S5. Time course for decomposition of **3c** under Shono's conditions

8.2 Direct electrolysis^[7]

A solution of **1a** or **1b** (0.2 mmol) and ⁿBu₄NBF₄ (329 mg, 1 mmol) in 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP, 10 mL) was electrolyzed under constant current (2.5 mA) at room temperature with graphite as anode and platinum wire as cathode. After 4.7 hrs (2.2 F/mol), the reaction was detected by ¹H NMR with *m*-xylene as internal standard and the results are shown in Scheme S2. The anode potential trace during electrolysis was 1.7-1.8 V vs Fc/Fc⁺ (Figure S6).

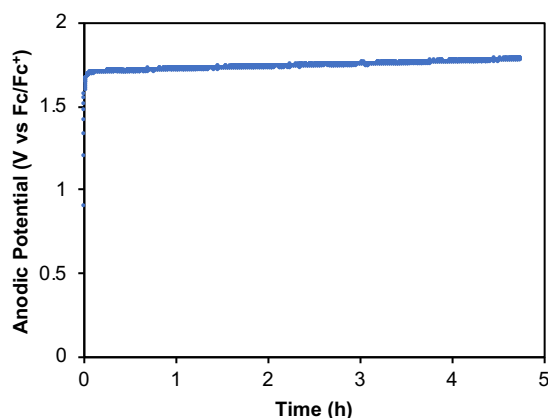


Figure S6. Anode potential trace during reaction of **1a** under ET-PT-ET conditions

An intermolecular additive experiment was also performed by testing the cyclization of **1a** with anisole as an additive (Figure S7). The result shown below revealed that oxidation of anisole happened exclusively without consumption of **1a**, which further reflected the poor functional group tolerance of this method.

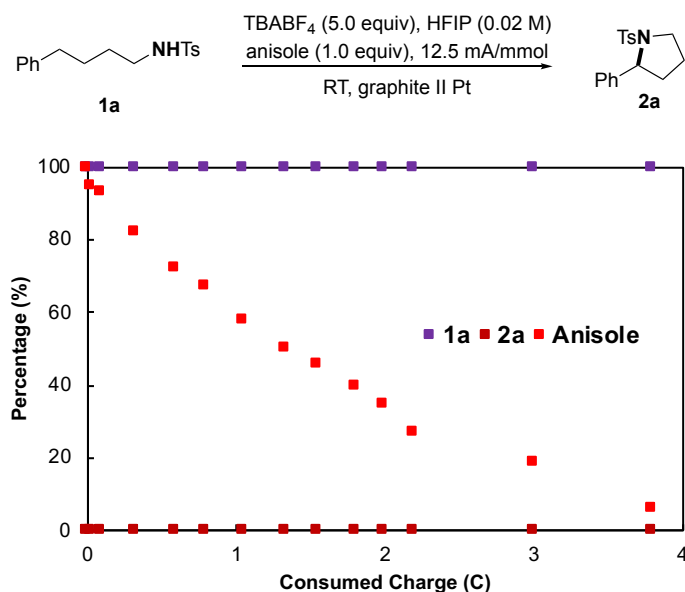


Figure S7. Time course of intermolecular additive screen under direct electrolysis

8.3 Acetate-promoted electrolysis^[8]

A solution of **1a** or **1b** (0.2 mmol), NaOAc (0.2 mmol) and ⁿBu₄NBF₄ (0.2 mmol) in a 2:1 mixture of DCE/HFIP (6 mL) was electrolyzed under constant current (7.5 mA) at room temperature with graphite as anode and platinum wire as cathode. After 4 hrs (5.6 F/mol), the reaction was detected by ¹H NMR with *m*-xylene as internal standard. The results shown in Scheme S2 demonstrated that this base-promoted electrolysis protocol still has functional group tolerance issues because of the high anode potential (Figure S8).

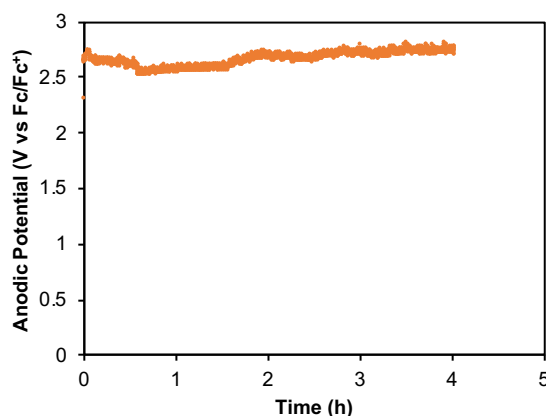
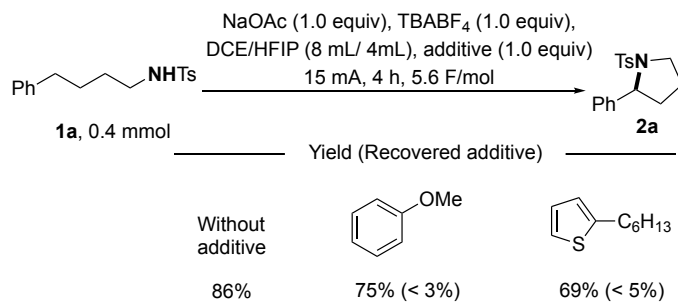


Figure S8. Anode potential trace during reaction of **1a** under PCET conditions

The intermolecular additive screen experiments were also conducted under the PCET conditions with anisole and **S4c** as additives, respectively, however, none of these additives could be recovered after bulk electrolysis (scheme S3).



Scheme S3. Intermolecular additive screen under OAc-promoted conditions

The time course of the reaction showed that anisole and substrate **1a** underwent oxidation simultaneously, where decomposition of anisole was faster than conversion of **1a** (Figure S9). This result revealed that this PCET protocol could shift the anodic potential negatively compared with ET-PT-ET method. However, such an improvement was not sufficient to exhibit good functional group tolerance.

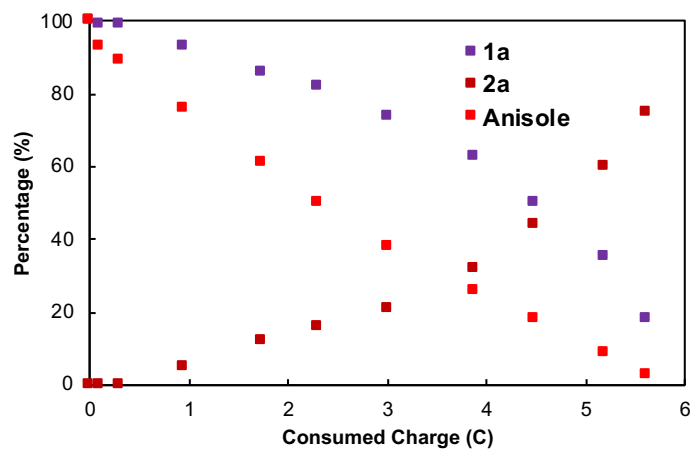
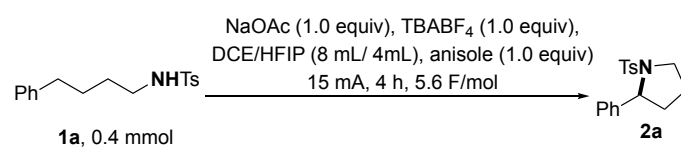
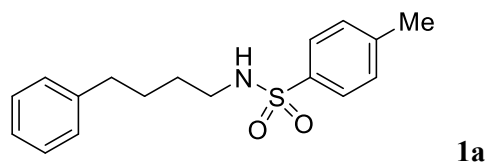


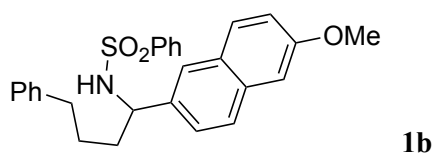
Figure S9. Time course of intermolecular additive screen under OAc-promoted conditions

9. Substrate and Product Characterization Data

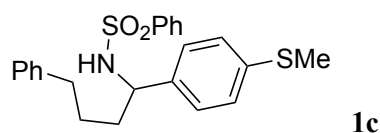
9.1 Characterization of substrates



Compound **1a** was prepared according to substrate synthesis procedure 2 and the spectral data are available in the literature.^[9] **¹H NMR** (500 MHz, CDCl₃) δ = 7.66 (d, J = 8.2 Hz, 2H), 7.19 (d, J = 8.1 Hz, 2H), 7.15 (t, J = 7.4 Hz, 2H), 7.07 (t, J = 7.3 Hz, 1H), 7.00 (d, J = 7.2 Hz, 2H), 4.80 (t, J = 6.0 Hz, 1H), 2.84 (q, J = 6.6 Hz, 2H), 2.45 (t, J = 7.6 Hz, 2H), 2.32 (s, 3H), 1.49 (dt, J = 12.1, 7.2 Hz, 2H), 1.43 – 1.35 ppm (m, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 143.2, 141.8, 136.8, 129.6, 128.3, 128.2, 127.0, 125.7, 42.9, 35.1, 29.0, 28.1, 21.4 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 304.1366, measured: 304.1362.

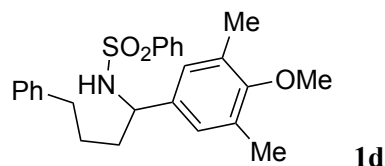


Compound **1b** was prepared according to substrate synthesis procedure 1. **¹H NMR** (400 MHz, CDCl₃) δ = 7.62 – 7.56 (m, 2H), 7.50 (t, J = 8.1 Hz, 2H), 7.31 – 7.25 (m, 2H), 7.22 (t, J = 7.3 Hz, 2H), 7.19 – 7.12 (m, 3H), 7.10 (dd, J = 8.9, 2.5 Hz, 1H), 7.07 – 7.00 (m, 4H), 5.02 (d, J = 7.4 Hz, 1H), 4.45 (q, J = 7.4 Hz, 1H), 3.90 (s, 3H), 2.55 (t, J = 7.6 Hz, 2H), 1.96 – 1.73 (m, 2H), 1.68 – 1.55 (m, 1H), 1.54 – 1.40 ppm (m, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ = 157.7, 141.6, 140.5, 135.3, 133.8, 132.1, 129.2, 128.5, 128.4, 128.34, 128.29, 127.3, 126.9, 125.8, 125.7, 124.4, 119.0, 105.5, 58.4, 55.3, 36.8, 35.2, 27.6 ppm. **HRMS (ESI)** Calculated for [M+NH₄⁺]: 463.2050, measured: 463.2046.

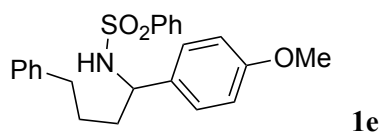


Compound **1c** was prepared according to substrate synthesis procedure 1. **¹H NMR** (400 MHz, CDCl₃) δ 7.65 – 7.59 (m, 2H), 7.42 (t, J = 7.4 Hz, 1H), 7.29 (t, J = 7.8 Hz, 2H), 7.22 (t, J = 7.3 Hz, 2H), 7.15 (t, J = 7.3 Hz, 1H), 7.04 (d, J = 7.1 Hz, 2H), 6.98 (d, J = 8.3 Hz, 2H), 6.87 (d, J = 8.3 Hz, 2H), 5.26 (d, J = 7.5 Hz, 1H), 4.27 (q, J = 7.3 Hz, 1H), 2.51 (t, J = 7.5 Hz, 2H), 2.40 (s, 3H), 1.78 (tdd, J = 10.0, 7.0,

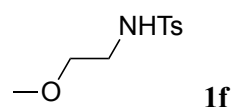
5.2 Hz, 1H), 1.72 – 1.50 (m, 2H), 1.49 – 1.35 ppm (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 141.6, 140.6, 137.5, 137.4, 132.1, 128.7, 128.29, 128.27, 126.97, 126.91, 126.5, 125.8, 57.8, 36.8, 35.1, 27.5, 15.8 ppm. HRMS (ESI) Calculated for $[\text{M}+\text{NH}_4^+]$: 429.1665, measured: 429.16653.



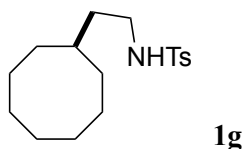
Compound **1d** was prepared according to substrate synthesis procedure 1. ^1H NMR (400 MHz, CDCl_3) δ = 7.66 – 7.58 (m, 2H), 7.34 (t, J = 7.4 Hz, 1H), 7.26 – 7.16 (m, 4H), 7.12 (t, J = 7.3 Hz, 1H), 7.04 (d, J = 7.1 Hz, 2H), 6.53 (s, 2H), 5.71 (d, J = 8.0 Hz, 1H), 4.20 (q, J = 7.6 Hz, 1H), 3.58 (s, 3H), 2.49 (t, J = 7.3 Hz, 2H), 2.05 (s, 6H), 1.82 – 1.70 (m, 1H), 1.69 – 1.53 (m, 2H), 1.53 – 1.36 ppm (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ = 155.7, 141.7, 140.8, 135.5, 131.8, 130.3, 128.23, 128.15, 128.11, 126.85, 126.83, 125.6, 59.4, 57.9, 36.7, 35.1, 27.5, 15.8 ppm. HRMS (ESI) Calculated for $[\text{M}+\text{NH}_4^+]$: 441.2206, measured: 441.2203.



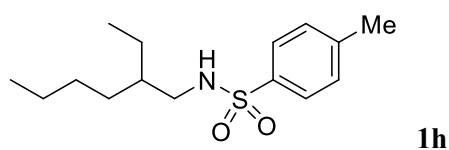
Compound **1e** was prepared according to substrate synthesis procedure 1. ^1H NMR (400 MHz, CDCl_3) δ = 7.63 (d, J = 7.8 Hz, 2H), 7.40 (t, J = 7.3 Hz, 1H), 7.28 (t, J = 7.7 Hz, 2H), 7.21 (t, J = 7.4 Hz, 2H), 7.14 (t, J = 7.2 Hz, 1H), 7.04 (d, J = 7.3 Hz, 2H), 6.87 (d, J = 8.5 Hz, 2H), 6.62 (d, J = 8.5 Hz, 2H), 5.37 (d, J = 7.5 Hz, 1H), 4.25 (q, J = 7.3 Hz, 1H), 3.71 (s, 3H), 2.50 (t, J = 7.5 Hz, 2H), 1.87 – 1.73 (m, 1H), 1.73 – 1.61 (m, 1H), 1.61 – 1.35 ppm (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ = 158.7, 141.7, 140.7, 132.7, 132.0, 128.6, 128.28, 128.22, 127.6, 126.9, 125.7, 113.7, 57.7, 55.2, 36.9, 35.2, 27.6 ppm. HRMS (ESI) Calculated for $[\text{M}+\text{NH}_4^+]$: 413.1893, measured: 413.1888.



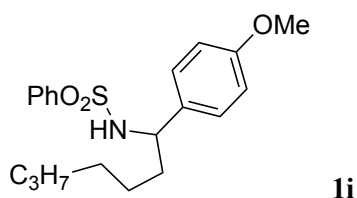
Compound **1f** was prepared according to substrate synthesis procedure 2. ^1H NMR (500 MHz, CDCl_3) δ = 7.72 (d, J = 8.2 Hz, 2H), 7.28 (d, J = 8.1 Hz, 2H), 5.02-5.11 (m, 1H), 3.37 (t, J = 5.1 Hz, 2H), 3.23 (s, 3H), 3.08 (t, J = 5.1 Hz, 2H), 2.39 ppm (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ = 143.3, 136.8, 129.6, 127.0, 70.4, 58.6, 42.7, 21.4 ppm. HRMS (ESI) Calculated for $[\text{M}-\text{H}]^-$: 228.0700, measured: 228.0703.



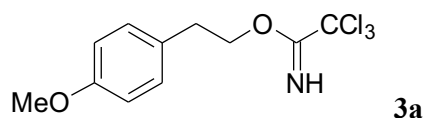
Compound **1g** was prepared according to substrate synthesis procedure 2. $^1\text{H NMR}$ (500 MHz, CDCl_3) $\delta = 7.75$ (d, $J = 8.3$ Hz, 2H), 7.30 (d, $J = 8.0$ Hz, 2H), 4.81 – 4.72 (m, 1H), 2.92 (dt, $J = 7.6, 6.3$ Hz, 2H), 2.41 (s, 3H), 1.64 – 1.29 (m, 15H), 1.21 – 1.12 ppm (m, 2H). $^{13}\text{C NMR}$ (125 MHz, CDCl_3) $\delta = 143.2, 136.9, 129.6, 127.1, 41.4, 37.5, 34.3, 31.9, 27.1, 26.1, 25.1, 21.5$ ppm. **HRMS (ESI)** Calculated for $[\text{M}-\text{H}]^-$: 308.1690, measured: 308.1691.



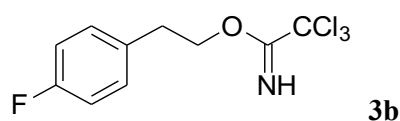
Compound **1h** was prepared according to substrate synthesis procedure 2 and the spectral data are available in the literature.^[10] $^1\text{H NMR}$ (500 MHz, CDCl_3) $\delta = 7.70$ (d, $J = 8.2$ Hz, 2H), 7.22 (d, $J = 8.2$ Hz, 2H), 5.06 (t, $J = 6.2$ Hz, 1H), 2.74 (t, $J = 6.1$ Hz, 2H), 2.33 (s, 3H), 1.33 – 1.01 (m, 9H), 0.74 (t, $J = 7.2$ Hz, 3H), 0.69 ppm (t, $J = 7.4$ Hz, 3H). $^{13}\text{C NMR}$ (125 MHz, CDCl_3) $\delta = 143.0, 136.9, 129.5, 127.0, 45.6, 39.0, 30.4, 28.5, 23.7, 22.7, 21.3, 13.9, 10.5$ ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}]^+$: 284.1679, measured: 284.1675.



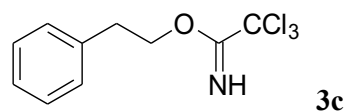
Compound **1i** was prepared according to substrate synthesis procedure 1. $^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta = 7.67 - 7.61$ (m, 2H), 7.44 (t, $J = 7.4$ Hz, 1H), 7.33 (t, $J = 7.7$ Hz, 2H), 6.91 (d, $J = 8.6$ Hz, 2H), 6.66 (d, $J = 8.7$ Hz, 2H), 4.86 (d, $J = 7.1$ Hz, 1H), 4.23 (q, $J = 7.3$ Hz, 1H), 3.74 (s, 3H), 1.81 – 1.58 (m, 2H), 1.28 – 1.01 (m, 8H), 0.83 ppm (t, $J = 7.0$ Hz, 3H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 158.8, 140.8, 132.9, 132.1, 128.6, 127.6, 127.0, 113.8, 57.9, 55.2, 37.5, 31.5, 28.7, 25.8, 22.5, 14.0$ ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{NH}_4]^+$: 379.2050, measured: 379.2046.



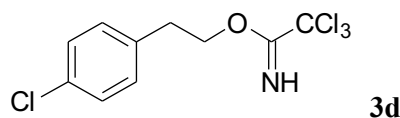
Compound **3a** was prepared according to substrate synthesis procedure 3 and the spectral data are available in the literature.^[3] **¹H NMR** (500 MHz, CDCl₃) δ = 8.28 (bs, 1H), 7.20 (d, J = 8.5 Hz, 2H), 6.85 (d, J = 8.5 Hz, 2H), 4.46 (t, J = 6.8 Hz, 2H), 3.79 (s, 3H), 3.03 ppm (t, J = 6.9 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.8, 158.3, 130.0, 129.6, 113.8, 91.4, 70.1, 55.2, 33.8 ppm.



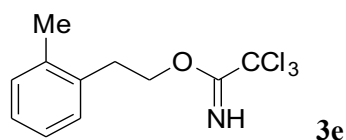
Compound **3b** was prepared according to substrate synthesis procedure 3 and the spectral data are available in the literature.^[3] **¹H NMR** (500 MHz, CDCl₃) δ = 8.29 (bs, 1H), 7.27 – 7.20 (m, 2H), 7.01 – 6.95 (m, 2H), 4.46 (t, J = 6.8 Hz, 2H), 3.05 ppm (t, J = 6.8 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.7, 161.7 (d, J = 244.3 Hz), 133.3 (d, J = 3.2 Hz), 130.5 (d, J = 7.9 Hz), 115.2 (d, J = 21.2 Hz), 91.3, 69.7 (d, J = 1.0 Hz), 33.9 ppm. **¹⁹F NMR** (377 MHz, CDCl₃) δ -116.47 ppm (s).



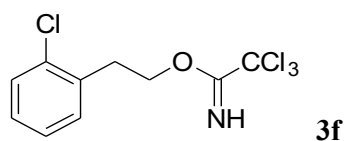
Compound **3c** was prepared according to substrate synthesis procedure 3 and the spectral data are available in the literature.^[3] **¹H NMR** (400 MHz, CDCl₃) δ = 8.28 (bs, 1H), 7.33 – 7.19 (m, 5H), 4.48 (t, J = 6.9 Hz, 2H), 3.08 ppm (t, J = 6.9 Hz, 2H). **¹³C NMR** (100 MHz, CDCl₃) δ = 162.7, 137.6, 129.0, 128.4, 126.6, 91.4, 69.8, 34.6 ppm.



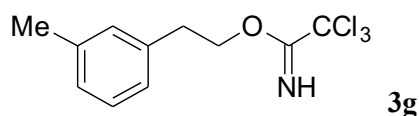
Compound **3d** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.21 (bs, 1H), 7.17 (d, J = 8.5 Hz, 2H), 7.11 (d, J = 8.4 Hz, 2H), 4.37 (t, J = 6.7 Hz, 2H), 2.95 ppm (t, J = 6.7 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.5, 136.1, 132.3, 130.3, 128.5, 91.3, 69.4, 34.0 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 299.9511, measured: 299.9509.



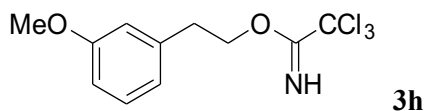
Compound **3e** was prepared according to substrate synthesis procedure 3 and the spectral data are available in the literature.^[3] **¹H NMR** (500 MHz, CDCl₃) δ = 8.29 (bs, 1H), 7.24 – 7.18 (m, 1H), 7.18 – 7.11 (m, 3H), 4.46 (t, *J* = 7.2 Hz, 2H), 3.09 (t, *J* = 7.2 Hz, 2H), 2.37 ppm (s, 3H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.8, 136.5, 135.6, 130.2, 129.7, 126.7, 126.0, 91.4, 69.1, 31.8, 19.4 ppm.



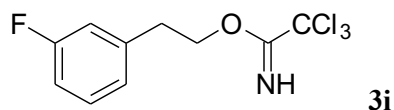
Compound **3f** was prepared according to substrate synthesis procedure 3 and the spectral data are available in the literature.^[3] **¹H NMR** (500 MHz, CDCl₃) δ = 8.30 (s, 1H), 7.40 – 7.35 (m, 1H), 7.35 – 7.31 (m, 1H), 7.23 – 7.15 (m, 2H), 4.53 (t, *J* = 6.7 Hz, 2H), 3.24 ppm (t, *J* = 6.7 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.7, 135.2, 134.2, 131.4, 129.5, 128.1, 126.7, 91.3, 68.1, 32.4 ppm.



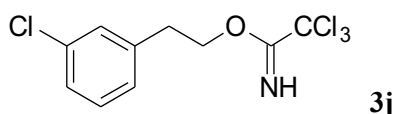
Compound **3g** was prepared according to substrate synthesis procedure 3 and the spectral data are available in the literature.^[3] **¹H NMR** (500 MHz, CDCl₃) δ = 8.29 (bs, 1H), 7.20 (t, *J* = 7.5 Hz, 1H), 7.11 (s, 1H), 7.08 (d, *J* = 7.6 Hz, 1H), 7.06 (d, *J* = 7.5 Hz, 1H), 4.49 (t, *J* = 7.0 Hz, 2H), 3.06 (t, *J* = 7.0 Hz, 2H), 2.34 ppm (s, 3H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.8, 138.0, 137.5, 129.9, 128.3, 127.3, 126.0, 91.5, 69.9, 34.6, 21.3 ppm.



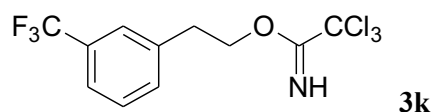
Compound **3h** was prepared according to substrate synthesis procedure 3 and the spectral data are available in the literature.^[3] **¹H NMR** (500 MHz, CDCl₃) δ = 8.29 (bs, 1H), 7.20 (t, *J* = 7.9 Hz, 1H), 6.87 – 6.80 (m, 2H), 6.77 (dd, *J* = 8.2, 2.5 Hz, 1H), 4.48 (t, *J* = 6.9 Hz, 2H), 3.77 (s, 3H), 3.05 ppm (t, *J* = 6.9 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.6, 159.6, 139.1, 129.3, 121.3, 114.6, 112.0, 91.4, 69.7, 55.0, 34.6 ppm.



