Electronic Supplementary Information

Organophotoredox-Catalyzed Sulfenylation of Biologically Active Maleimides, Acrylates and Cyclohexenones with Thiols under Ambient Conditions

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1. General Information

All the commercial reagents were used without purification unless otherwise noted. All reactions were performed in oven-dried glass apparatus and were stirred with Teflon-coated magnetic stirring bars. Liquid reagents and solvents were transferred *via* syringe using standard Schlenk techniques. Petroleum ether was used in the boiling range of 60-80 °C. The reactions were irradiated using a regular blue light-emitting diode (LED) strip purchased from market (240 LEDs per meter, 14 Lumens per LED, 12V strip light at 460 nm). Thin layer chromatography was performed using silica gel 60 F-254 pre-coated plates and viewed under UV irradiation. Column chromatography was performed using 100-200 mesh silica gel and eluted with petroleum ether/ethyl acetate solvent mixture. ^{1}H NMR spectra were recorded on a 400 MHz and 300 MHz spectrometers in $CDCl_3$ with TMS as internal standard. ^{13}C NMR spectra were recorded on a 100 MHz and 75 MHz spectrometer. Chemical shifts (δ) are reported in parts per million (ppm) relative to the residual solvent $CDCl_3$ signal at 7.26 ppm for ^{1}H NMR, multiplicities (abbreviations are as follows: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets, m = multiplet, br = broad) and at 77.16 ppm for ^{13}C NMR. Data for ^{1}H NMR spectra are reported as follows: chemical shift (multiplicity, number of hydrogen and coupling constants). Mass spectra were obtained using Q-TOF-HRMS spectrometer using electron spray ionization. Density Functional Theory (DFT) computations were accomplished by employing the Gaussian 16 program suite.

2. Experimental

2.1 Experimental Setup

12 W blue LED light strip (5 meters) attached with a 12 V; 12 Amp AC adaptor was purchased from commercial source. The reaction was carried out in 15 mL borosilicate glass vial with cap and rice bead magnets purchased from Tarsons (Figure S1). At the time of reaction, the distance of LED lights from the borosilicate glass vials was kept 3.5-4 cm and the room temperature (25-30 ^{o} C) was maintained for all the reactions.

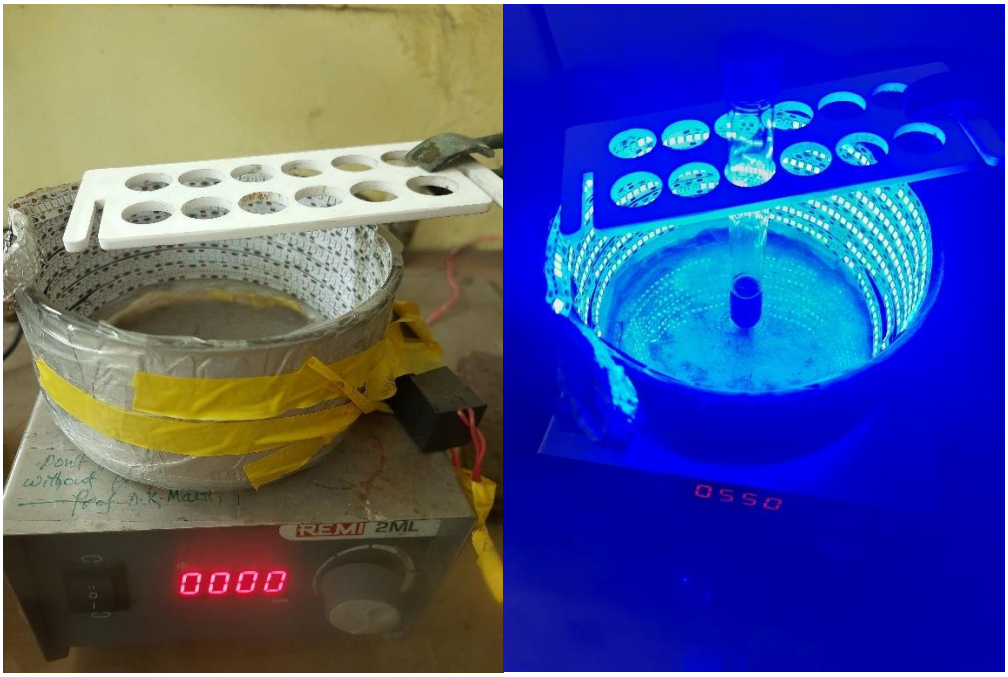


Figure S1. Laboratory Light Setup used for Irradiation of the Reaction.

2.2 On/Off Experiment

An on/off irradiation experiment was conducted using visible light with starting materials **1a** and **2a** under optimized conditions to confirm the effect of photo-irradiation. From this experiment it was evident that continuous irradiation of visible light is important for the reaction to progress (Figure S2).

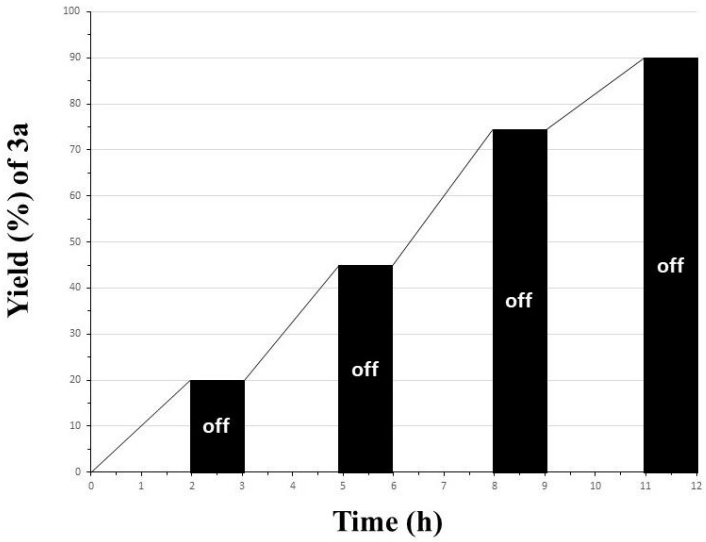


Figure S2. On-Off experiment.

2.3 Fluorescence Quenching Experiments

Formulation of solution: 4-Chlorothiophenol **2a** (7.2 mg) was dissolved in CH_3OH in a 5 mL volumetric flask to obtain a 0.01 M solution. N-phenyl maleimide **1a** (43.3 mg) was dissolved in CH_3OH in a 25 mL volumetric flask to obtain 0.01 M solution. Product **3a** (15.8 mg) was dissolved in CH_3OH in a 5 mL volumetric flask to obtain a 0.01 M solution. The photocatalyst Eosin Y (3.5 mg) was dissolved in CH_3OH in a 25 mL volumetric flask, shaken well, then 5 mL of the solution taken out in a volumetric flask and the volume was made up to 25 mL with CH_3OH, setting the concentration to 0.08 mM.

Experimental procedure: The fluorescence emission intensities were recorded on a fluorescence spectrophotometer. The resulting 0.08 mM solution (5 µL) was added to cuvette to obtain different concentrations of catalyst solution. This solution was then diluted to a volume of 2.0 mL by adding CH_3OH to prepare a 0.2 µM solution. 100.0 µL of a 4-chlorothiolphenol solution was added successively and uniformly stirred, and the resulting mixture was irradiated at 525 nm. Fluorescence emission spectra of 0 µL, 100 µL, 200 µL, 300 µL, etc. fluorescence intensities were recorded. This method was followed and made changes to the amount to obtain the Stern–Volmer relationship in turn. The solution was excited at λ= 524 nm.

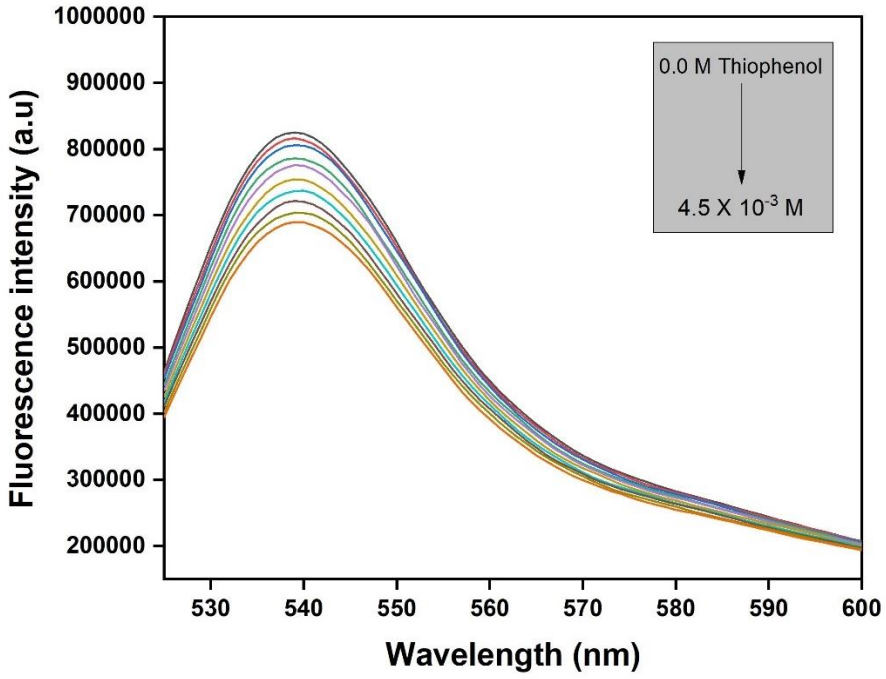


Figure S3. Fluorescence Emission Quenching of Eosin Y in the Presence of *p*-Chlorothiophenol (**2a**).

A fluorescence quenching phenomenon of Eosin Y under different concentrations of 4-chlorothiophenol was demonstrated in a curve of $[I_0/I]$ vs $C_{[Thiophenol]}$, as shown in Figure S4.

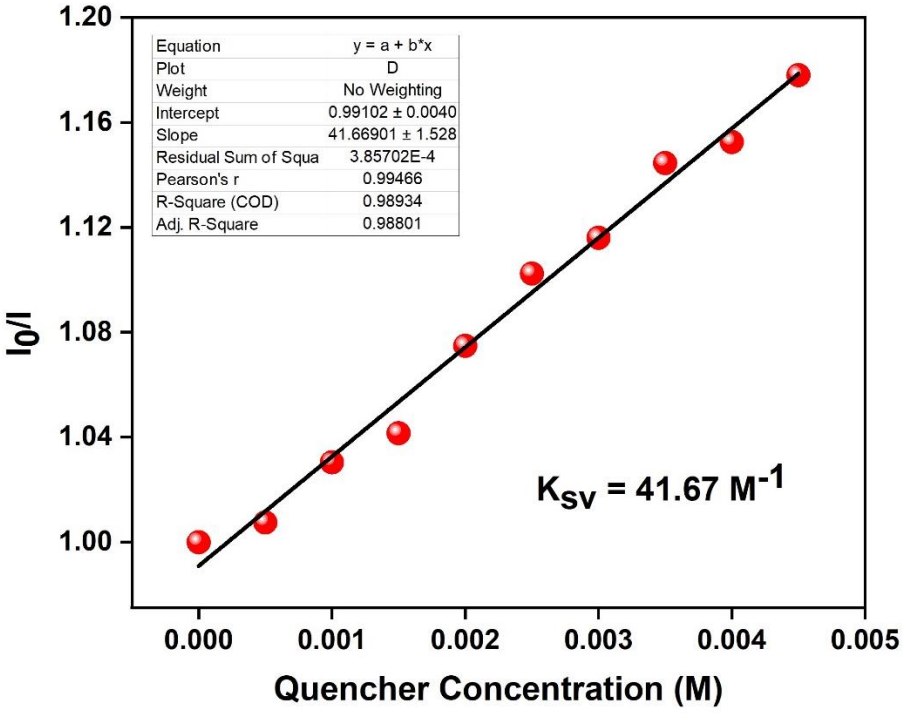


Figure S4. Stern-Volmer Plots for Quenching of Eosin Y Fluorescence Emission in the Presence of *p*-Chlorothiophenol (**2a**).

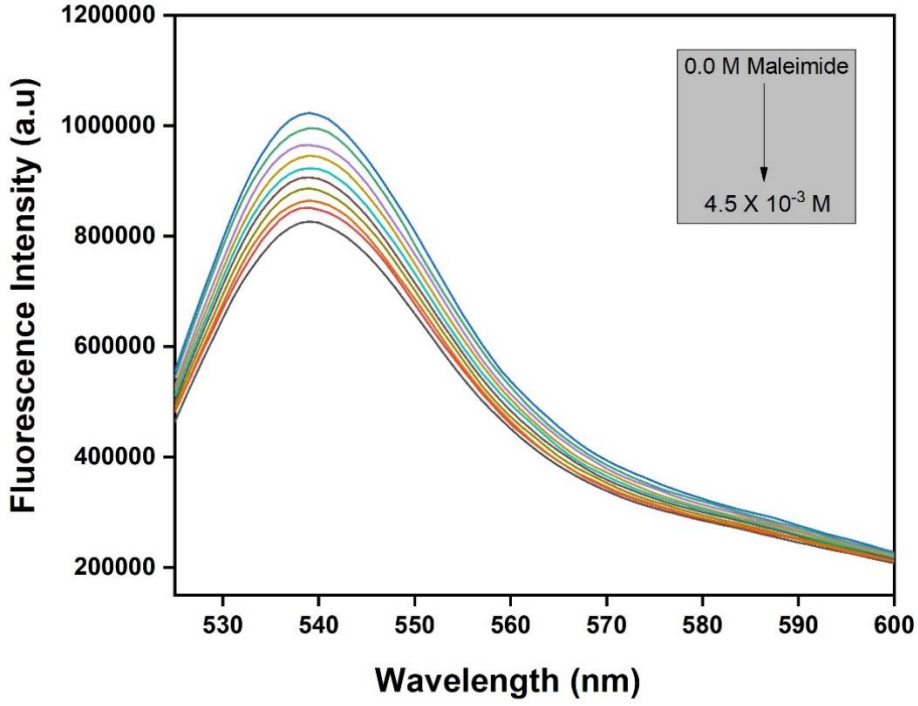


Figure S5. Quenching of Eosin Y Fluorescence Emission in the Presence of Maleimide.

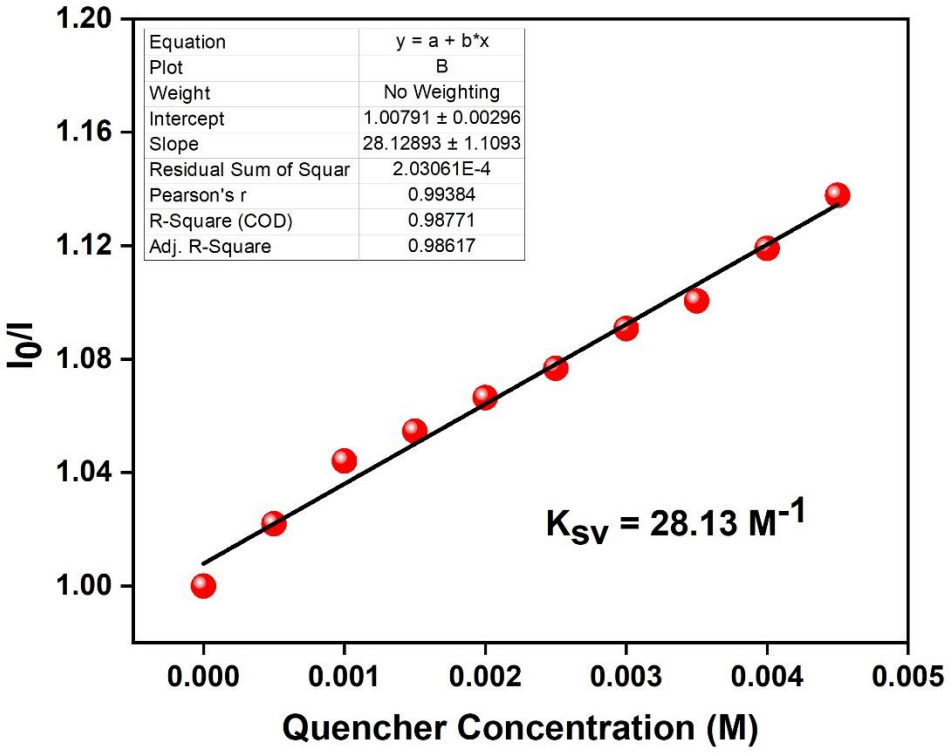


Figure S6. Stern-Volmer Plots for Quenching of Eosin Y Fluorescence Emission in the Presence of Maleimide.

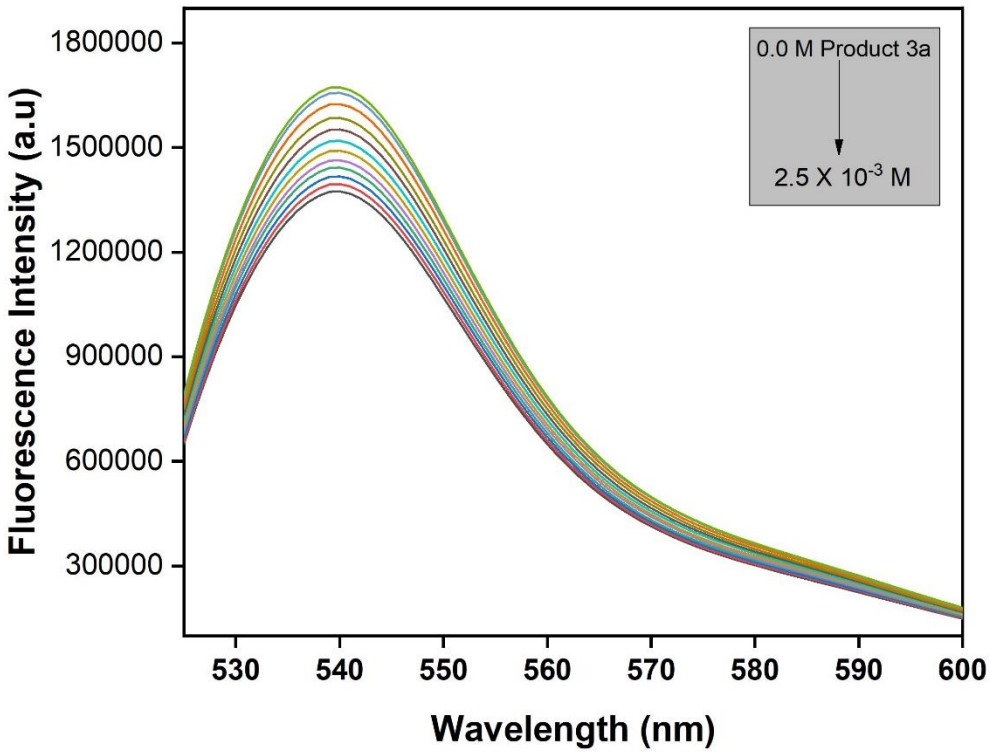


Figure S7. Quenching of Eosin Y Fluorescence Emission in the Presence of Product **3a**.

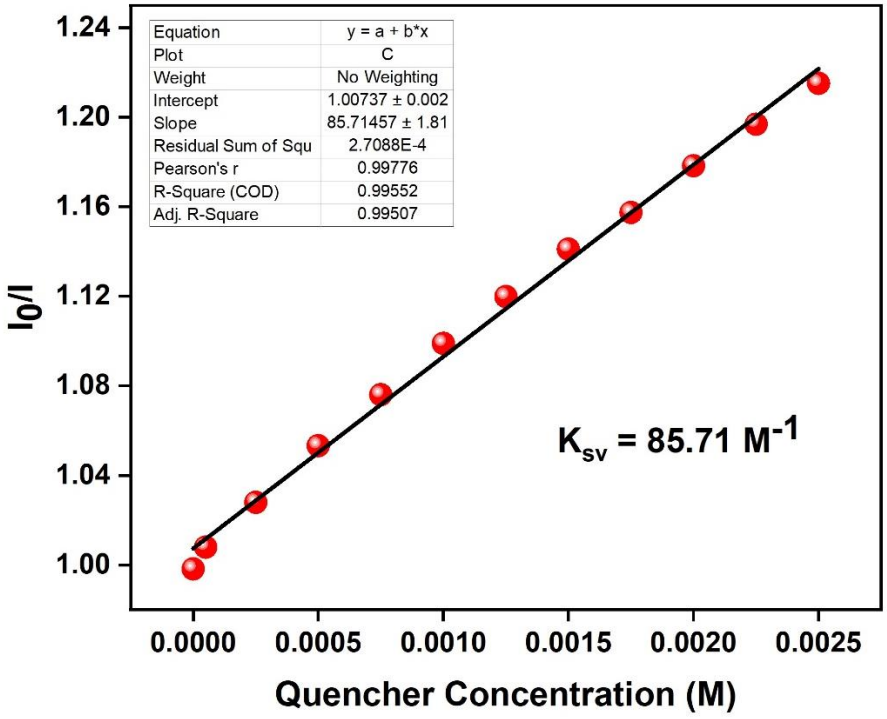


Figure S8. Stern-Volmer Plots for Quenching of Eosin Y Fluorescence Emission in the Presence of Product **3a**.

2.4 Density Functional Theory (DFT) Computational Details

Density Functional Theory (DFT) computations were accomplished by employing the Gaussian 16 program suite.[1] Ground state level energy minimizations of all the geometries of the reactants, products and intermediates were achieved by the B3LYP-D3 functional. Different elements like H, C, O, S are depicted by using localized 6-31+G(d) basis set.[2] Applying the Polarizable Continuum Model (PCM) solvation was introduced implicitly, as implemented in Gaussian 16 package with acetonitrile as a solvent. In solution phase all geometry optimizations were carried out without symmetry restrictions. Different thermodynamic parameters such as enthalpy corrections and free energy were evaluated at 298.15 K and 1 atm pressure, incorporating zero-point energy corrections (ZPE). Vibration frequency calculation is carried out to confirm that the minimum energy structure on the potential energy surfaces.

Results and discussion: Density Functional Theory (DFT) calculations were performed to validate the proposed mechanism. The free energy profiles, along with the optimized

geometries of the key intermediates, are shown in Figure S9. In the photocatalytic cycle, the reduction of EY to EY•− leads to the formation of radical cation **A**, which undergoes deprotonation to yield thiyl radical **B**, with an associated free energy change of -73.71 kcal/mol. The subsequent reaction between **1a** and **B** formed the radical intermediate **C**, accompanied by a free energy change of -68.13 kcal/mol. This radical intermediate C then readily protonates to form the desired thiolated product **3a**, with a significant free energy change of -149.14 kcal/mol. The negative free energy values confirm the thermodynamic favorability of these processes.

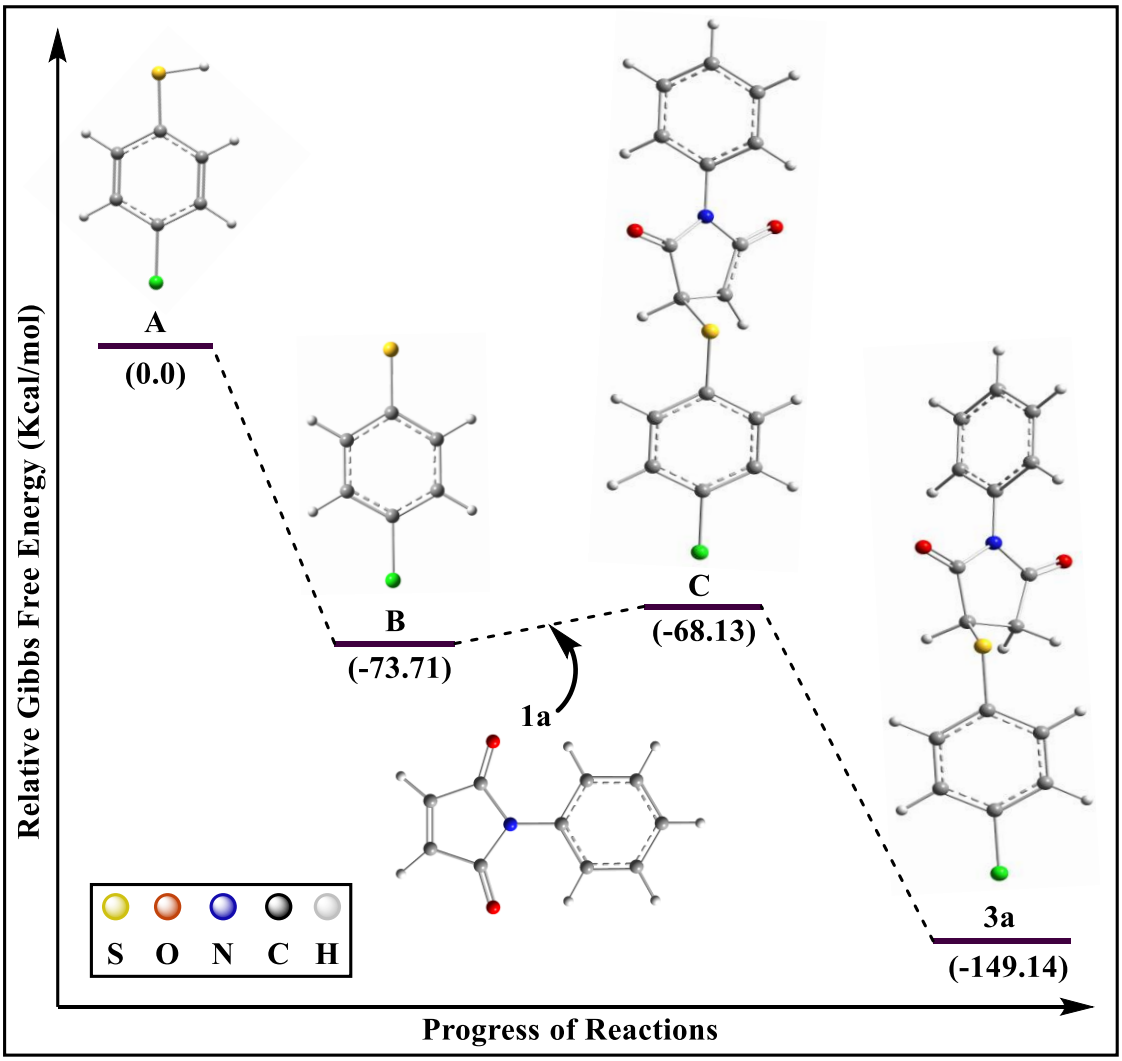
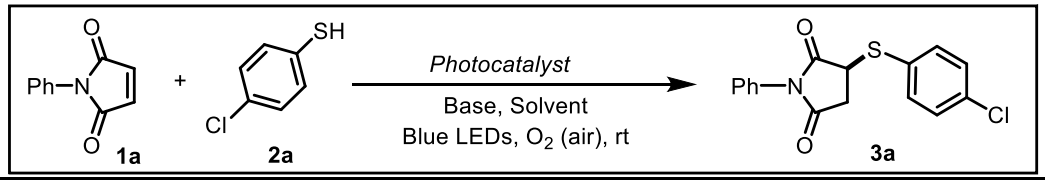
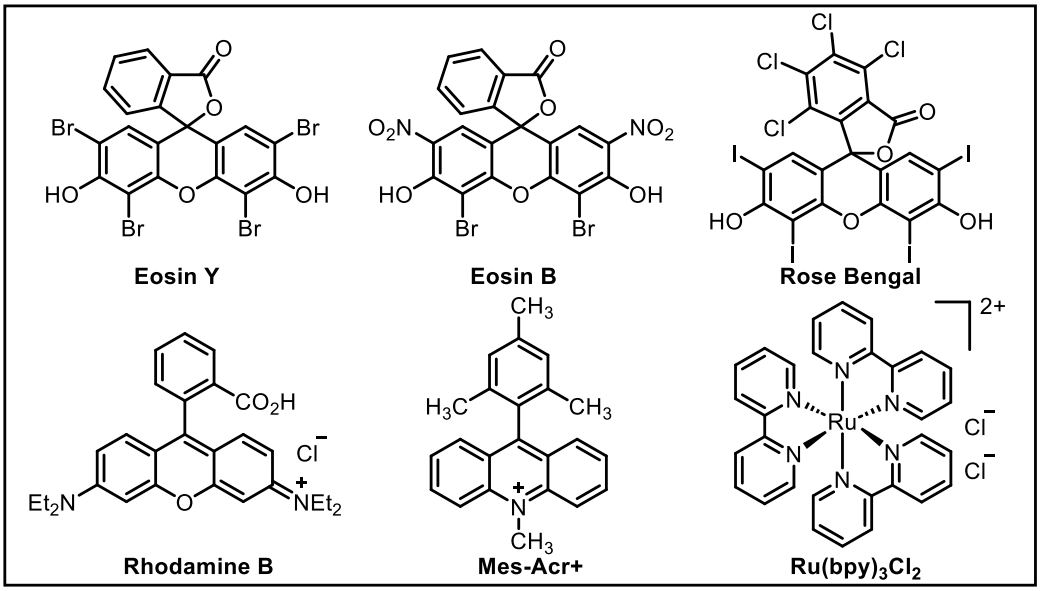


Figure S9. Computed free energy profiles (kcal/mol) of the catalytic cycle for the sulfenylation of maleimides in CH_3CN.