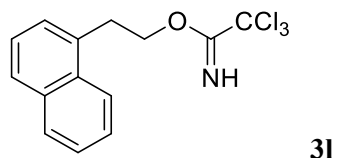
Compound **3i** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.22 (bs, 1H), 7.18 – 7.13 (m, 1H), 6.95 (d, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 9.8 Hz, 1H), 6.83 (td, *J* = 8.5, 2.4 Hz, 1H), 4.40 (t, *J* = 6.7 Hz, 2H), 2.98 (t, *J* = 6.7 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.8 (d, *J* = 245.6 Hz), 162.6, 140.1 (d, *J* = 7.4 Hz), 129.8 (d, *J* = 8.3 Hz), 124.6 (d, *J* = 2.8 Hz), 116.0 (d, *J* = 21.2 Hz), 113.5 (d, *J* = 21.0 Hz), 91.3, 69.3, 34.3 (d, *J* = 1.7 Hz). **¹⁹F NMR** (377 MHz, CDCl₃) δ = -113.37 ppm (s). **HRMS (ESI)** Calculated for [M+H⁺]: 283.9807, measured: 283.9803.



Compound **3j** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.34 (bs, 1H), 7.32 (s, 1H), 7.29 – 7.21 (m, 2H), 7.21 – 7.15 (m, 1H), 4.50 (t, *J* = 6.7 Hz, 2H), 3.09 ppm (t, *J* = 6.7 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.6, 139.6, 134.1, 129.6, 129.3, 127.2, 126.8, 91.3, 69.3, 34.3 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 299.9511, measured: 299.9508.

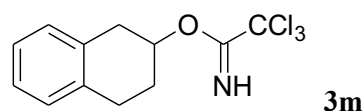


Compound **3k** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.32 (bs, 1H), 7.57 (s, 1H), 7.53 – 7.45 (m, 2H), 7.42 (t, *J* = 7.6 Hz, 1H), 4.51 (t, *J* = 6.5 Hz, 2H), 3.15 ppm (t, *J* = 6.5 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.7, 138.7, 132.5 (q, *J* = 1.0 Hz), 130.7 (q, *J* = 32.1 Hz), 128.9, 126.0 (q, *J* = 3.8 Hz), 124.1 (q, *J* = 272.3 Hz), 123.5 (q, *J* = 3.8 Hz), 91.2, 69.2, 34.5 ppm. **¹⁹F NMR** (377 MHz, CDCl₃) δ = -62.59 ppm (s). **HRMS (ESI)** Calculated for [M+H⁺]: 333.9775, measured: 333.9769.

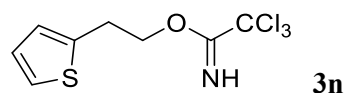


Compound **3l** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.36 (bs, 1H), 8.18 (d, *J* = 8.4 Hz, 1H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.78 (dd, *J* = 7.2, 1.9 Hz, 1H), 7.59 – 7.54 (m, 1H), 7.51 (t, *J* = 7.1 Hz, 1H), 7.46 – 7.41 (m, 2H), 4.66 (t, *J* = 7.3 Hz, 2H), 3.59 ppm (t, *J* = 7.3 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.7, 133.8, 133.4, 132.0, 128.8, 127.5, 127.3, 126.2,

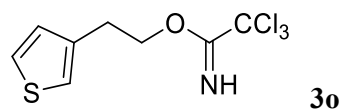
125.6, 125.5, 123.7, 91.4, 69.2, 31.6 ppm. **HRMS (ESI)** Calculated for $[M+H^+]$: 316.0057, measured: 316.0054.



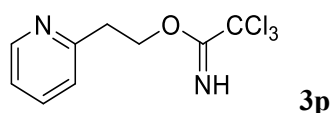
Compound **3m** was prepared according to substrate synthesis procedure 3 and the spectral data are available in the literature.^[4] **¹H NMR** (500 MHz, CDCl₃) δ = 8.35 (bs, 1H), 7.18 – 7.07 (m, 4H), 5.42 – 5.33 (m, 1H), 3.26 (dd, J = 16.7, 5.1 Hz, 1H), 3.10 – 2.99 (m, 2H), 2.94 – 2.83 (m, 1H), 2.23 – 2.09 ppm (m, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.2, 135.5, 133.4, 129.3, 128.5, 126.0, 125.9, 91.8, 74.7, 33.8, 27.1, 26.3 ppm.



Compound **3n** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.33 (bs, 1H), 7.17 (dd, J = 5.0, 1.1 Hz, 1H), 6.97 – 6.92 (m, 2H), 4.52 (t, J = 6.7 Hz, 2H), 3.31 ppm (t, J = 6.7 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.7, 139.5, 126.9, 125.8, 124.0, 91.3, 69.5, 28.9 ppm. **HRMS (ESI)** Calculated for $[M+H^+]$: 271.9465, measured: 271.9462.

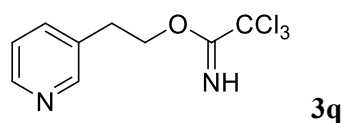


Compound **3o** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ 8.32 (bs, 1H), 7.27 (dd, J = 4.9, 3.0 Hz, 1H), 7.11 (d, J = 2.1 Hz, 1H), 7.05 (d, J = 4.9 Hz, 1H), 4.51 (t, J = 6.7 Hz, 2H), 3.13 ppm (t, J = 6.7 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.7, 137.7, 128.4, 125.4, 121.8, 91.3, 69.2, 29.1 ppm. **HRMS (ESI)** Calculated for $[M+H^+]$: 271.9465, measured: 271.9463.

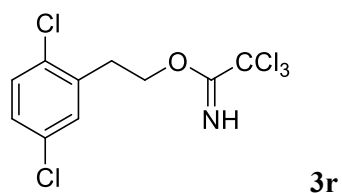


Compound **3p** was prepared according to substrate synthesis procedure 3 and the spectral data are available in the literature.^[3]

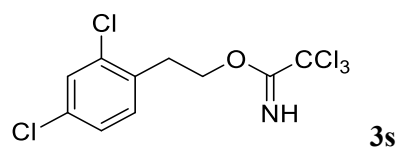
¹H NMR (500 MHz, CDCl₃) δ = 8.52 (d, J = 4.8 Hz, 1H), 8.28 (bs, 1H), 7.57 (t, J = 7.7 Hz, 1H), 7.22 (d, J = 7.8 Hz, 1H), 7.11 (dd, J = 7.3, 5.1 Hz, 1H), 4.65 (t, J = 6.6 Hz, 2H), 3.23 ppm (t, J = 6.6 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.6, 157.7, 149.4, 136.2, 123.6, 121.6, 91.3, 68.4, 36.9 ppm.



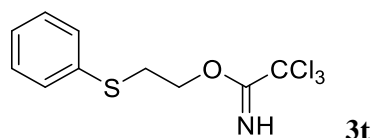
Compound **3q** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.52 (d, J = 1.9 Hz, 1H), 8.47 (dd, J = 4.8, 1.4 Hz, 1H), 8.30 (bs, 1H), 7.62 – 7.57 (m, 1H), 7.21 (dd, J = 7.7, 4.8 Hz, 1H), 4.48 (t, J = 6.5 Hz, 2H), 3.07 ppm (t, J = 6.5 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.6, 150.4, 148.2, 136.6, 133.3, 123.3, 91.2, 69.1, 31.9 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 266.9853, measured: 266.9854.



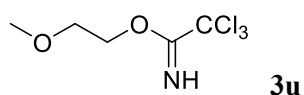
Compound **3r** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.31 (bs, 1H), 7.34 (d, J = 2.5 Hz, 1H), 7.29 (dd, J = 8.5, 3.6 Hz, 1H), 7.16 (dd, J = 8.5, 2.5 Hz, 1H), 4.51 (t, J = 6.5 Hz, 2H), 3.20 ppm (t, J = 6.5 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.6, 137.0, 132.4, 132.3, 131.5, 130.5, 128.2, 91.2, 67.6, 32.2 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 333.9121, measured: 333.9118.



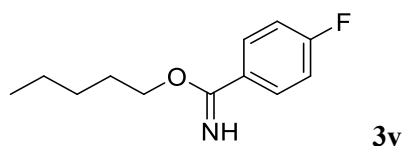
Compound **3s** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.30 (s, 1H), 7.38 (s, 1H), 7.26 (d, J = 8.2 Hz, 1H), 7.18 (dd, J = 8.2, 1.4 Hz, 1H), 4.50 (t, J = 6.5 Hz, 2H), 3.20 ppm (t, J = 6.5 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.6, 134.8, 133.9, 133.2, 132.2, 129.3, 127.0, 91.2, 67.8, 31.9 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 333.9121, measured: 333.9117.



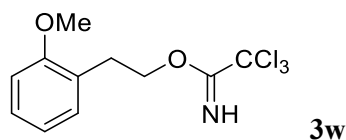
Compound **3t** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.35 (s, 1H), 7.44 (d, J = 7.4 Hz, 2H), 7.31 (t, J = 7.7 Hz, 2H), 7.22 (t, J = 7.4 Hz, 1H), 4.46 (t, J = 7.2 Hz, 2H), 3.29 ppm (t, J = 7.1 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.4, 135.0, 129.8, 129.0, 126.5, 91.1, 67.5, 31.5 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 297.9621, measured: 297.9619.



Compound **3u** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.32 (s, 1H), 4.48 – 4.42 (m, 2H), 3.76 – 3.71 (m, 2H), 3.43 ppm (s, 3H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.9, 91.3, 70.0, 68.7, 59.2 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 219.9693, measured: 219.9693.

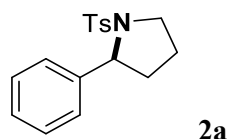


Compound **3v** was prepared according to substrate synthesis procedure 4. **¹H NMR** (400 MHz, CDCl₃) δ = 7.66 (dd, J = 7.6, 5.7 Hz, 2H), 7.46 (bs, 1H), 6.97 (t, J = 8.6 Hz, 2H), 4.12 (t, J = 6.5 Hz, 2H), 1.79 – 1.62 (m, 2H), 1.40 – 1.22 (m, 4H), 0.83 ppm (t, J = 7.1 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ = 166.3, 164.1 (d, J = 251.0 Hz), 129.0 (d, J = 3.1 Hz), 128.8 (d, J = 8.7 Hz), 115.2 (d, J = 21.8 Hz), 65.9, 28.2, 28.2, 22.3, 13.8 ppm. **¹⁹F NMR** (377 MHz, CDCl₃) δ = -109.58 ppm (s). **HRMS (ESI)** Calculated for [M+H⁺]: 210.1289, measured: 210.1287.

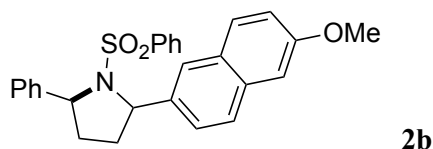


Compound **3w** was prepared according to substrate synthesis procedure 3. **¹H NMR** (500 MHz, CDCl₃) δ = 8.26 (s, 1H), 7.26 – 7.18 (m, 2H), 6.93 – 6.82 (m, 2H), 4.50 (t, J = 7.0 Hz, 2H), 3.84 (s, 3H), 3.13 ppm (t, J = 7.0 Hz, 2H). **¹³C NMR** (125 MHz, CDCl₃) δ = 162.9, 157.6, 130.9, 127.9, 125.7, 120.3, 110.1, 91.5, 68.8, 55.2, 29.4 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 296.0006, measured: 296.0000.

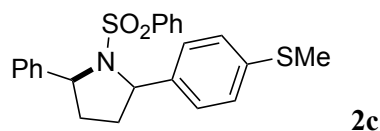
9.2 Characterization of products



The reaction was conducted in a 0.5 mmol scale following the general procedure A and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (8:1) to afford the product **2a** (107.1 mg, 71% yield) as white solid. **¹H NMR** (400 MHz, CDCl₃) δ = 7.58 (d, *J* = 8.2 Hz, 2H), 7.26 – 7.09 (m, 7H), 4.69 (dd, *J* = 7.9, 3.6 Hz, 1H), 3.52 (ddd, *J* = 10.0, 6.7, 4.7 Hz, 1H), 3.32 (dt, *J* = 10.2, 7.2 Hz, 1H), 2.32 (s, 3H), 1.96 – 1.82 (m, 1H), 1.80 – 1.65 (m, 2H), 1.60 – 1.48 ppm (m, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ = 143.2, 143.0, 134.9, 129.5, 128.2, 127.3, 126.9, 126.0, 63.1, 49.3, 35.6, 23.8, 21.4 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 302.1209, measured: 302.1207.

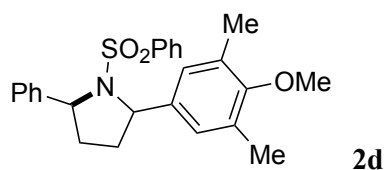


The reaction was conducted in a 0.5 mmol scale following the general procedure A and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (3:1) to afford the product **2b** (159.8 mg, 72% yield) as light yellow solid. *Isomer 1*: **¹H NMR** (400 MHz, CDCl₃) δ = 7.70 – 7.18 (m, 12H), 7.11 (d, *J* = 2.4 Hz, 1H), 7.07 (d, *J* = 2.1 Hz, 1H), 7.00 (t, *J* = 7.9 Hz, 2H), 5.42 (d, *J* = 7.4 Hz, 1H), 5.36 (d, *J* = 7.4 Hz, 1H), 3.91 (s, 3H), 2.75 – 2.59 (m, 2H), 1.94 – 1.79 ppm (m, 2H). **¹³C NMR** (101 MHz, CDCl₃) δ = 157.6, 143.0, 141.3, 137.5, 133.7, 131.4, 129.4, 128.4, 128.3, 128.0, 127.0, 126.9, 126.7, 126.2, 125.3, 125.0, 118.8, 105.5, 64.9, 64.8, 55.3, 33.6, 33.2 ppm. *Isomer 2*: **¹H NMR** (400 MHz, CDCl₃) δ = 7.76 (s, 1H), 7.71 – 7.11 (m, 15H), 5.11 (t, *J* = 6.1 Hz, 1H), 5.00 (t, *J* = 6.2 Hz, 1H), 3.92 (s, 3H), 2.17 – 2.05 ppm (m, 4H). **¹³C NMR** (100 MHz, CDCl₃) δ = 157.7, 141.9, 138.3, 136.8, 133.8, 132.5, 128.7, 128.6, 128.4, 127.7, 127.3, 127.1, 127.1, 125.8, 125.5, 118.9, 105.6, 65.28, 65.27, 55.3, 34.0, 33.9 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 444.1628, measured: 444.1620.

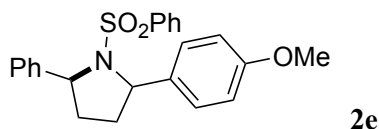


The reaction was conducted in a 0.5 mmol scale following the general procedure A and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (8:1 – 5:1) to afford the product **2c** (133.1 mg, 65% yield) as light yellow solid. *Isomer 1*: **¹H NMR** (400 MHz, CDCl₃) δ = 7.36

– 7.07 (m, 14H), 5.29 (d, $J = 7.3$ Hz, 1H), 5.24 (d, $J = 7.3$ Hz, 1H), 2.68 – 2.54 (m, 2H), 2.47 (s, 3H), 1.87 – 1.72 ppm (m, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 142.6, 141.4, 139.8, 137.1, 131.5, 128.3, 128.1, 127.1, 126.9, 126.8, 126.6, 126.3, 64.8, 64.4, 33.4, 33.3, 16.0$ ppm. *Isomer 2*: $^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta = 7.65 – 7.59$ (m, 2H), 7.53 (t, $J = 7.4$ Hz, 1H), 7.44 – 7.40 (m, 2H), 7.40 – 7.19 (m, 9H), 4.94 (t, $J = 6.8$ Hz, 1H), 4.90 (t, $J = 6.6$ Hz, 1H), 2.49 (s, 3H), 2.12 – 1.96 ppm (m, 4H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 141.9, 138.8, 138.1, 137.4, 132.6, 128.8, 128.4, 127.7, 127.5, 127.3, 126.9, 126.6, 65.2, 64.8, 34.0, 33.9, 15.9$ ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 410.1243, measured: 410.1238.

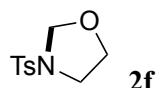


The reaction was conducted in a 0.5 mmol scale following the general procedure A and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (3:1) to afford the product **2d** (166.7 mg, 79% yield) as light yellow viscous oil. *Isomer 1*: $^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta = 7.41 – 7.19$ (m, 8H), 7.15 (t, $J = 7.8$ Hz, 2H), 6.70 (s, 2H), 5.28 (d, $J = 7.8$ Hz, 1H), 5.19 (d, $J = 8.0$ Hz, 1H), 3.68 (s, 3H), 2.70 – 2.49 (m, 2H), 2.14 (s, 6H), 1.80 (dd, $J = 11.6, 5.9$ Hz, 1H), 1.74 ppm (dd, $J = 11.8, 6.0$ Hz, 1H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 155.9, 143.4, 141.5, 137.3, 131.4, 130.3, 128.3, 127.9, 127.0, 127.0, 126.8, 126.0, 64.9, 64.3, 59.6, 33.7, 33.0, 16.1$ ppm. *Isomer 2*: $^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta = 7.61 – 7.55$ (m, 2H), 7.50 (t, $J = 7.4$ Hz, 1H), 7.46 (d, $J = 7.3$ Hz, 2H), 7.41 – 7.23 (m, 5H), 6.99 (s, 2H), 4.97 (t, $J = 6.3$ Hz, 1H), 4.85 (t, $J = 5.8$ Hz, 1H), 3.70 (s, 3H), 2.22 (s, 6H), 2.08 – 1.94 ppm (m, 4H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 156.0, 142.0, 138.4, 136.8, 132.4, 130.4, 128.5, 128.3, 127.7, 127.4, 127.3, 127.1, 64.98, 64.92, 59.6, 34.3, 33.7, 16.2$ ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 422.1784, measured: 422.1778.

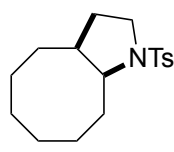


The reaction was conducted in a 0.5 mmol scale following the general procedure A and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (5:1) to afford the product **2e** (161.3 mg, 82% yield) as light yellow solid. *Isomer 1*: $^1\text{H NMR}$ (400 MHz, CDCl_3) $\delta = 7.43 – 7.05$ (m, 12H), 6.71 (d, $J = 8.7$ Hz, 2H), 5.27 (d, $J = 7.6$ Hz, 1H), 5.24 (d, $J = 7.5$ Hz, 1H), 3.77 (s, 3H), 2.67 – 2.50 (m, 2H), 1.86 – 1.70 ppm (m, 2H). $^{13}\text{C NMR}$ (100 MHz, CDCl_3) $\delta = 158.6, 142.9, 141.5, 134.7,$

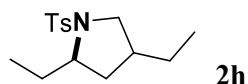
131.4, 128.2, 128.0, 127.6, 127.0, 126.7, 126.2, 113.6, 64.7, 64.2, 55.2, 33.5, 33.3 ppm. *Isomer 2*: **¹H NMR** (400 MHz, CDCl₃) δ = 7.65 – 7.58 (m, 2H), 7.50 (t, J = 7.4 Hz, 1H), 7.40 (t, J = 7.6 Hz, 2H), 7.37 – 7.05 (m, 7H), 6.85 (d, J = 8.6 Hz, 2H), 4.93 (t, J = 6.4 Hz, 1H), 4.88 (t, J = 5.9 Hz, 1H), 3.79 (s, 3H), 2.08 – 1.93 ppm (m, 4H). **¹³C NMR** (100 MHz, CDCl₃) δ = 158.7, 142.0, 138.1, 133.8, 132.4, 128.6, 128.3, 128.1, 127.6, 127.2, 126.8, 113.7, 65.0, 64.7, 55.2, 34.0, 33.8 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 394.1471, measured: 394.1468.



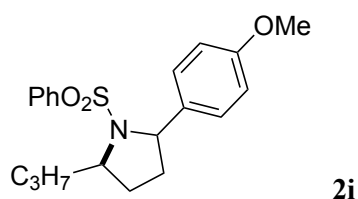
The reaction was conducted in a 0.5 mmol scale following the general procedure A and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (3:1) to afford the product **2f** (64.9 mg, 57% yield) as white solid. The spectral data are available in the literature.^[8] **¹H NMR** (500 MHz, CDCl₃) δ = 7.73 (d, J = 8.4 Hz, 2H), 7.32 (d, J = 8.1 Hz, 2H), 4.83 (s, 2H), 3.65 (t, J = 6.7 Hz, 2H), 3.42 (t, J = 6.7 Hz, 2H), 2.42 ppm (s, 3H). **¹³C NMR** (125 MHz, CDCl₃) δ = 144.2, 134.0, 129.8, 127.7, 80.7, 66.0, 46.0, 21.5 ppm.



The reaction was conducted in a 0.5 mmol scale following the general procedure A and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (3:1) to afford the product **2g** (67.7 mg, 44% yield) as white solid. *Major isomer*: **¹H NMR** (400 MHz, CDCl₃) δ = 7.70 (d, J = 8.2 Hz, 3H), 7.30 (d, J = 8.0 Hz, 2H), 3.70 (dd, J = 9.9, 6.1 Hz, 1H), 3.37 – 3.31 (m, 1H), 2.84 – 2.79 (m, 1H), 2.41 (s, 3H), 1.97 – 1.90 (m, 1H), 1.84 – 1.25 (m, 12H), 1.14 – 1.03 ppm (m, 2H). **¹³C NMR** (100 MHz, CDCl₃) δ = 143.0, 134.5, 129.5, 127.4, 63.8, 46.3, 42.6, 33.0, 32.0, 29.6, 28.1, 25.50, 25.49, 25.2, 21.4 ppm. *Minor isomer*: **¹H NMR** (400 MHz, CDCl₃) δ = 7.70 (d, J = 8.2 Hz, 2H), 7.29 (d, J = 7.9 Hz, 2H), 3.59 – 3.47 (m, 2H), 3.11 (td, J = 12.3, 5.0 Hz, 1H), 2.41 (s, 3H), 2.30 – 2.21 (m, 1H), 2.06 – 1.97 (m, 1H), 1.84 – 1.22 (11, H), 0.81 – 0.59 ppm (m, 2H). **¹³C NMR** (100 MHz, CDCl₃) δ = 143.0, 136.1, 129.5, 127.2, 64.6, 47.3, 44.3, 37.1, 35.3, 34.8, 26.7, 26.6, 24.2, 22.2 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 308.1679, measured: 308.1677.



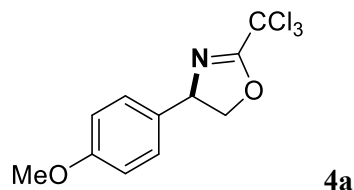
The reaction was conducted in a 0.5 mmol scale following the general procedure A and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (5:1) to afford the product **2h** (94.3 mg, 67% yield) as colorless oil. **¹H NMR** (400 MHz, CDCl₃) δ 7.69 (d, *J* = 8.2 Hz, 2H), 7.28 (d, *J* = 8.2 Hz, 2H), 3.64 – 3.42 (m, 2H), 2.83 (dd, *J* = 11.3, 10.5 Hz, 0.5H), 2.59 (t, *J* = 9.5 Hz, 0.5H), 2.40 (s, 3H), 2.15 – 1.92 (m, 1.5H), 1.82 (dq, *J* = 15.1, 7.5, 4.2 Hz, 0.5H), 1.69 (ddd, *J* = 12.4, 6.2, 1.4 Hz, 0.5H), 1.61 – 1.40 (m, 1H), 1.37 – 0.97 (m, 3.5H), 0.87 (t, *J* = 7.4 Hz, 1.5H), 0.86 (t, *J* = 7.4 Hz, 1.5H), 0.80 (t, *J* = 7.6 Hz, 1.5H), 0.76 ppm (t, *J* = 7.4 Hz, 1.5H). **¹³C NMR** (100 MHz, CDCl₃) δ (143.05, 143.03), (135.4, 134.4), (129.50, 129.42), (127.4, 127.2), (62.2, 61.7), (54.5, 54.0), (39.7, 38.7), (37.7, 35.7), (29.4, 29.3), (25.8, 25.3), 21.4, (12.5, 12.4), (10.5, 9.7) ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 282.1522, measured: 282.1521.



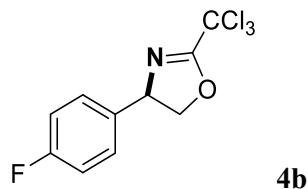
The reaction was conducted in a 0.5 mmol scale following the general procedure A and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (8:1) to afford the product **2i** (113.5mg, 63% yield) as colorless viscous oil.

Major isomer (cis): **¹H NMR** (400 MHz, CDCl₃) δ = 7.83 – 7.75 (m, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.27 (d, *J* = 8.6 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 4.66 (t, *J* = 6.8 Hz, 1H), 3.85 – 3.80 (m, 1H), 3.79 (s, 3H), 2.07 – 1.96 (m, 1H), 1.92 – 1.79 (m, 2H), 1.65 – 1.48 (m, 3H), 1.46 – 1.31 (m, 2H), 0.97 ppm (t, *J* = 7.3 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ = 158.6, 138.2, 134.7, 132.4, 128.9, 127.6, 127.4, 113.7, 64.1, 62.1, 55.2, 39.0, 34.3, 29.7, 19.9, 14.0 ppm.

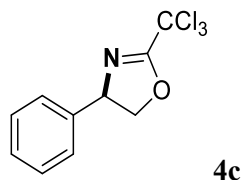
Minor isomer (trans): **¹H NMR** (400 MHz, CDCl₃) δ = 7.45 – 7.41 (m, 2H), 7.38 (t, *J* = 7.4 Hz, 1H), 7.24 (t, *J* = 7.8 Hz, 2H), 6.89 (d, *J* = 8.6 Hz, 2H), 6.61 (d, *J* = 8.7 Hz, 2H), 4.93 (d, *J* = 8.2 Hz, 1H), 4.15 – 4.05 (m, 1H), 3.74 (s, 3H), 2.47 – 2.32 (m, 1H), 2.24 – 2.04 (m, 2H), 1.78 (dd, *J* = 12.7, 7.1 Hz, 1H), 1.71 (dd, *J* = 12.4, 7.1 Hz, 1H), 1.58 – 1.46 (m, 1H), 1.37 – 1.23 (m, 2H), 0.94 ppm (t, *J* = 7.3 Hz, 3H). **¹³C NMR** (100 MHz, CDCl₃) δ = 158.5, 141.9, 134.3, 131.4, 128.2, 127.9, 126.7, 113.4, 63.3, 61.7, 55.2, 36.9, 33.4, 28.2, 19.7, 14.0 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 360.1628, measured: 360.1624.



The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (20:1) with 2% Et₃N to afford the product **4a** (107.1 mg, 73% yield) as colorless oil. ¹H NMR (500 MHz, CDCl₃) δ = 7.18 (d, *J* = 8.7 Hz, 2H), 6.91 (d, *J* = 8.7 Hz, 2H), 5.37 (dd, *J* = 10.0, 8.2 Hz, 1H), 4.95 (dd, *J* = 10.0, 8.6 Hz, 1H), 4.48 (t, *J* = 8.3 Hz, 1H), 3.80 ppm (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ = 163.3, 159.5, 132.3, 127.8, 114.3, 86.6, 78.3, 69.6, 55.3 ppm. HRMS (ESI) Calculated for [M+H⁺]: 293.9850, measured: 293.9847.

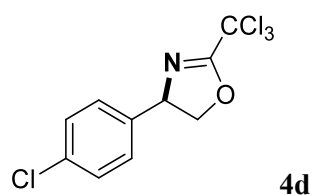


The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (30:1) with 2% Et₃N to afford the product **4b** (105.6 mg, 75% yield) as light yellow oil. ¹H NMR (400 MHz, CDCl₃) δ = 7.28 – 7.20 (m, 2H), 7.13 – 7.03 (m, 2H), 5.42 (dd, *J* = 10.1, 8.2 Hz, 1H), 4.99 (dd, *J* = 10.1, 8.6 Hz, 1H), 4.47 ppm (t, *J* = 8.4 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ = 163.7, 162.5 (d, *J* = 247.0 Hz), 135.9 (d, *J* = 3.2 Hz), 128.3 (d, *J* = 8.3 Hz), 115.9 (d, *J* = 21.7 Hz), 86.4, 78.2, 69.4 ppm. ¹⁹F NMR (377 MHz, CDCl₃) δ = -113.69 ppm (s). HRMS (ESI) Calculated for [M+H⁺]: 281.9650, measured: 281.9648.

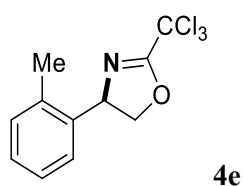


The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (30:1) with 2% Et₃N to afford the product **4c** (112.4 mg, 85% yield) as light yellow oil. ¹H NMR (400 MHz, CDCl₃) δ = 7.34 – 7.15 (m, 5H), 5.35 (dd, *J* = 10.1, 8.2 Hz, 1H), 4.91 (dd, *J* = 10.1, 8.6 Hz, 1H), 4.43 ppm (t, *J* = 8.4 Hz,

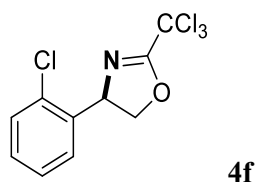
1H). ^{13}C NMR (100 MHz, CDCl_3) δ = 163.5, 140.1, 128.9, 128.2, 126.5, 86.5, 78.2, 70.0 ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 263.9744, measured: 263.9744.



The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (40:1) with 2% Et_3N to afford the product **4d** (90.6 mg, 61% yield) as yellow oil. ^1H NMR (400 MHz, CDCl_3) δ = 7.45 – 7.31 (m, 2H), 7.26 – 7.11 (m, 2H), 5.41 (dd, J = 10.1, 8.2 Hz, 1H), 4.98 (dd, J = 10.1, 8.7 Hz, 1H), 4.46 ppm (t, J = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ = 163.9, 138.6, 134.1, 129.2, 127.9, 86.4, 78.0, 69.4 ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 297.9355, measured: 297.9355.

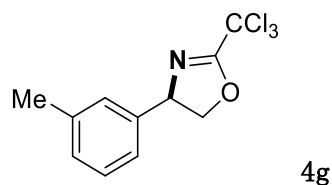


The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (30:1) with 2% Et_3N to afford the product **4e** (92.9 mg, 67% yield) as light yellow oil. ^1H NMR (500 MHz, CDCl_3) δ = 7.28 – 7.15 (m, 4H), 5.63 (dd, J = 10.2, 8.7 Hz, 1H), 5.03 (dd, J = 10.3, 8.4 Hz, 1H), 4.37 ppm (t, J = 8.5 Hz, 1H), 2.32 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ = 163.5, 138.6, 134.5, 130.5, 127.9, 126.7, 125.9, 86.5, 77.6, 67.1, 19.5 ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 277.9901, measured: 277.9899.

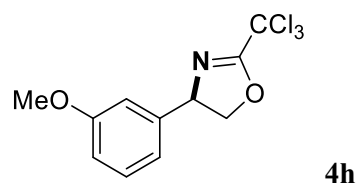


The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (40:1) with 2% Et_3N to afford the product **4f** (111.6 mg, 75% yield) as colorless oil. ^1H NMR (400 MHz, CDCl_3) δ = 7.42 – 7.23 (m, 4H), 5.78 (dd, J = 10.3, 8.6 Hz, 1H), 5.14 (dd, J = 10.4, 8.6 Hz, 1H), 4.37 ppm (t, J = 8.6 Hz,

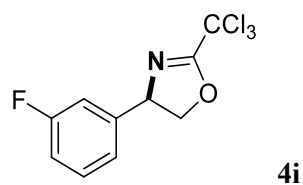
1H). ^{13}C NMR (100 MHz, CDCl_3) δ = 164.2, 138.4, 132.0, 129.4, 129.2, 127.5, 127.4, 86.4, 77.6, 67.3 ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 297.9355, measured: 297.9353.



The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (30:1) with 2% Et_3N to afford the product **4g** (99.7 mg, 72% yield) as colorless oil. ^1H NMR (500 MHz, CDCl_3) δ = 7.27 (t, J = 7.7 Hz, 1H), 7.14 (d, J = 7.6 Hz, 1H), 7.07 (s, 1H), 7.06 (d, J = 7.4 Hz, 1H), 5.39 (dd, J = 10.0, 8.4 Hz, 1H), 4.98 (dd, J = 10.1, 8.6 Hz, 1H), 4.51 (t, J = 8.4 Hz, 1H), 2.37 ppm (s, 3H). ^{13}C NMR (125 MHz, CDCl_3) δ = 163.5, 140.0, 138.7, 129.0, 128.9, 127.2, 123.6, 86.6, 78.2, 70.0, 21.4 ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 277.9901, measured: 277.9900.

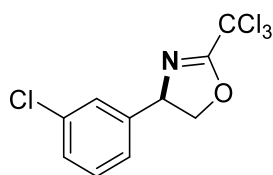


The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (20:1) with 2% Et_3N to afford the product **4h** (110.9 mg, 76% yield) as light yellow oil. ^1H NMR (400 MHz, CDCl_3) δ = 7.21 (t, J = 7.9 Hz, 1H), 6.80 – 6.74 (m, 2H), 6.73 – 6.69 (m, 1H), 5.31 (dd, J = 10.1, 8.1 Hz, 1H), 4.88 (dd, J = 10.1, 8.6 Hz, 1H), 4.42 (t, J = 8.3 Hz, 1H), 3.72 ppm (s, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ = 163.6, 160.0, 141.7, 130.0, 118.7, 113.5, 112.2, 86.5, 78.1, 69.9, 55.2 ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 293.9850, measured: 293.9848.



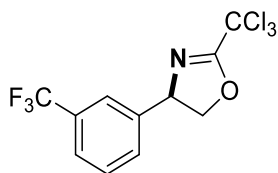
The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (20:1) with 2% Et_3N to

afford the product **4i** (104.3 mg, 74% yield) as light yellow oil. **¹H NMR** (500 MHz, CDCl₃) δ = 7.35 (td, *J* = 8.0, 5.9 Hz, 1H), 7.07 – 6.95 (m, 3H), 5.43 (dd, *J* = 10.1, 8.3 Hz, 1H), 5.00 (dd, *J* = 10.2, 8.7 Hz, 1H), 4.48 ppm (t, *J* = 8.4 Hz, 1H). **¹³C NMR** (125 MHz, CDCl₃) δ = 164.0, 163.1 (d, *J* = 247.2 Hz), 142.6 (d, *J* = 7.0 Hz), 130.6 (d, *J* = 8.3 Hz), 122.2 (d, *J* = 3.0 Hz), 115.2 (d, *J* = 21.1 Hz), 113.6 (d, *J* = 22.2 Hz), 86.4, 78.0, 69.5 ppm (d, *J* = 1.9 Hz). **¹⁹F NMR** (377 MHz, CDCl₃) δ = -111.83 ppm (s). **HRMS (ESI)** Calculated for [M+H⁺]: 281.9650, measured: 281.9648.



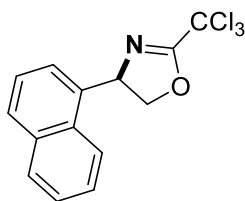
4j

The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (40:1) with 2% Et₃N to afford the product **4j** (99.8 mg, 67% yield) as yellow oil. **¹H NMR** (400 MHz, CDCl₃) δ = 7.30 – 7.04 (m, 4H), 5.33 (dd, *J* = 10.1, 8.4 Hz, 1H), 4.92 (dd, *J* = 10.1, 8.7 Hz, 1H), 4.39 ppm (t, *J* = 8.5 Hz, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ = 164.0, 142.0, 134.8, 130.3, 128.4, 126.8, 124.7, 86.3, 77.9, 69.5 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 297.9355, measured: 297.9354.



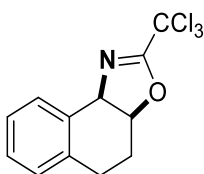
4k

The reaction was conducted in a 0.5 mmol scale following the general procedure B with 2,6-lutidine instead of pyridine, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (100:1) with 2% Et₃N to afford the product **4k** (86.2 mg, 52% yield) as colorless oil. **¹H NMR** (500 MHz, CDCl₃) δ = 7.60 (d, *J* = 7.8 Hz, 1H), 7.57 – 7.50 (m, 2H), 7.46 (d, *J* = 7.6 Hz, 1H), 5.50 (dd, *J* = 10.0, 8.4 Hz, 1H), 5.04 (dd, *J* = 10.0, 8.9 Hz, 1H), 4.50 ppm (t, *J* = 8.5 Hz, 1H). **¹³C NMR** (125 MHz, CDCl₃) δ = 164.3, 141.1, 131.3 (q, *J* = 32.5 Hz), 129.9 (q, *J* = 0.9 Hz), 129.6, 125.2 (q, *J* = 3.7 Hz), 123.8 (q, *J* = 272.4 Hz), 123.5 (q, *J* = 3.8 Hz), 86.3, 77.9, 69.6 ppm. **¹⁹F NMR** (377 MHz, CDCl₃) δ = -62.71 ppm (s). **HRMS (ESI)** Calculated for [M+H⁺]: 331.9618, measured: 331.9616.



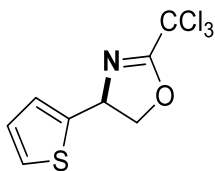
4l

The reaction was conducted in a 0.5 mmol scale following the general procedure B with RVC as working electrode, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (10:1) with 2% Et₃N to afford the product **4l** (129.8 mg, 83% yield) as light yellow oil. **¹H NMR** (500 MHz, CDCl₃) δ = 7.94 – 7.91 (m, 1H), 7.84 (dd, *J* = 7.3, 1.7 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.59 – 7.49 (m, 4H), 6.15 (dd, *J* = 10.2, 8.7 Hz, 1H), 5.24 (dd, *J* = 10.4, 8.4 Hz, 1H), 4.44 ppm (t, *J* = 8.5 Hz, 1H). **¹³C NMR** (125 MHz, CDCl₃) δ = 163.6, 136.3, 133.8, 130.1, 129.2, 128.4, 126.6, 125.8, 125.7, 123.4, 122.3, 86.6, 77.9, 66.9 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 313.9901, measured: 313.9898.



4m

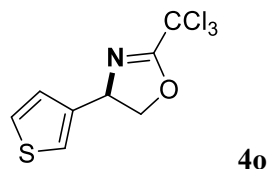
The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (40:1) with 1% Et₃N to afford the product **4m** (99.8 mg, 69% yield) as white solid. **¹H NMR** (400 MHz, CDCl₃) δ = 7.54 – 7.47 (m, 1H), 7.31 – 7.19 (m, 2H), 7.15 (d, *J* = 7.3 Hz, 1H), 5.48 – 5.39 (m, 2H), 2.82 (ddd, *J* = 15.2, 11.5, 3.6 Hz, 1H), 2.63 (dt, *J* = 15.6, 4.5 Hz, 1H), 2.30 (ddt, *J* = 13.9, 4.9, 3.5 Hz, 1H), 1.96 – 1.85 ppm (m, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ = 162.5, 138.3, 132.5, 130.0, 128.4, 127.8, 127.2, 86.8, 82.8, 67.8, 28.1, 24.3 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 289.9901, measured: 289.9899.



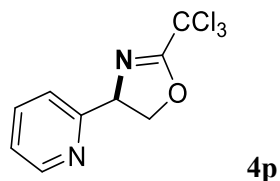
4n

The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (15:1) with 2% Et₃N to afford the product **4n** (82.3 mg, 61% yield) as light yellow oil. **¹H NMR** (500 MHz, CDCl₃) δ = 7.29 (dd, *J* = 4.8, 1.3 Hz, 1H), 7.04 – 6.96 (m, 2H), 5.68 (dd, *J* = 9.8, 7.9 Hz, 1H), 4.96 (dd, *J* = 9.7, 8.9 Hz,

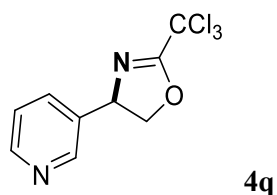
1H), 4.61 ppm (t, $J = 8.2$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3) $\delta = 163.6, 142.9, 127.1, 125.5, 124.9, 86.4, 78.0, 65.9$ ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 269.9308, measured: 269.9308.



The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (15:1) with 2% Et_3N to afford the product **4o** (106.3 mg, 79% yield) as light yellow oil. ^1H NMR (500 MHz, CDCl_3) $\delta = 7.36$ (dd, $J = 5.0, 3.0$ Hz, 1H), 7.22 (d, $J = 2.7$ Hz, 1H), 7.01 (d, $J = 5.0$ Hz, 1H), 5.51 (dd, $J = 9.9, 7.9$ Hz, 1H), 4.92 (dd, $J = 9.9, 8.6$ Hz, 1H), 4.53 ppm (t, $J = 8.2$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3) $\delta = 163.4, 140.7, 127.1, 125.5, 122.1, 86.5, 77.4, 66.0$ ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 269.9308, measured: 269.9305.

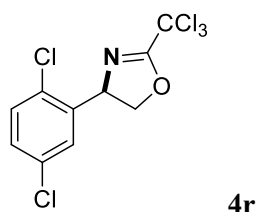


The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (5:1) with 2% Et_3N to afford the product **4p** (113.6 mg, 86% yield) as light yellow oil. ^1H NMR (500 MHz, CDCl_3) $\delta = 8.60 - 8.53$ (m, 1H), 7.70 (td, $J = 7.7, 1.7$ Hz, 1H), 7.41 (d, $J = 7.8$ Hz, 1H), 7.23 - 7.20 (m, 1H), 5.56 (dd, $J = 10.3, 8.3$ Hz, 1H), 5.03 (dd, $J = 10.3, 8.6$ Hz, 1H), 4.86 ppm (t, $J = 8.4$ Hz, 1H). ^{13}C NMR (125 MHz, CDCl_3) $\delta = 164.0, 159.0, 149.6, 137.0, 122.9, 121.5, 86.5, 76.5, 70.9$ ppm. **HRMS (ESI)** Calculated for $[\text{M}+\text{H}^+]$: 264.9697, measured: 264.9696.

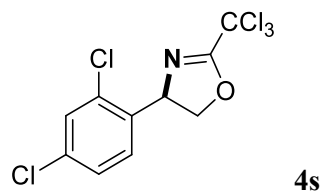


The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (5:1) with 2% Et_3N to afford

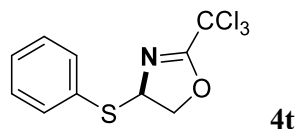
the product **4q** (98.8 mg, 75% yield) as yellow oil. **¹H NMR** (400 MHz, CDCl₃) δ = 8.56 (d, J = 4.7 Hz, 1H), 8.52 (d, J = 1.5 Hz, 1H), 7.57 (d, J = 7.9 Hz, 1H), 7.31 (dd, J = 7.9, 4.8 Hz, 1H), 5.45 (dd, J = 9.9, 8.4 Hz, 1H), 5.01 (dd, J = 10.3, 8.6 Hz, 1H), 4.47 ppm (t, J = 8.4 Hz, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ = 164.2, 149.7, 148.2, 135.6, 134.0, 123.8, 86.2, 77.7, 67.7 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 264.9697, measured: 264.9696.



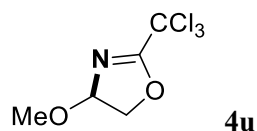
The reaction was conducted in a 0.5 mmol scale following the general procedure B with 2,6-lutidine instead of pyridine, and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (80:1) with 2% Et₃N to afford the product **4r** (111.3 mg, 67% yield) as colorless oil. **¹H NMR** (500 MHz, CDCl₃) δ = 7.34 (s, 1H), 7.34 (d, J = 9.9 Hz, 2H), 7.26 (dd, J = 8.5, 2.5 Hz, 1H), 5.72 (dd, J = 10.2, 8.9 Hz, 1H), 5.16 (dd, J = 10.4, 8.8 Hz, 1H), 4.35 ppm (t, J = 8.7 Hz, 1H). **¹³C NMR** (125 MHz, CDCl₃) δ = 164.7, 140.1, 133.5, 130.6, 130.2, 129.3, 127.7, 86.3, 77.2, 67.1 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 331.8965, measured: 331.8964.



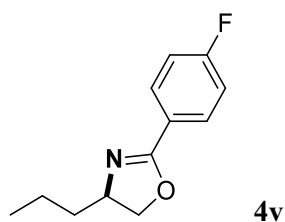
The reaction was conducted in a 0.5 mmol scale following the general procedure B with 2,6-lutidine instead of pyridine, and the residue was purified by column chromatography on silica gel with hexane/ethyl acetate (80:1) with 2% Et₃N to afford the product **4s** (117.8 mg, 71% yield) as yellow oil. **¹H NMR** (500 MHz, CDCl₃) δ = 7.42 (s, 1H), 7.32 – 7.27 (m, 2H), 5.71 (dd, J = 10.4, 8.6 Hz, 1H), 5.13 (dd, J = 10.4, 8.7 Hz, 1H), 4.34 ppm (t, J = 8.6 Hz, 1H). **¹³C NMR** (125 MHz, CDCl₃) δ = 164.5, 137.0, 134.5, 132.6, 129.3, 128.4, 127.7, 86.3, 77.4, 66.9 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 331.8965, measured: 331.8963.



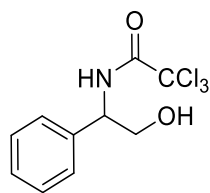
The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (30:1) with 2% Et₃N to afford the product **4t** (88.9 mg, 60% yield) as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ = 7.57 – 7.50 (m, 2H), 7.37 – 7.29 (m, 3H), 5.52 (dd, *J* = 9.7, 6.4 Hz, 1H), 4.85 (t, *J* = 9.7 Hz, 1H), 4.55 ppm (dd, *J* = 9.7, 6.4 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ = 163.7, 135.0, 129.3, 129.1, 129.0, 85.9, 75.7, 72.2 ppm. HRMS (ESI) Calculated for [M+H⁺]: 295.9465, measured: 295.9464.



The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (12:1) with 2% Et₃N to afford the product **4u** (70.5 mg, 65% yield) as colorless oil. ¹H NMR (500 MHz, CDCl₃) δ = 5.53 (dd, *J* = 7.4, 5.0 Hz, 1H), 4.61 (dd, *J* = 10.1, 7.4 Hz, 1H), 4.41 (dd, *J* = 10.1, 5.0 Hz, 1H), 3.54 ppm (s, 3H). ¹³C NMR (125 MHz, CDCl₃) δ = 165.4, 97.8, 86.6, 75.7, 56.3 ppm. HRMS (ESI) Calculated for [M+H⁺]: 217.9537, measured: 217.9537.

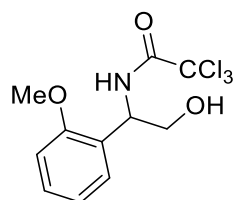


The reaction was conducted in a 0.5 mmol scale following the general procedure B, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (15:1) with 2% Et₃N to afford the product **4v** (53.9 mg, 52% yield) as colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.93 (dd, *J* = 8.4, 5.6 Hz, 2H), 7.07 (t, *J* = 8.4 Hz, 2H), 4.47 (td, *J* = 8.4, 0.8 Hz, 1H), 4.31 – 4.21 (m, 1H), 4.01 (td, *J* = 8.0, 0.6 Hz, 1H), 1.78 – 1.65 (m, 1H), 1.59 – 1.34 (m, 3H), 0.96 ppm (t, *J* = 7.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃) δ 164.6 (d, *J* = 251.4 Hz), 162.4, 130.4 (d, *J* = 8.8 Hz), 124.2 (d, *J* = 3.2 Hz), 115.3 (d, *J* = 21.9 Hz), 72.7, 66.6, 38.1, 19.1, 14.1 ppm. ¹⁹F NMR (377 MHz, CDCl₃) δ -108.58 ppm (s). HRMS (ESI) Calculated for [M+H⁺]: 208.1132, measured: 208.1131.



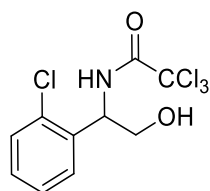
5a

The reaction was conducted in a 0.5 mmol scale following the general procedure C, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (5:1) to afford the product **5a** (92.9 mg, 66% yield) as white solid. **¹H NMR** (400 MHz, CDCl₃) δ = 7.51 (bs, 1H), 7.43 – 7.36 (m, 2H), 7.36 – 7.28 (m, 3H), 5.03 (dt, *J* = 8.0, 4.0 Hz, 1H), 4.04 – 3.87 (m, 2H), 2.16 ppm (bs, 1H). **¹³C NMR** (100 MHz, CDCl₃) δ = 161.8, 137.5, 129.0, 128.2, 126.4, 92.5, 65.3, 56.7 ppm. **HRMS (ESI)** Calculated for [M+Na⁺]: 303.9669, measured: 303.9669.