2.5. Table S1. Optimization of the Reaction Conditions[a]





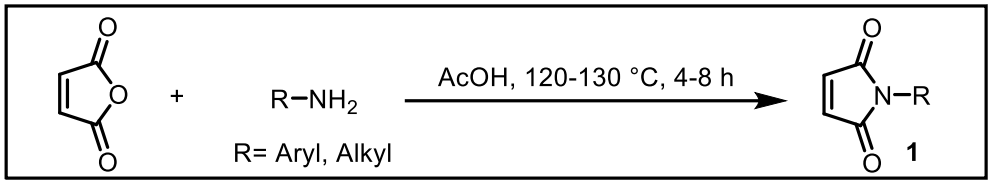
entry	photocatalyst (mol%)	solvent	base	yield (%)[b] (**3a**)
1	Eosin Y (2)	DCE	TMEDA	76
2	Mes-Acr+ (2)	DCE	TMEDA	25
3	$Ru(bpy)_3Cl_2$ (2)	DCE	TMEDA	55
4	Eosin B (2)	DCE	TMEDA	28
5	Rose Bengal (2)	DCE	TMEDA	35
6	Rhodamine B (2)	DCE	TMEDA	NR
7	**Eosin Y (5)**	**DCE**	**TMEDA**	**90**
8	Eosin Y (8)	DCE	TMEDA	82
9	Eosin Y (5)	EtOH	TMEDA	trace
10	Eosin Y (5)	MeOH	TMEDA	trace
11	Eosin Y (5)	DMF	TMEDA	trace
12	Eosin Y (5)	MeCN	TMEDA	35
13	Eosin Y (5)	DMSO	TMEDA	25
14	Eosin Y (5)	DCM	TMEDA	trace
15	Eosin Y (5)	DCE	DIPEA	36
16	Eosin Y (5)	DCE	DBU	20

17	Eosin Y (5)	DCE	Et_3N	26
18	Eosin Y (5)	DCE	NaOAc	27
19	Eosin Y (5)	DCE	Na_2CO_3	20
20	Eosin Y (5)	DCE	KOAc	22
21	Eosin Y (5)	DCE	TMEDA	35[c]
22	Eosin Y (5)	DCE	TMEDA	20[d]
23	–	DCE	TMEDA	–
24	Eosin Y (5)	DCE	–	trace
25	Eosin Y (5)	DCE	TMEDA	88[e]
26	Eosin Y (5)	DCE	TMEDA	Trace[f]

[a]Reaction conditions: **1a** (0.2 mmol), **2a** (0.24 mmol), TMEDA (0.3 mmol), photocatalyst (5 mol%), solvent (1.5 mL), blue LED irradiation in presence of O_2 (air) at room temperature for 12 h. [b]Yield was determined through ^1H NMR analysis using internal standard 1,3,5-trimethoxybenzene. [c]Under Green LED irradiation. [d]Irradiated under White LEDs. [e]O_2 balloon was used instead of air. [f]N_2 atmosphere. NR= no reaction.

2.6. *General Procedure for the synthesis of N-aryl/N-alkyl maleimide derivatives (1):*

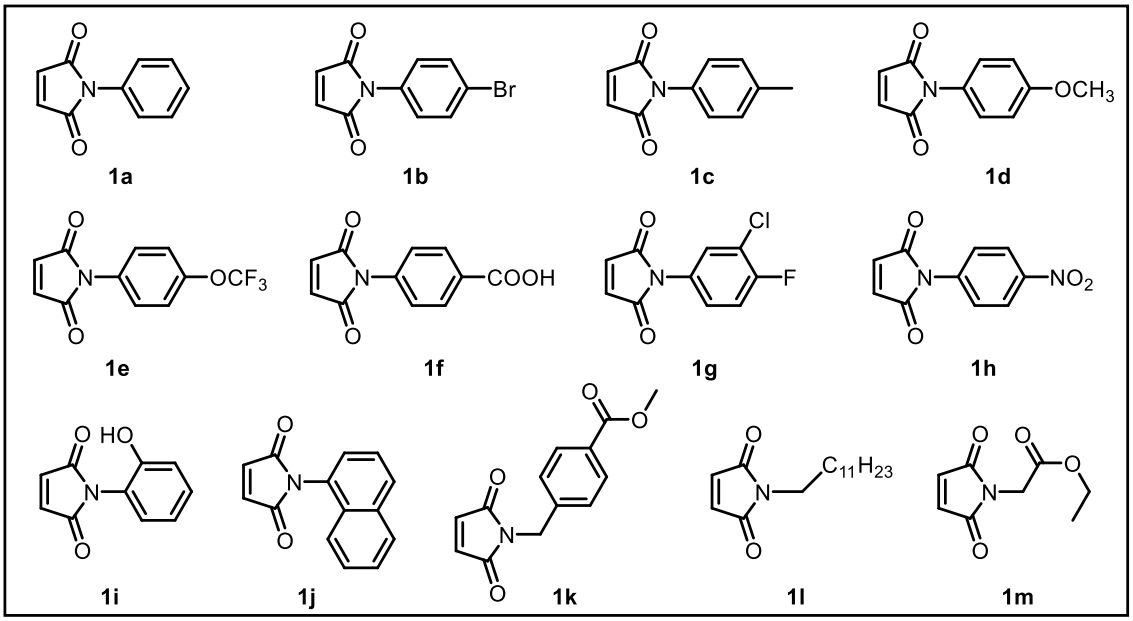
N-Aryl/Alkyl maleimide were prepared using the modified reported procedure mentioned below from the corresponding primary aniline.[3]



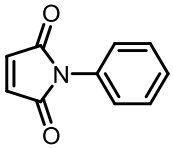
Primary amine (1.0 equiv.) and maleic anhydride (2.0 equiv.) were taken in a round bottomed flask with acetic acid (1.5 mL per mmol of amine). This mixture was stirred until the maleic anhydride dissolved completely and then refluxed for 4-8 h (depending on the type of amine) at 120-130 °C. After completion of the reaction (often marked by the reaction mixture turning homogeneous), the mixture was allowed to cool down to room temperature and then transferred to a 500 mL beaker. Saturated sodium bicarbonate aqueous solution was added to the beaker containing the reaction mixture until effervescence stops. The aqueous mixture was extracted with ethyl acetate (3x20 mL). The organic layer was further washed with brine solution (30 mL). The excess solvent was removed under reduced pressure and the residue was purified by

flash column chromatography using ethyl acetate/ hexane to get highly pure maleimide in good yield (60-90%).

Synthesized maleimide derivatives:

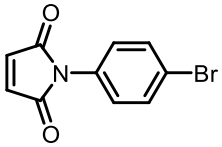


2.7. Characterization Data for Synthesized maleimide derivatives



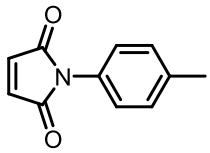
1-phenyl-1H-pyrrole-2,5-dione (1a)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 (t, *J* = 7.4 Hz, 2H), 7.39 – 7.32 (m, 3H), 6.83 (s, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 169.57, 134.22, 131.24, 129.18, 128.00, 126.11.



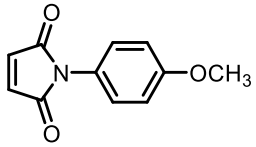
1-(4-Bromophenyl)-1H-pyrrole-2,5-dione (1b) [3]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60–7.58 (m, 2H), 7.27 (s, 1H), 7.25 (s, 1H), 6.86 (s, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 169.1, 134.3, 132.3, 130.3, 127.4, 121.6.



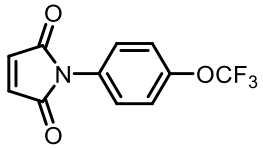
1-(p-Tolyl)-1H-pyrrole-2,5-dione (1c)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.5 Hz, 2H), 6.81 (s, 2H), 2.37 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 169.7, 138.0, 134.1, 129.6, 128.5, 126.0, 21.0.



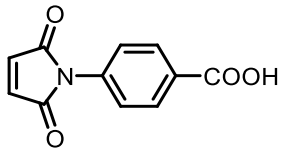
1-(4-Methoxyphenyl)-1H-pyrrole-2,5-dione (1d)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (d, *J* = 9.0 Hz, 2H), 6.97 (d, *J* = 9.0 Hz, 2H), 6.81 (d, *J* = 1.0 Hz, 2H), 3.81 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 169.8, 159.1, 134.1, 127.6, 123.7, 114.5, 55.5.



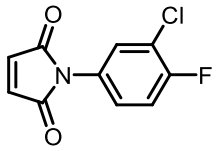
1-(4-(Trifluoromethoxy)phenyl)-1H-pyrrole-2,5-dione (1e)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 (dd, *J* = 9.0, 2.2 Hz, 2H), 7.32 (d, *J* = 7.8 Hz, 2H), 6.88 (d, *J* = 2.0 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 169.1, 148.2, 134.3, 129.7, 127.2, 125.5, 122.1, 121.6, 118.6, 115.2.



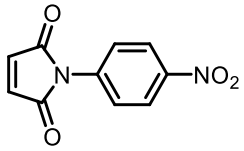
4-(2,5-Dioxo-2,5-dihydro-1H-pyrrol-1-yl)benzoic acid (1f)[3]

^{1}H NMR (300 MHz, DMSO) δ 10.60 (s, 1H), 7.91 (d, *J* = 8.7 Hz, 2H), 7.74 (d, *J* = 8.8 Hz, 2H), 6.35 – 6.27 (m, 2H). **^{13}C NMR (75 MHz, DMSO)** δ 170.0, 167.1, 135.9, 135.3, 130.3, 130.0, 126.6.



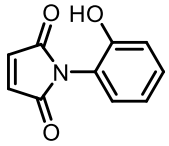
1-(3-Chloro-4-fluorophenyl)-1H-pyrrole-2,5-dione (1g)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 (dd, *J* = 6.5, 2.3 Hz, 1H), 7.27 – 7.22 (m, 2H), 6.86 (s, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 168.9, 158.9, 155.6, 134.3, 128.2, 127.7, 125.8, 125.7, 121.7, 121.4, 117.1, 116.8.



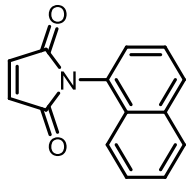
1-(4-Nitrophenyl)-1H-pyrrole-2,5-dione (1h)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.33 (d, *J* = 9.3 Hz, 2H), 7.68 (d, *J* = 9.3 Hz, 2H), 6.93 (s, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 168.5, 146.1, 137.1, 134.6, 125.5, 124.4.



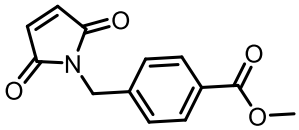
1-(2-Hydroxyphenyl)-1H-pyrrole-2,5-dione (1i)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.91(br, 1H), 7.25 – 7.20 (m, 1H), 7.10 (d, *J* = 6.1 Hz, 1H), 6.94 (t, *J* = 7.2 Hz, 2H), 6.80 (d, *J* = 6.2 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.1, 134.6, 134.5, 130.5, 129.5, 120.4, 118.4, 117.4.



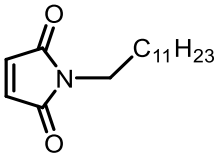
1-(Naphthalen-1-yl)-1H-pyrrole-2,5-dione (1j)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 (t, *J* = 9.3 Hz, 2H), 7.59 – 7.53 (m, 4H), 7.38 (d, *J* = 7.2 Hz, 1H), 6.94 (s, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.0, 134.5, 134.4, 130.3, 130.0, 128.6, 127.6, 127.2, 127.0, 126.6, 125.4, 122.2.



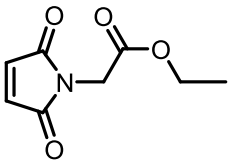
Methyl 4-((2,5-dioxo-2,5-dihydro-1H-pyrrol-1-yl)methyl)benzoate (1k)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.38 (d, *J* = 8.3 Hz, 2H), 6.73 (s, 2H), 4.71 (s, 2H), 3.89 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.2, 166.6, 141.0, 134.3, 130.0, 129.7, 128.1, 52.1, 41.0.



1-Dodecyl-1H-pyrrole-2,5-dione (1l)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 6.67 (s, 2H), 3.49 (t, *J* = 7.3 Hz, 2H), 1.56 (t, *J* = 7.2 Hz, 2H), 1.24 (s, 18H), 0.87 (t, *J* = 6.5 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 170.9, 134.0, 37.9, 31.9, 29.6, 29.5, 29.5, 29.3, 29.1, 28.5, 26.7, 22.7, 14.1.

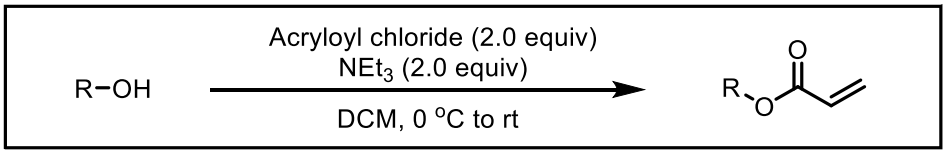


Ethyl 2-(2,5-dioxo-2,5-dihydro-1H-pyrrol-1-yl)acetate (1m)[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 (d, *J* = 1.8 Hz, 2H), 4.70 (d, *J* = 2.7 Hz, 2H), 4.63 (t, *J* = 7.2 Hz, 2H), 1.71 (q, *J* = 4.6 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 169.8, 167.2, 134.4, 61.7, 38.5, 13.9.

2.8 *General Procedure for the synthesis of acrylate derivatives (4):*

Acrylate derivatives were prepared using the modified reported procedure mentioned below from the corresponding primary alcohols.[4]



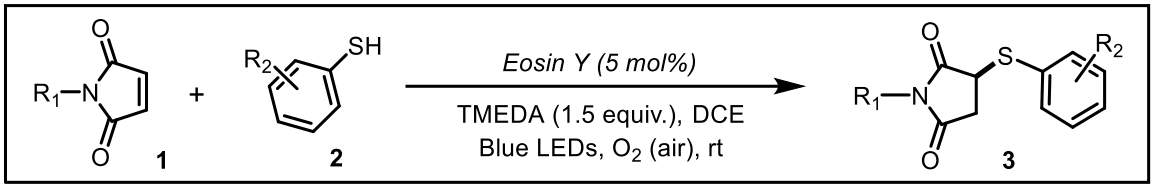
To a solution of R–OH and Et_3N (20 mmol, 2.0 equiv) in CH_2Cl_2 (25 mL) at 0 °C acryloyl chloride (20 mmol, 2.0 equiv) was added dropwise. The reaction mixture was warmed to rt and

stirred for 12 h. The reaction mixture was quenched with water (15 mL) and the mixture was extracted with CH_2Cl_2 (15 mL× 3). The combined organic layer was dried over $MgSO_4$, and then concentrated under reduced pressure. The residue was purified by flash silica gel column chromatography to afford the acrylate.

2.9 General Synthetic Procedures

Method A. General procedure for the synthesis of sulfenylated maleimide (3):

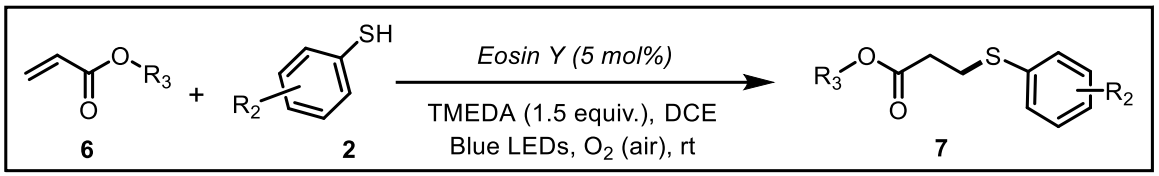
To an oven dried 15 mL borosilicate reaction tube, *N*-substitited maleimides **1** (0.2 mmol, 1 equiv), thiophenols **2** (0.24 mmol, 1.2 equiv), 1.5 equiv of base TMEDA and 5.0 mol% photocatalyst Eosin Y were taken in solvent DCE (1.5 mL), then the system was stirred under the irradiation of blue LEDs at room temperature for 12 h. The progress of the reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was extracted with chloroform and washed with water. To the aqueous layer brine solution was added and again extracted with ethyl acetate. The combined organic layers were dried over Na_2SO_4 and concentrated under vacuum. The crude mixture was purified by silica gel column chromatography using petroleum ether/ethyl acetate as eluent (10:1 to 5:1 v/v) to afford the pure desired product maleimide sulfides (**3**).



Method B. General procedure for the synthesis of sulfenylated acrylate (7):

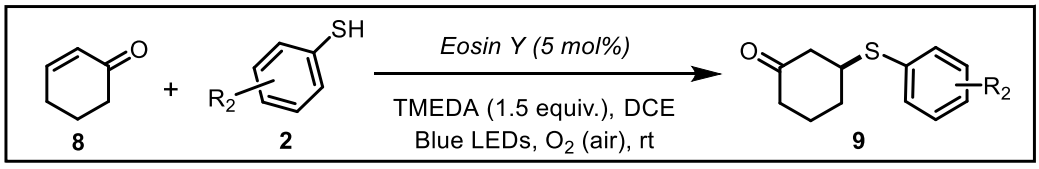
To an oven dried 15 mL borosilicate reaction tube, alkyl acrylayes **6** (0.2 mmol, 1 equiv), thiophenols **2** (0.24 mmol, 1.2 equiv), 1.5 equiv of base TMEDA and 5.0 mol% photocatalyst Eosin Y were taken in solvent DCE (1.5 mL), then the system was stirred under the irradiation of blue LEDs at room temperature for 12 h. The progress of the reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was extracted with chloroform and washed with water. To the aqueous layer brine solution was added and again extracted with ethyl acetate. The combined organic layers were dried over Na_2SO_4 and concentrated

under vacuum. The crude mixture was purified by silica gel column chromatography using petroleum ether/ethyl acetate as eluent (10:1 to 5:1 v/v) to afford the pure desired product *acrylate sulfides* (**7**).

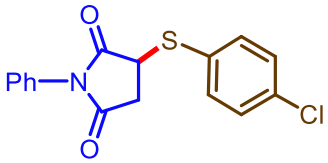


Method C. General procedure for the synthesis of sulfenylated cyclohexenone (9):

To an oven dried 15 mL borosilicate reaction tube, cyclohexenone **8** (0.2 mmol, 1 equiv), thiophenols **2** (0.24 mmol, 1.2 equiv), 1.5 equiv of base TMEDA and 5.0 mol% photocatalyst Eosin Y were taken in solvent DCE (1.5 mL), then the system was stirred under the irradiation of blue LEDs at room temperature for 6 h. The progress of the reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was extracted with chloroform and washed with water. To the aqueous layer brine solution was added and again extracted with ethyl acetate. The combined organic layers were dried over Na_2SO_4 and concentrated under vacuum. The crude mixture was purified by silica gel column chromatography using petroleum ether/ethyl acetate as eluent (10:1 to 5:1 v/v) to afford the pure desired product *cyclohexenone sulfides* (**9**).



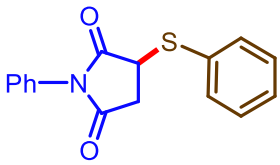
3. Characterization Data for all the Synthesized Compounds



3-((4-Chlorophenyl)thio)-1-phenylpyrrolidine-2,5-dione (3a).

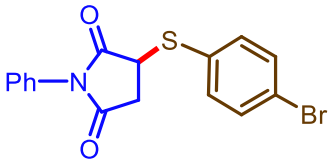
Yield 57.2 mg, 90%; White solid; R_f = 0.2 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.35 (d, *J* = 8.5 Hz, 2H), 7.31 – 7.22 (m, 3H), 7.17 (d, *J* = 8.5 Hz, 2H),

6.93 (d, *J* = 6.9 Hz, 2H), 3.96 (dd, *J* = 9.3, 4.0 Hz, 1H), 3.21 – 3.12 (m, 1H), 2.72 – 2.64 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.3, 173.2, 136.1, 131.4, 129.7, 129.2, 128.9, 128.9, 128.6, 126.2, 44.1, 36.2. **ESI-MS (m/z) for $C_{16}H_{13}ClNO_2S$ $[M+H]^+$:** Calculated 318.0350, found 318.0345.



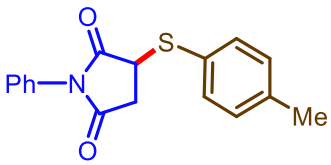
1-Phenyl-3-(phenylthio)pyrrolidine-2,5-dione (3b).

Yield 51.0 mg, 90%; White solid; R_f = 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.58 (dd, *J* = 7.7, 1.8 Hz, 2H), 7.46 – 7.34 (m, 6H), 7.05 (dd, *J* = 8.3, 1.6 Hz, 2H), 4.14 (dd, *J* = 9.3, 3.9 Hz, 1H), 3.37 – 3.28 (m, 1H), 2.93 (d, *J* = 3.9 Hz, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.5, 173.5, 135.1, 131.5, 129.8, 129.7, 129.5, 129.1, 128.8, 126.3, 44.1, 36.4. **ESI-MS (m/z) for $C_{16}H_{14}NO_2S$ $[M+H]^+$:** Calculated 284.0740, found 284.0736.



3-((4-Bromophenyl)thio)-1-phenylpyrrolidine-2,5-dione (3c).

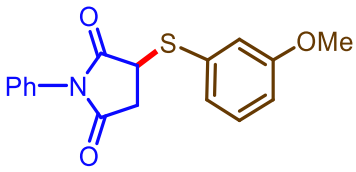
Yield 64.5 mg, 89%; White solid; R_f= 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.51 – 7.39 (m, 7H), 7.09 (d, *J* = 6.8 Hz, 2H), 4.14 (dd, *J* = 9.3, 4.0 Hz, 1H), 3.38 – 3.29 (m, 1H), 2.89 – 2.81 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.3, 173.2, 136.2, 132.6, 131.4, 129.3, 129.2, 128.9, 126.2, 124.2, 44.0, 36.2. **ESI-MS (m/z) for $C_{16}H_{13}BrNO_2S$ $[M+H]^+$:** Calculated 361.9845, found 361.9841.



1-Phenyl-3-(p-tolylthio)pyrrolidine-2,5-dione (3d).

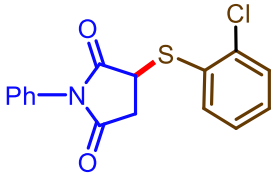
Yield 54.1 mg, 91%; White solid; R_f = 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.47 – 7.36 (m, 5H), 7.17 (d, *J* = 7.7 Hz, 2H), 7.04 (d, *J* = 7.4 Hz, 2H), 4.07 (dd, *J* = 9.5, 3.8 Hz, 1H), 3.33 – 3.24 (m, 1H), 2.92 – 2.84 (m, 1H), 2.36 (s, 3H). **^{13}C NMR**

(75 MHz, $CDCl_3$) δ 174.6, 173.6, 140.2, 135.5, 131.6, 130.3, 129.1, 128.7, 126.3, 125.8, 44.3, 36.3, 21.2. **ESI-MS (m/z) for $C_{17}H_{16}NO_2S$ $[M+H]^+$:** Calculated 298.0896, found 298.0891.



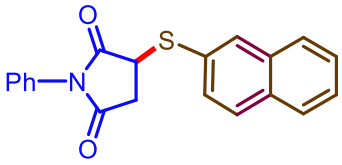
3-((3-Methoxyphenyl)thio)-1-phenylpyrrolidine-2,5-dione (3e).

Yield 57.6 mg, 92%; Yellowish solid; R_f = 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.46 – 7.28 (m, 4H), 7.16 – 7.08 (m, 4H), 6.94 (dd, *J* = 8.3, 2.5 Hz, 1H), 4.17 (dd, *J* = 9.4, 3.8 Hz, 1H), 3.76 (d, *J* = 2.0 Hz, 3H), 3.38 – 3.29 (m, 1H), 2.95 – 2.88 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.6, 173.5, 160.0, 131.5, 130.9, 130.2, 129.1, 128.8, 126.8, 126.3, 119.7, 115.6, 55.3, 44.1, 36.4. **ESI-MS (m/z) for $C_{17}H_{16}NO_3S$ $[M+H]^+$:** Calculated 314.0845, found 314.0851.



3-((2-Chlorophenyl)thio)-1-phenylpyrrolidine-2,5-dione (3f).

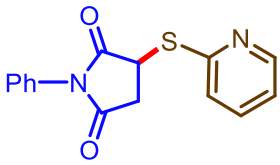
Yield 53.6 mg, 84%; solid; R_f= 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.63 (d, *J* = 7.4 Hz, 1H), 7.46 – 7.36 (m, 4H), 7.31 – 7.18 (m, 4H), 4.37 – 4.32 (m, 1H), 3.32 – 3.23 (m, 1H), 2.86 – 2.78 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.2, 173.3, 137.6, 135.4, 131.5, 130.5, 130.3, 130.3, 129.2, 128.8, 127.6, 126.3, 42.6, 35.8. **ESI-MS (m/z) for $C_{16}H_{13}ClNO_2S$ $[M+H]^+$:** Calculated 318.0350, found 318.0347.



3-(Naphthalen-2-ylthio)-1-phenylpyrrolidine-2,5-dione (3g).

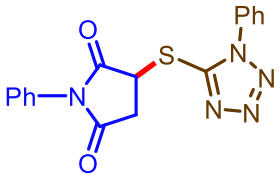
Yield 58.0 mg, 87%; White crystalline solid; R_f= 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.13 (d, *J* = 1.8 Hz, 1H), 7.86 – 7.78 (m, 3H), 7.61 – 7.52 (m, 3H), 7.38 – 7.35 (m, 3H), 7.01 (dd, *J* = 7.7, 2.0 Hz, 2H), 4.26 (dd, *J* = 9.3, 4.0 Hz, 1H), 3.38 – 3.29 (m, 1H), 2.98 – 2.90 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.6, 173.4, 134.7, 133.5,

133.3, 131.5, 130.9, 129.2, 129.1, 128.8, 127.8, 127.8, 127.3, 127.2, 127.0, 126.3, 44.2, 36.3. **ESI-MS (m/z) for $C_{20}H_{16}NO_2S$ $[M+H]^+$:** Calculated 334.0896, found 334.0890.



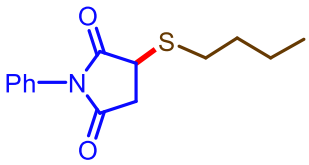
1-Phenyl-3-(pyridin-2-ylthio)pyrrolidine-2,5-dione (3h).

Yield 46.6 mg, 82%; Yellow solid; R_f= 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.28 (d, *J* = 4.9 Hz, 1H), 7.54 – 7.33 (m, 6H), 7.20 (d, *J* = 8.1 Hz, 1H), 7.00 (dd, *J* = 7.4, 5.1 Hz, 1H), 4.25 (dd, *J* = 9.6, 5.5 Hz, 1H), 3.42 – 3.32 (m, 1H), 3.14 – 3.06 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 175.1, 174.5, 155.8, 149.1, 136.7, 132.5, 129.2, 128.6, 126.5, 122.1, 120.3, 40.9, 36.5. **ESI-MS (m/z) for $C_{15}H_{13}N_2O_2S$ $[M+H]^+$:** Calculated 285.0692, found 285.0687.



1-Phenyl-3-((1-phenyl-1H-tetrazol-5-yl)thio)pyrrolidine-2,5-dione (3i).

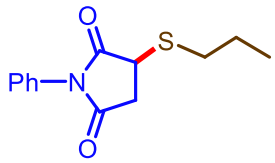
Yield 54.8 mg, 78%; Yellow solid; R_f= 0.2 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.58 (s, 5H), 7.55 – 7.37 (m, 5H), 4.62 (dd, *J* = 9.6, 5.7 Hz, 1H), 3.61 – 3.51 (m, 1H), 3.34 – 3.26 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 172.8, 172.5, 151.2, 133.1, 131.8, 130.6, 130.0, 129.3, 129.1, 126.6, 123.7, 42.8, 36.4. **ESI-MS (m/z) for $C_{17}H_{14}N_5O_2S$ $[M+H]^+$:** Calculated 352.0863, found 352.0867.



3-(Butylthio)-1-phenylpyrrolidine-2,5-dione (3j).

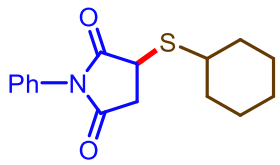
Yield 37.9 mg, 72%; Yellowish sticky solid; R_f= 0.2 (8% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.50 – 7.39 (m, 3H), 7.29 (d, *J* = 7.8 Hz, 2H), 3.88 – 3.84 (m, 1H), 3.34 – 3.25 (m, 1H), 3.00 – 2.91 (m, 1H), 2.86 – 2.76 (m, 1H), 2.71 – 2.63 (m, 1H), 1.65 (q, *J* = 6.9 Hz, 2H), 1.44 (q, *J* = 7.3 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)**

δ 175.5, 173.8, 131.6, 129.2, 128.7, 126.4, 39.1, 36.2, 31.5, 31.0, 21.9, 13.6. **ESI-MS (m/z) for $C_{14}H_{18}NO_2S$ $[M+H]^+$:** Calculated 264.1053, found 264.1046.



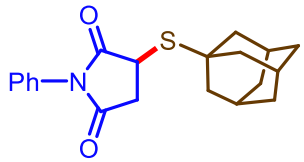
1-Phenyl-3-(propylthio)pyrrolidine-2,5-dione (3k).

Yield 34.9 mg, 70%; Brown liquid; R_f = 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.45 (t, *J* = 7.5 Hz, 2H), 7.37 (t, *J* = 7.3 Hz, 1H), 7.27 – 7.25 (m, 2H), 3.81 (dd, *J* = 9.1, 3.6 Hz, 1H), 3.28 – 3.19 (m, 1H), 2.92 – 2.85 (m, 1H), 2.77 – 2.68 (m, 1H), 2.65 – 2.58 (m, 1H), 1.76 – 1.57 (m, 2H), 1.00 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 175.6, 173.9, 131.7, 129.2, 128.7, 126.4, 39.1, 36.2, 33.7, 22.4, 13.4. **ESI-MS (m/z) for $C_{13}H_{16}NO_2S$ $[M+H]^+$:** Calculated 250.0896, found 250.0890.



3-(Cyclohexylthio)-1-phenylpyrrolidine-2,5-dione (3l).

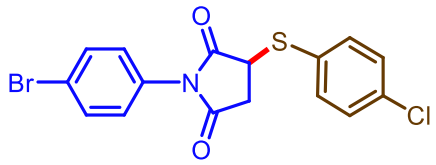
Yield 39.9 mg, 69%; Pale white solid; R_f = 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, CDCl3)** δ 7.48 (t, *J* = 7.4 Hz, 2H), 7.41 (d, *J* = 7.2 Hz, 1H), 7.32 – 7.27 (m, 2H), 3.96 (dd, *J* = 9.1, 3.6 Hz, 1H), 3.35 – 3.25 (m, 1H), 3.25 (d, *J* = 3.3 Hz, 1H), 2.71 – 2.63 (m, 1H), 2.21 – 2.15 (m, 1H), 1.99 – 1.92 (m, 1H), 1.81 – 1.77 (m, 2H), 1.67 – 1.69 (m, 1H), 1.41 – 1.24 (m, 5H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 175.8, 173.9, 131.7, 129.2, 128.7, 126.4, 43.8, 37.7, 36.5, 33.5, 32.9, 25.9, 25.7, 25.6. **ESI-MS (m/z) for $C_{16}H_{20}NO_2S$ $[M+H]^+$:** Calculated 290.1209, found 290.1203.



3-(((3s,5s,7s)-Adamantan-1-yl)thio)-1-phenylpyrrolidine-2,5-dione (3m).

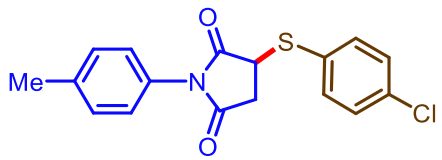
Yield 44.3 mg, 65%; White solid; R_f = 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.46 (dd, *J* = 8.3, 6.5 Hz, 2H), 7.41 – 7.35 (m, 1H), 7.28 (dd, *J* = 7.3, 2.1 Hz, 2H), 4.01 (dd, *J* = 9.4, 4.5 Hz, 1H), 3.41 – 3.31 (m, 1H), 2.85 – 2.77 (m, 1H), 2.11 – 2.09 (m, 3H), 1.98 (s, 6H), 1.71 (s, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 176.3, 174.1, 131.9, 129.1,

126.4, 47.4, 43.8, 39.2, 36.7, 36.0, 29.8. **ESI-MS (m/z) for $C_{20}H_{24}NO_2S$ $[M+H]^+$:** Calculated 342.1522, found 342.1515.



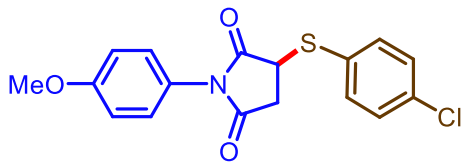
1-(4-Bromophenyl)-3-((4-chlorophenyl)thio)pyrrolidine-2,5-dione (3n).

Yield 61.9 mg, 78%; Off-white solid; R_f= 0.2 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.59 – 7.50 (m, 4H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.00 (d, *J* = 8.8 Hz, 2H), 4.13 (dd, *J* = 9.3, 3.9 Hz, 1H), 3.40 – 3.30 (m, 1H), 2.90 – 2.83 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.9, 172.8, 136.2, 136.1, 132.4, 130.3, 129.7, 128.4, 127.7, 122.7, 44.1, 36.2. **ESI-MS (m/z) for $C_{16}H_{12}BrClNO_2S$ $[M+H]^+$:** Calculated 395.9455, found 395.9448.



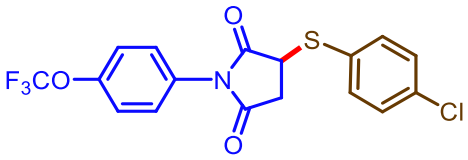
3-((4-Chlorophenyl)thio)-1-(p-tolyl)pyrrolidine-2,5-dione (3o).

Yield 52.4 mg, 79%; White solid; R_f= 0.2 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.53 (d, *J* = 8.5 Hz, 2H), 7.35 (d, *J* = 8.5 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 6.98 (d, *J* = 8.3 Hz, 2H), 4.14 (dd, *J* = 9.3, 4.0 Hz, 1H), 3.39 – 3.29 (m, 1H), 2.89 – 2.82 (m, 1H), 2.39 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.4, 173.4, 139.0, 136.1, 129.8, 129.6, 129.3, 128.7, 128.6, 126.0, 44.1, 36.2, 21.2. **ESI-MS (m/z) for $C_{17}H_{15}ClNO_2S$ $[M+H]^+$:** Calculated 332.0507, found 332.0501.



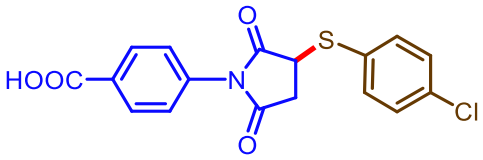
3-((4-Chlorophenyl)thio)-1-(4-methoxyphenyl)pyrrolidine-2,5-dione (3p).

Yield 59.8 mg, 86%; White solid; R_f= 0.2 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.51 (d, *J* = 8.5 Hz, 2H), 7.33 (d, *J* = 8.5 Hz, 2H), 6.97 (q, *J* = 9.1 Hz, 4H), 4.11 (dd, *J* = 9.3, 3.9 Hz, 1H), 3.81 (s, 3H), 3.36 – 3.26 (m, 1H), 2.87 – 2.79 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.5, 173.5, 159.6, 136.0, 129.6, 128.7, 127.5, 123.9, 114.5, 55.5, 44.1, 36.2. **ESI-MS (m/z) for $C_{17}H_{15}ClNO_3S$ $[M+H]^+$:** Calculated 348.0456, found 348.0449.



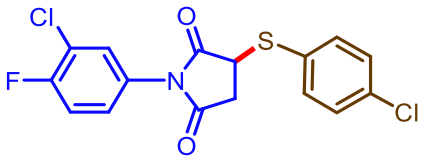
3-((4-Chlorophenyl)thio)-1-(4-(trifluoromethoxy)phenyl)pyrrolidine-2,5-dione (3q).

Yield 60.3 mg, 75%; White solid; R_f= 0.2 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.51 (d, *J* = 8.5 Hz, 2H), 7.35 – 7.26 (m, 4H), 7.17 (d, *J* = 9.1 Hz, 2H), 4.14 (dd, *J* = 9.3, 3.9 Hz, 1H), 3.40 – 3.31 (m, 1H), 2.91 – 2.83 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.0, 172.9, 148.9, 148.9, 136.2, 136.1, 129.7, 128.4, 127.7, 122.0, 121.6, 118.6, 44.1, 36.2. **^{19}F NMR (377 MHz, $CDCl_3$)** δ -57.87. **ESI-MS (m/z) for $C_{17}H_{12}ClF_3NO_3S$ $[M+H]^+$:** Calculated 402.0173, found 402.0168.



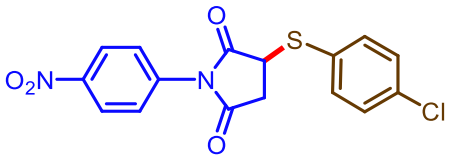
4-(3-((4-Chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoic acid (3r).

Yield 55.7 mg, 77%; Pinkish solid; R_f = 0.2 (30% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, DMSO)** δ 13.18 (s, 1H), 8.04 (d, *J* = 8.2 Hz, 2H), 7.59 – 7.46 (m, 4H), 7.29 (d, *J* = 8.2 Hz, 2H), 4.59 (dd, *J* = 9.3, 4.3 Hz, 1H), 3.49 – 3.40 (m, 1H), 2.89 – 2.82 (m, 1H). **^{13}C NMR (75 MHz, DMSO)** δ 175.1, 174.1, 167.0, 136.3, 134.7, 133.9, 131.3, 131.0, 130.3, 129.7, 127.2, 44.2, 36.6. **ESI-MS (m/z) for $C_{17}H_{13}ClNO_4S$ $[M+H]^+$:** Calculated 362.0248, found 362.0242.



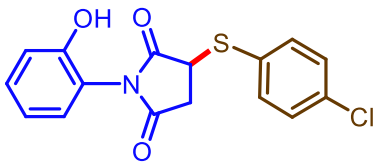
1-(3-Chloro-4-fluorophenyl)-3-((4-chlorophenyl)thio)pyrrolidine-2,5-dione (3s).

Yield 57.8 mg, 78%; Pale yellow solid; R_f= 0.25 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.50 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.23 – 7.15 (m, 2H), 7.03 – 6.98 (m, 1H), 4.13 (dd, *J* = 9.3, 3.8 Hz, 1H), 3.39 – 3.30 (m, 1H), 2.90 – 2.83 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.8, 172.7, 159.5, 156.1, 136.3, 136.1, 129.7, 128.7, 128.4, 127.7, 127.7, 126.2, 126.1, 121.9, 121.6, 117.1, 116.8, 44.1, 36.2. **^{19}F NMR (377 MHz, $CDCl_3$)** δ -113.73. **ESI-MS (m/z) for $C_{16}H_{11}Cl_2FNO_2S$ $[M+H]^+$:** Calculated 369.9866, found 369.9860.



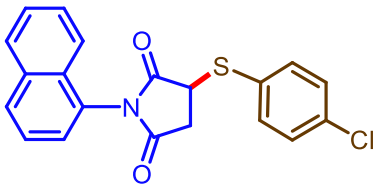
3-((4-Chlorophenyl)thio)-1-(4-nitrophenyl)pyrrolidine-2,5-dione (3t).

Yield 53.7 mg, 74%; Yellow solid; R_f= 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.29 (d, *J* = 9.1 Hz, 2H), 7.51 (d, *J* = 8.5 Hz, 2H), 7.41 – 7.33 (m, 4H), 4.18 (dd, *J* = 9.3, 3.9 Hz, 1H), 3.45 – 3.36 (m, 1H), 2.94 – 2.87 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.6, 172.3, 147.1, 136.8, 136.4, 136.1, 129.8, 128.3, 126.7, 124.4, 44.1, 36.2. **ESI-MS (m/z) for $C_{16}H_{12}ClN_2O_4S$ $[M+H]^+$:** Calculated 363.0201, found 363.0194.



3-((4-Chlorophenyl)thio)-1-(2-hydroxyphenyl)pyrrolidine-2,5-dione (3u).

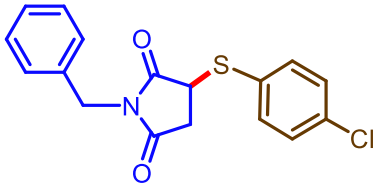
Yield 51.4 mg, 77%; Brownish semi-solid; R_f= 0.2 (30% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.91 (s, 1H), 7.48 (d, *J* = 6.4 Hz, 2H), 7.29 – 7.26 (m, 2H), 7.20 (t, *J* = 7.8 Hz, 1H), 6.93 (d, *J* = 8.3 Hz, 1H), 6.84 (t, *J* = 7.6 Hz, 1H), 6.74 (d, *J* = 8.1 Hz, 1H), 4.13 (dd, *J* = 8.8, 4.1 Hz, 1H), 3.32 (d, *J* = 8.4 Hz, 1H), 2.34 – 2.27 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.8, 173.8, 152.8, 135.6, 135.4, 130.8, 129.5, 129.4, 128.6, 119.9, 118.8, 117.5, 44.5, 36.3. **ESI-MS (m/z) for $C_{16}H_{13}ClNO_3S$ $[M+H]^+$:** Calculated 334.0299, found 334.0292.



4-((4-Chlorophenyl)thio)-1-(naphthalen-1-yl)pyrrolidin-2-one (3v).

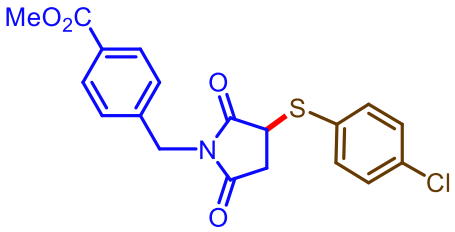
Yield 59.6 mg, 81%; Light brownish liquid; R_f = 0.2 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.96 – 7.89 (m, 2H, isomers mixture), 7.58 (dd, *J* = 8.5, 2.1 Hz, 2H, isomers mixture), 7.51 (d, *J* = 7.9 Hz, 2H, isomers mixture), 7.45 (t, *J* = 7.6 Hz, 1H, isomers mixture), 7.40 – 7.35 (m, 2H, isomers mixture), 7.29 (d, *J* = 7.2 Hz, 1H, isomers mixture), 6.87 (d, *J* = 8.3 Hz, 1H, isomers mixture), 4.30 – 4.23 (m, 1H, isomers mixture), 3.57 – 3.39 (m, 1H, isomers mixture), 3.09 – 2.96 (m, 1H, isomers mixture). **^{13}C NMR (75 MHz,**

$CDCl_3$) δ 174.5, 174.4, 173.6, 173.6, 136.9, 136.4, 136.3, 136.2, 135.7, 134.4, 134.3, 130.6, 130.2, 129.9, 129.7, 129.0, 124.0, 128.7, 128.6, 128.4, 128.1, 128.0, 127.3, 127.2, 126.7, 126.6, 126.2, 126.0, 125.3, 125.3, 121.7, 121.4, 44.8, 44.5, 36.6, 36.0. **ESI-MS (m/z) for $C_{20}H_{15}ClNO_2S$ $[M+H]^+$:** Calculated 368.0507, found 368.0502.



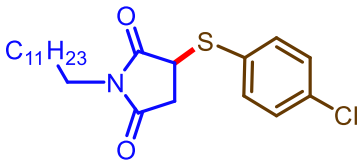
1-Benzyl-3-((4-chlorophenyl)thio)pyrrolidine-2,5-dione (3w).

Yield 48.4 mg, 73%; Pale brownish solid; R_f= 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.34 – 7.28 (m, 7H), 7.13 (d, *J* = 8.5 Hz, 2H), 4.58 (s, 2H), 3.96 (dd, *J* = 9.3, 4.2 Hz, 1H), 3.20 – 3.10 (m, 1H), 2.68 – 2.60 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 175.0, 173.8, 135.8, 135.7, 135.1, 129.5, 128.9, 128.6, 128.3, 128.1, 43.8, 42.8, 35.7. **ESI-MS (m/z) for $C_{17}H_{15}ClNO_2S$ $[M+H]^+$:** Calculated 332.0507, found 332.0509.



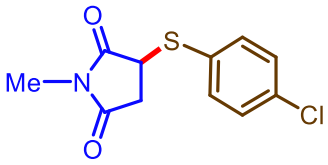
Methyl 4-((3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)methyl)benzoate (3x).

Yield 56.1 mg, 72%; White solid; R_f = 0.2 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.95 (d, *J* = 8.2 Hz, 2H), 7.31 (t, *J* = 8.1 Hz, 4H), 7.14 (d, *J* = 8.4 Hz, 2H), 4.60 (s, 2H), 3.99 (dd, *J* = 9.3, 4.1 Hz, 1H), 3.90 (s, 3H), 3.23 – 3.13 (m, 1H), 2.71 – 2.64 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.8, 173.7, 166.6, 139.9, 135.9, 135.8, 129.9, 129.9, 129.5, 128.7, 128.3, 52.2, 43.8, 42.3, 35.8. **ESI-MS (m/z) for $C_{19}H_{17}ClNO_4S$ $[M+H]^+$:** Calculated 390.0561, found 390.0554.



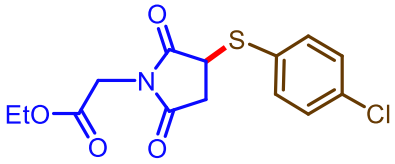
4-((4-Chlorophenyl)thio)-1-dodecylpyrrolidin-2-one (3y).

Yield 56.6 mg, 69%; Off-white solid; R_f= 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.45 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.1 Hz, 2H), 3.96 (dd, *J* = 9.3, 4.1 Hz, 1H), 3.39 (t, *J* = 7.4 Hz, 2H), 3.17 – 3.08 (m, 1H), 2.68 – 2.61 (m, 1H), 1.43 – 1.38 (m, 2H), 1.23 (s, 18H), 0.86 (t, *J* = 6.5 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 175.2, 174.2, 135.7, 135.7, 129.5, 128.9, 43.9, 39.2, 35.9, 31.9, 29.6, 29.5, 29.4, 29.3, 29.1, 27.5, 26.7, 22.6, 14.1. **ESI-MS (m/z) for $C_{22}H_{33}ClNO_2S$ $[M+H]^+$:** Calculated 410.1915, found 410.1921.



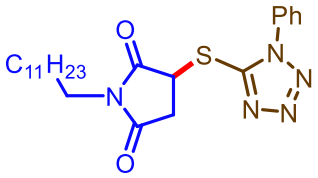
3-((4-Chlorophenyl)thio)-1-methylpyrrolidine-2,5-dione (3z).

Yield 35.8 mg, 70%; White solid; R_f= 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.44 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 4.00 (dd, *J* = 9.1, 4.0 Hz, 1H), 3.19 – 3.10 (m, 1H), 2.90 (s, 3H), 2.68 – 2.61 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 175.4, 174.2, 135.7, 135.4, 129.5, 129.1, 44.1, 36.0, 25.1. **ESI-MS (m/z) for $C_{11}H_{11}ClNO_2S$ $[M+H]^+$:** Calculated 256.0194, found 256.0199.



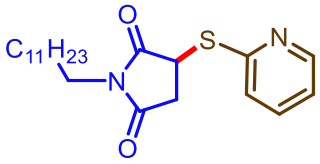
Ethyl 2-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)acetate (3aa).

Yield 48.5 mg, 74%; Pale yellowish solid; R_f= 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.46 (d, *J* = 8.1 Hz, 2H), 7.31 (d, *J* = 8.1 Hz, 2H), 4.19 (d, *J* = 6.1 Hz, 2H), 4.10 (dd, *J* = 9.2, 4.4 Hz, 1H), 3.27 – 3.18 (m, 1H), 2.74 –2.66 (m, 1H), 1.27 (q, *J* = 6.7 Hz, 5H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.6, 173.3, 166.2, 135.5, 135.2, 129.5, 129.3, 62.0, 44.0, 39.8, 35.7, 14.0. **ESI-MS (m/z) for $C_{14}H_{15}ClNO_4S$ $[M+H]^+$:** Calculated 328.0405, found 328.0401.