5b

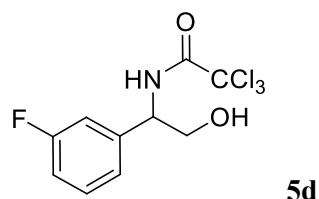
The reaction was conducted in a 0.5 mmol scale following the general procedure C, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (3:1) to afford the product **5b** (90.2 mg, 58% yield) as light yellow solid. **¹H NMR** (500 MHz, CDCl₃) δ = 8.09 (d, *J* = 7.7 Hz, 1H), 7.32 (td, *J* = 8.2, 1.6 Hz, 1H), 7.24 (dd, *J* = 7.5, 1.4 Hz, 1H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.95 (d, *J* = 8.3 Hz, 1H), 5.22 (dt, *J* = 8.4, 5.9 Hz, 1H), 3.93 (dd, *J* = 11.3, 6.3 Hz, 1H), 3.91 – 3.86 (m, 4H), 2.05 ppm (bs, 1H). **¹³C NMR** (125 MHz, CDCl₃) δ = 161.7, 156.9, 129.6, 129.1, 124.6, 121.2, 111.20 (s), 92.8, 64.4, 55.5, 55.5 ppm. **HRMS (ESI)** Calculated for [M+H⁺]: 311.9956, measured: 311.9954.



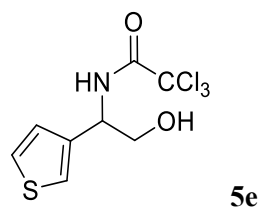
5c

The reaction was conducted in a 0.5 mmol scale following the general procedure C, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (5:1) to afford the product **5c** (105.5 mg, 67% yield) as white solid. **¹H NMR** (500 MHz, CD₃CN) δ = 7.91 (s, 1H), 7.47 – 7.40 (m, 2H), 7.37 – 7.28 (m, 2H), 5.29 (td, *J* = 7.1, 4.5 Hz, 1H), 3.86 (dt, *J* = 11.6, 4.7 Hz, 1H), 3.77 (dt, *J* = 11.6, 6.5 Hz, 1H), 3.28 ppm (t, *J* = 6.0 Hz, 1H). **¹³C NMR** (125 MHz, CD₃CN) δ = 162.5, 137.2,

133.7, 130.7, 130.4, 128.9, 128.4, 93.4, 63.4, 56.0 ppm. **HRMS (ESI)** Calculated for $[M+Na^+]$: 337.9280, measured: 337.9278.



The reaction was conducted in a 0.5 mmol scale following the general procedure C, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (5:1) to afford the product **5d** (80.6 mg, 54% yield) as white solid. **¹H NMR** (400 MHz, CDCl₃) δ 7.52 (d, $J = 5.3$ Hz, 1H), 7.36 (td, $J = 7.8, 6.2$ Hz, 1H), 7.11 (d, $J = 7.7$ Hz, 1H), 7.07 – 6.98 (m, 2H), 5.02 (dt, $J = 7.4, 4.2$ Hz, 1H), 4.02 (dt, $J = 11.4, 4.3$ Hz, 1H), 3.95 (dt, $J = 11.3, 4.0$ Hz, 1H), 1.94 ppm (t, $J = 5.2$ Hz, 1H). **¹³C NMR** (125 MHz, CDCl₃) δ 163.1 (d, $J = 247.3$ Hz), 161.7, 140.2 (d, $J = 6.9$ Hz), 130.6 (d, $J = 8.3$ Hz), 122.1 (d, $J = 3.0$ Hz), 115.2 (d, $J = 21.1$ Hz), 113.6 (d, $J = 22.4$ Hz), 92.4, 65.1, 56.1 ppm (d, $J = 1.7$ Hz). **¹⁹F NMR** (377 MHz, CDCl₃) δ -111.70 ppm (s). **HRMS (ESI)** Calculated for $[M+Na^+]$: 321.9575, measured: 321.9576.

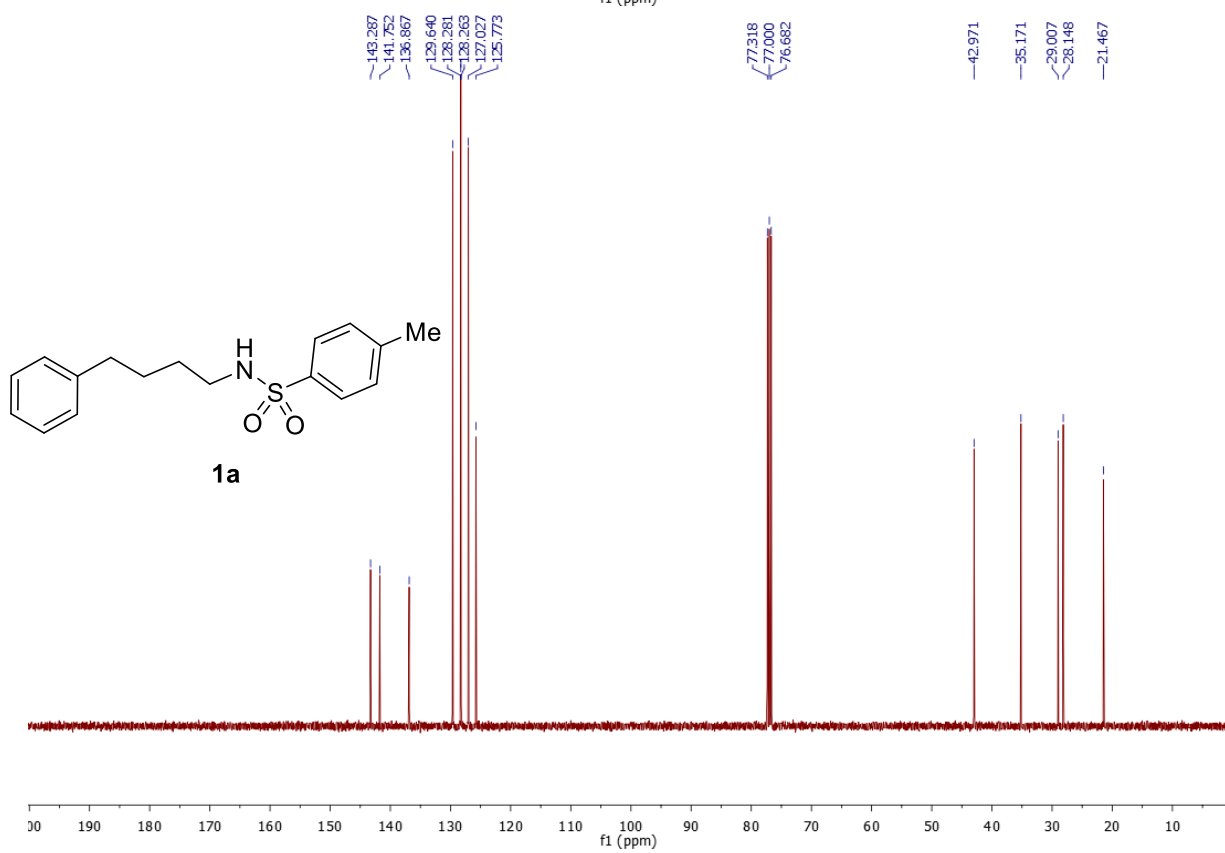
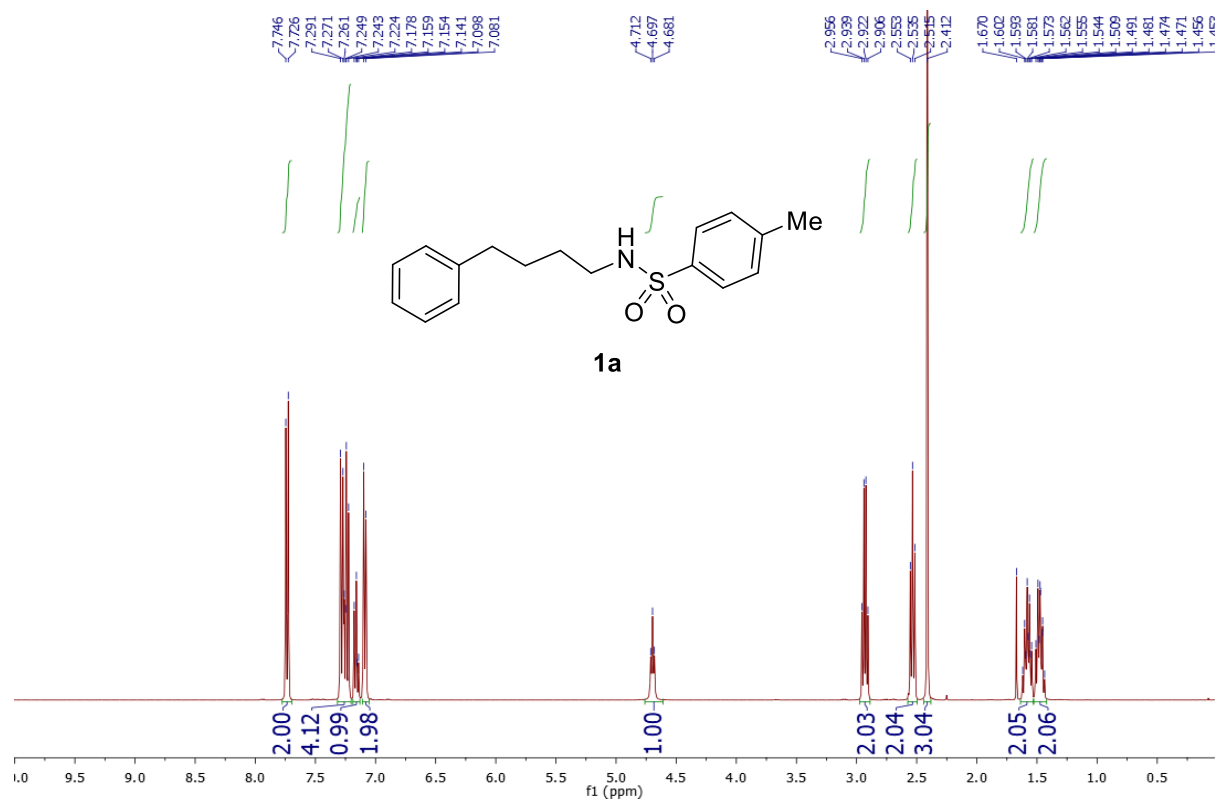


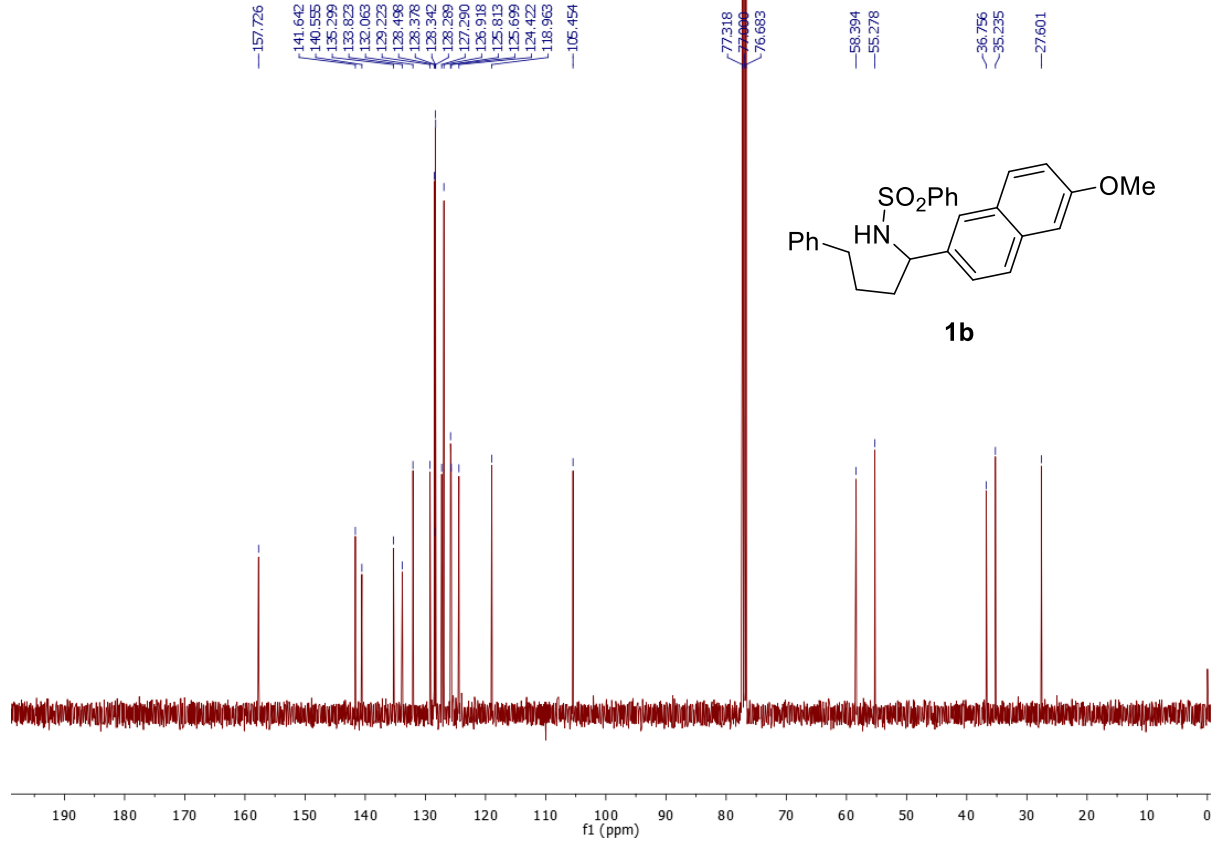
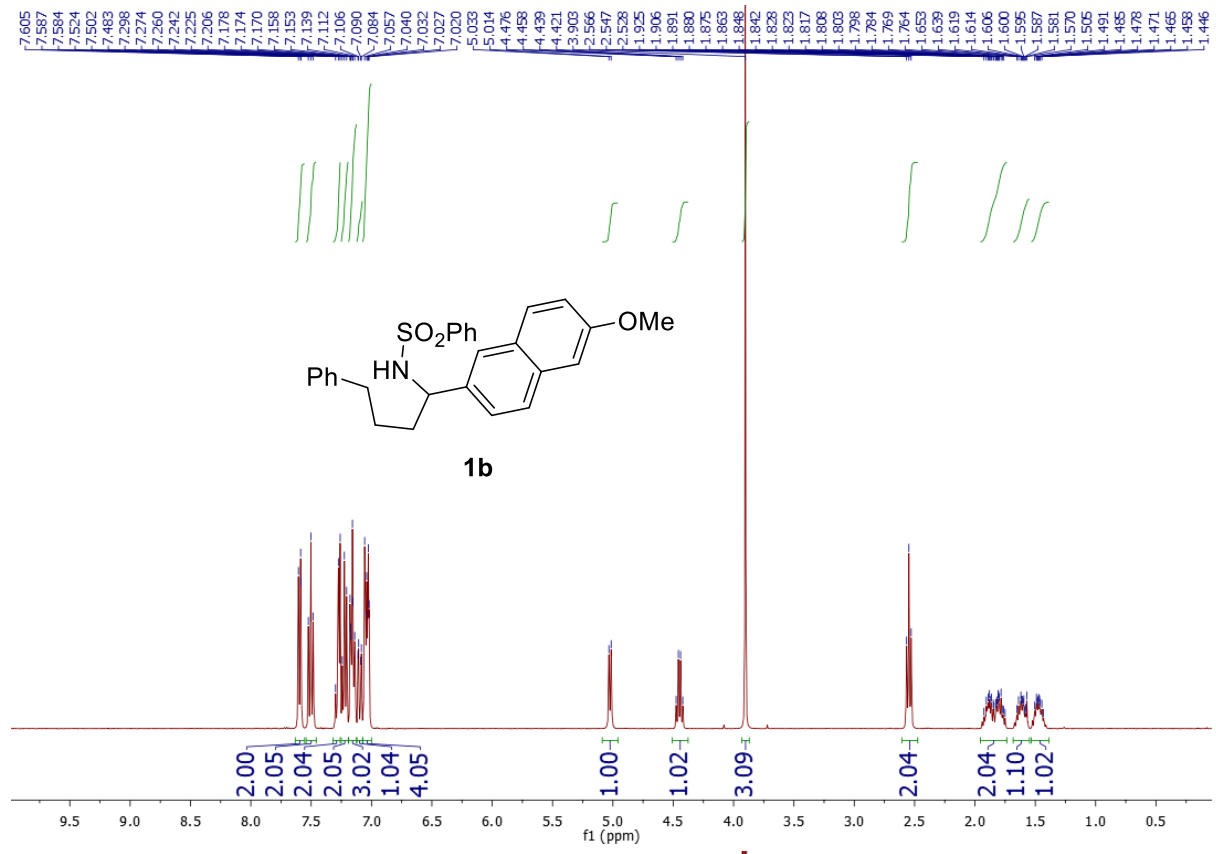
The reaction was conducted in a 0.5 mmol scale following the general procedure C, and the residue was purified by column chromatography on silica gel with hexane/ ethyl acetate (4:1) to afford the product **5e** (84.8 mg, 59% yield) as light yellow solid. **¹H NMR** (500 MHz, CDCl₃) δ = 7.43 (d, $J = 6.7$ Hz, 1H), 7.36 (dd, $J = 5.0, 3.0$ Hz, 1H), 7.25 – 7.23 (m, 1H), 7.06 (dd, $J = 5.0, 1.0$ Hz, 1H), 5.15 (dt, $J = 8.1, 4.2$ Hz, 1H), 4.18 – 3.75 (m, 2H), 2.30 ppm (bs, 1H). **¹³C NMR** (125 MHz, CDCl₃) δ = 161.7, 138.3, 127.1, 125.9, 122.2, 92.5, 64.6, 52.9 ppm. **HRMS (ESI)** Calculated for $[M+Na^+]$: 309.9234, measured: 309.9232.

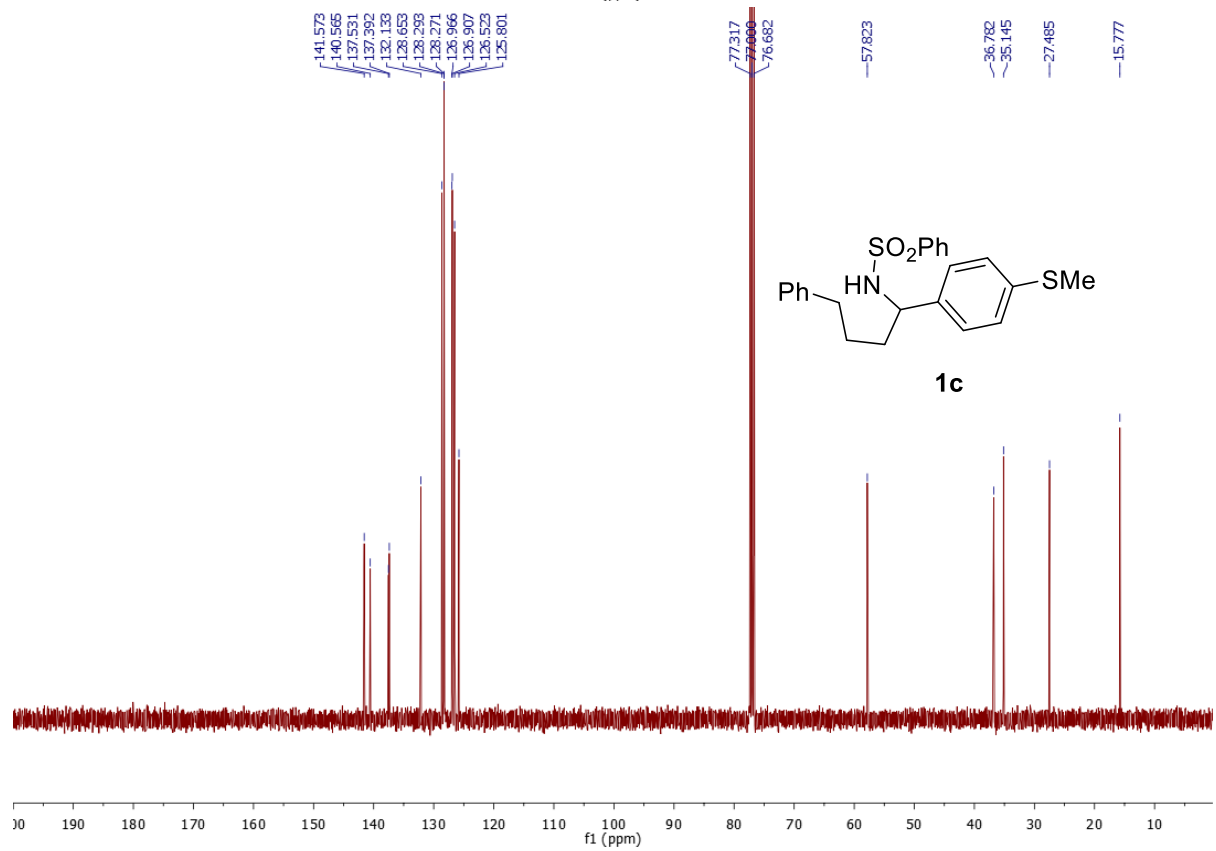
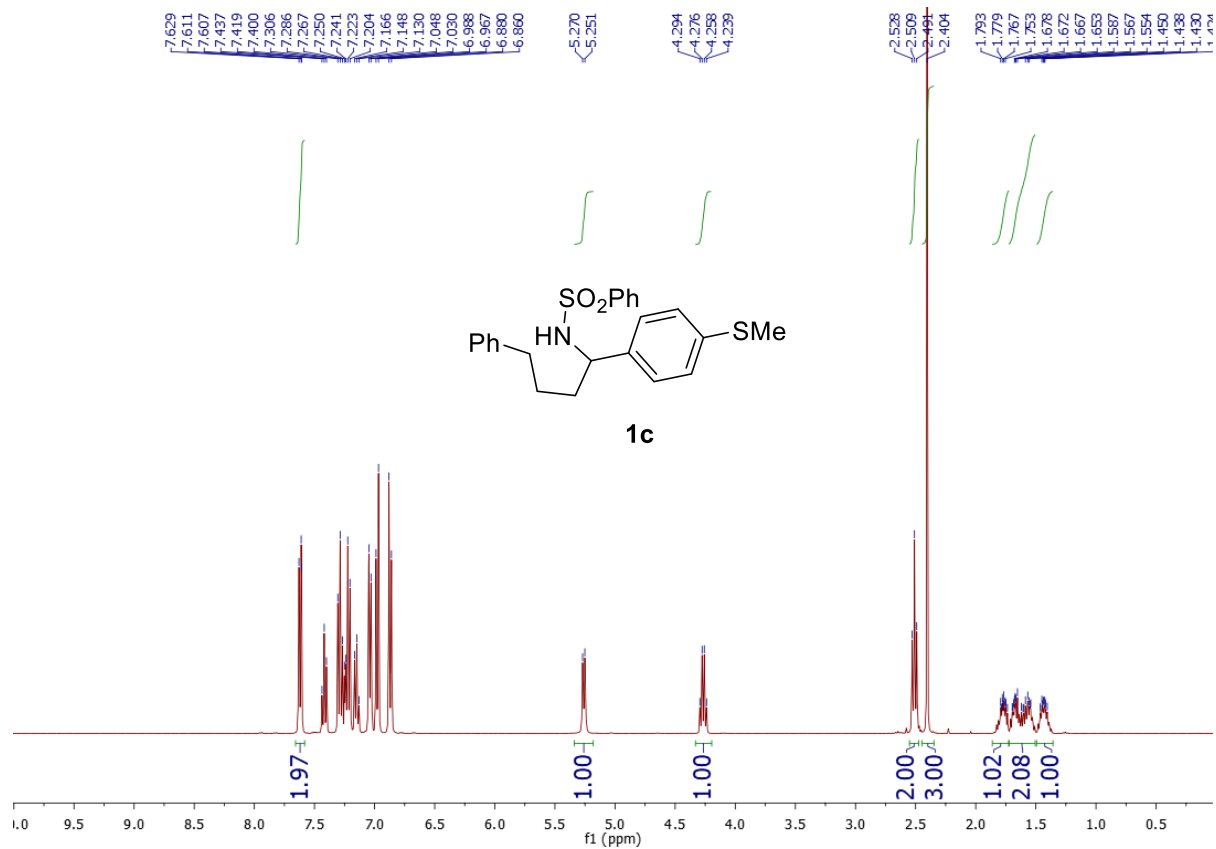
10. References