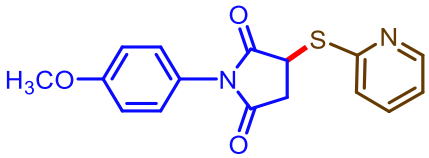
1-Dodecyl-3-((1-phenyl-1H-tetrazol-5-yl)thio)pyrrolidine-2,5-dione (3ab).

Yield 64.8 mg, 73%; Yellow solid; R_f= 0.2 (20% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.56 (s, 5H), 4.55 (dd, *J* = 9.3, 5.5 Hz, 1H), 3.59 – 3.54 (m, 2H), 3.45 – 3.35 (m, 1H), 3.12 – 3.04 (m, 1H), 1.60 (s, 2H), 1.28 – 1.24 (m, 18H), 0.85 (t, *J* = 6.5 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.84, 173.33, 151.43, 133.30, 130.65, 130.11, 123.84, 42.90, 40.03, 36.63, 32.00, 29.71, 29.66, 29.56, 29.43, 29.21, 27.45, 26.96, 22.77, 14.22. **ESI-MS (m/z) for $C_{23}H_{34}N_5O_2S$ $[M+H]^+$:** Calculated 444.2428, found 444.2434.



1-Dodecyl-3-(pyridin-2-ylthio)pyrrolidine-2,5-dione (3ac).

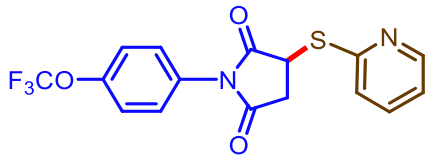
Yield 57.2 mg, 76%; Reddish brown liquid; R_f= 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.20 (d, *J* = 5.0 Hz, 1H), 7.52 – 7.46 (m, 1H), 7.17 (d, *J* = 8.1 Hz, 1H), 6.98 (dd, *J* = 7.6, 4.7 Hz, 1H), 4.20 (dd, *J* = 9.4, 5.5 Hz, 1H), 3.58 – 3.53 (m, 2H), 3.24 – 3.15 (m, 1H), 2.97 – 2.89 (m, 1H), 1.63 – 1.59 (m, 2H), 1.30 – 1.24 (m, 18H), 0.86 (t, *J* = 6.6 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 175.8, 175.4, 155.9, 148.9, 136.5, 122.1, 120.2, 40.7, 39.5, 36.5, 31.9, 29.6, 29.6, 29.5, 29.5, 29.3, 29.2, 27.5, 26.9, 22.6, 14.1. **ESI-MS (m/z) for $C_{21}H_{33}N_2O_2S$ $[M+H]^+$:** Calculated 377.2257, found 377.2253.



1-(4-Methoxyphenyl)-3-(pyridin-2-ylthio)pyrrolidine-2,5-dione (3ad).

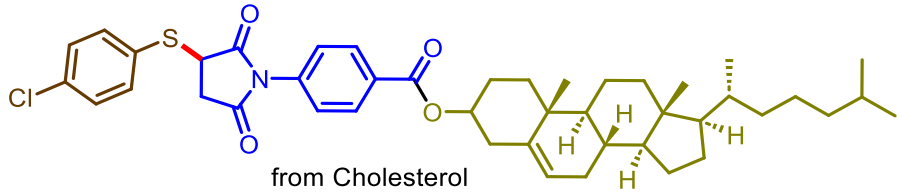
Yield 49.7 mg, 79%; Off-white solid; R_f = 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.31 (d, *J* = 4.4 Hz, 1H), 7.55 (t, *J* = 7.7 Hz, 1H), 7.29 – 7.27 (m, 2H), 7.23 (d, *J* = 8.1 Hz, 1H), 7.02 (t, *J* = 9.0 Hz, 3H), 4.28 (dd, *J* = 9.6, 5.5 Hz, 1H), 3.84 (s, 3H), 3.43 – 3.34 (m, 1H), 3.16 – 3.08 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 175.3, 174.8, 159.5,

155.9, 149.1, 136.7, 127.6, 125.1, 122.2, 120.3, 114.5, 55.5, 40.9, 36.5. **ESI-MS (m/z) for $C_{16}H_{15}N_2O_3S$ $[M+H]^+$:** Calculated 315.0798, found 315.0791.



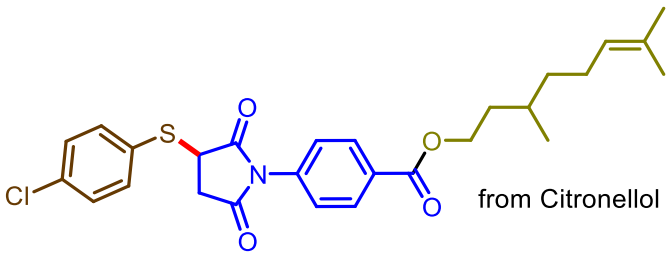
3-(Pyridin-2-ylthio)-1-(4-(trifluoromethoxy)phenyl)pyrrolidine-2,5-dione (3ae).

Yield 57.4 mg, 78%; Off-white solid; R_f= 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.26 (d, *J* = 5.0 Hz, 1H), 7.57 – 7.51 (m, 1H), 7.44 – 7.41 (m, 2H), 7.33 (d, *J* = 9.2 Hz, 2H), 7.22 (d, *J* = 8.2 Hz, 1H), 7.005 – 7.00 (m, 1H), 4.27 (dd, *J* = 9.7, 5.5 Hz, 1H), 3.44 – 3.35 (m, 1H), 3.16 – 3.08 (m, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 174.9, 174.2, 155.7, 149.0, 136.8, 130.8, 127.8, 122.2, 122.1, 121.6, 120.4, 118.6, 40.9, 36.4. **^{19}F NMR (377 MHz, $CDCl_3$)** δ -57.83. **ESI-MS (m/z) for $C_{16}H_{12}F_3N_2O_3S$ $[M+H]^+$:** Calculated 369.0515, found 369.0522.



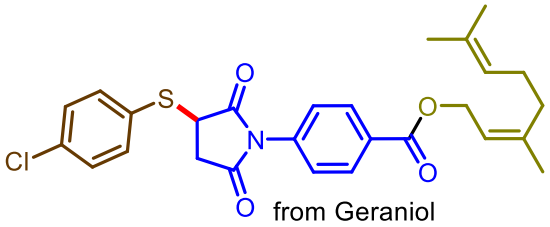
(8S,9S,10R,13R,14S,17R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 4-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoate (5a).

Yield 105.2 mg, 72%; Yellow ochre solid; R_f= 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.11 (d, *J* = 8.6 Hz, 2H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.22 (d, *J* = 8.6 Hz, 2H), 5.42 (d, *J* = 4.1 Hz, 1H), 4.91 – 4.81 (m, 1H), 4.15 (dd, *J* = 9.3, 3.9 Hz, 1H), 3.42 – 3.32 (m, 1H), 2.91 (d, *J* = 3.9 Hz, 1H), 2.45 (d, *J* = 7.7 Hz, 2H), 2.04 – 1.94 (m, 4H), 1.60 – 1.46 (m, 7H), 1.34 (d, *J* = 8.1 Hz, 3H), 1.25 (s, 2H), 1.17 – 1.11 (m, 5H), 1.06 (s, 3H), 1.02 (d, *J* = 8.8 Hz, 4H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.86 (d, *J* = 6.6 Hz, 7H), 0.69 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.9, 172.7, 164.9, 139.5, 136.3, 136.1, 135.1, 131.0, 130.4, 129.7, 128.4, 125.9, 122.9, 75.0, 56.7, 56.1, 50.0, 44.1, 42.3, 39.7, 39.5, 38.1, 37.0, 36.6, 36.2, 36.1, 35.8, 31.9, 31.8, 28.2, 28.0, 27.8, 24.3, 23.8, 22.8, 22.5, 21.0, 19.3, 18.7, 11.8. **ESI-MS (m/z) for $C_{44}H_{57}ClNO_4S$ $[M+H]^+$:** Calculated 730.3691, found 730.3682.



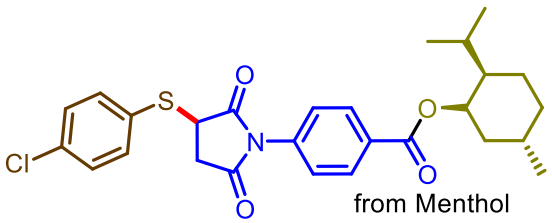
3,7-Dimethyloct-6-en-1-yl 4-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoate (5b).

Yield 70.0 mg, 70%; Pale white solid; R_f= 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.11 (d, *J* = 8.6 Hz, 2H), 7.51 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.22 (d, *J* = 8.6 Hz, 2H), 5.09 (t, *J* = 7.1 Hz, 1H), 4.38 – 4.35 (m, 2H), 4.15 (dd, *J* = 9.4, 3.9 Hz, 1H), 3.42 – 3.32 (m, 1H), 2.92 – 2.84 (m, 1H), 2.04 – 1.98 (m, 2H), 1.67 (s, 3H), 1.60 (s, 3H), 1.31 (s, 1H), 1.25 (s, 2H), 0.96 (dd, *J* = 6.4, 4.2 Hz, 3H), 0.86 (d, *J* = 6.6 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.9, 172.7, 165.6, 136.3, 136.1, 135.2, 131.4, 130.6, 130.4, 129.7, 128.4, 126.0, 124.5, 63.9, 44.1, 36.9, 36.2, 35.4, 29.5, 25.7, 25.4, 19.4, 17.7. **ESI-MS (m/z) for $C_{27}H_{31}ClNO_4S$ $[M+H]^+$:** Calculated 500.1657, found 500.1649.



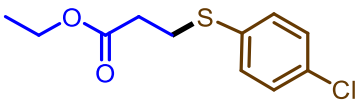
(Z)-3,7-dimethylocta-2,6-dien-1-yl 4-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoate (5c).

Yield 67.7 mg, 68%; Pale yellow solid; R_f= 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.12 (d, *J* = 8.6 Hz, 2H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.22 (d, *J* = 8.6 Hz, 2H), 5.45 (t, *J* = 7.1 Hz, 1H), 5.09 (t, *J* = 6.8 Hz, 1H), 4.84 (d, *J* = 7.1 Hz, 2H), 4.15 (dd, *J* = 9.3, 3.9 Hz, 1H), 3.42 – 3.32 (m, 1H), 2.92 – 2.84 (m, 1H), 2.15 – 2.03 (m, 4H), 1.76 (s, 3H), 1.67 (s, 3H), 1.61 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.9, 172.7, 165.6, 142.8, 136.3, 136.1, 135.2, 131.9, 130.7, 130.4, 129.7, 128.4, 125.9, 123.7, 118.1, 62.2, 44.1, 39.5, 36.2, 26.3, 25.7, 17.7, 16.6. **ESI-MS (m/z) for $C_{14}H_{15}O_2S$ $[M+H]^+$:** Calculated 500.1657, found 247.0798. **ESI-MS (m/z) for $C_{27}H_{29}ClNO_4S$ $[M+H]^+$:** Calculated 498.1500, found 498.1492.



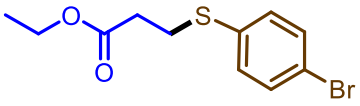
2-Isopropyl-5-methylcyclohexyl 4-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoate (5d).

Yield 73.0 mg, 73%; Pale yellow solid; R_f= 0.2 (12% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.12 (d, *J* = 8.6 Hz, 2H), 7.52 (d, *J* = 8.6 Hz, 2H), 7.34 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.7 Hz, 2H), 4.97 – 4.88 (m, 1H), 4.15 (dd, *J* = 9.3, 3.9 Hz, 1H), 3.42 – 3.33 (m, 1H), 2.91 – 2.84 (m, 1H), 2.12– 2.08 (m, 1H), 1.92 (t, *J* = 7.1 Hz, 1H), 1.72 (d, *J* = 9.6 Hz, 3H), 1.59 – 1.55 (m, 2H), 1.15 – 1.07 (m, 2H), 0.92 (t, *J* = 6.4 Hz, 6H), 0.78 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.9, 172.8, 165.0, 136.2, 136.1, 135.1, 131.0, 130.4, 129.8, 129.7, 126.0, 75.2, 47.2, 44.1, 40.9, 36.2, 34.2, 31.4, 26.5, 23.6, 22.0, 20.7, 16.4. **ESI-MS (m/z) for $C_{27}H_{31}ClNO_4S$ $[M+H]^+$:** Calculated 500.1657, found 500.1648.



Ethyl 3-((4-chlorophenyl)thio)propanoate (7a).

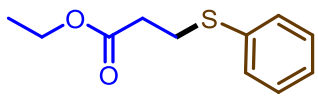
Yield 40.1 mg, 82%; Yellow liquid; R_f= 0.2 (2% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 – 7.24 (m, 4H), 4.14 (q, *J* = 7.1 Hz, 2H), 3.14 (t, *J* = 7.4 Hz, 2H), 2.60 (t, *J* = 7.4 Hz, 2H), 1.25 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.5, 133.8, 132.5, 131.4, 129.1, 60.7, 34.2, 29.2, 14.1. **ESI-MS (m/z) for $C_{11}H_{14}ClO_2S$ $[M+H]^+$:** Calculated 245.0398, found 245.0393.



Ethyl 3-((4-bromophenyl)thio)propanoate (7b).

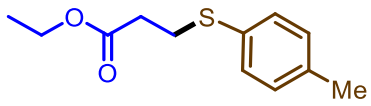
Yield 46.8 mg, 81%; Pale yellow solid; R_f= 0.2 (3% EtOAc in petroleum ether, TLC). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 (dt, *J* = 8.8, 3.2 Hz, 2H), 7.19 (dt, *J* = 8.8, 3.1 Hz, 2H), 4.11 (q, *J* = 7.1 Hz, 2H), 3.11 (t, *J* = 7.4 Hz, 2H), 2.57 (t, *J* = 7.4 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H). **^{13}C**

NMR (101 MHz, $CDCl_3$) δ 171.49, 134.61, 132.06, 131.48, 120.41, 60.80, 34.26, 29.06, 14.2. **ESI-MS (m/z) for $C_{11}H_{14}BrO_2S$ $[M+H]^+$:** Calculated 288.9892, found 288.9897.



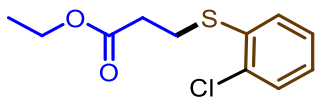
Ethyl 3-(phenylthio)propanoate (7c).

Yield 33.6 mg, 80%; Yellow liquid; R_f= 0.2 (2% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.32 (d, *J* = 7.4 Hz, 2H), 7.26 – 7.19 (m, 2H), 7.14 (t, *J* = 6.5 Hz, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.11 (t, *J* = 7.4 Hz, 2H), 2.56 (t, *J* = 7.4 Hz, 2H), 1.21 – 1.15 (m, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.4, 135.4, 129.8, 128.9, 126.4, 60.5, 34.3, 28.9, 14.1. **ESI-MS (m/z) for $C_{11}H_{15}O_2S$ $[M+H]^+$:** Calculated 211.0787, found 211.0783.



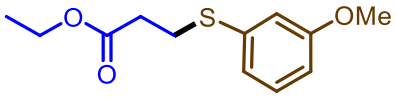
Ethyl 3-(p-tolylthio)propanoate (7d).

Yield 38.1 mg, 85%; Light yellow liquid; R_f= 0.2 (3% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.28 (d, *J* = 8.3 Hz, 2H), 7.10 (d, *J* = 7.9 Hz, 2H), 4.12 (q, *J* = 7.1 Hz, 2H), 3.10 (t, *J* = 7.4 Hz, 2H), 2.58 (t, *J* = 7.4 Hz, 2H), 2.31 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.82, 136.82, 131.42, 131.05, 129.81, 60.67, 34.55, 29.79, 21.05, 14.2. **ESI-MS (m/z) for $C_{12}H_{17}O_2S$ $[M+H]^+$:** Calculated 225.0944, found 225.0949.



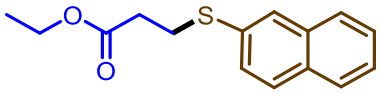
Ethyl 3-((2-chlorophenyl)thio)propanoate (7e).

Yield 36.7 mg, 75%; Whitish liquid; R_f= 0.2 (2% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.38 – 7.30 (m, 2H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.13 (t, *J* = 7.6 Hz, 1H), 4.14 (q, *J* = 7.1 Hz, 2H), 3.19 (t, *J* = 7.5 Hz, 2H), 2.64 (t, *J* = 7.5 Hz, 2H), 1.25 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.5, 134.7, 134.2, 129.8, 129.3, 127.2, 127.1, 60.8, 33.9, 27.6, 14.1. **ESI-MS (m/z) for $C_{11}H_{14}ClO_2S$ $[M+H]^+$:** Calculated 245.0398, found 245.0392.



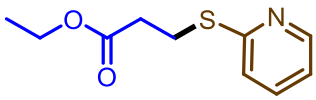
Ethyl 3-((3-methoxyphenyl)thio)propanoate (7f).

Yield 39.9 mg, 83%; Light yellowish liquid; R_f= 0.2 (2% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.19 (t, *J* = 8.0 Hz, 1H), 6.92 (d, *J* = 7.7 Hz, 1H), 6.89 (s, 1H), 6.73 (dd, *J* = 8.7, 3.0 Hz, 1H), 4.13 (q, *J* = 7.1 Hz, 2H), 3.78 (s, 3H), 3.16 (t, *J* = 7.4 Hz, 2H), 2.62 (t, *J* = 7.4 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.7, 159.8, 136.6, 129.8, 121.8, 115.1, 112.1, 60.7, 55.2, 34.4, 28.7, 14.1. **ESI-MS (m/z) for $C_{12}H_{17}O_3S$ $[M+H]^+$:** Calculated 241.0893, found 241.0897.



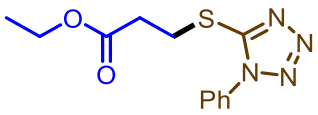
Ethyl 3-(naphthalen-2-ylthio)propanoate (7g).

Yield 42.2 mg, 81%; Light pink liquid; R_f= 0.2 (2% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.82 – 7.75 (m, 4H), 7.51 – 7.43 (m, 3H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.29 (t, *J* = 7.4 Hz, 2H), 2.68 (t, *J* = 7.4 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.7, 133.7, 132.8, 132.0, 128.6, 128.0, 127.8, 127.7, 127.2, 126.6, 125.9, 60.7, 34.4, 28.9, 14.2. **ESI-MS (m/z) for $C_{15}H_{17}O_2S$ $[M+H]^+$:** Calculated 261.0944, found 261.0941.



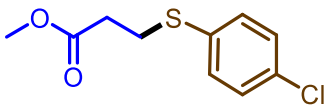
Ethyl 3-(pyridin-2-ylthio)propanoate (7h).

Yield 30.4 mg, 72%; Deep yellowish liquid; R_f= 0.2 (2% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 8.43 (d, *J* = 4.4 Hz, 1H), 7.49 (t, *J* = 7.7 Hz, 1H), 7.17 (d, *J* = 8.1 Hz, 1H), 7.01 – 6.97 (m, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.44 (t, *J* = 7.1 Hz, 2H), 2.77 (t, *J* = 7.1 Hz, 2H), 1.28 – 1.23 (m, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 172.1, 158.1, 149.1, 136.3, 122.5, 119.5, 60.7, 34.6, 25.1, 14.2. **ESI-MS (m/z) for $C_{10}H_{14}NO_2S$ $[M+H]^+$:** Calculated 212.0740, found 212.0735.



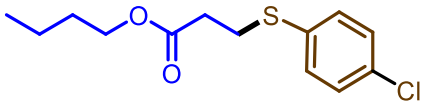
Ethyl 3-((1-phenyl-1H-tetrazol-5-yl)thio)propanoate (7i).

Yield 39.5 mg, 71%; Deep yellowish liquid; R_f= 0.2 (15% EtOAc in petroleum ether, TLC). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.53 (p, *J* = 3.6 Hz, 5H), 4.62 (dd, *J* = 6.6, 3.9 Hz, 1H), 4.23 – 4.18 (m, 2H), 3.90 – 3.86 (m, 2H), 3.73 – 3.67 (m, 1H), 1.25 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 172.2, 153.9, 133.4, 130.3, 129.8, 123.9, 69.2, 62.4, 37.3, 14.1. **ESI-MS (m/z) for $C_{12}H_{15}N_4O_2S$ $[M+H]^+$:** Calculated 279.0910, found 279.0915.



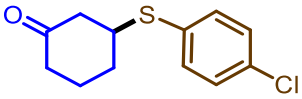
Methyl 3-((4-chlorophenyl)thio)propanoate (7j).

Yield 31.8 mg, 69%; Yellow liquid; R_f= 0.2 (2% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.33 – 7.25 (m, 4H), 3.68 (s, 3H), 3.28 – 3.21 (m, 1H), 2.95 – 2.87 (m, 2H), 2.69 (q, *J* = 7.0 Hz, 1H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 175.1, 134.2, 132.5, 131.4, 129.1, 51.9, 39.6, 37.6. **ESI-MS (m/z) for $C_{10}H_{12}ClO_2S$ $[M+H]^+$:** Calculated 231.0241, found 231.0236.



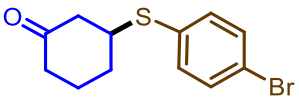
Butyl 3-((4-chlorophenyl)thio)propanoate (7k).

Yield 41.5 mg, 76%; Yellow liquid; R_f= 0.2 (2% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.31 – 7.24 (m, 4H), 4.08 (t, *J* = 6.7 Hz, 2H), 3.14 (t, *J* = 7.3 Hz, 2H), 2.60 (t, *J* = 7.3 Hz, 2H), 1.65 – 1.55 (m, 2H), 1.43 – 1.33 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 171.6, 133.8, 132.6, 131.4, 129.1, 64.6, 34.3, 30.6, 29.3, 19.1, 13.6. **ESI-MS (m/z) for $C_{13}H_{18}ClO_2S$ $[M+H]^+$:** Calculated 273.0711, found 273.0703.



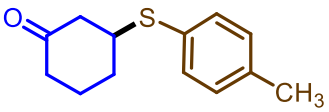
3-((4-Chlorophenyl)thio)cyclohexan-1-one (9a).

Yield 36.6 mg, 76%; Yellow sticky liquid; R_f = 0.2 (5% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.35 – 7.25 (m, 4H), 3.43 – 3.34 (m, 1H), 2.68 – 2.62 (m, 1H), 2.38 – 2.28 (m, 3H), 2.12 (t, *J* = 7.6 Hz, 2H), 1.78 – 1.66 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 208.4, 134.5, 134.0, 131.5, 129.2, 47.6, 46.3, 40.8, 31.1, 23.9. **ESI-MS (m/z) for $C_{12}H_{14}ClOS$ $[M+H]^+$:** Calculated 241.0448, found 241.0443.



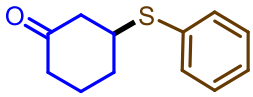
3-((4-Bromophenyl)thio)cyclohexan-1-one (9b).

Yield 42.8 mg, 75%; Brownish yellow liquid; R_f= 0.2 (6% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.46 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 3.47 – 3.38 (m, 1H), 2.73 – 2.66 (m, 1H), 2.42 – 2.32 (m, 3H), 2.21 – 2.13 (m, 2H), 1.77 – 1.70 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 208.4, 134.7, 132.2, 129.4, 122.1, 47.6, 46.2, 40.8, 31.1, 23.9. **ESI-MS (m/z) for $C_{12}H_{14}BrOS$ $[M+H]^+$:** Calculated 284.9943, found 284.9949.



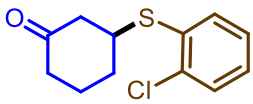
3-(p-Tolylthio)cyclohexan-1-one (9c).

Yield 34.8 mg, 79%; Light brownish liquid; R_f= 0.2 (5% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.28 (d, *J* = 8.2 Hz, 2H), 7.07 (d, *J* = 8.1 Hz, 2H), 3.35 – 3.26 (m, 1H), 2.64 – 2.57 (m, 1H), 2.34 – 2.24 (m, 6H), 2.13 – 2.04 (m, 2H), 1.69 – 1.62 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 208.8, 138.0, 133.9, 129.8, 129.1, 47.7, 46.4, 40.8, 31.1, 24.0, 21.1. **ESI-MS (m/z) for $C_{13}H_{17}OS$ $[M+H]^+$:** Calculated 221.0995, found 221.0990.



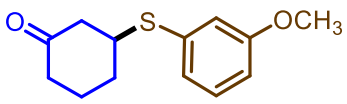
3-(Phenylthio)cyclohexan-1-one (9d).

Yield 31.4 mg, 76%; Yellow liquid; R_f= 0.2 (5% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.35 (d, *J* = 5.9 Hz, 2H), 7.25 – 7.16 (m, 3H), 3.37 (t, *J* = 4.0 Hz, 1H), 2.63 – 2.57 (m, 1H), 2.33 – 2.20 (m, 3H), 2.07 – 2.00 (m, 2H), 1.71 – 1.58 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 208.5, 133.1, 133.0, 129.0, 127.7, 47.6, 46.0, 40.8, 31.1, 23.9. **ESI-MS (m/z) for $C_{12}H_{15}OS$ $[M+H]^+$:** Calculated 207.0838, found 207.0834.



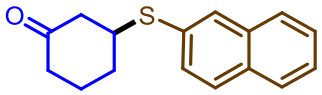
3-((2-Chlorophenyl)thio)cyclohexan-1-one (9e).

Yield 33.2 mg, 69%; Yellow-brownish liquid; R_f= 0.2 (10% EtOAc in petroleum ether, TLC). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.37 (td, *J* = 7.7, 3.2 Hz, 2H), 7.17 – 7.14 (m, 2H), 3.57 – 3.51 (m, 1H), 2.66 – 2.62 (m, 1H), 2.40 – 2.28 (m, 3H), 2.11 (dd, *J* = 9.9, 4.5 Hz, 2H), 1.74 – 1.69 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 208.2, 136.6, 133.4, 132.5, 130.1, 128.6, 127.2, 47.4, 44.7, 40.8, 30.9, 24.0. **ESI-MS (m/z) for $C_{12}H_{14}ClOS$ $[M+H]^+$:** Calculated 241.0448, found 241.0445.



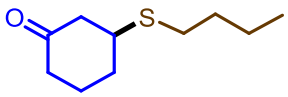
3-((3-Methoxyphenyl)thio)cyclohexan-1-one (9f).

Yield 36.4 mg, 77%; Light-yellowish liquid; R_f= 0.2 (75.15% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.13 (t, *J* = 7.0 Hz, 1H), 6.92 – 6.88 (m, 2H), 6.73 (d, *J* = 8.3 Hz, 1H), 3.70 (s, 3H), 3.40 – 3.36 (m, 1H), 2.63 – 2.57 (m, 1H), 2.34 – 2.20 (m, 3H), 2.07 – 2.02 (m, 2H), 1.72 – 1.58 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 208.5, 159.7, 134.3, 129.8, 124.8, 118.1, 113.2, 55.2, 47.5, 45.8, 40.8, 31.0, 23.9. **ESI-MS (m/z) for $C_{13}H_{17}O_2S$ $[M+H]^+$:** Calculated 237.0944, found 237.0948.



3-(Naphthalen-2-ylthio)cyclohexan-1-one (9g).

Yield 36.9 mg, 72%; Brownish liquid; R_f= 0.2 (4% EtOAc in petroleum ether, TLC). **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.91 (d, *J* = 1.7 Hz, 1H), 7.82 – 7.76 (m, 3H), 7.50 – 7.47 (m, 3H), 3.59 – 3.49 (m, 1H), 2.76 – 2.70 (m, 1H), 2.46 – 2.30 (m, 3H), 2.20 – 2.12 (m, 2H), 1.83 – 1.69 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 208.7, 133.6, 132.5, 132.1, 130.4, 130.2, 128.7, 127.7, 127.5, 126.6, 126.5, 47.7, 46.1, 40.9, 31.2, 24.0. **ESI-MS (m/z) for $C_{16}H_{17}OS$ $[M+H]^+$:** Calculated 257.0995, found 257.0991.



3-(Butylthio)cyclohexan-1-one (9h).

Yield 25.7 mg, 69%; Light yellow liquid; R_f= 0.2 (7% EtOAc in petroleum ether, TLC). **^{1}H NMR (400 MHz, $CDCl_3$)** δ 2.92 (t, *J* = 7.4 Hz, 2H), 2.59 (t, *J* = 6.0 Hz, 2H), 2.51 – 2.48 (m, 3H), 2.04 – 2.00 (m, 2H), 1.62 (t, *J* = 7.6 Hz, 2H), 1.47 – 1.40 (m, 4H), 0.93 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 190.0, 35.1, 32.7, 32.1, 30.3, 29.7, 28.2, 22.5, 21.8, 13.6. **ESI-MS (m/z) for $C_{10}H_{19}OS$ $[M+H]^+$:** Calculated 187.1151, found 187.1148.

4. References

1. Frisch, M.; Trucks, G.; Schlegel, H.; Scuseria, G.; Robb, M.; Cheeseman, J.; Scalmani, G.; Barone, V.; Petersson, G.; Nakatsuji, H., Rev. B. 01 Gaussian16, Gaussian. Inc., Wallingford CT 2016.

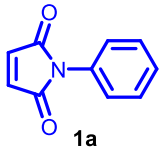
2. Ghosh, D.; Molla, S. A.; Ghosh, N. N.; Khamarui, S.; Maiti, D. K., CuII-Catalyzed cis-Selective Synthesis of Ketoepoxides from Phenacyl Bromides and Water. *J. Org. Chem.* **2023**, *88*, 9657-9667.

3. (a) Mandal, R.; Emayavaramban, B.; Sundararaju, B. Cp*Co(III)-Catalyzed C–H Alkylation with Maleimides Using Weakly Coordinating Carbonyl Directing Groups. *Org. Lett.* **2018**, *20*, 2835-2838.

4. García-Domínguez, A.; Mondal, R.; Nevado, C. Dual Photoredox/Nickel-Catalyzed Three-Component Carbofunctionalization of Alkenes. *Angew. Chem. Int. Ed.* **2019**, *58*, 12286–12290.

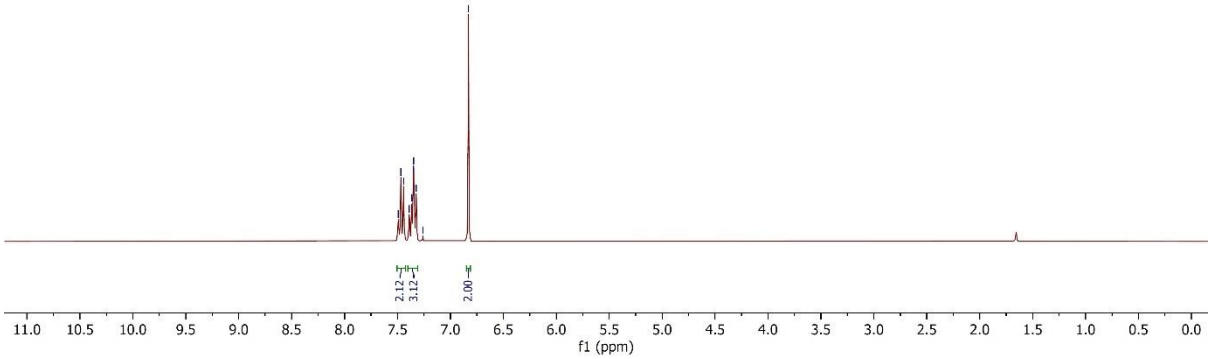
5. ^{1}H and ^{13}C NMR Spectra for all the Synthesized Compounds

GS-90-DB-1.1.fid



1a

^{1}H NMR (300 MHz, $CDCl_3$)



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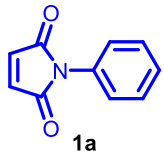
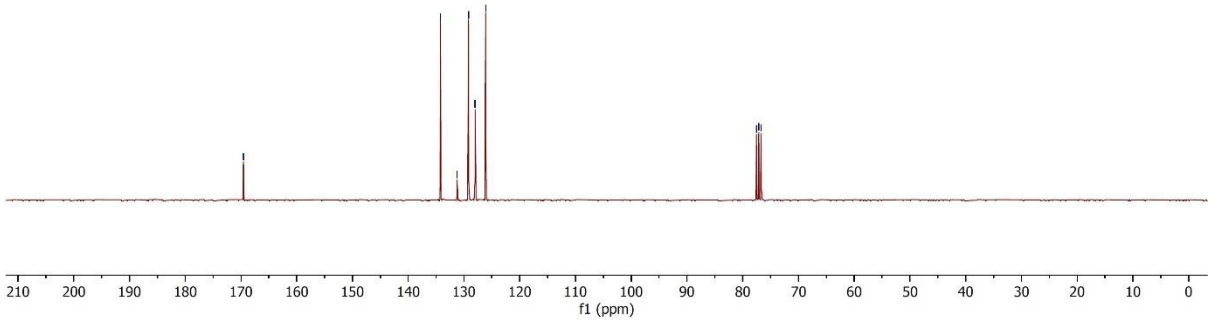


1a

^{13}C NMR (75 MHz, $CDCl_3$)



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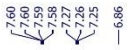
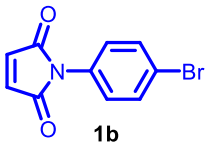
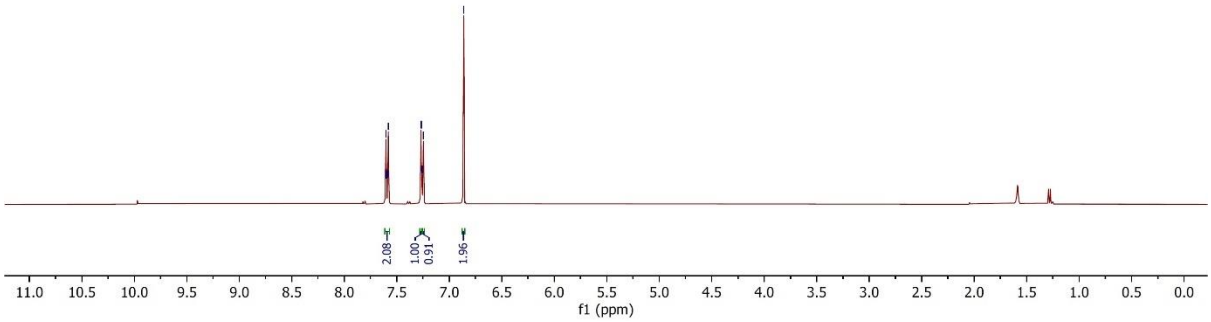




1b

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



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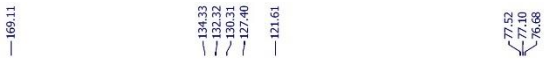
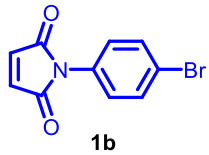
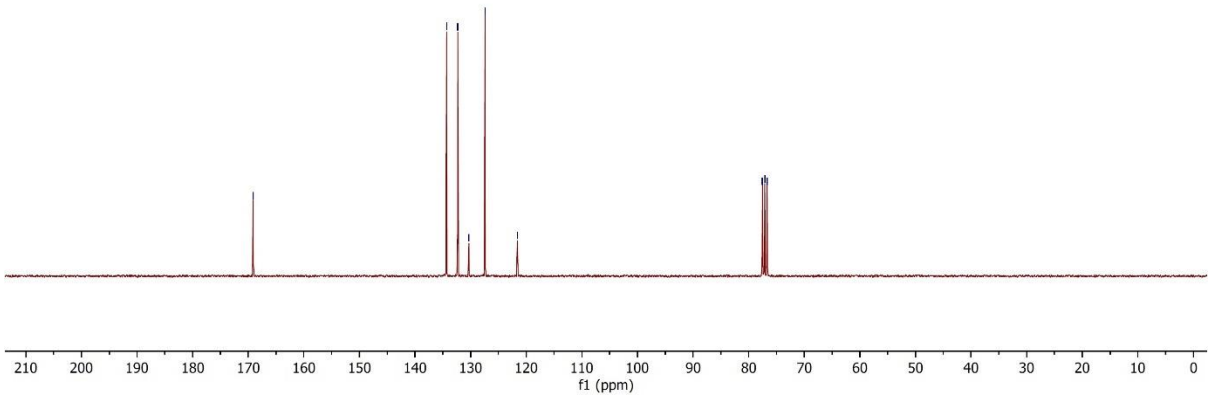




1b

^{13}C NMR (75 MHz, $CDCl_3$)



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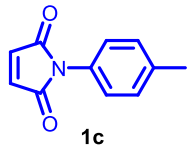
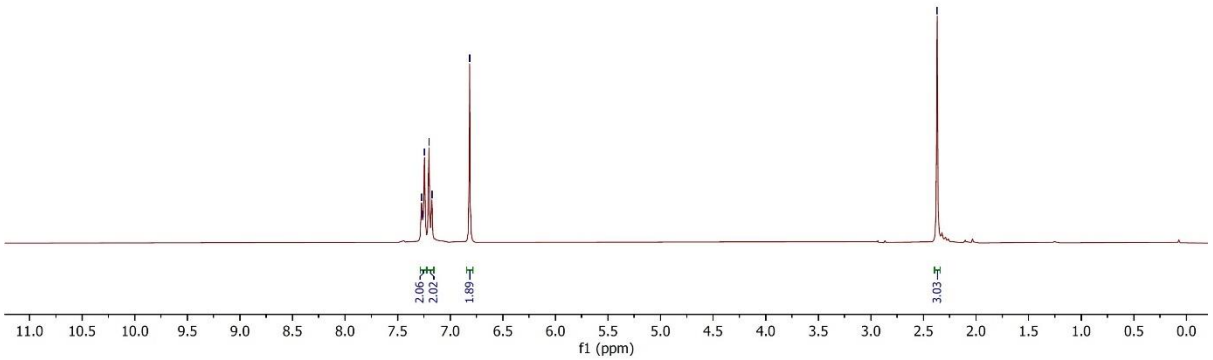


1c

^{1}H NMR (300 MHz, $CDCl_3$)



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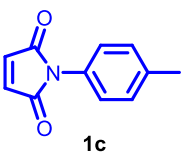
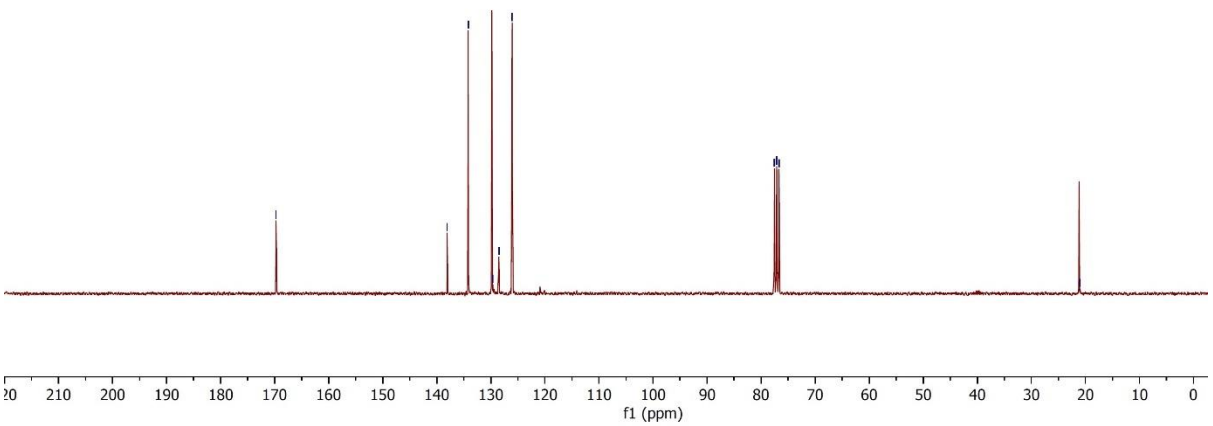


1c

^{13}C NMR (75 MHz, $CDCl_3$)



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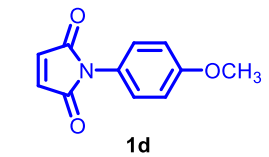
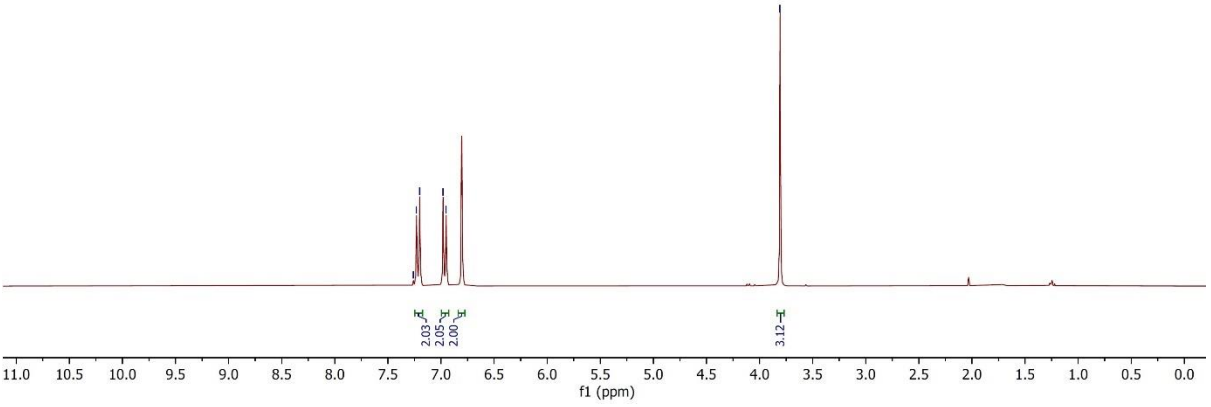
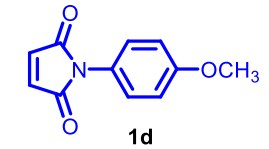


1d

^{1}H NMR (300 MHz, $CDCl_3$)



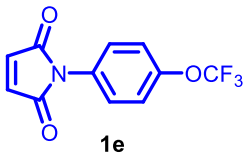
DKM-DJB-174 13C.2.fid



1d

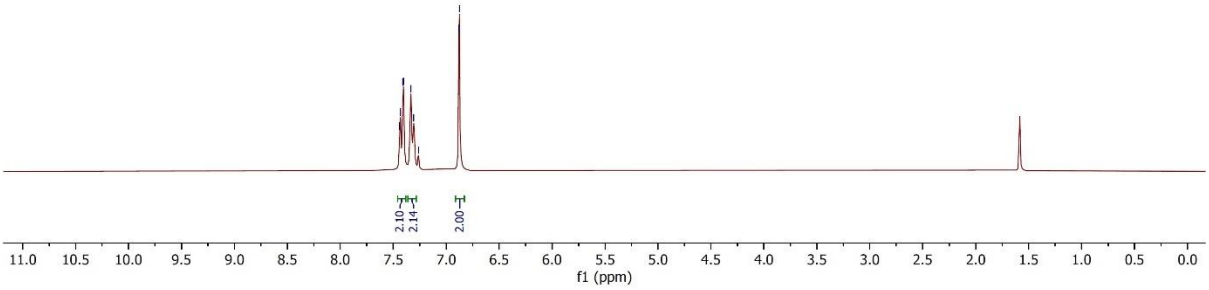
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-177.1.fid



1e

^{1}H NMR (300 MHz, $CDCl_3$)



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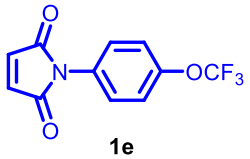
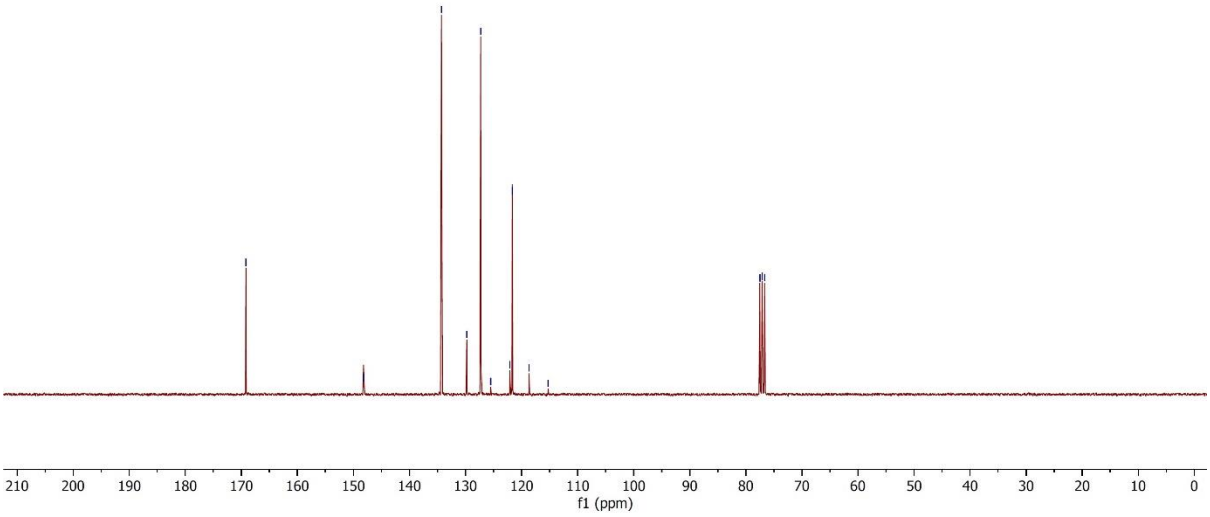
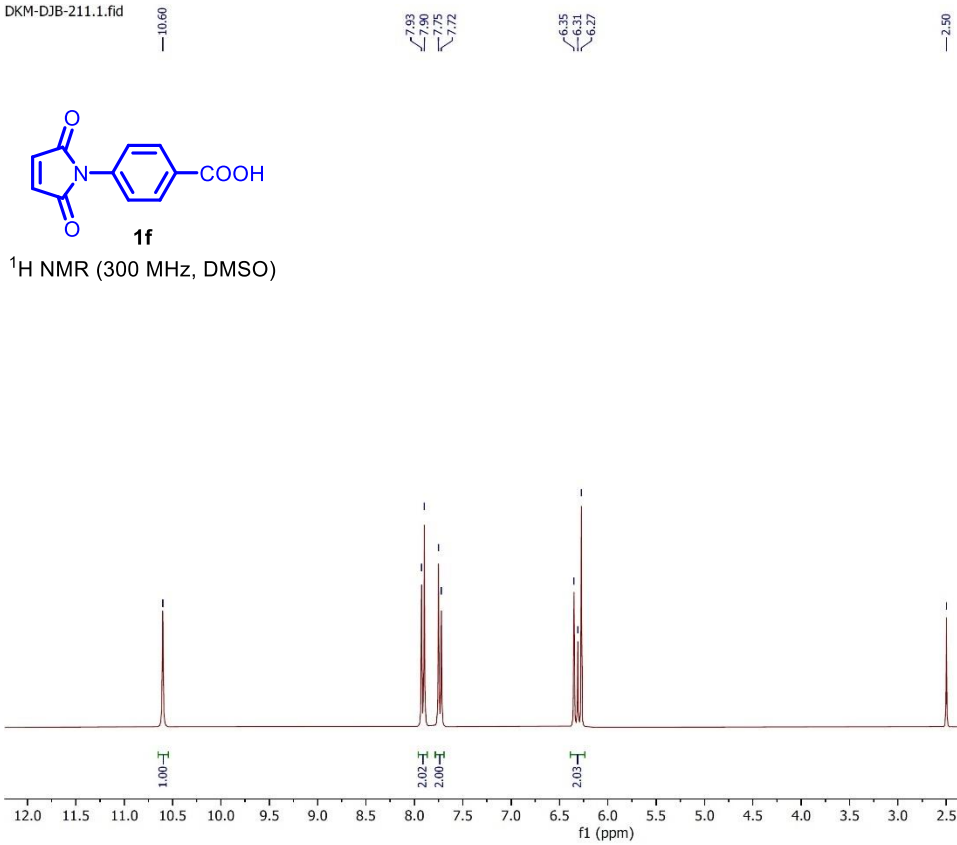


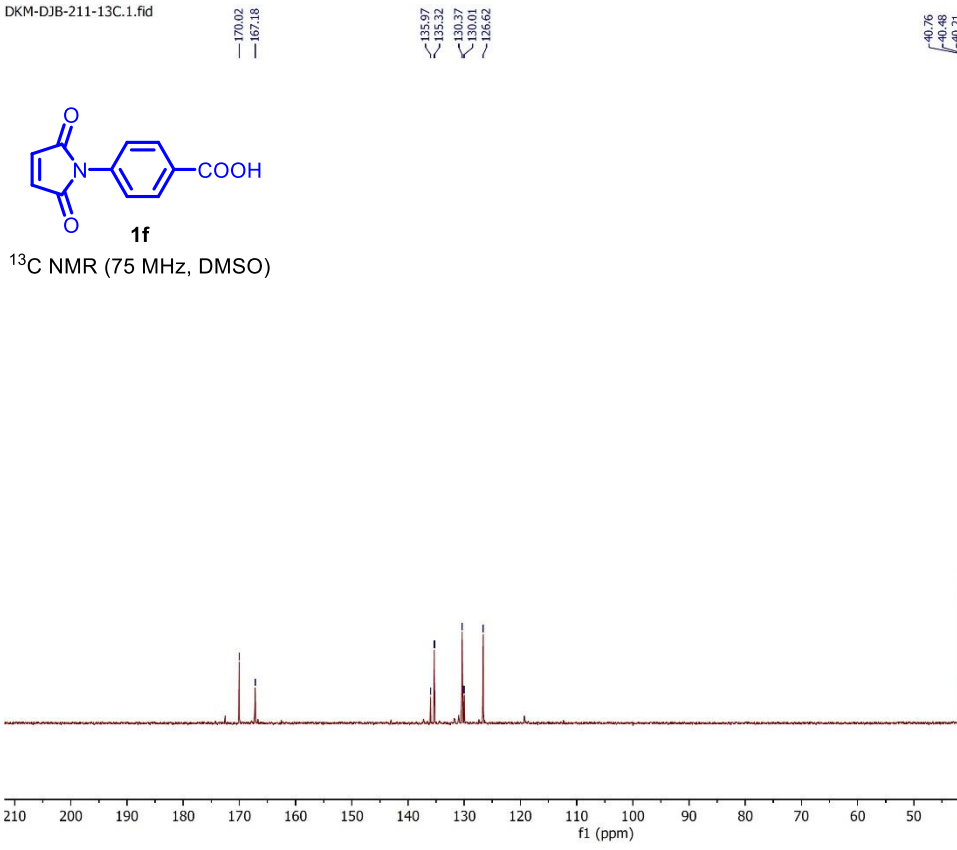
1e

^{13}C NMR (75 MHz, $CDCl_3$)



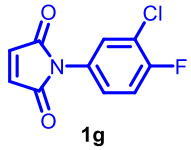


^{1}H NMR (300 MHz, DMSO)