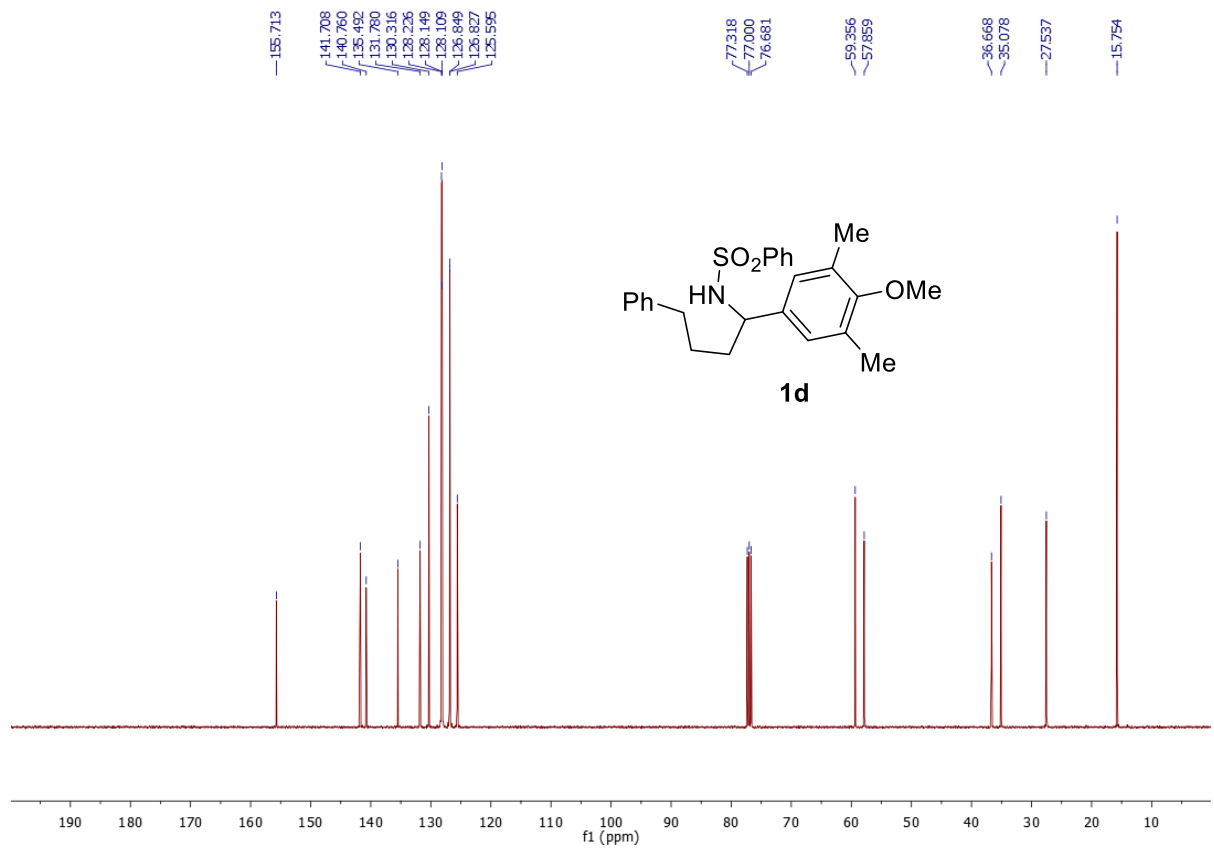
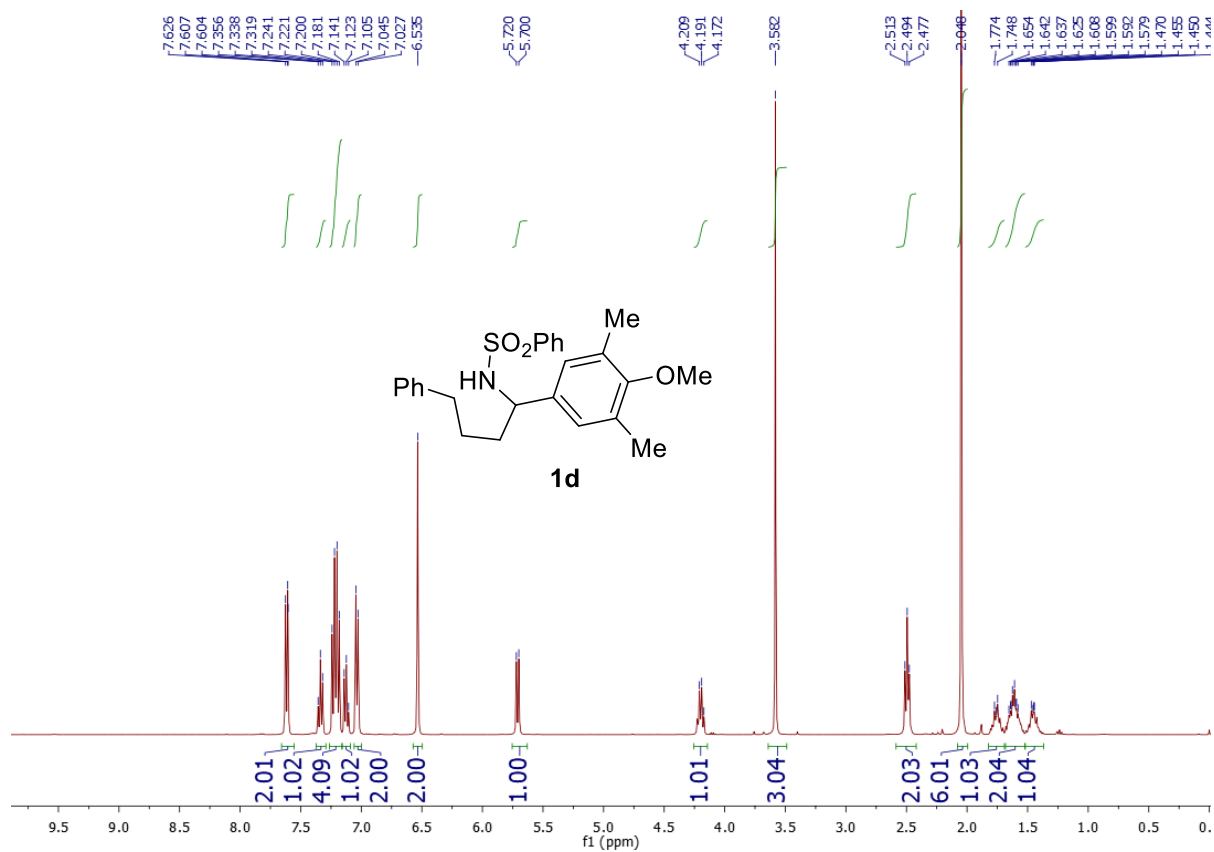
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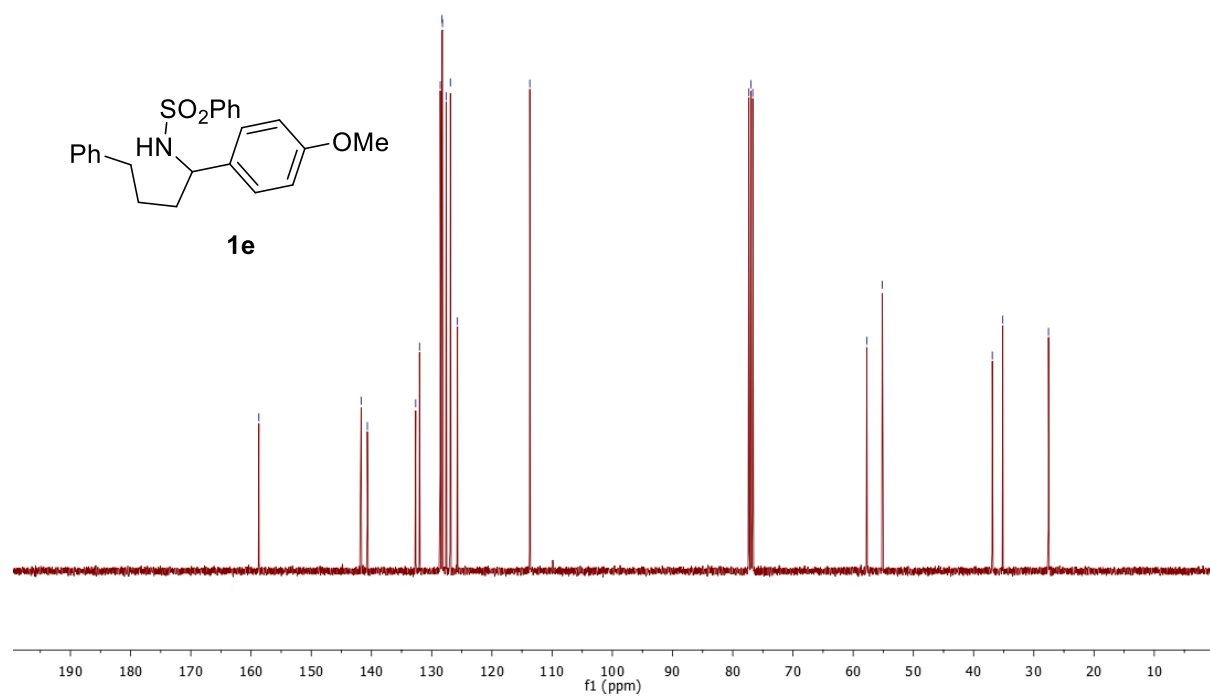
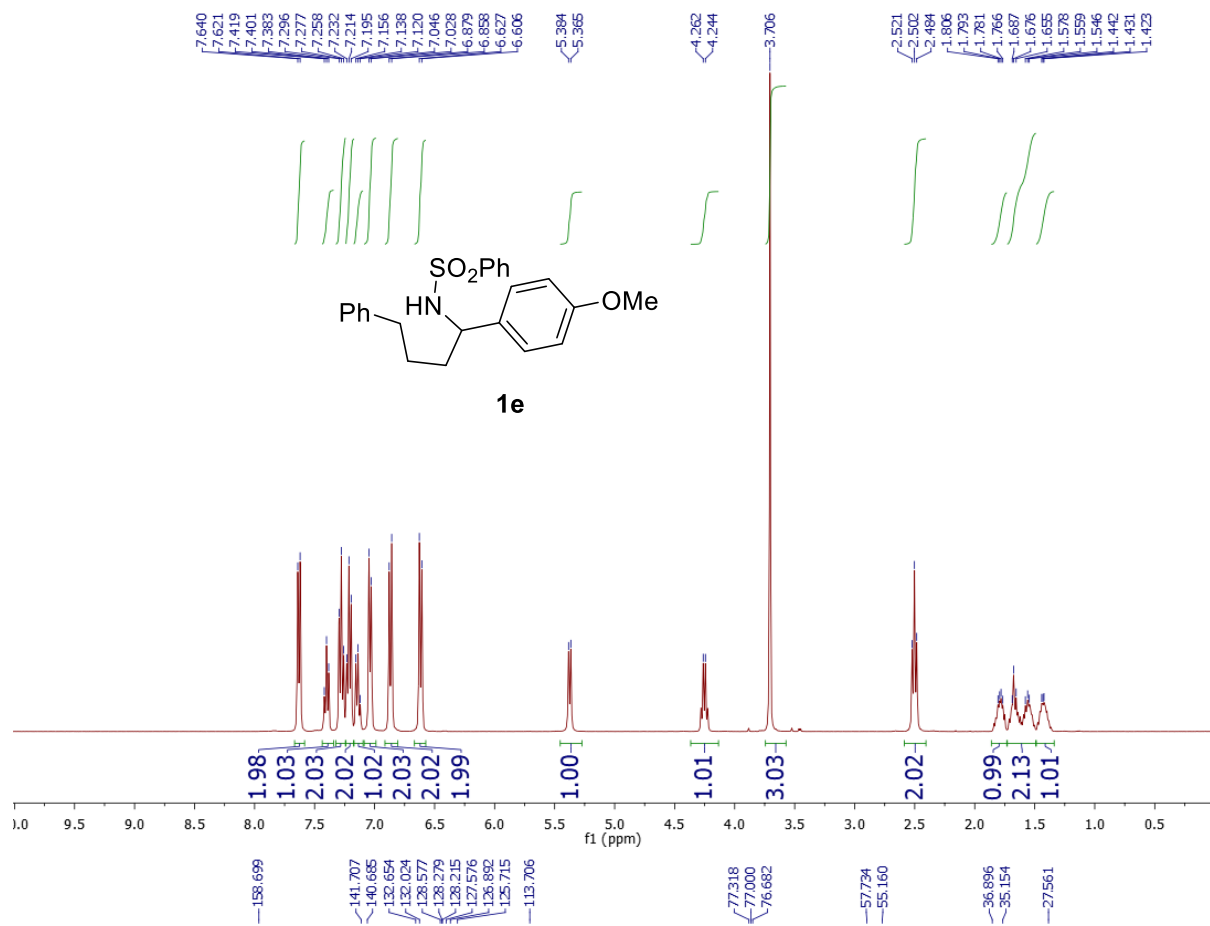
11. NMR Spectra

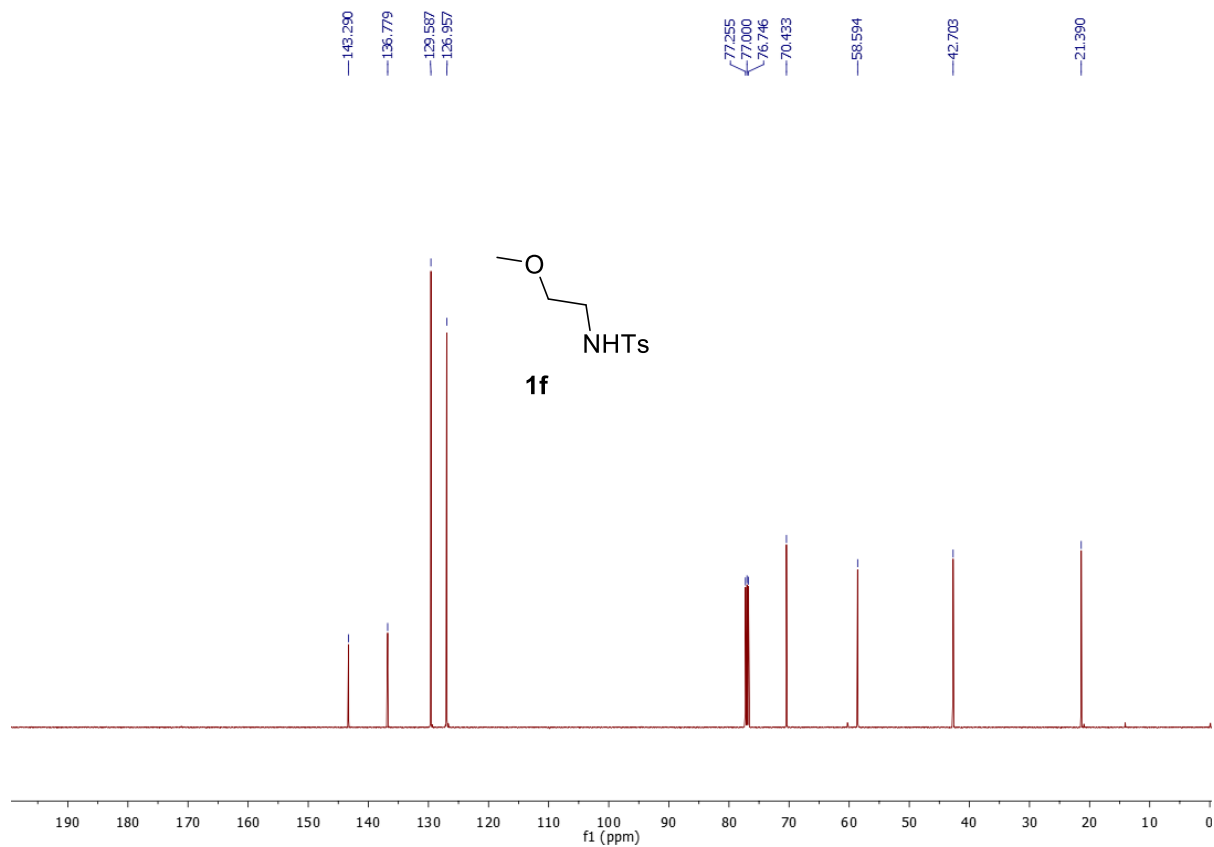
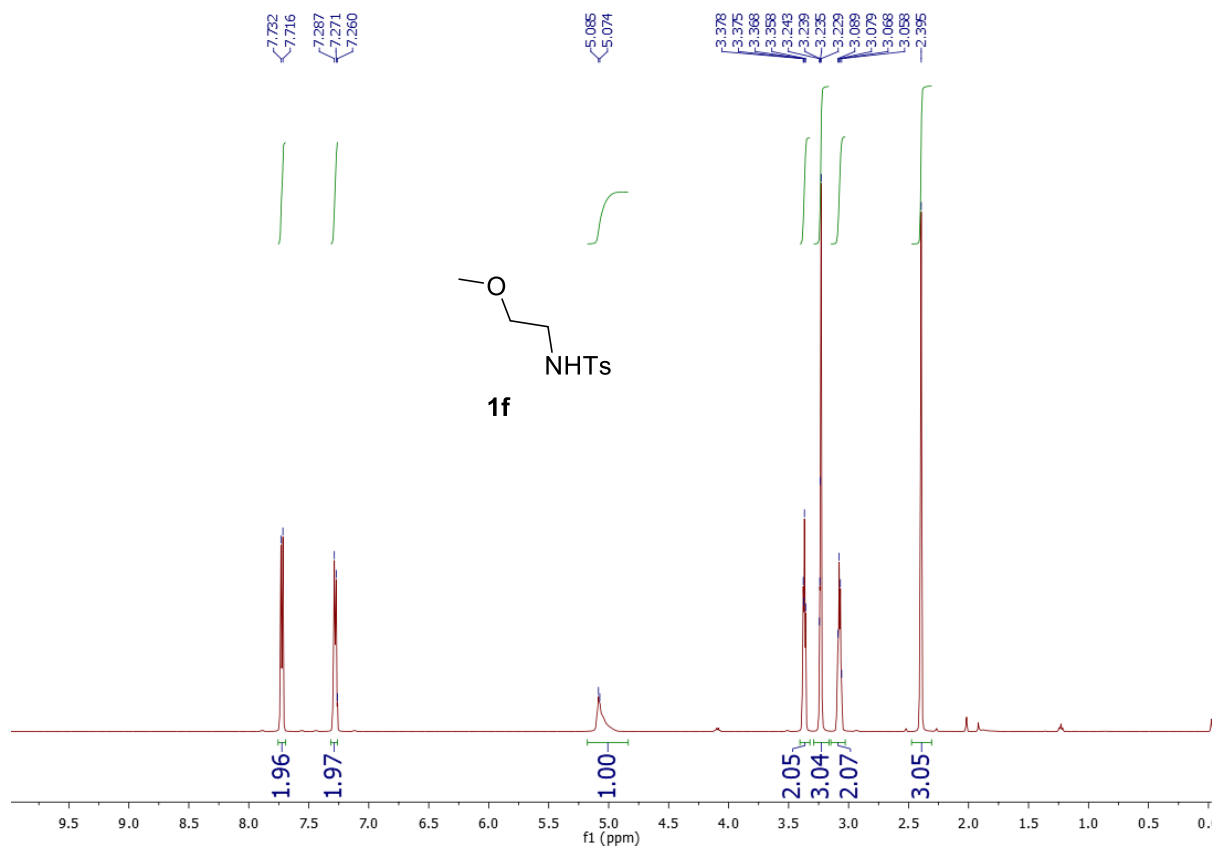


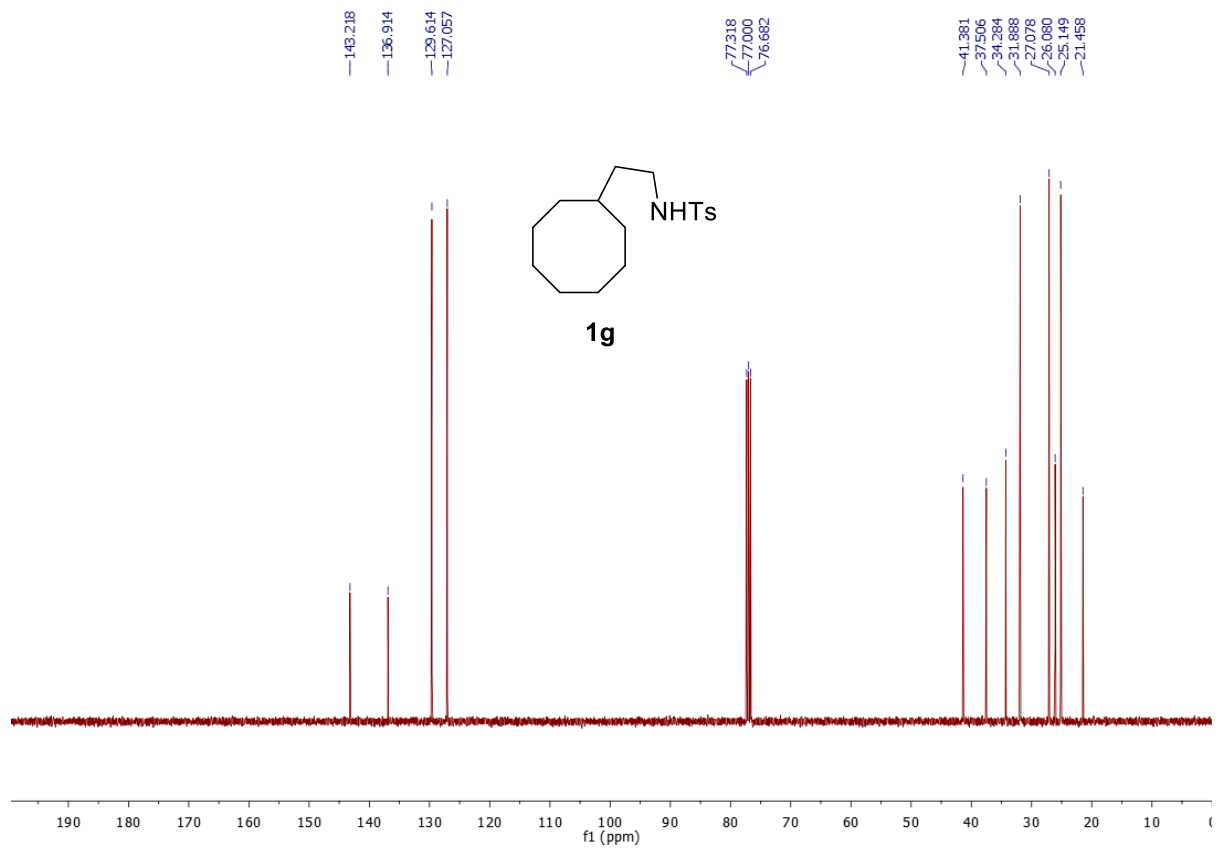
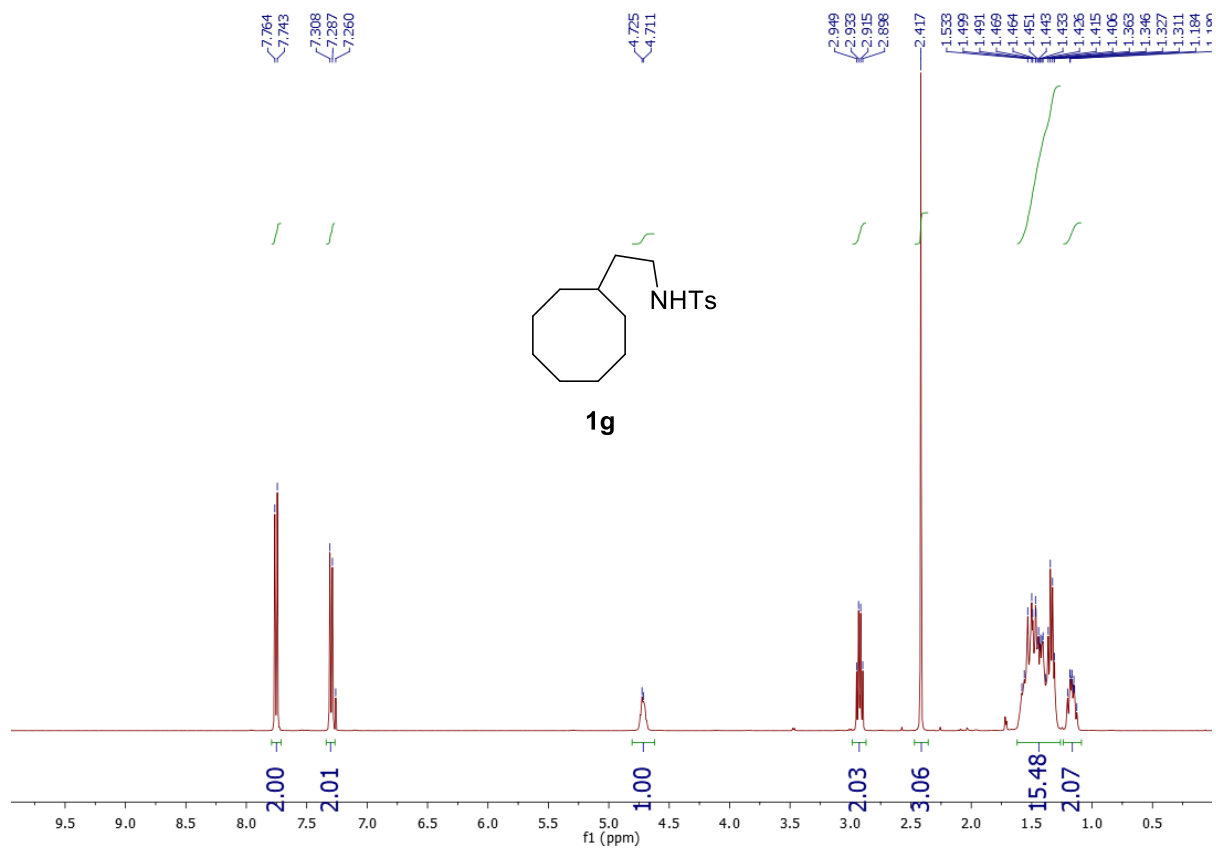