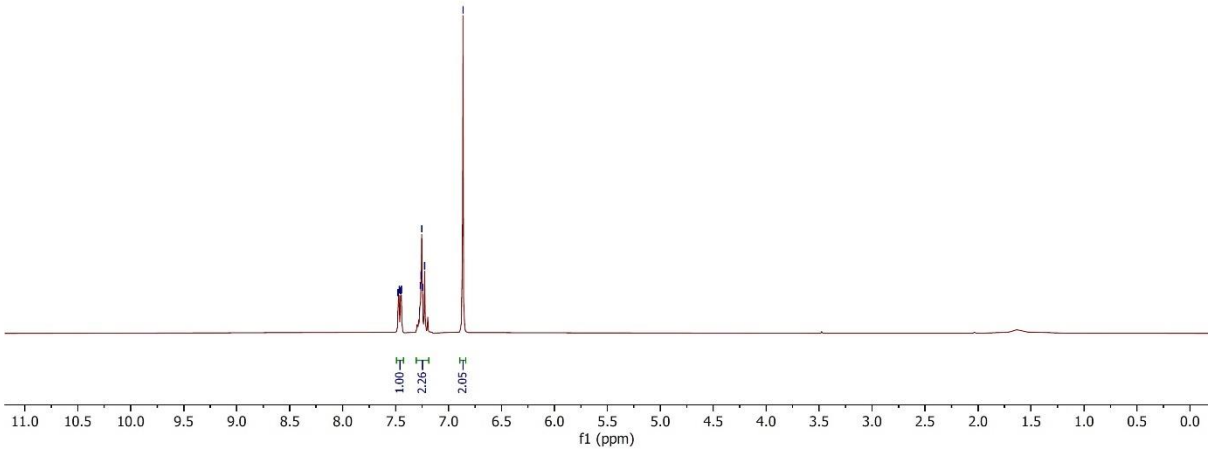
^{13}C NMR (75 MHz, DMSO)

DKM-DJB-180.1.fid



1g

^{1}H NMR (300 MHz, $CDCl_3$)



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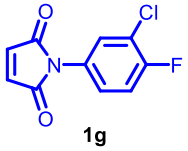
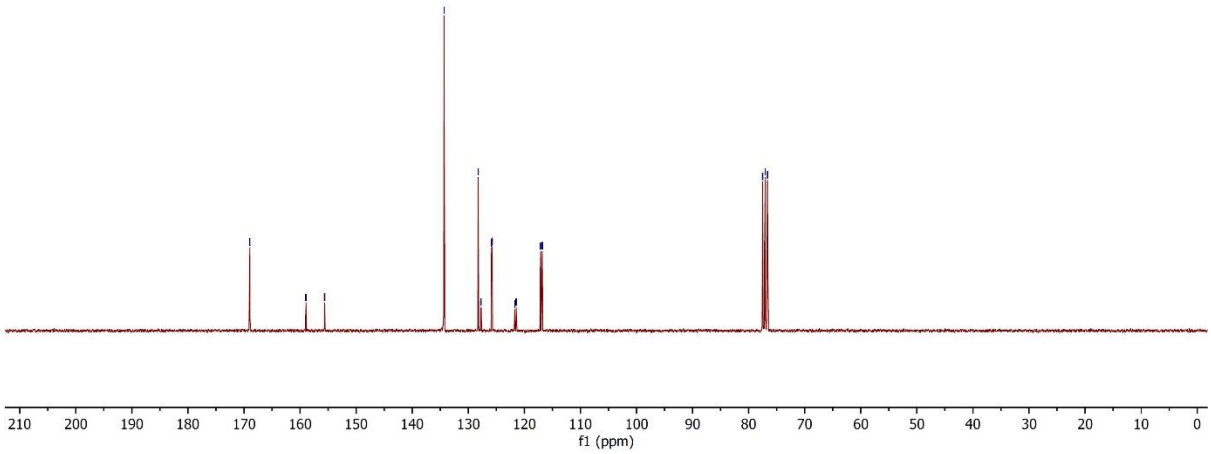


1g

^{13}C NMR (75 MHz, $CDCl_3$)



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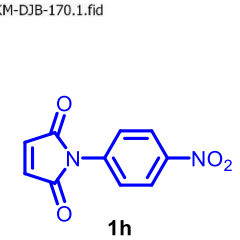
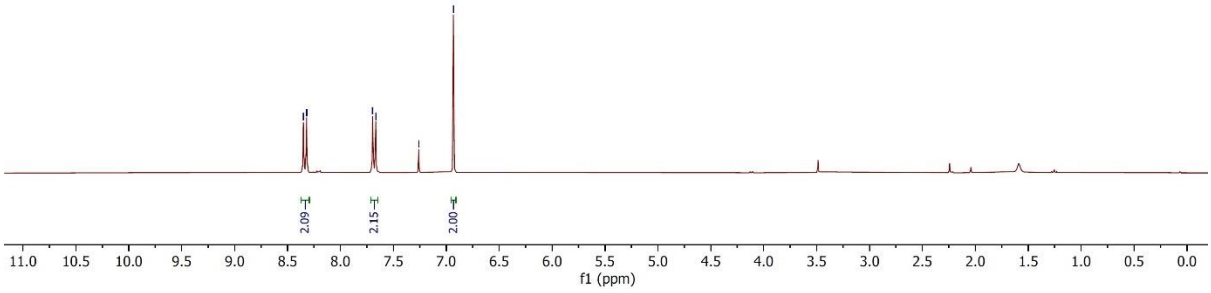
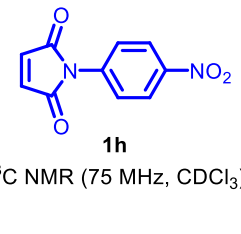


1h

^{1}H NMR (300 MHz, $CDCl_3$)

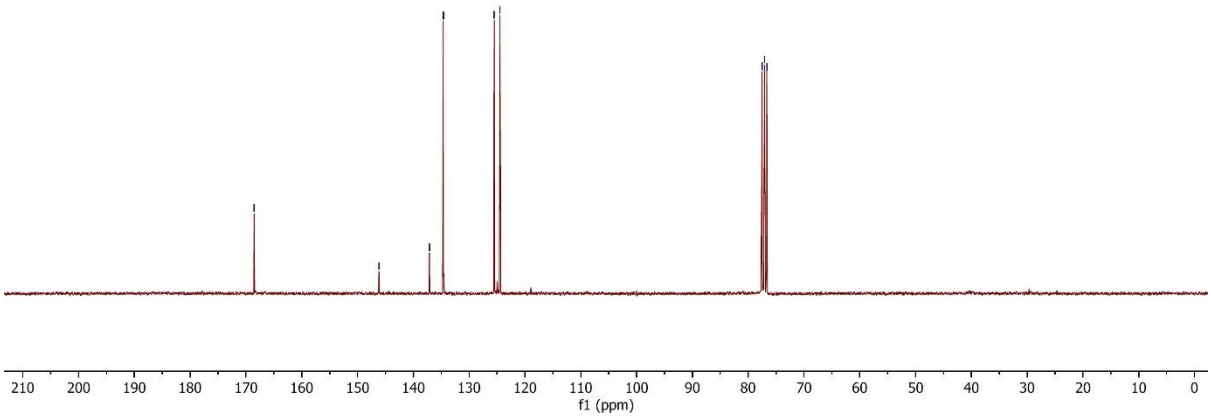


DKM-DJB-170 13C.2.fid

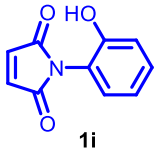


1h

^{13}C NMR (75 MHz, $CDCl_3$)

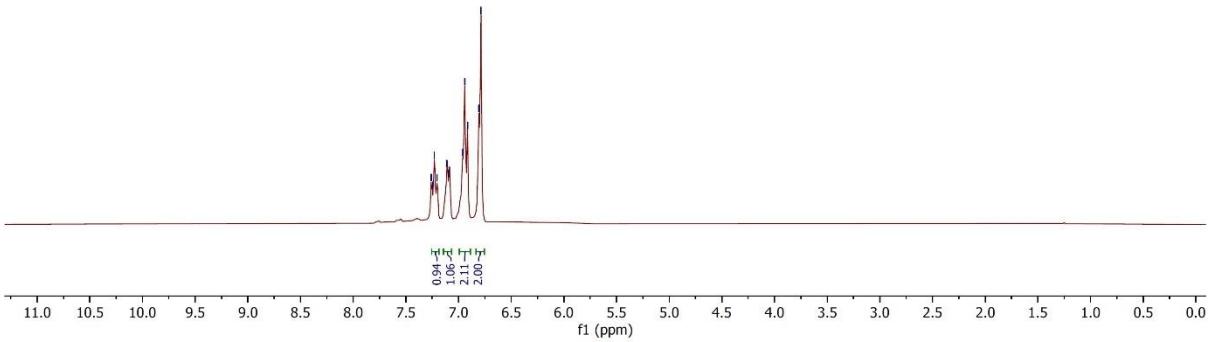


DKM-DJB-213.2.fid

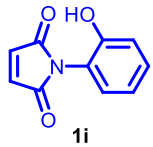


1i

^{1}H NMR (300 MHz, $CDCl_3$)

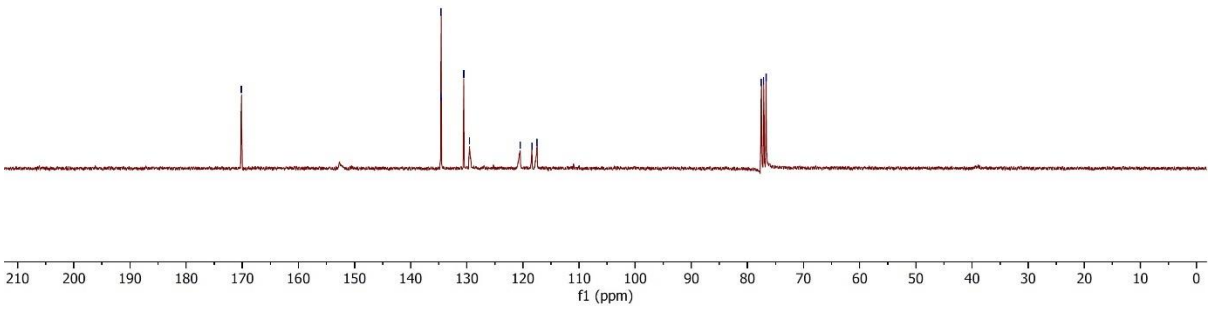


DKM-DJB-213-13C.1.fid

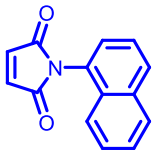


1i

^{13}C NMR (75 MHz, $CDCl_3$)

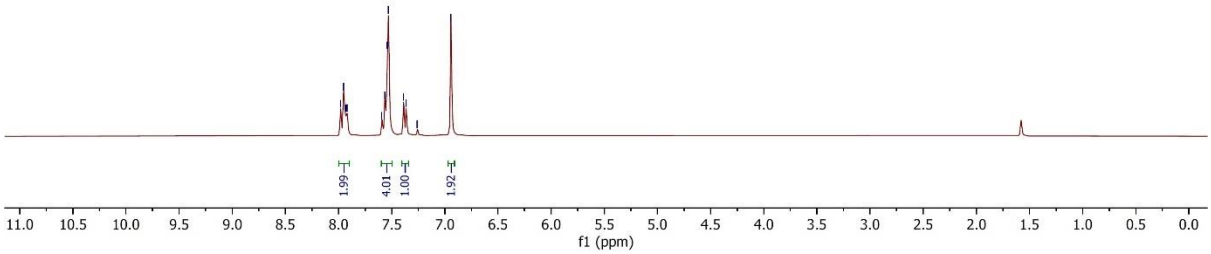


DKM-DJB-137.1.fid

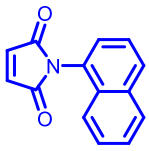


1j

^{1}H NMR (300 MHz, $CDCl_3$)

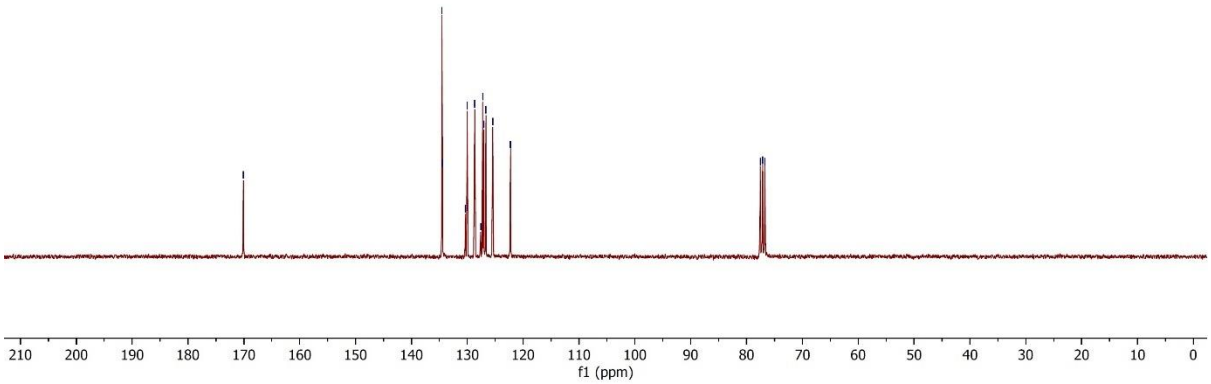


DKM-DJB-137-13C.1.fid

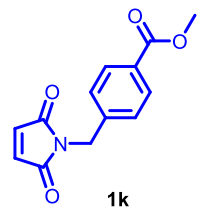


1j

^{13}C NMR (75 MHz, $CDCl_3$)

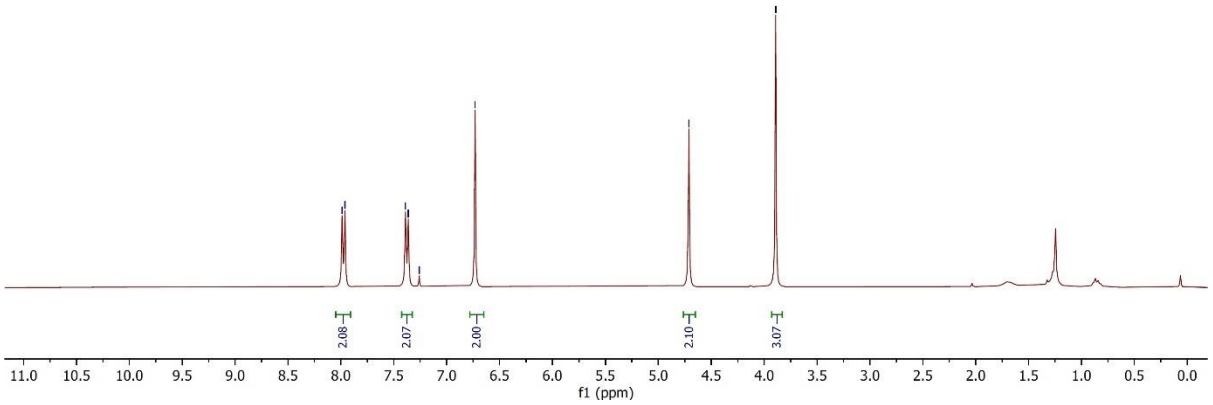


DKM-DJB-179.1.fid

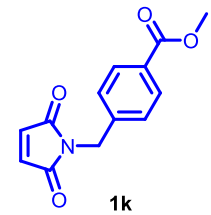


1k

^{1}H NMR (300 MHz, $CDCl_3$)

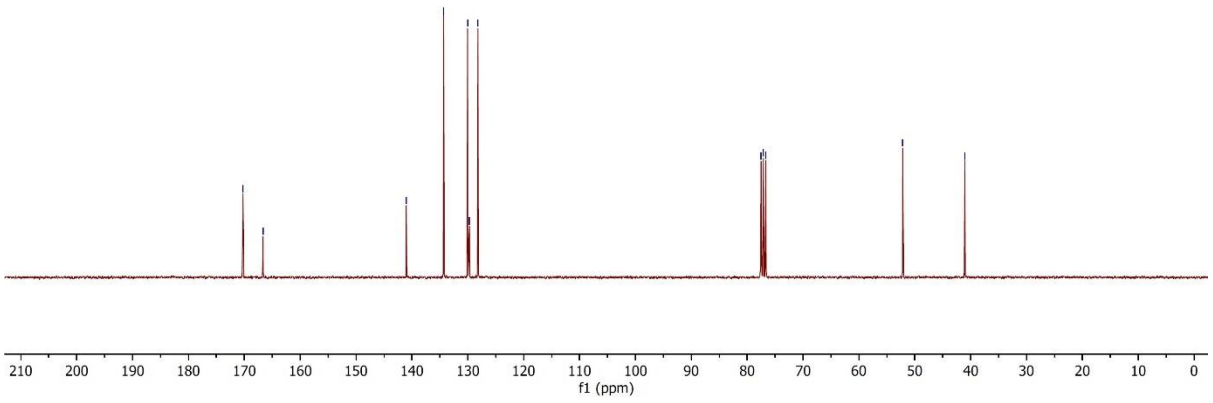


DKM-DJB-179-13C.1.fid

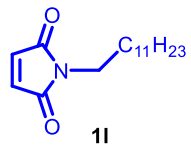


1k

^{13}C NMR (75 MHz, $CDCl_3$)



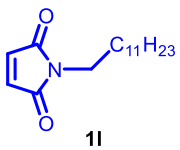
DKM-DJB-138.1.fid



1l

^{1}H NMR (300 MHz, $CDCl_3$)

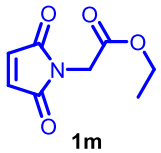
DKM-DJB-138 13C.23.fid



1l

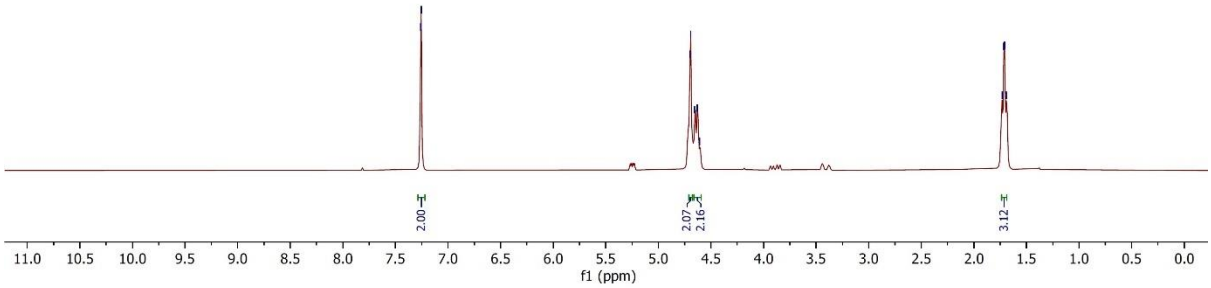
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-153.2.fid

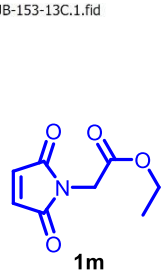


1m

^{1}H NMR (300 MHz, $CDCl_3$)



DKM-DJB-153-13C.1.fid

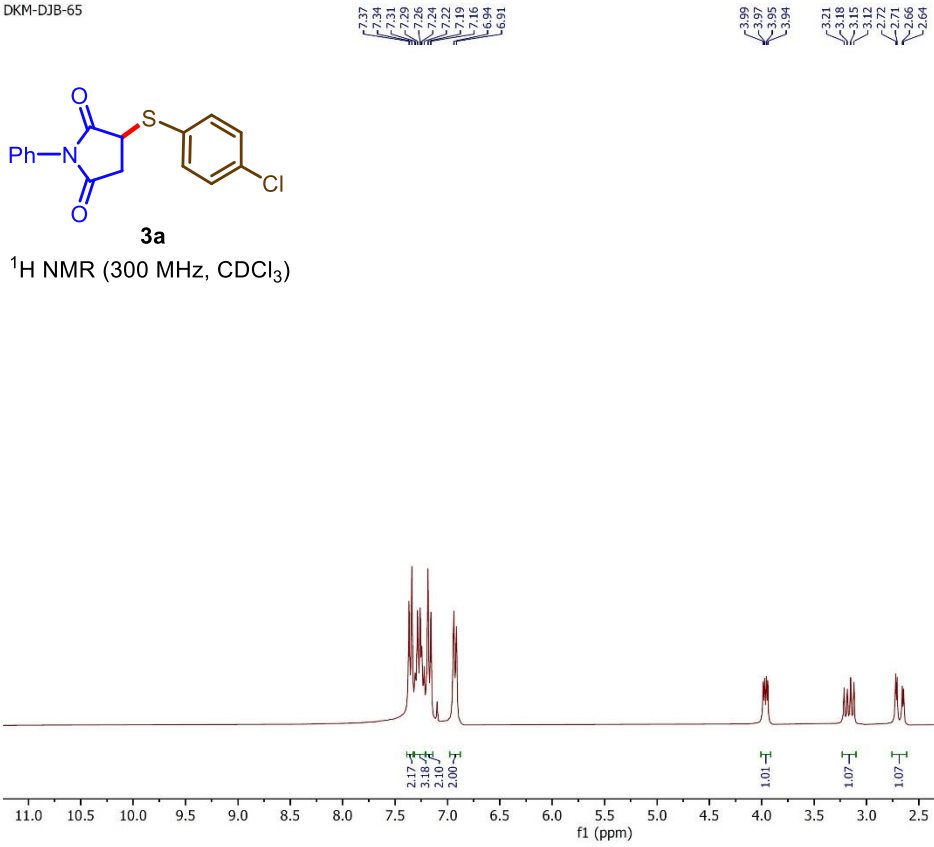


1m

^{13}C NMR (75 MHz, $CDCl_3$)

^{1}H and ^{13}C NMR Spectra for all the Synthesized Compounds

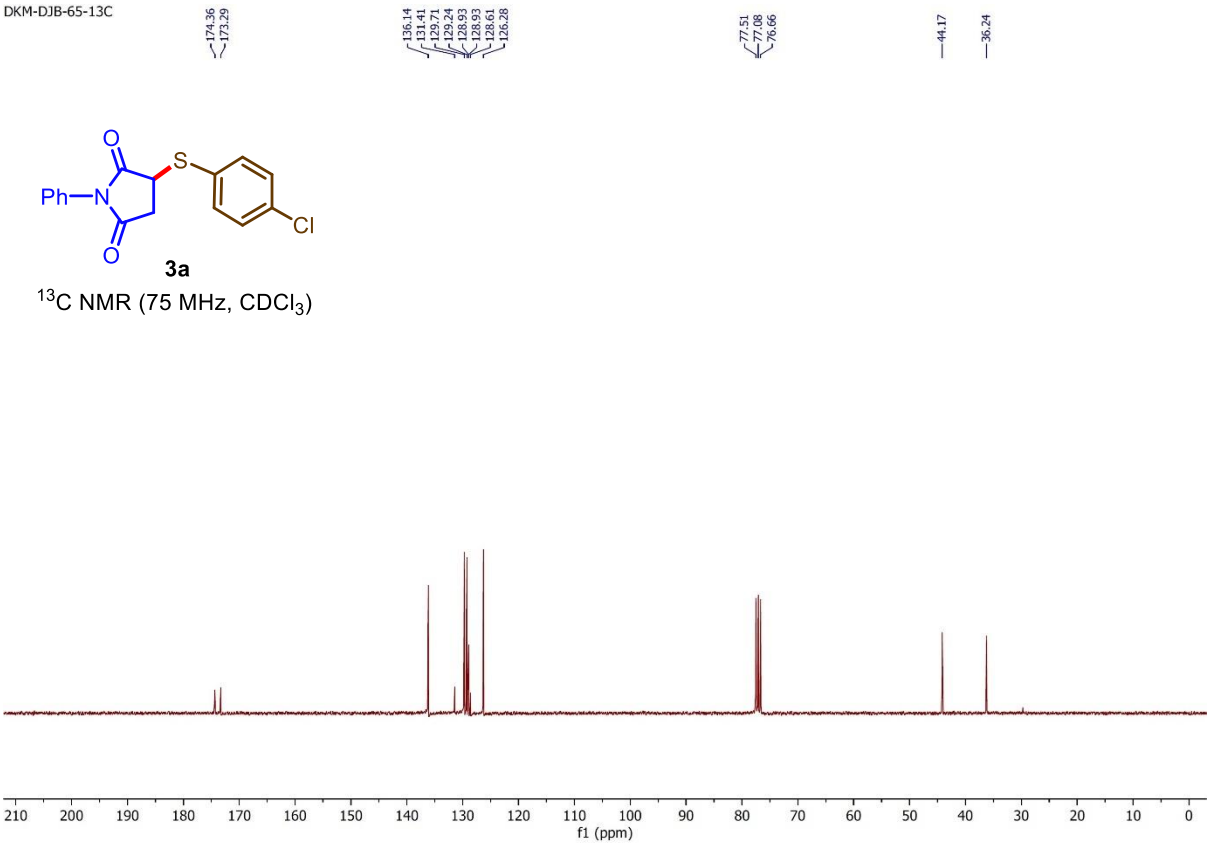
DKM-DJB-65



3a

^{1}H NMR (300 MHz, $CDCl_3$)

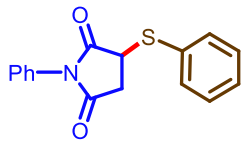
DKM-DJB-65-13C



3a

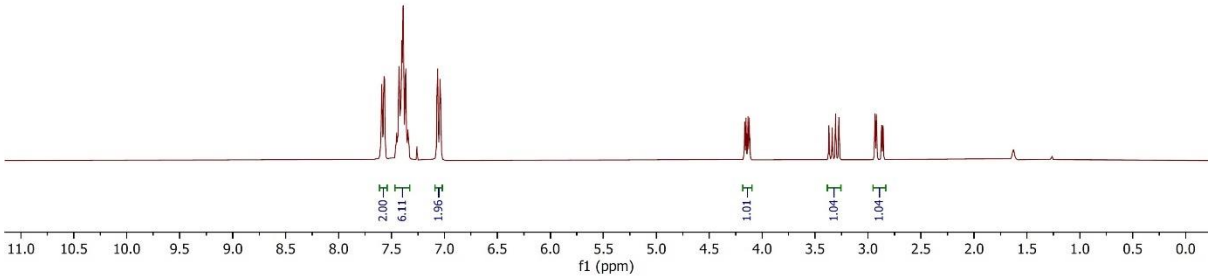
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-133

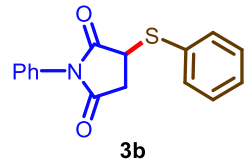


3b

^{1}H NMR (300 MHz, $CDCl_3$)