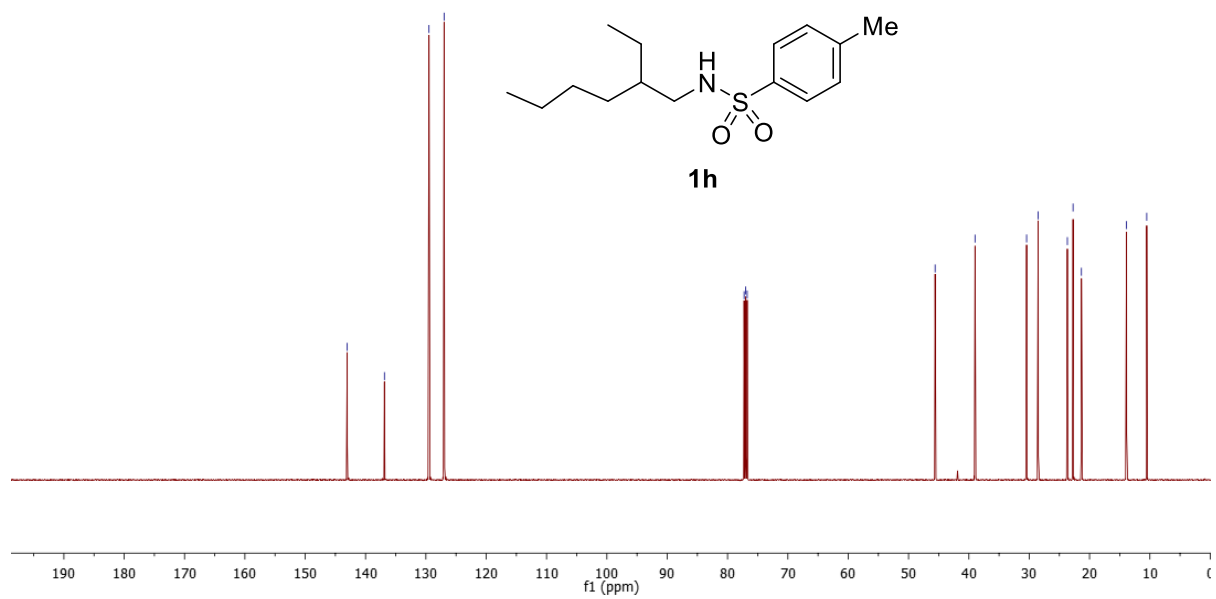
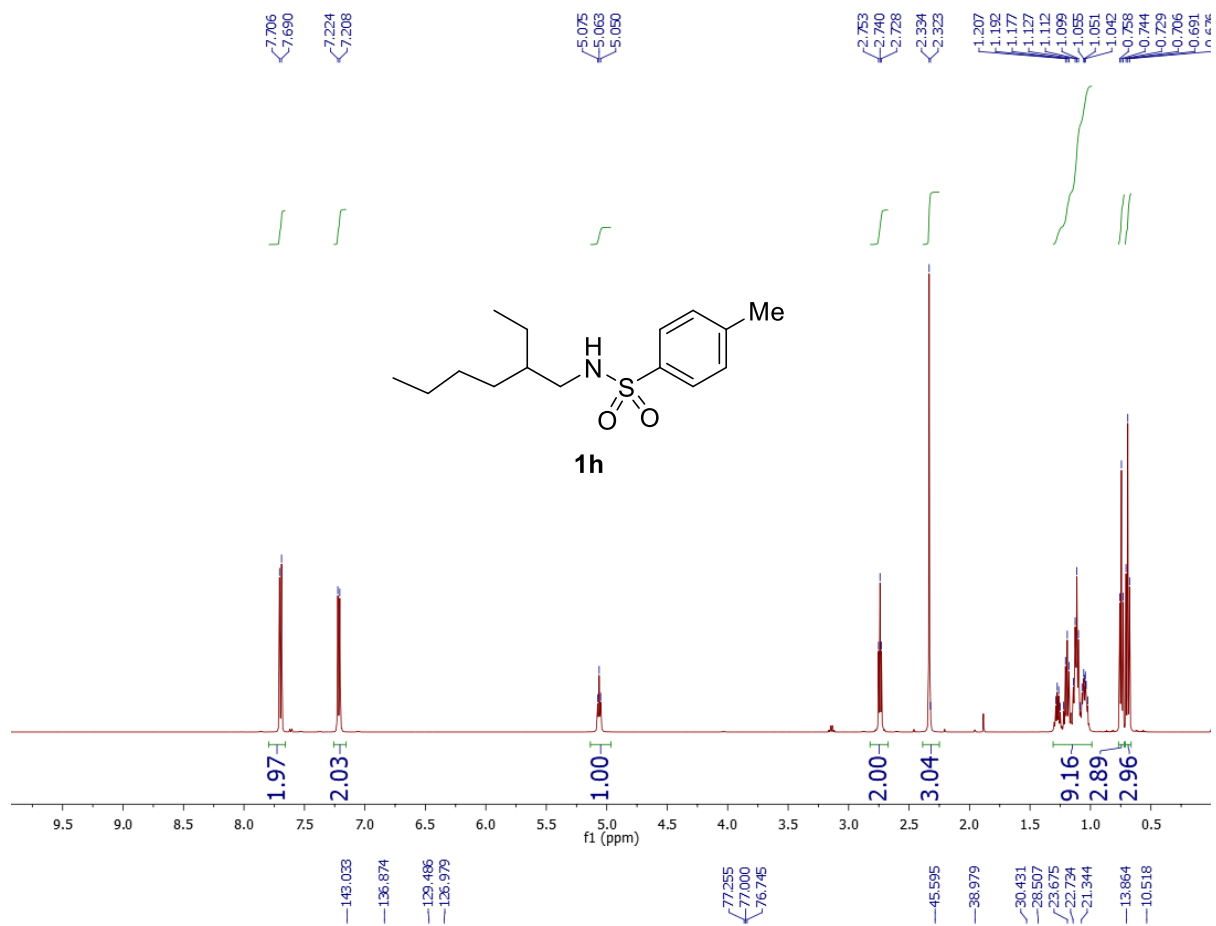


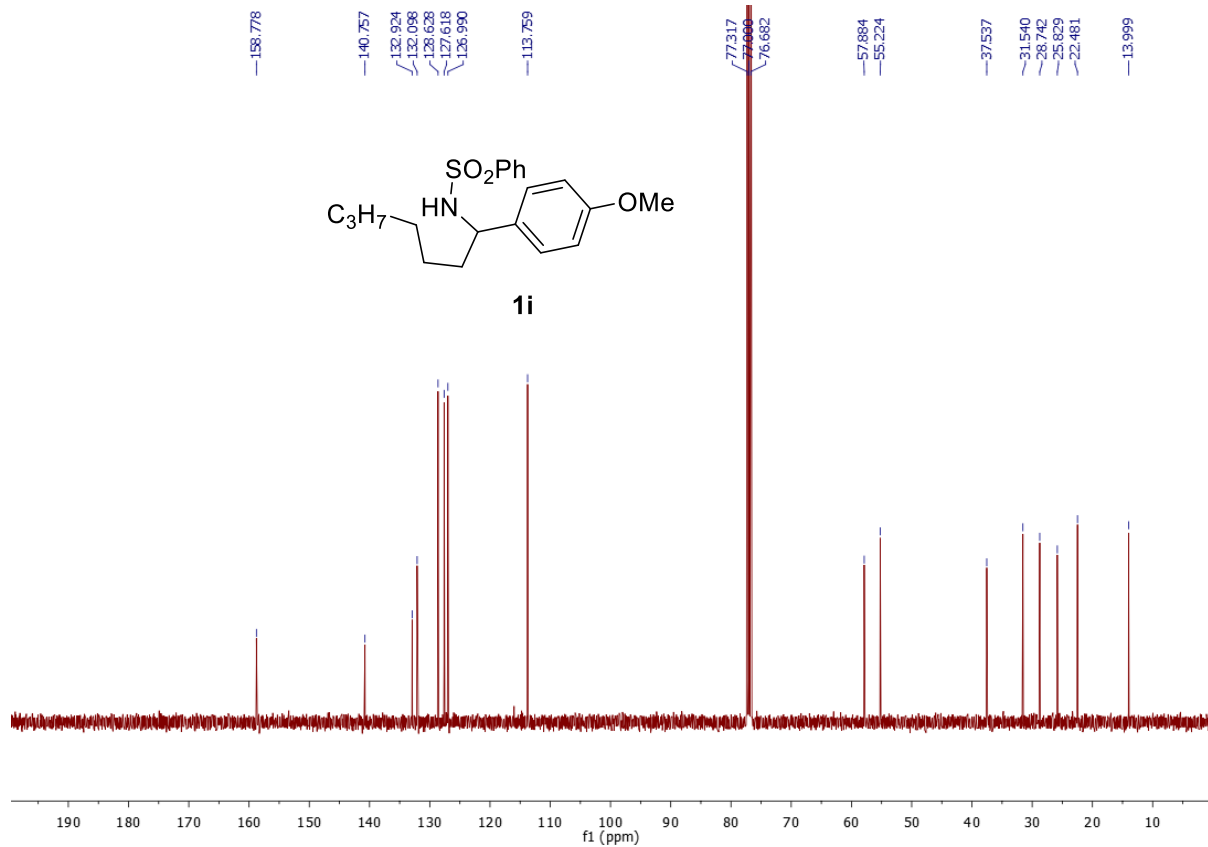
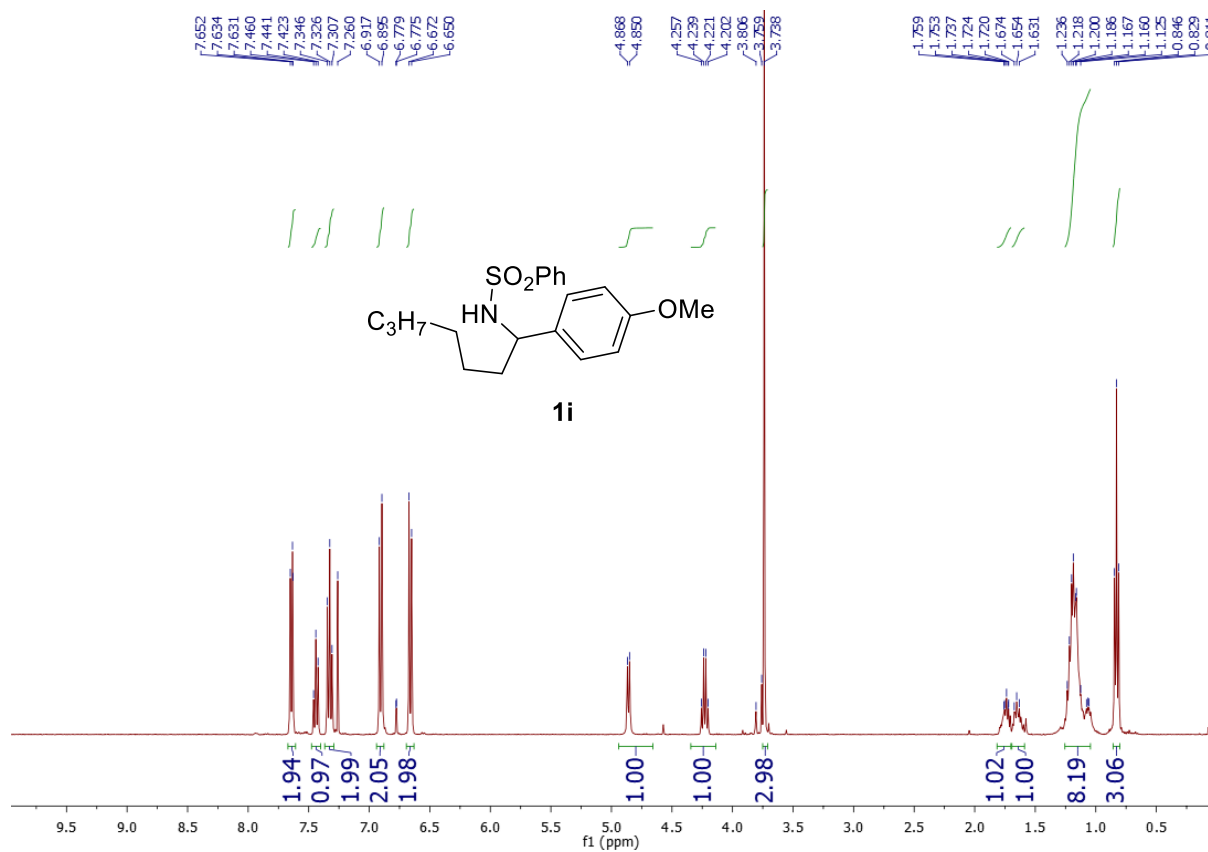


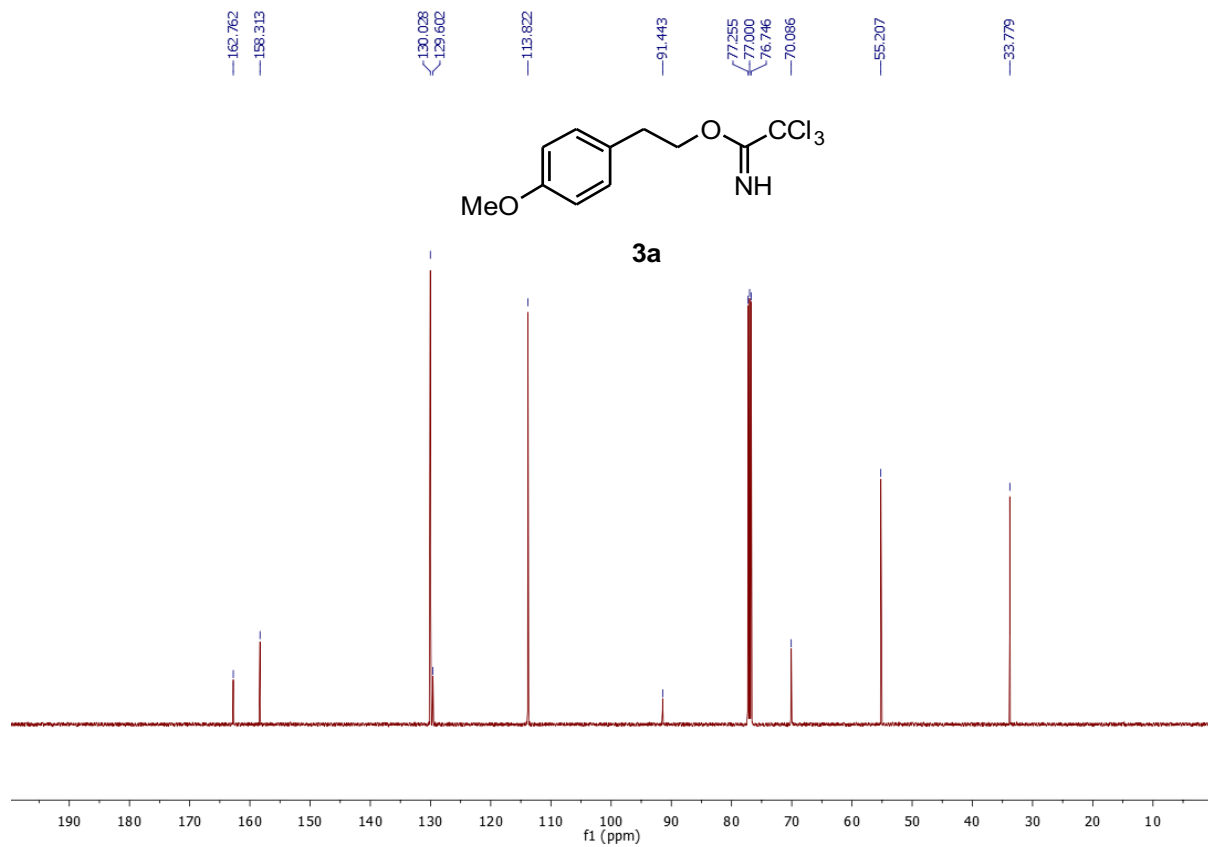
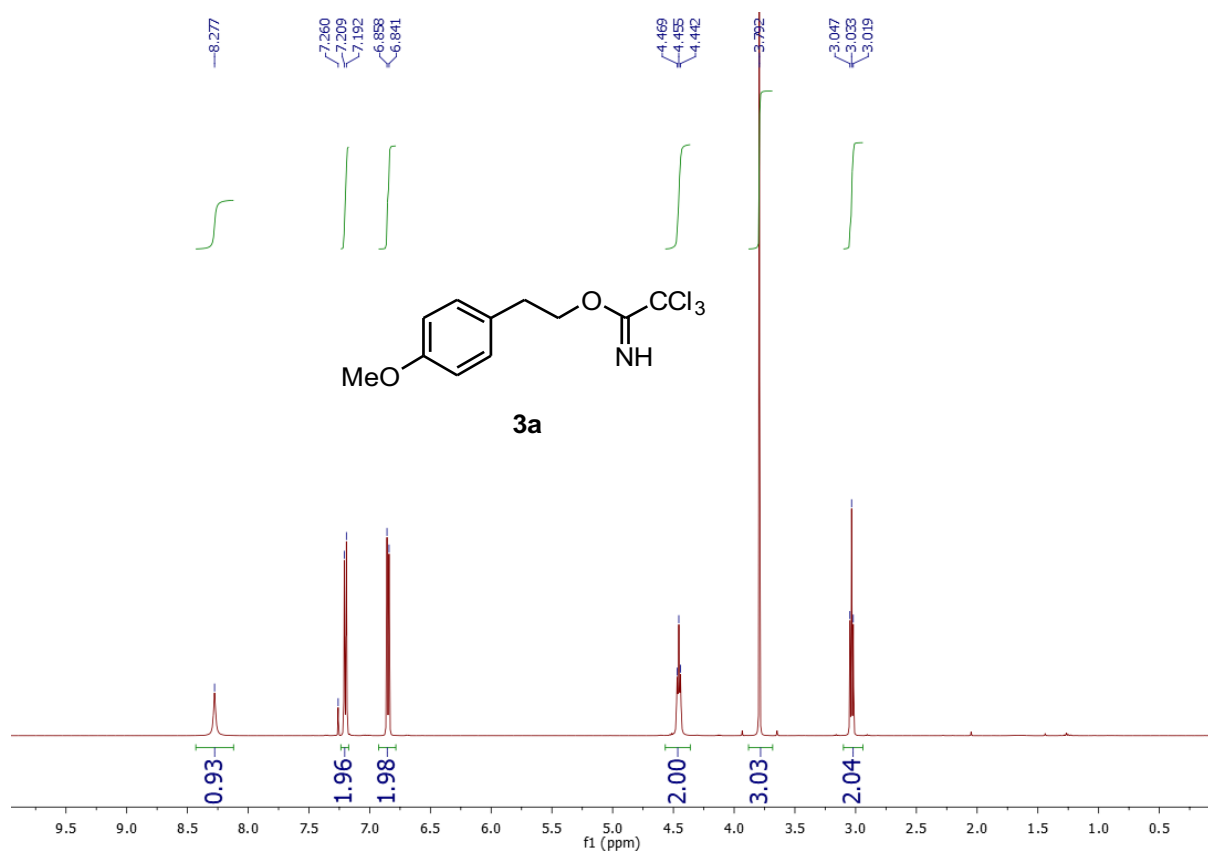


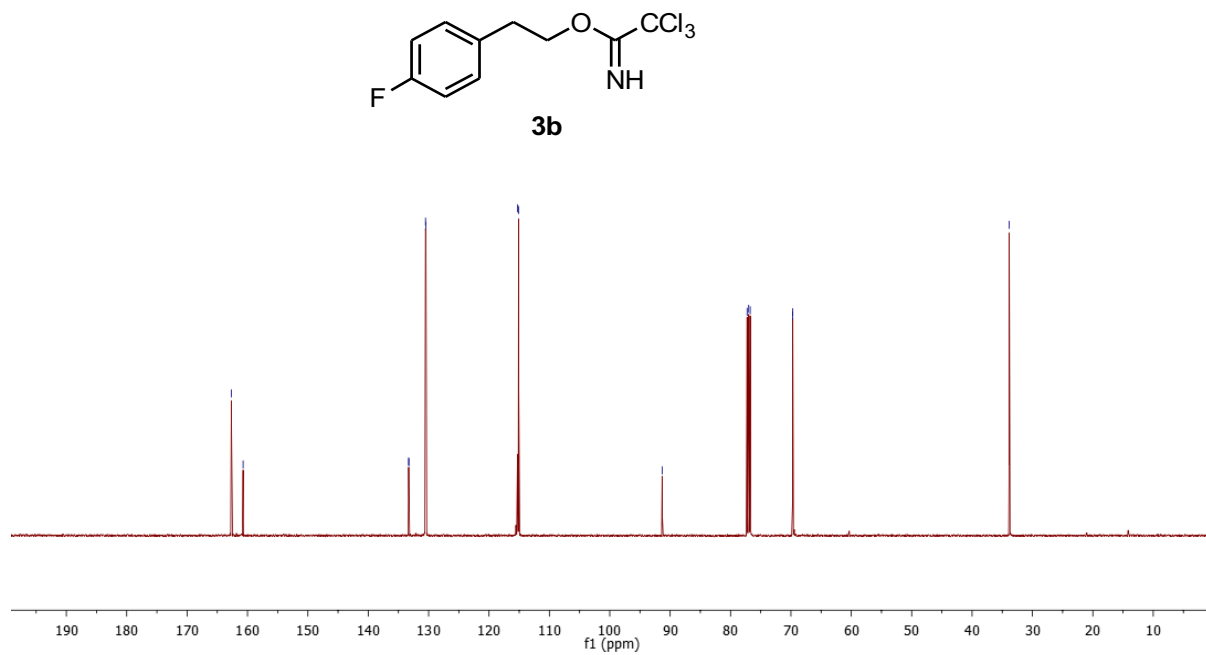
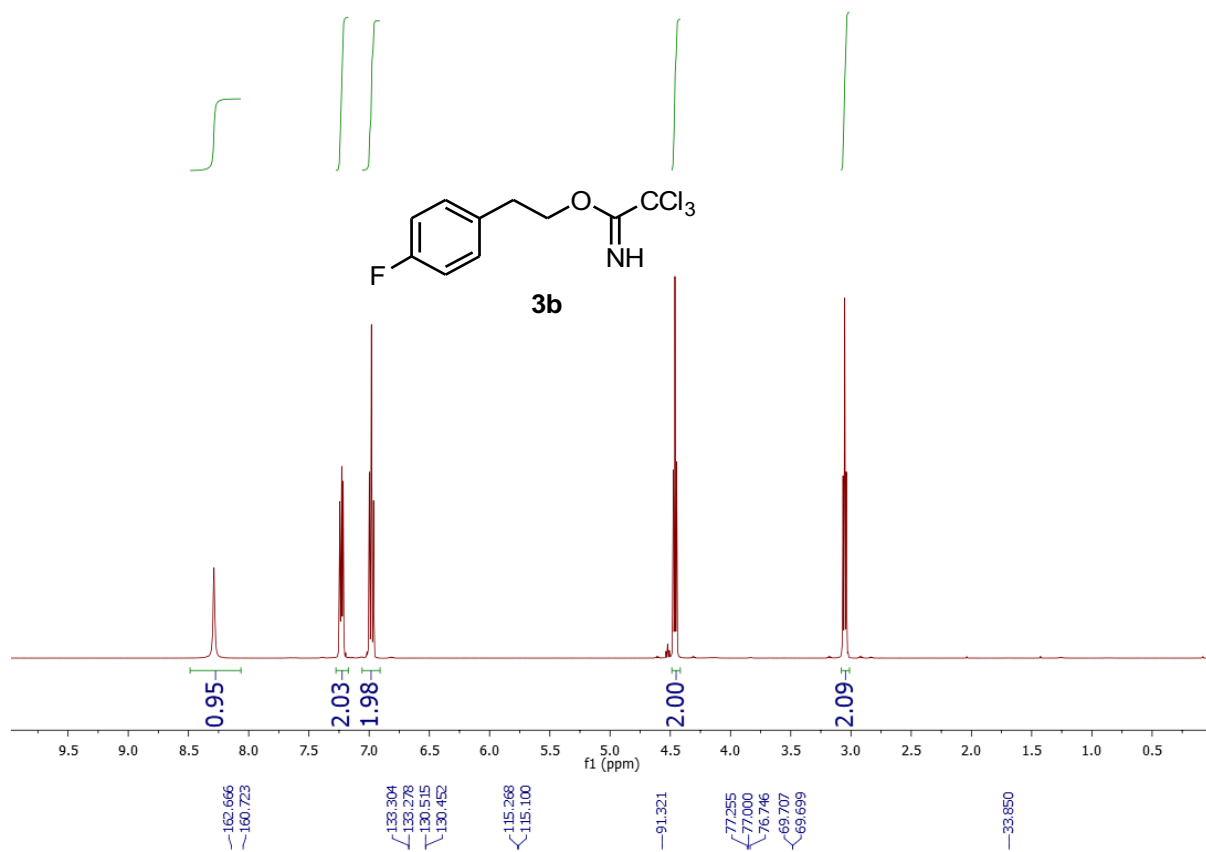


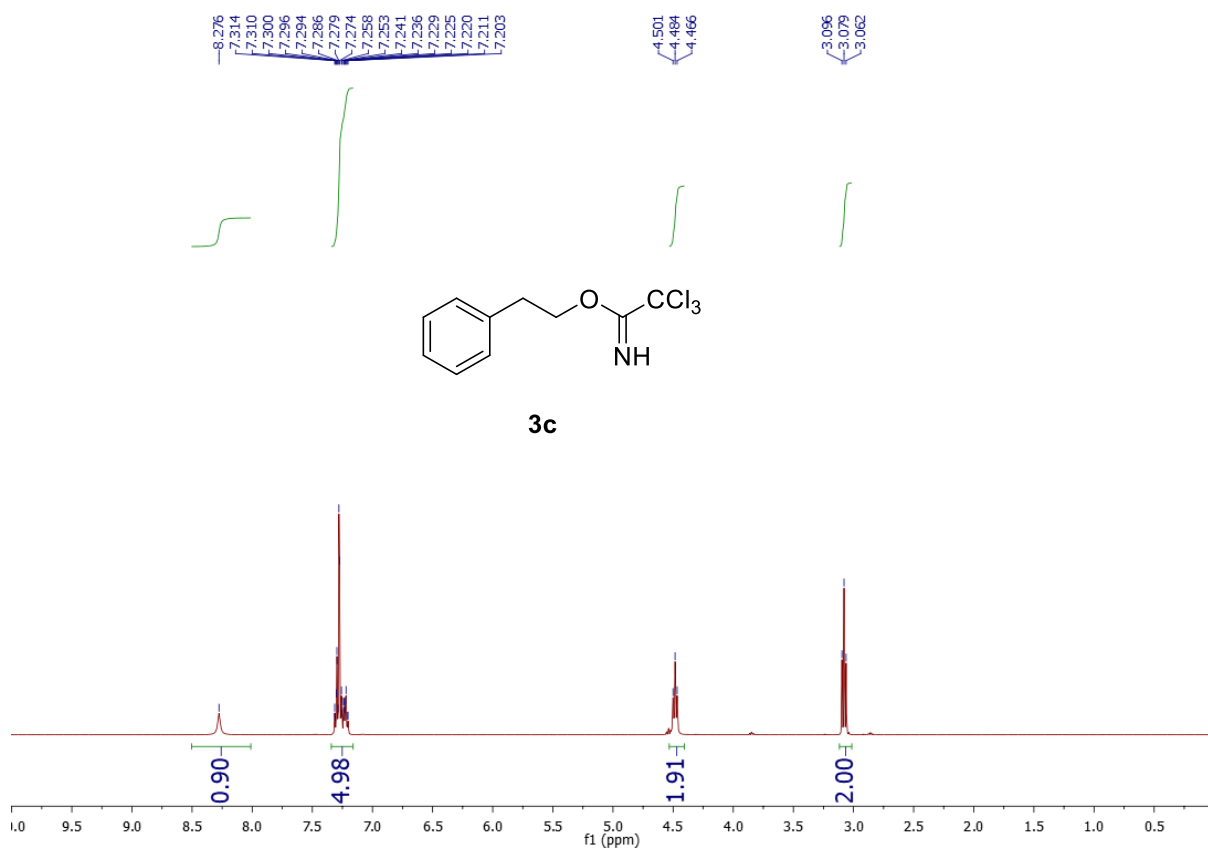
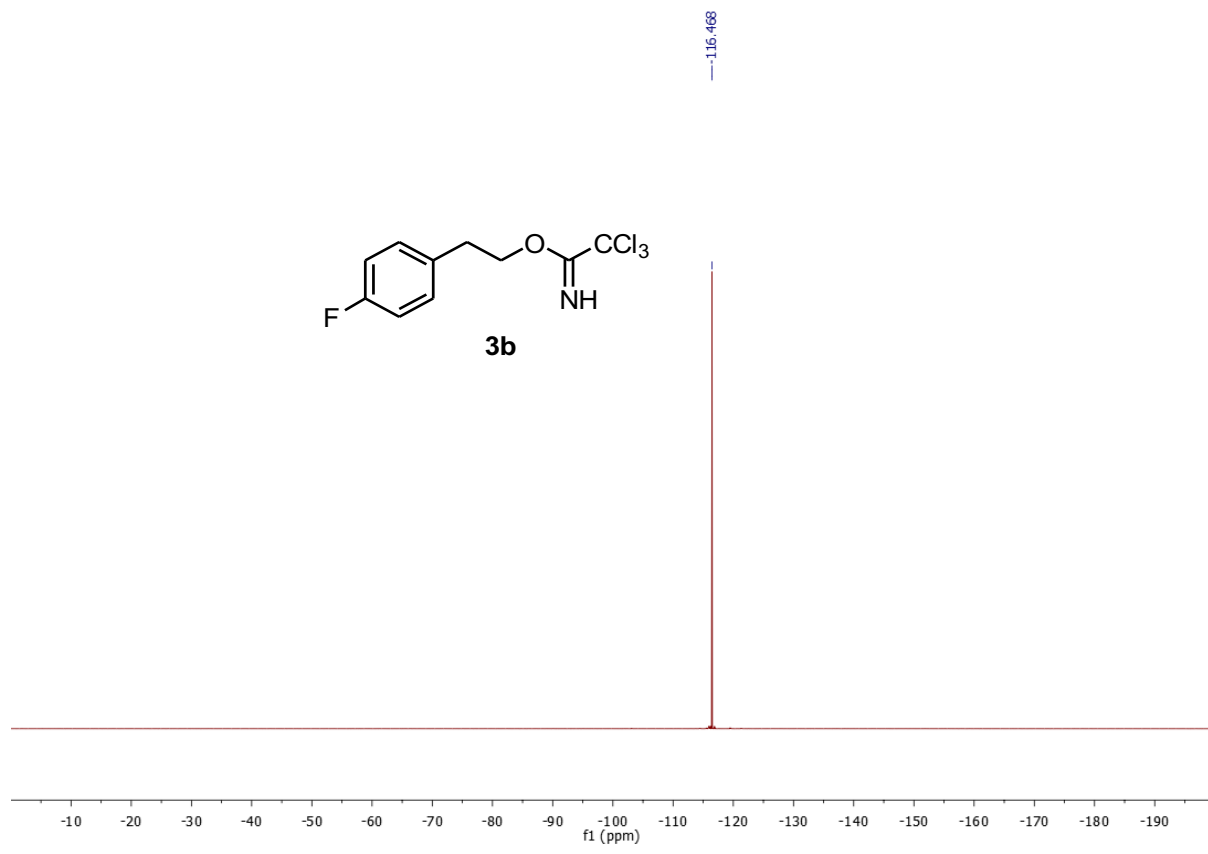


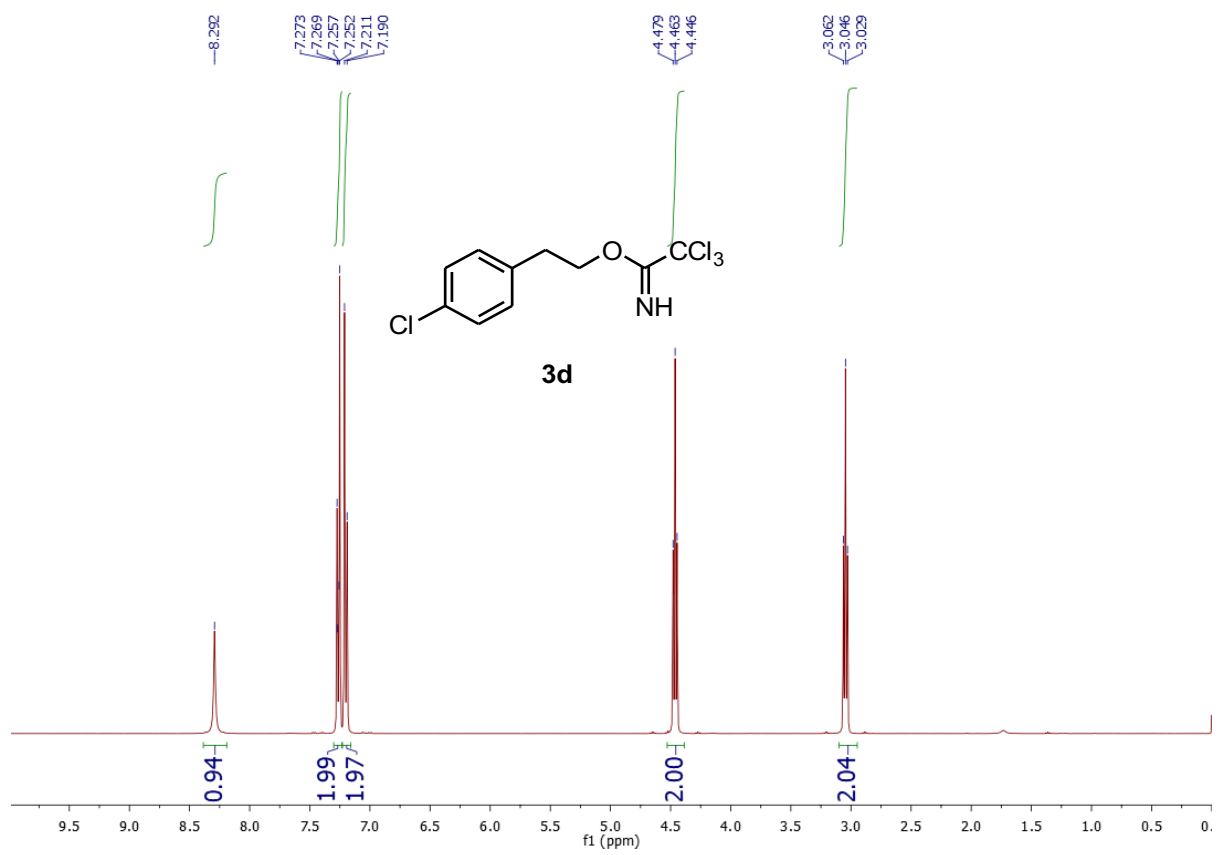
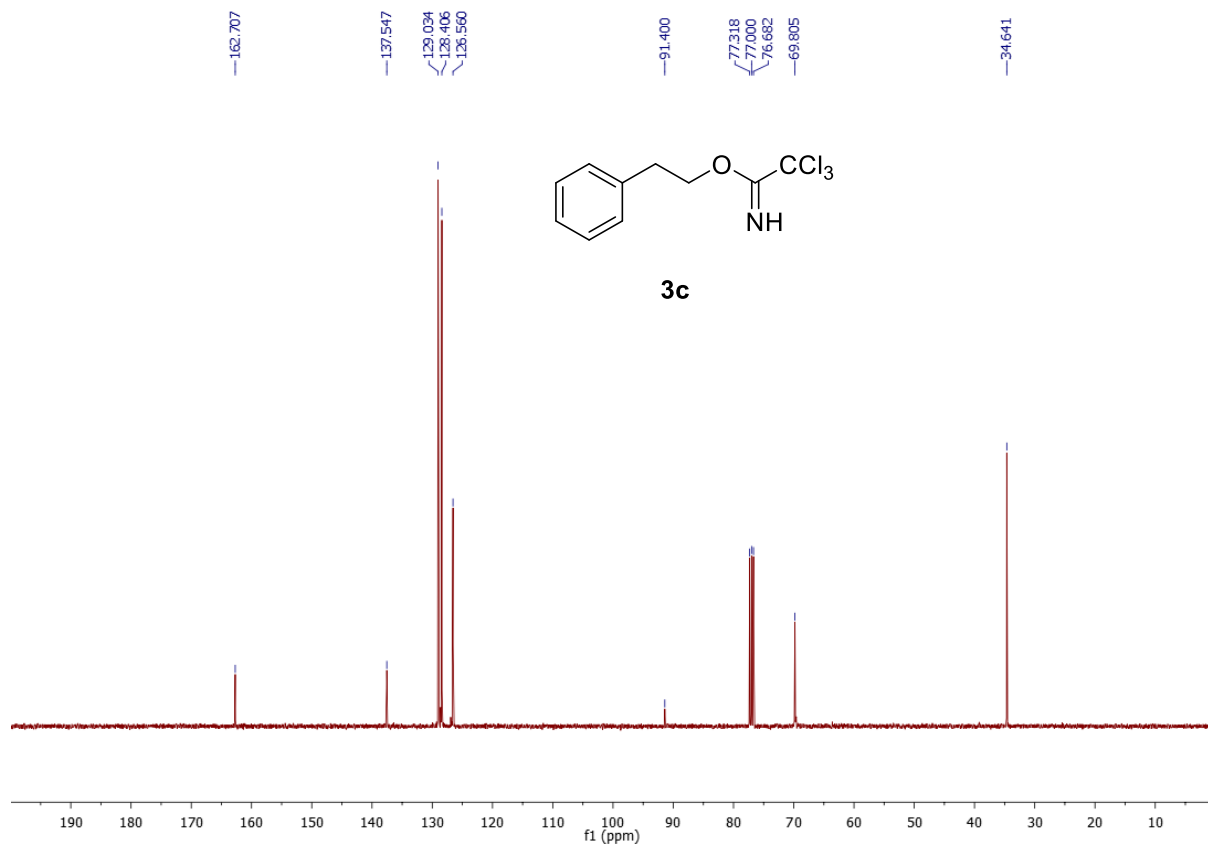


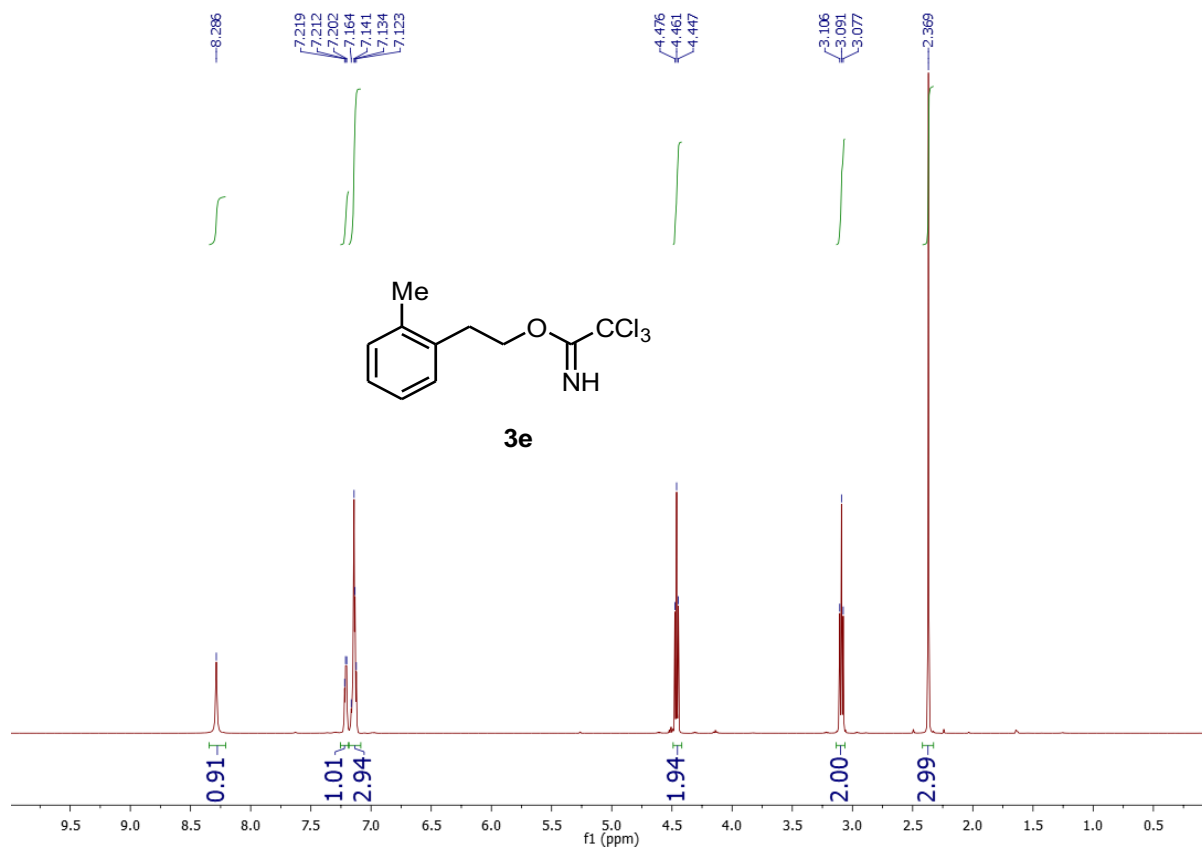
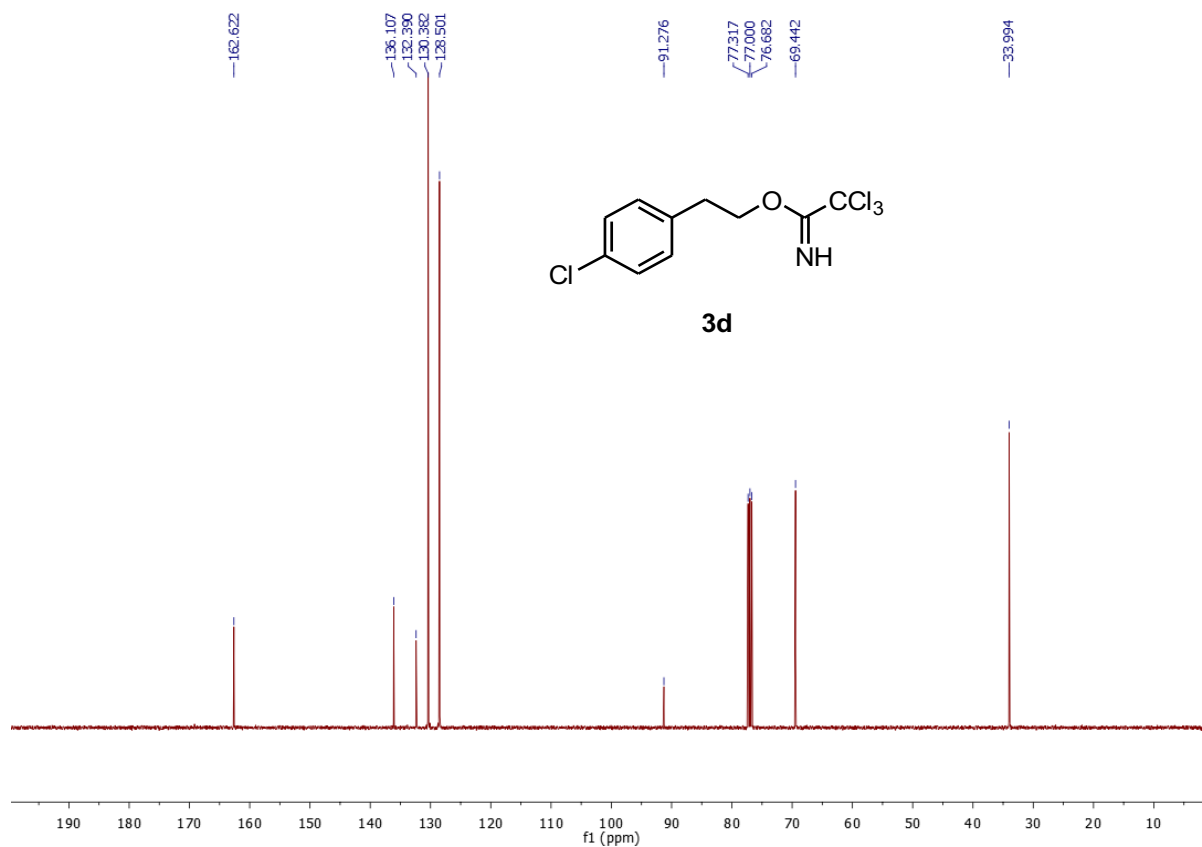


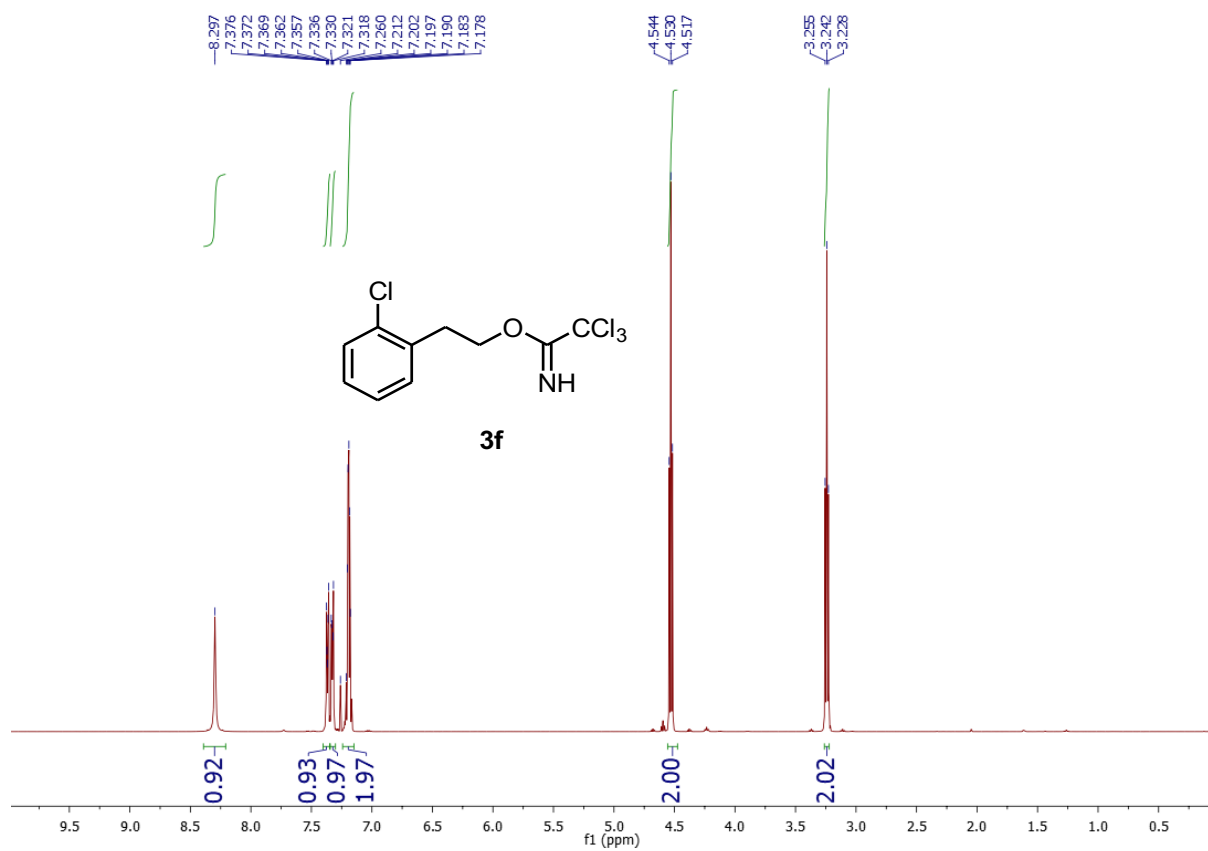
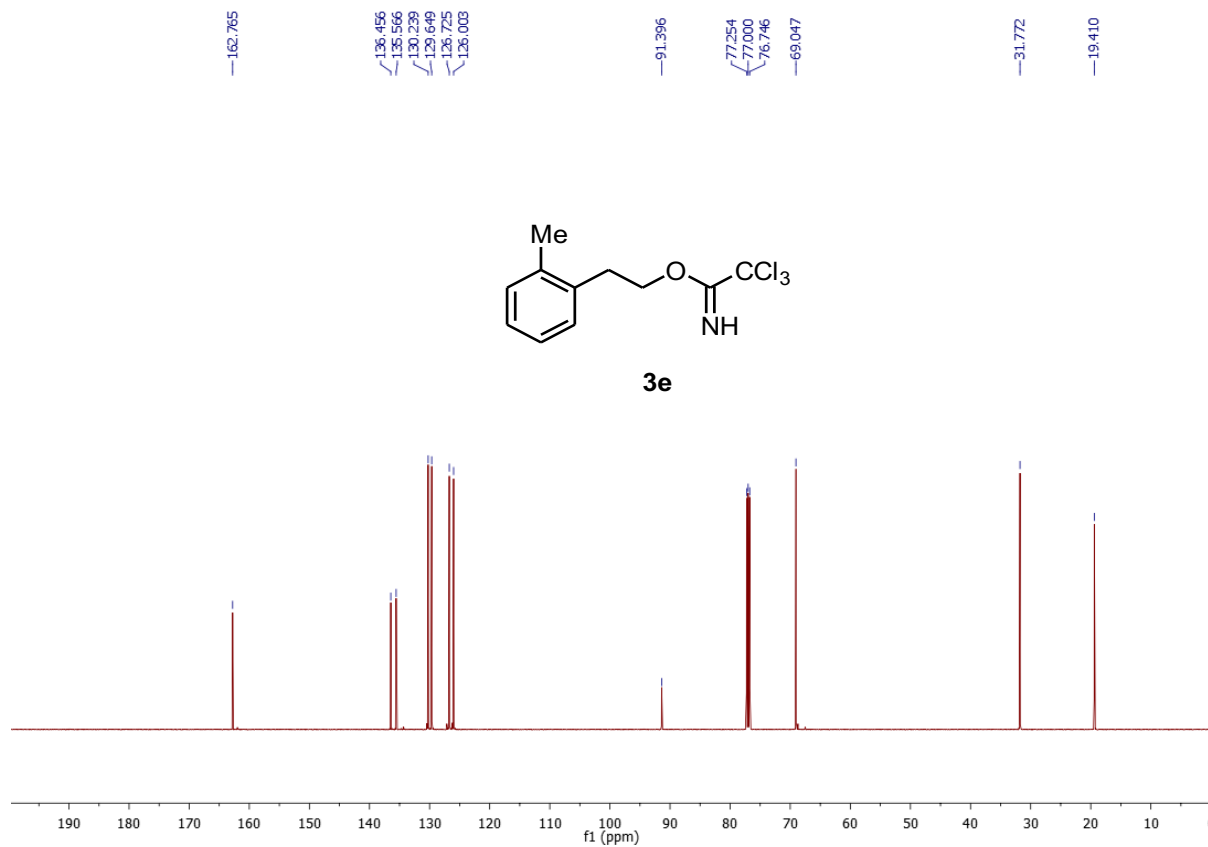