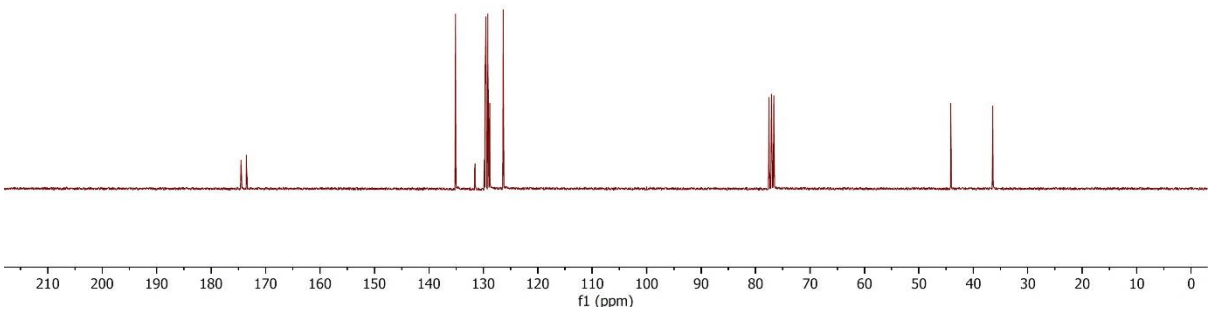


DKM-DJB-133-13C

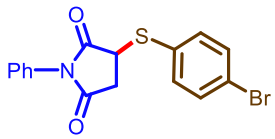


3b

^{13}C NMR (75 MHz, $CDCl_3$)

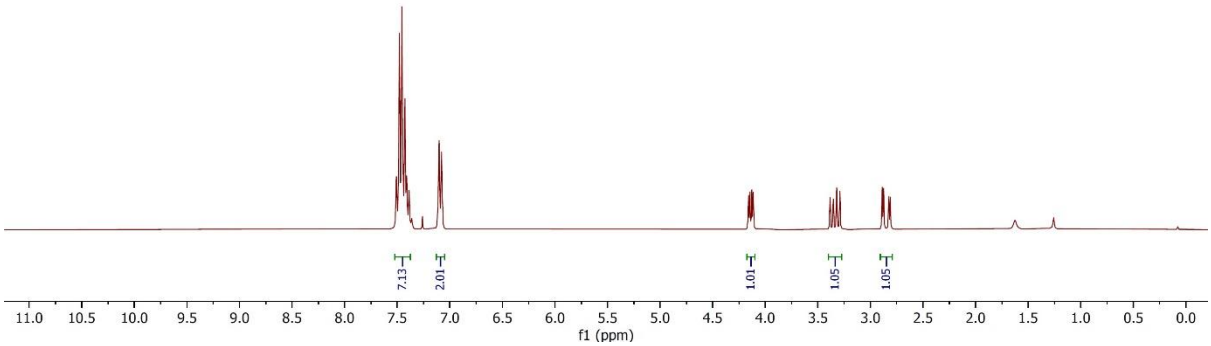


DKM-DJB-131

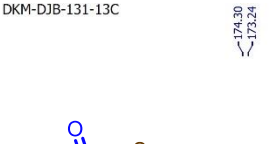


3c

^{1}H NMR (300 MHz, $CDCl_3$)



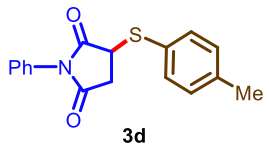
DKM-DJB-131-13C



3c

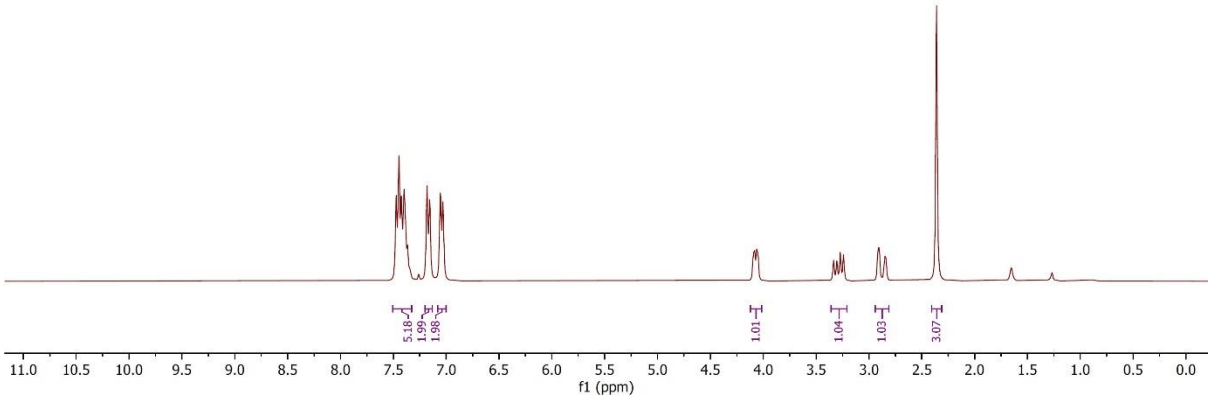
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-71

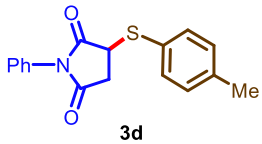


3d

^{1}H NMR (300 MHz, $CDCl_3$)

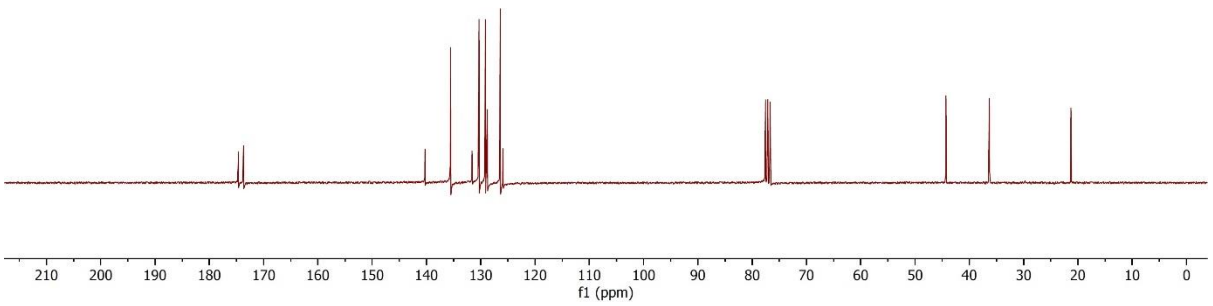


DKM-DJB-71-13C

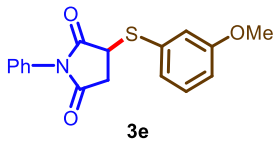


3d

^{13}C NMR (75 MHz, $CDCl_3$)

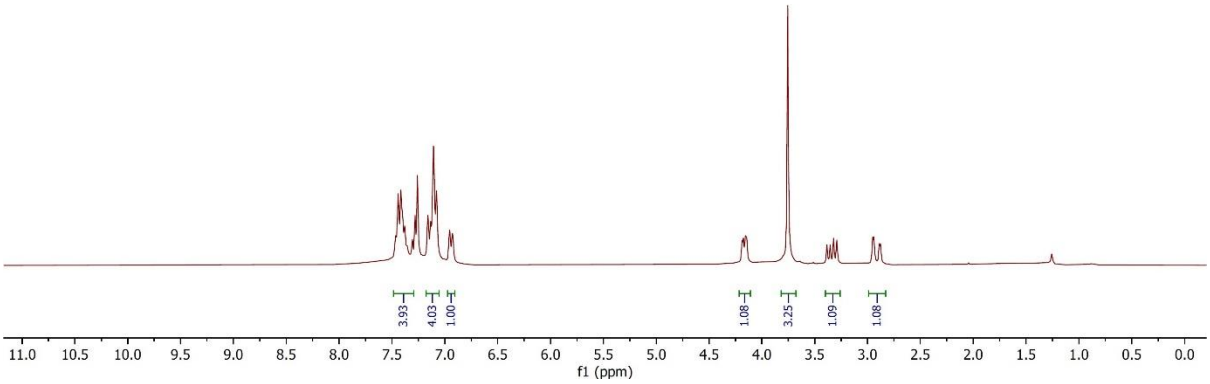


DKM-DJB-70

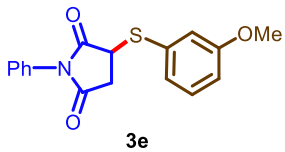


3e

^{1}H NMR (300 MHz, $CDCl_3$)

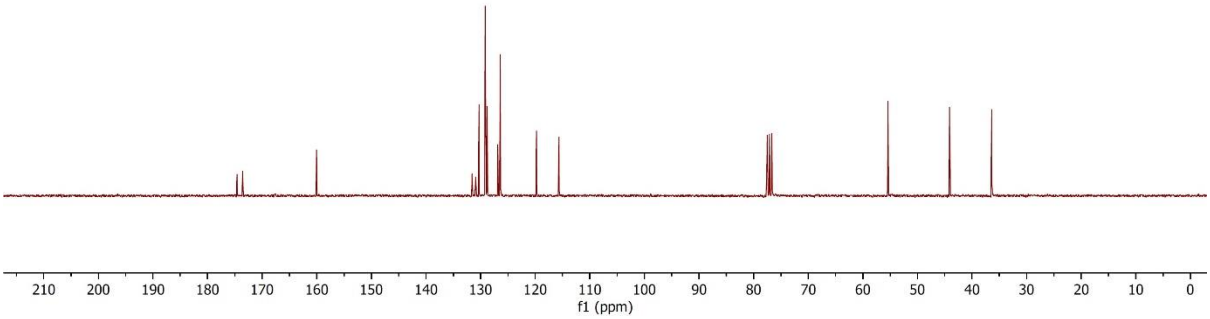


DKM-DJB-70-13C

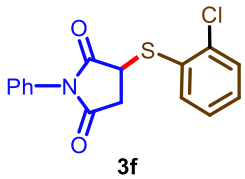


3e

^{13}C NMR (75 MHz, $CDCl_3$)

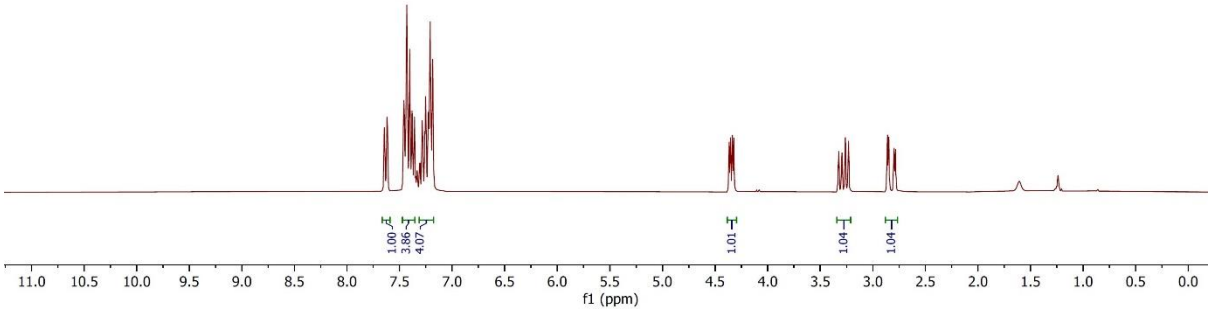


DKM-DJB-134

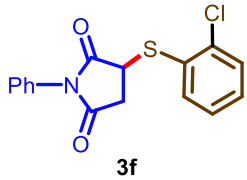


3f

^{1}H NMR (300 MHz, $CDCl_3$)

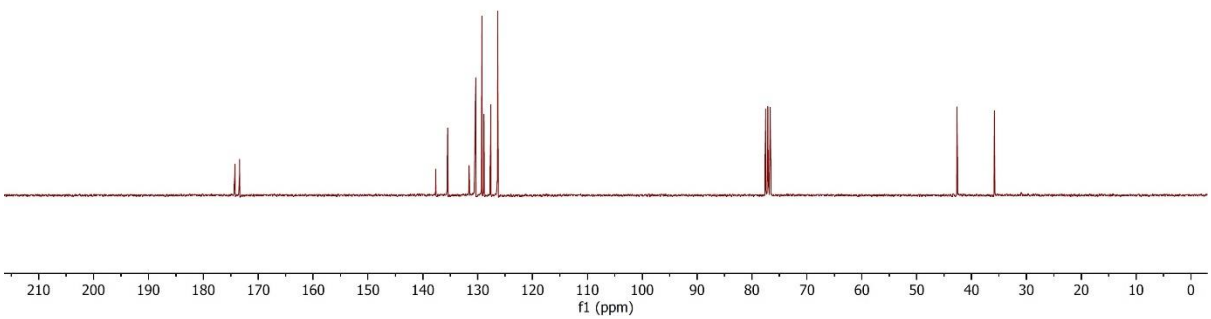


DKM-DJB-134-13C

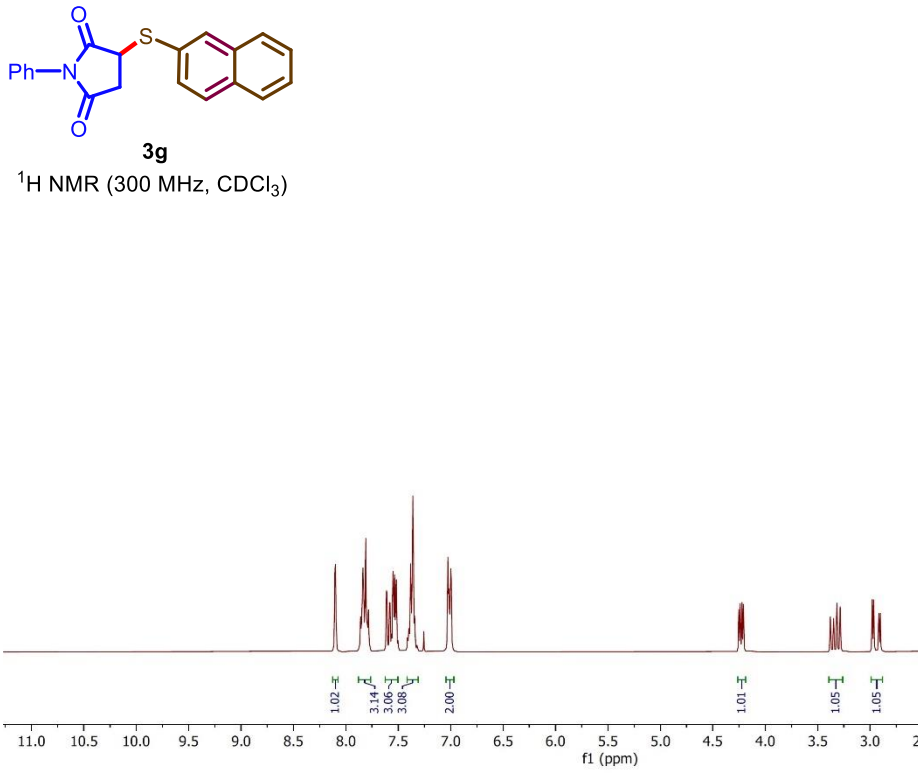


3f

^{13}C NMR (75 MHz, $CDCl_3$)



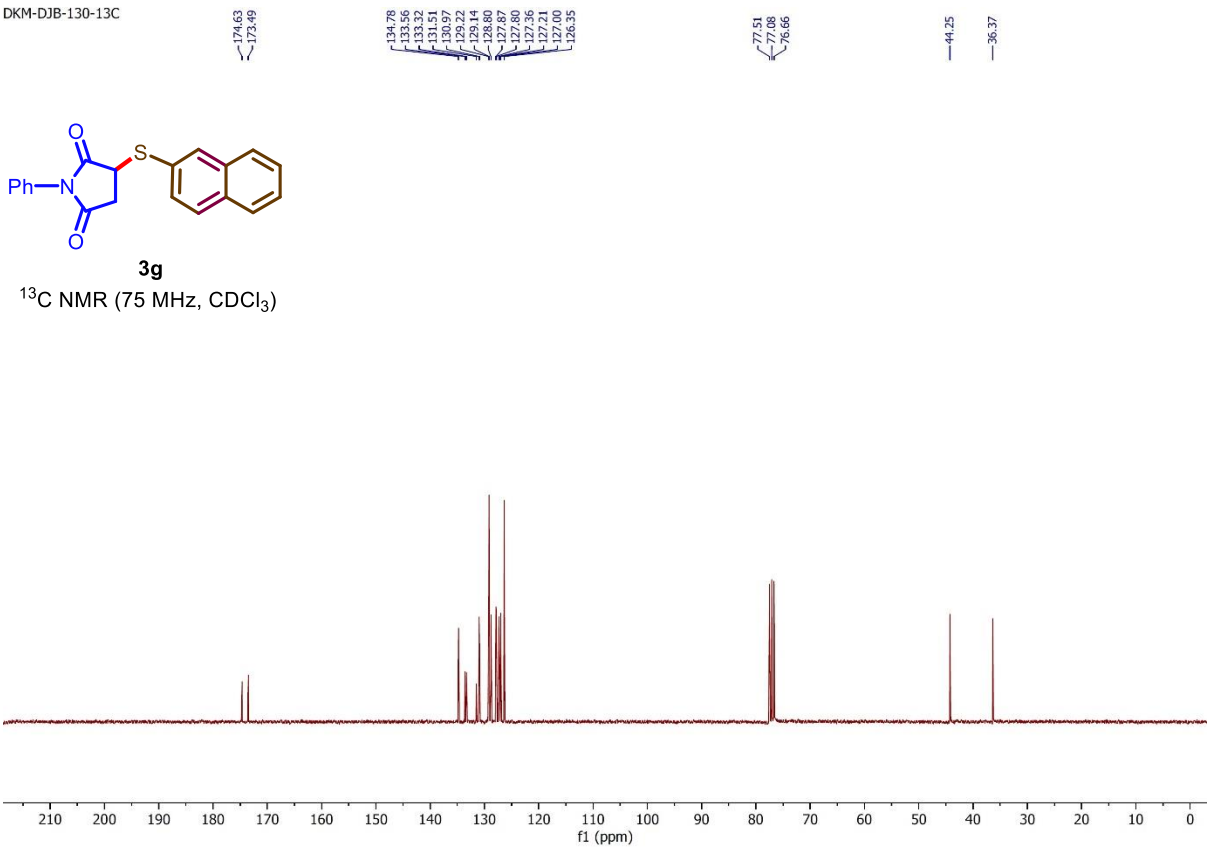
DKM-DJB-130



3g

^{1}H NMR (300 MHz, $CDCl_3$)

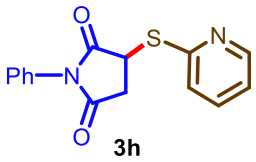
DKM-DJB-130-13C



3g

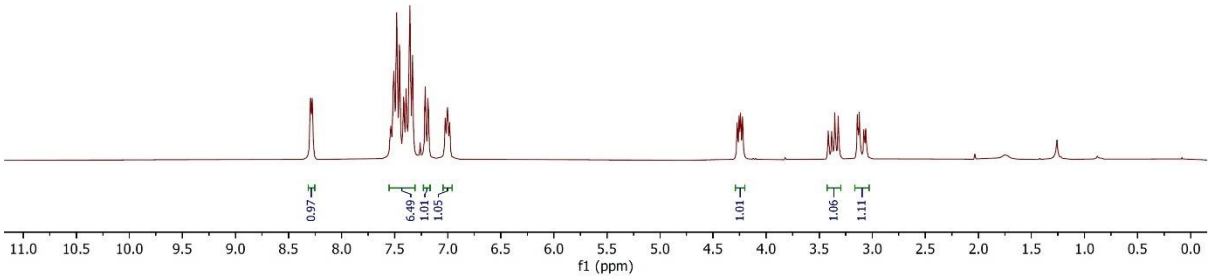
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-185

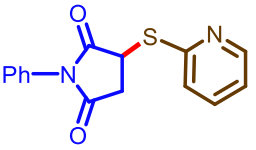


3h

^{1}H NMR (300 MHz, $CDCl_3$)

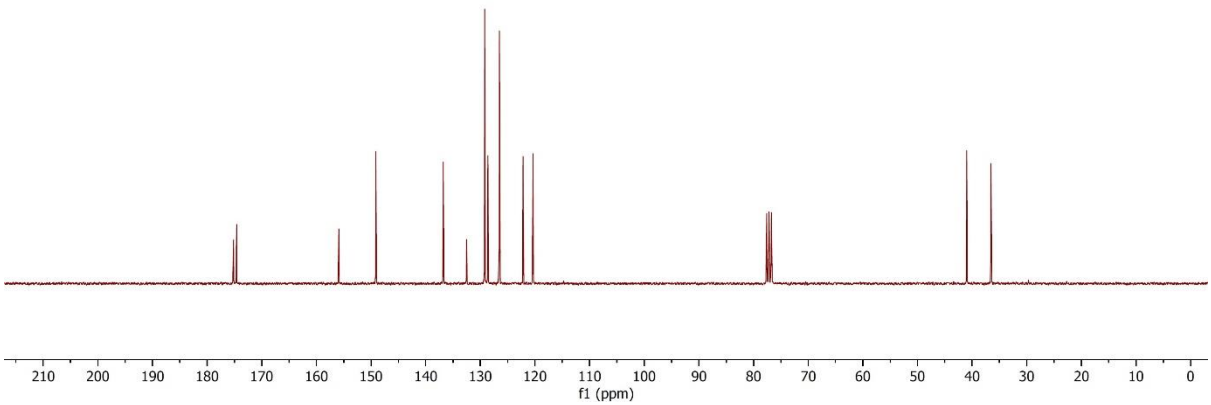


DKM-DJB-185-13C

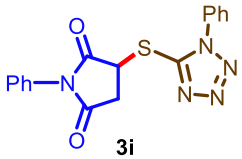


3h

^{13}C NMR (75 MHz, $CDCl_3$)

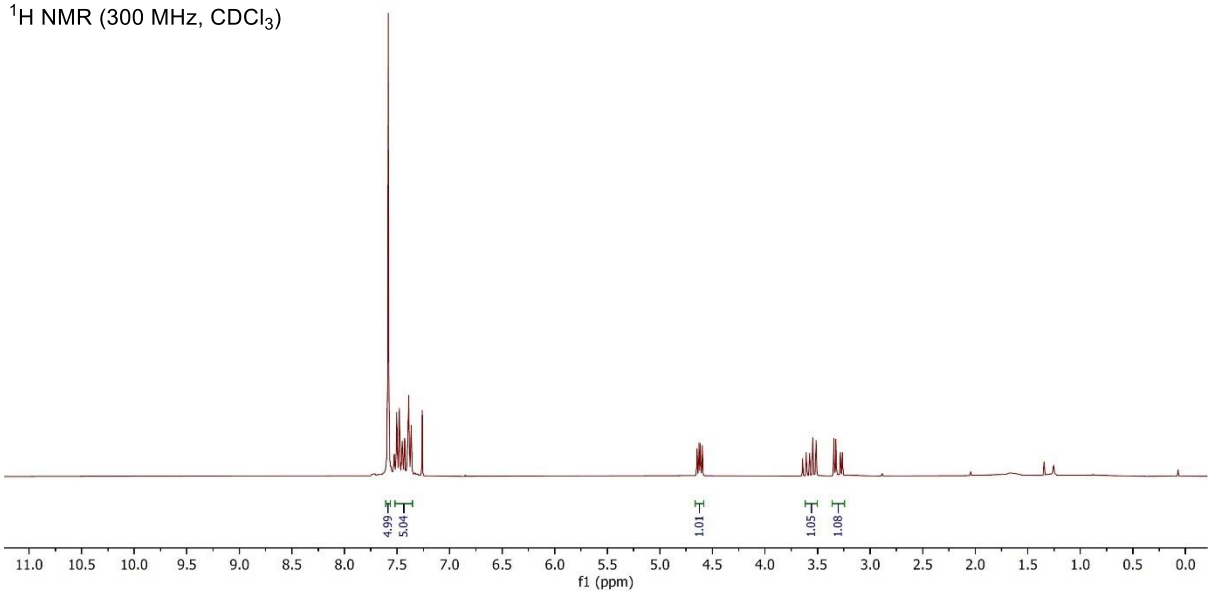


DKM-DJB-169LS

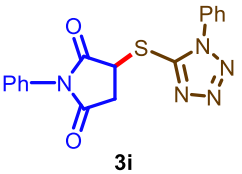


3i

^{1}H NMR (300 MHz, $CDCl_3$)



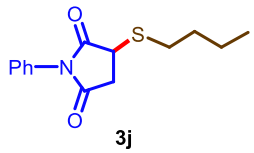
DKM-DJB-169LS-13C



3i

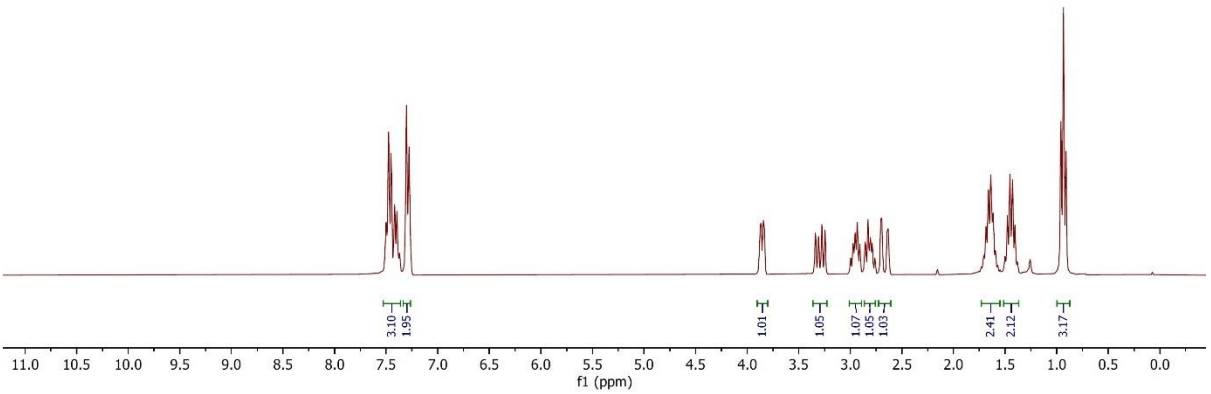
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-136