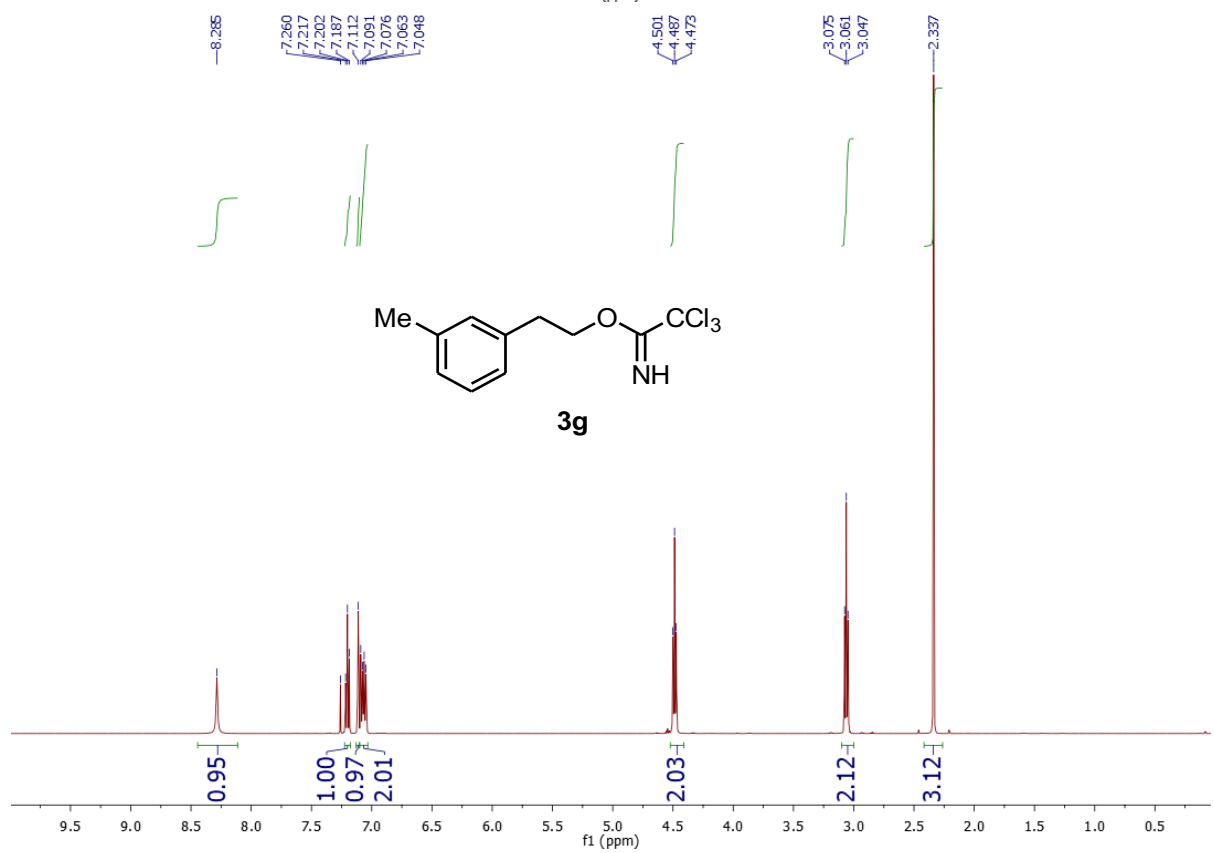
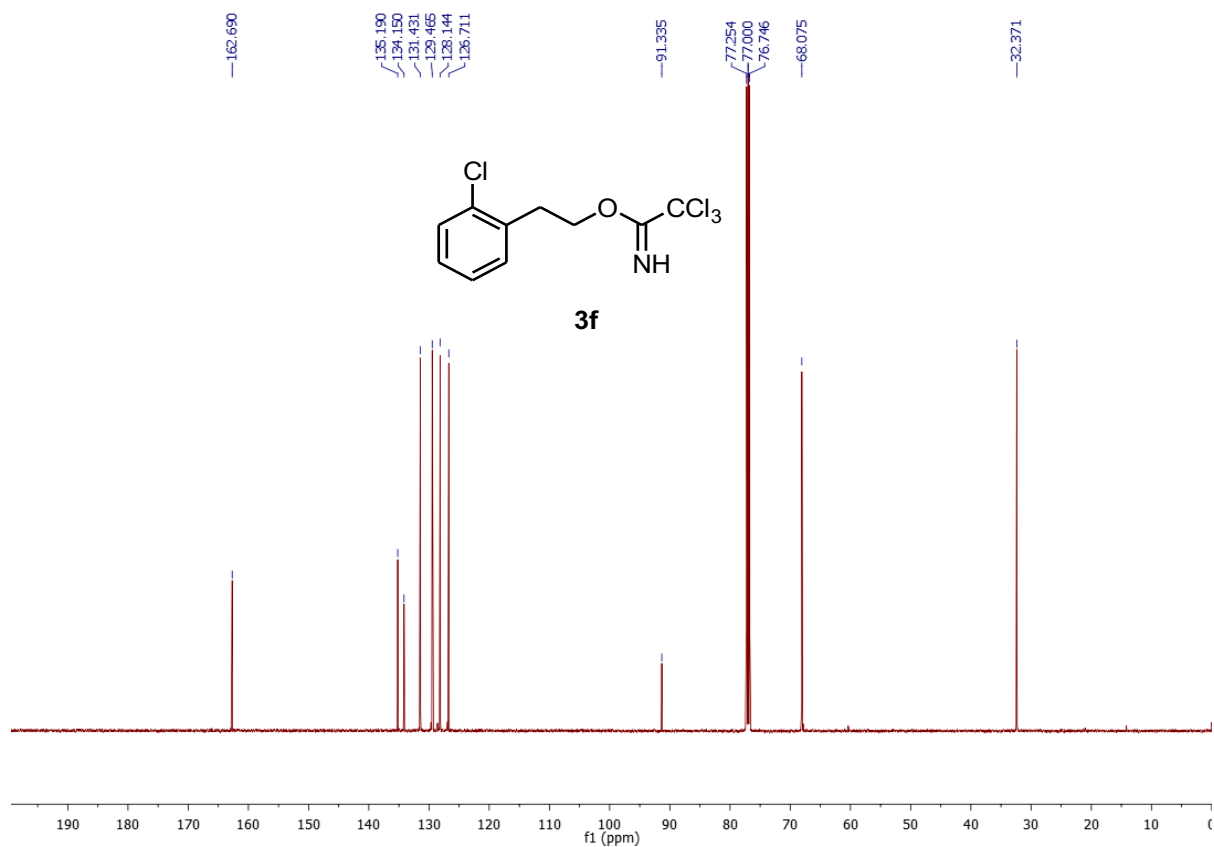


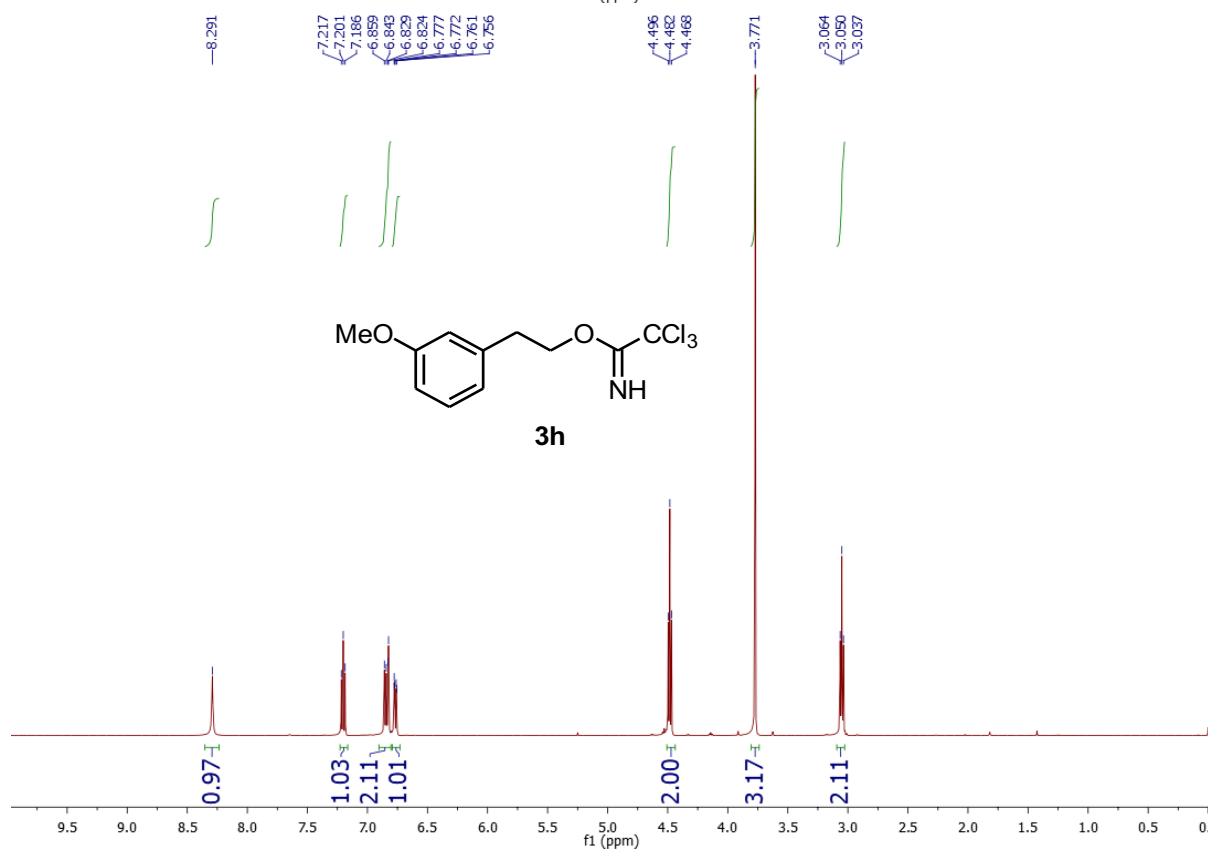
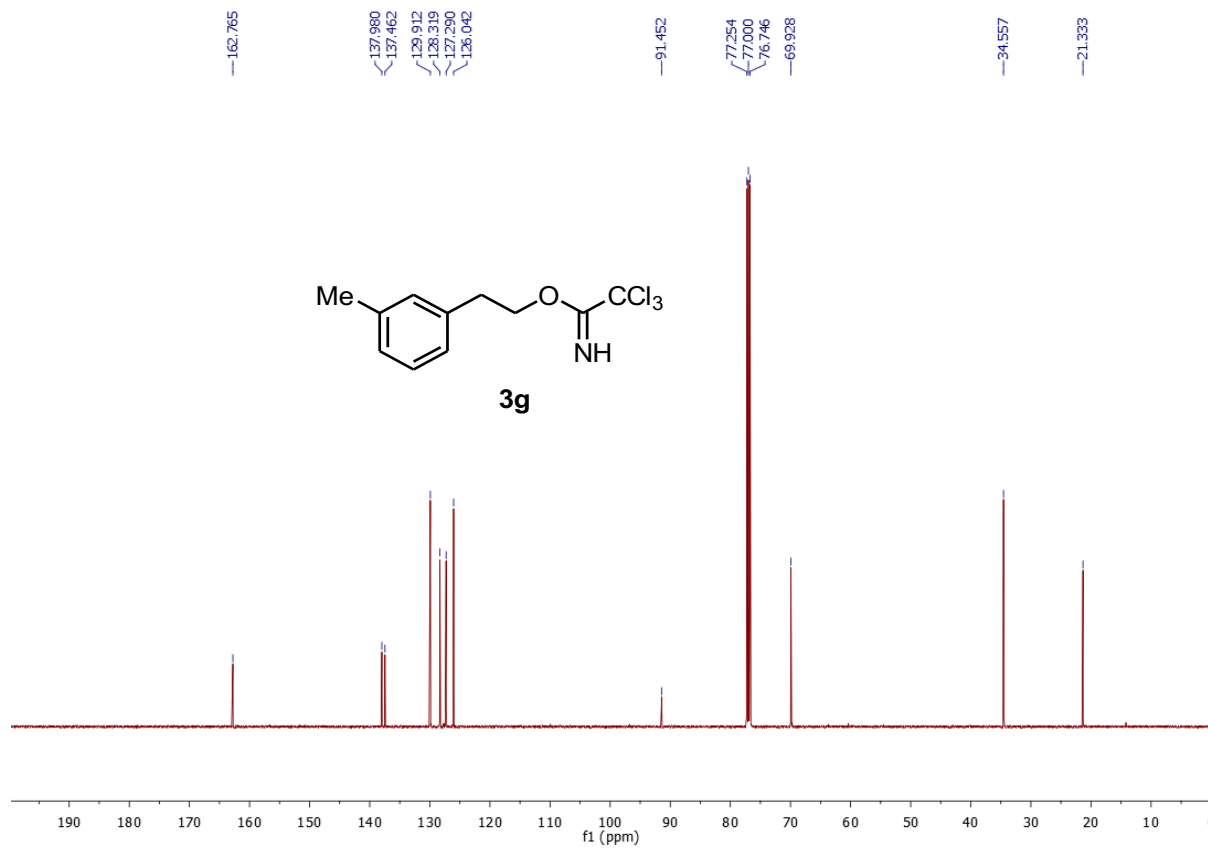


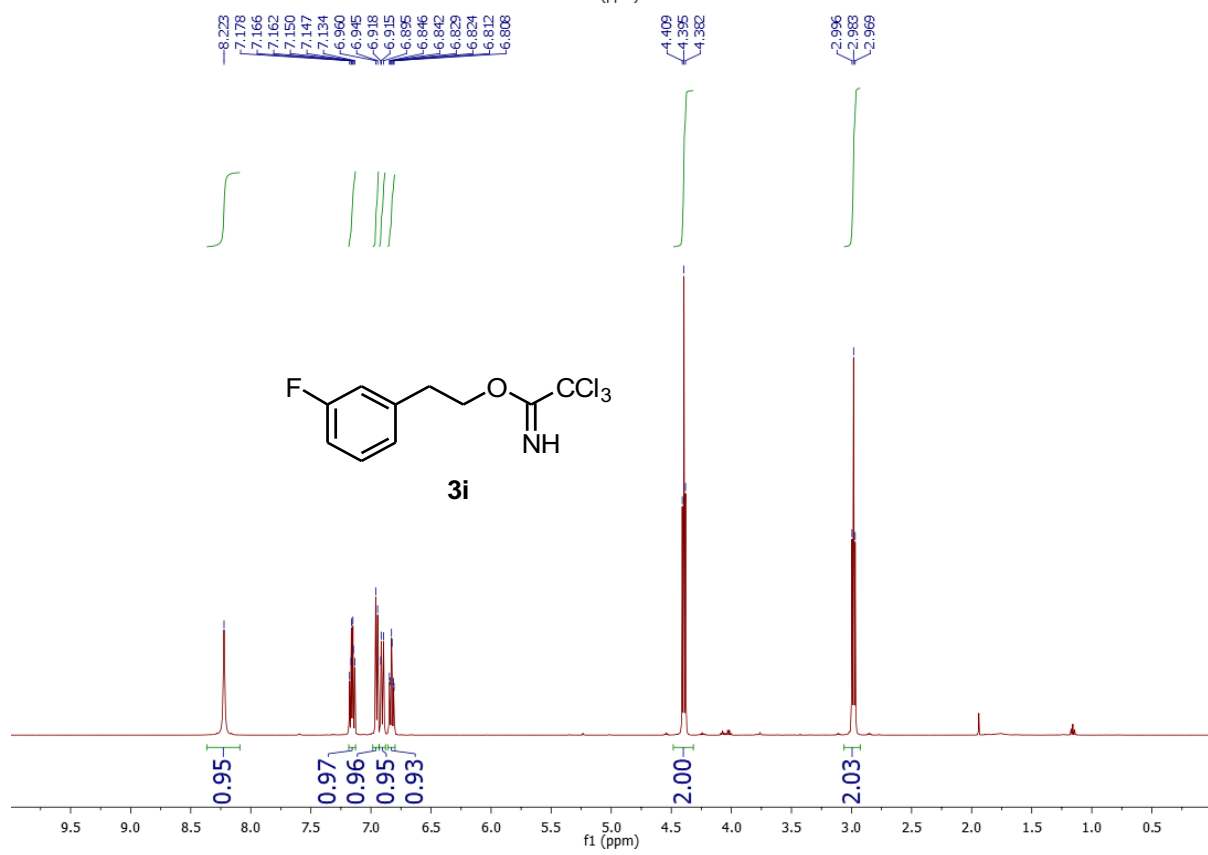
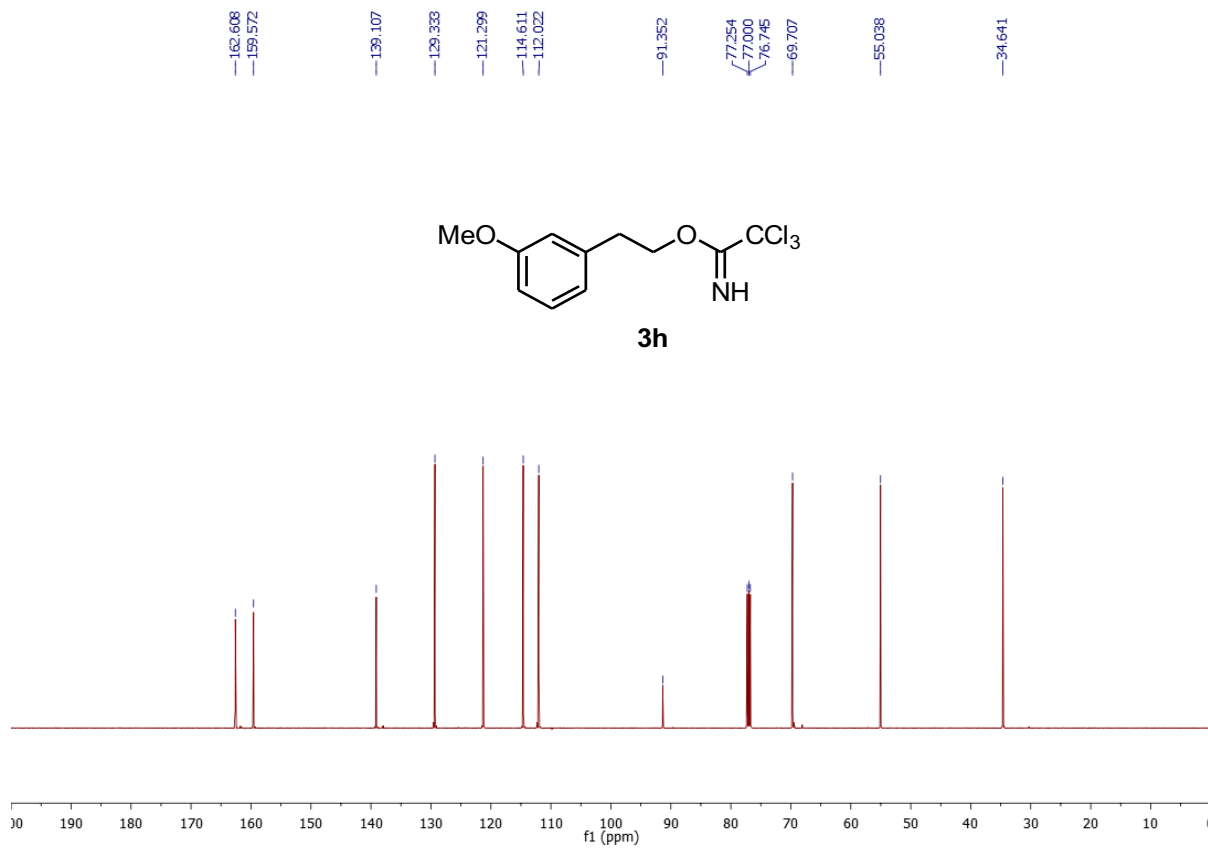












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161.780

140.158
140.099

129.826
129.760
124.647
124.624

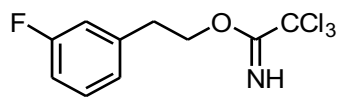
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91.259

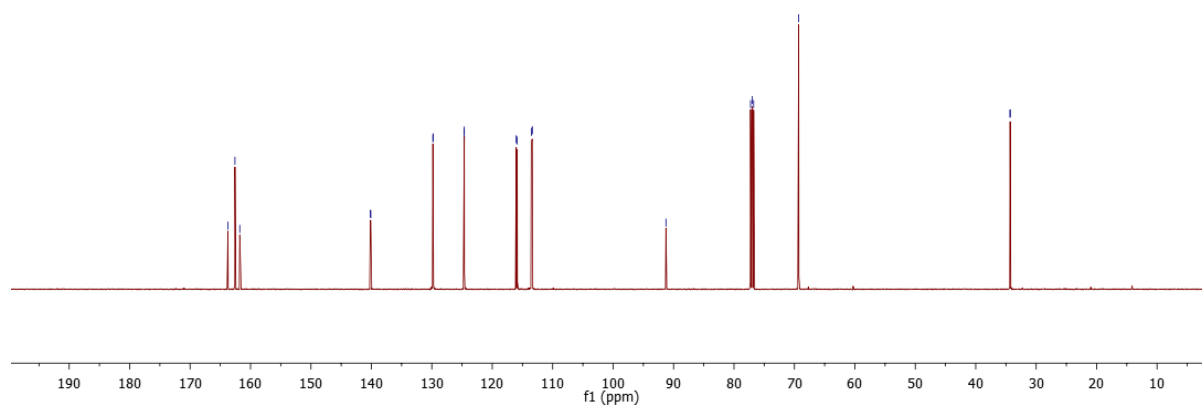
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76.746

69.293

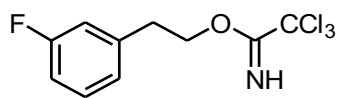
34.320
34.306



3i



113.365



3i