3j

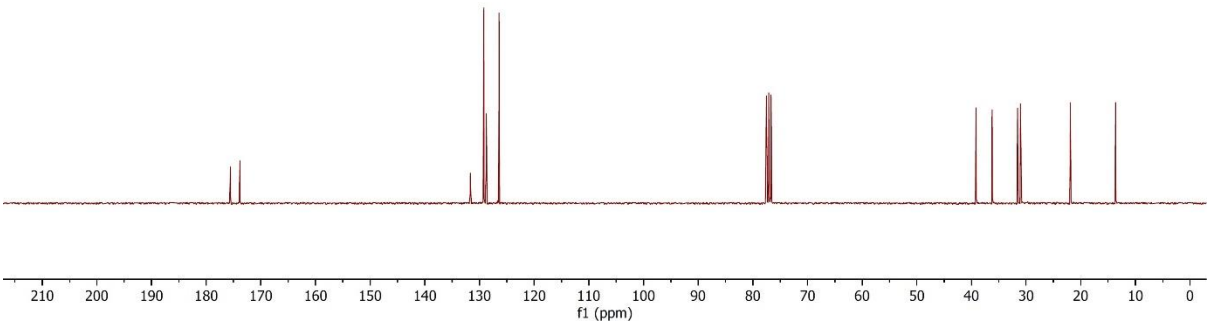
^{1}H NMR (300 MHz, $CDCl_3$)



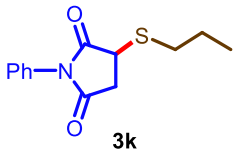
DKM-DJB-136-13C

3j

^{13}C NMR (75 MHz, $CDCl_3$)

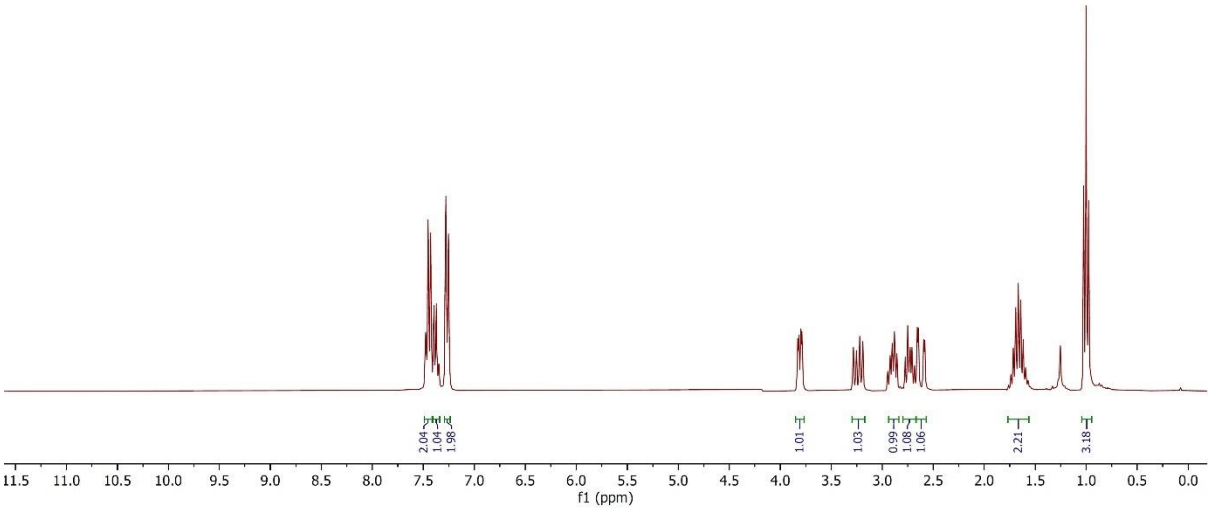


DKM-DJB-343
1H CDCl3 DKM-DJB-343 30/08/2024

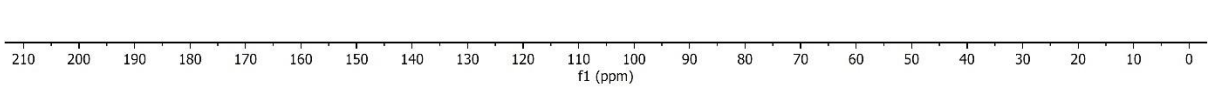


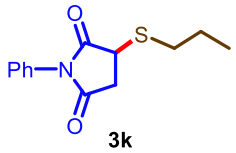
3k

^{1}H NMR (300 MHz, $CDCl_3$)



DKM-DJB-343-13C.1.fid

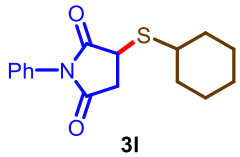




3k

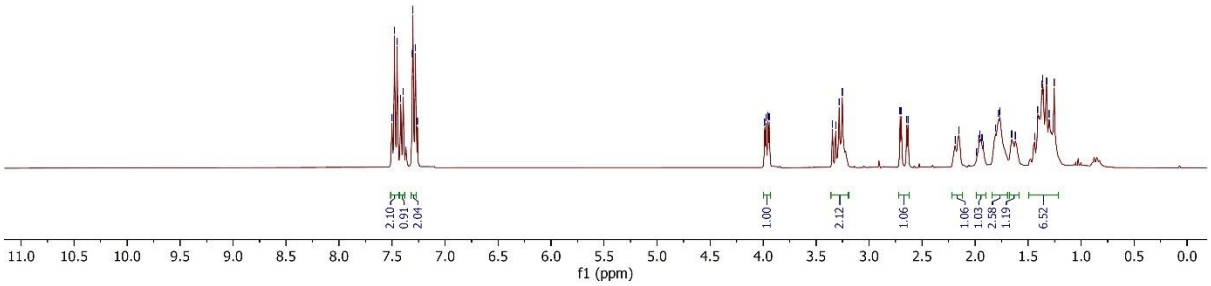
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-344.1.fid

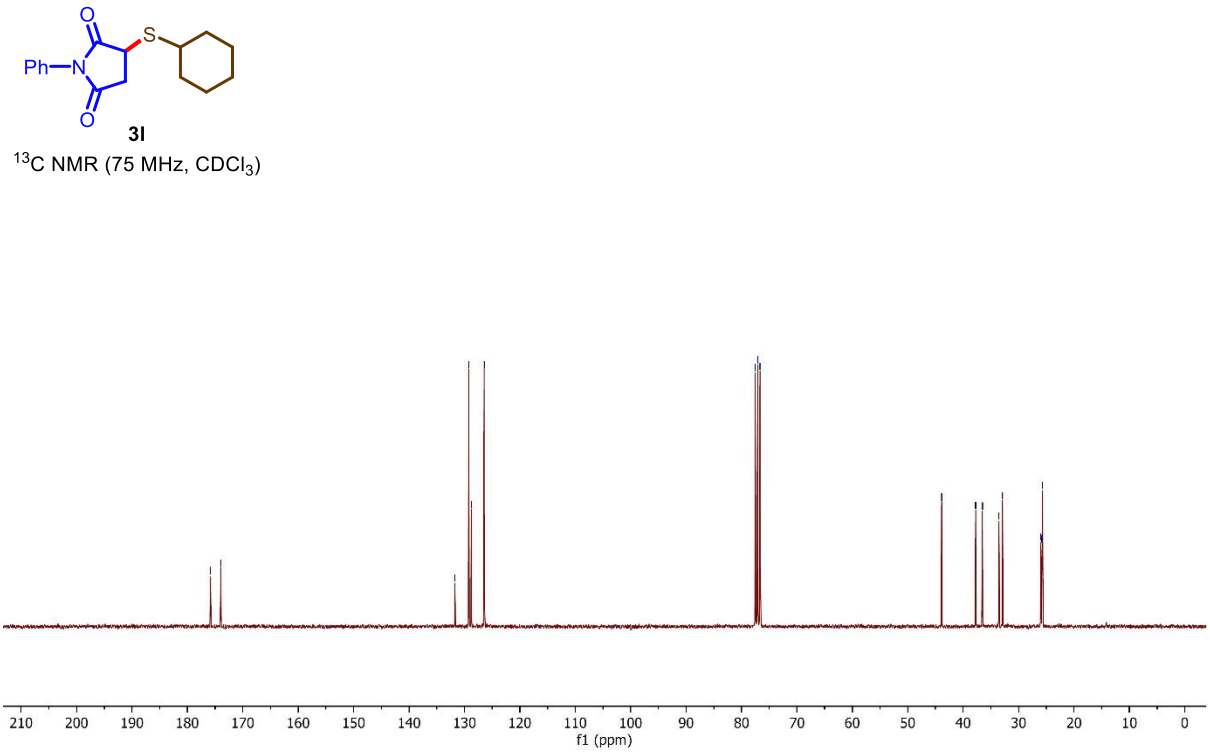


3l

^{1}H NMR (300 MHz, $CDCl_3$)



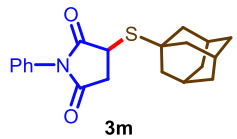
DKM-DJB-344-13C.2.fid



3l

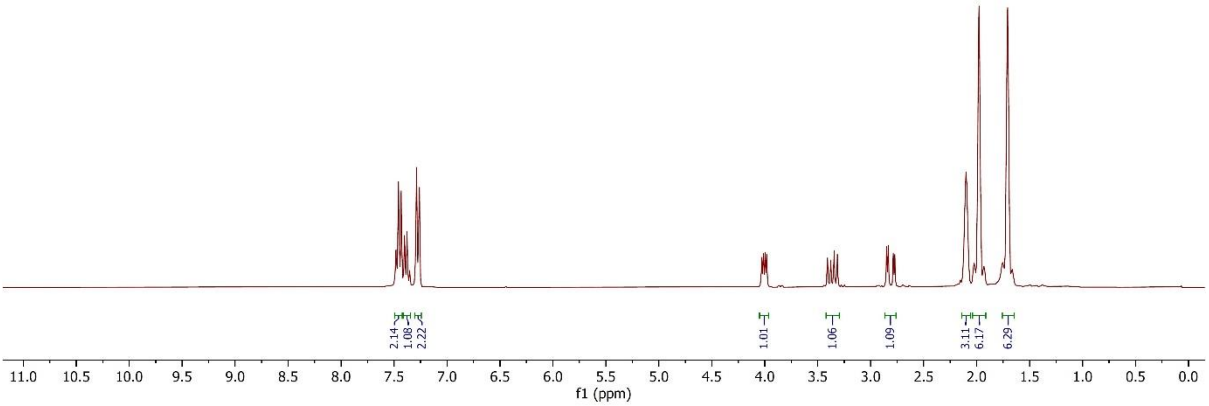
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-345
1H CDCl3 DKM-DJB-345 30/08/2024

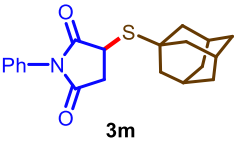


3m

^{1}H NMR (300 MHz, $CDCl_3$)

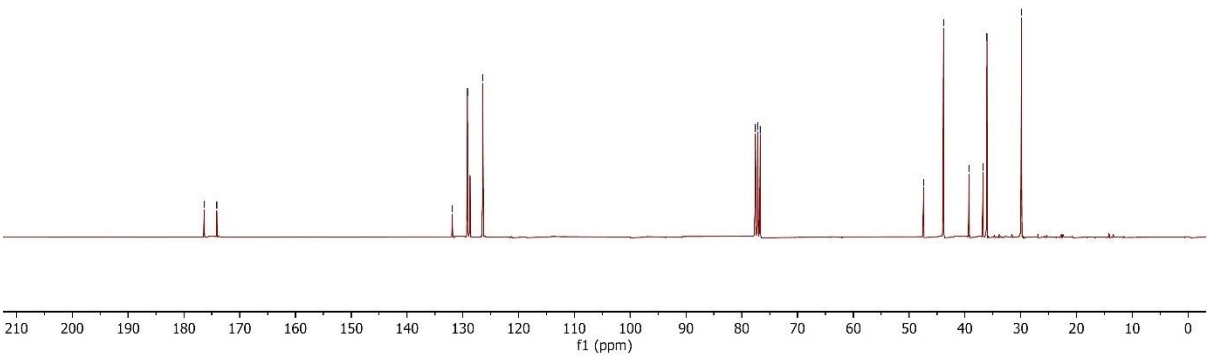


DKM-DJB-345-13C.1.fid

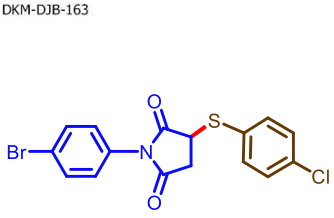


3m

^{13}C NMR (75 MHz, $CDCl_3$)

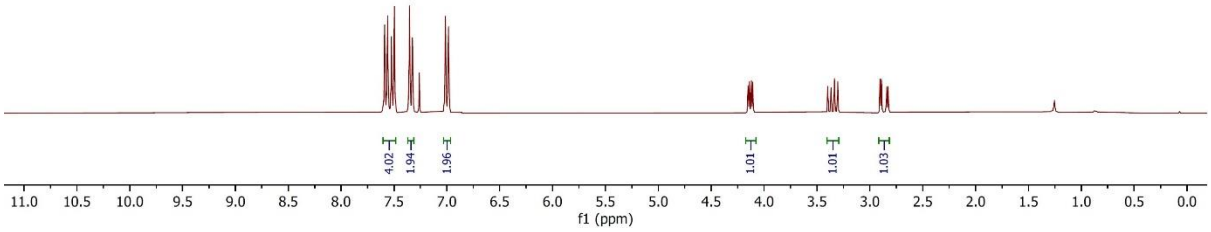


DKM-DJB-163

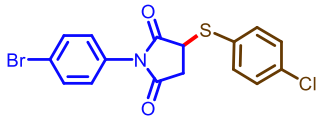


3n

^{1}H NMR (300 MHz, $CDCl_3$)

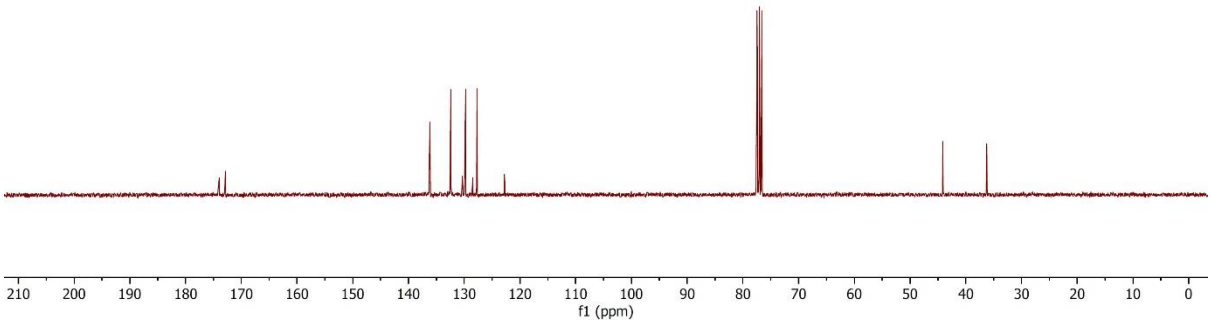


DKM-DJB-163-13C

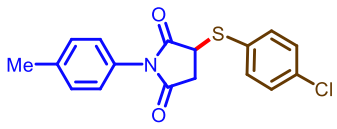


3n

^{13}C NMR (75 MHz, $CDCl_3$)

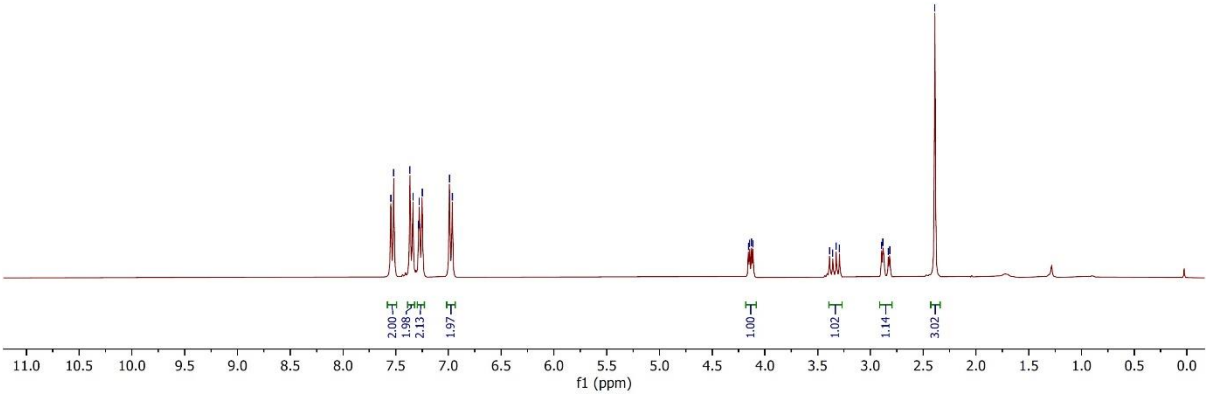


DKM-DJB-216.1.fid

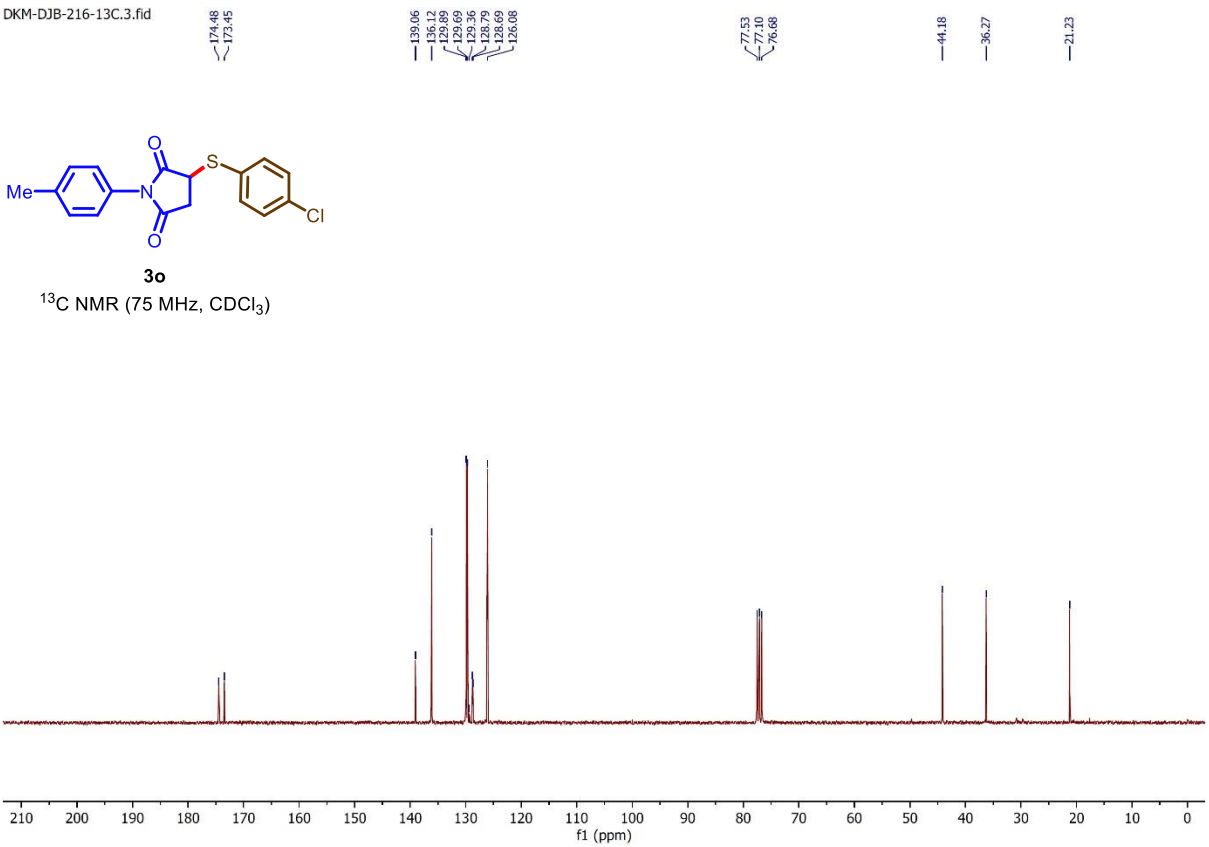


3o

^{1}H NMR (300 MHz, $CDCl_3$)



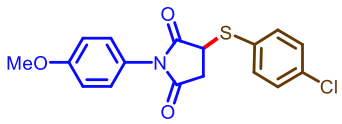
DKM-DJB-216-13C.3.fid



3o

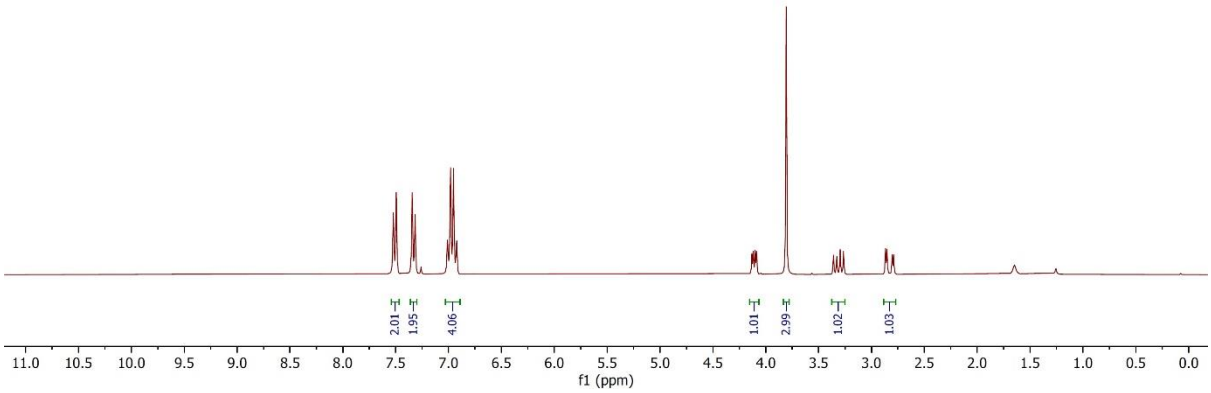
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-181

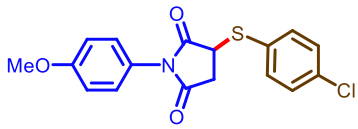


3p

^{1}H NMR (300 MHz, $CDCl_3$)



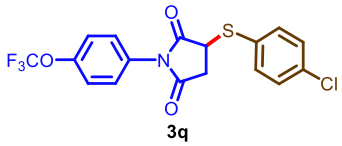
DKM-DJB-181-13C



3p

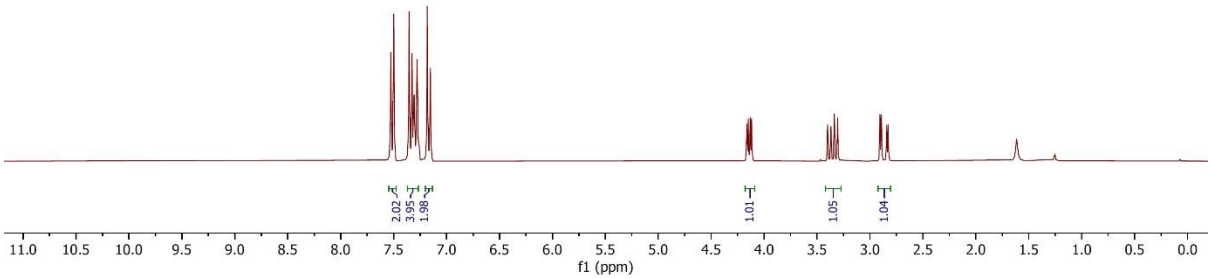
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-182

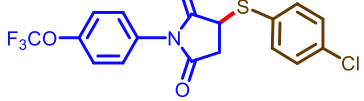


3q

^{1}H NMR (300 MHz, $CDCl_3$)

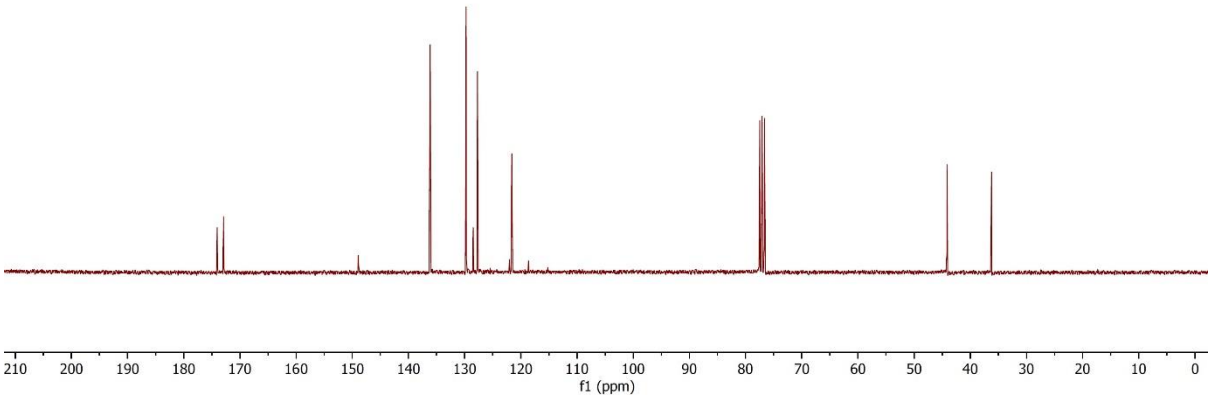


DKM-DJB-182-13C

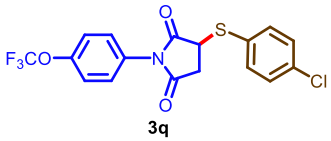


3q

^{13}C NMR (75 MHz, $CDCl_3$)

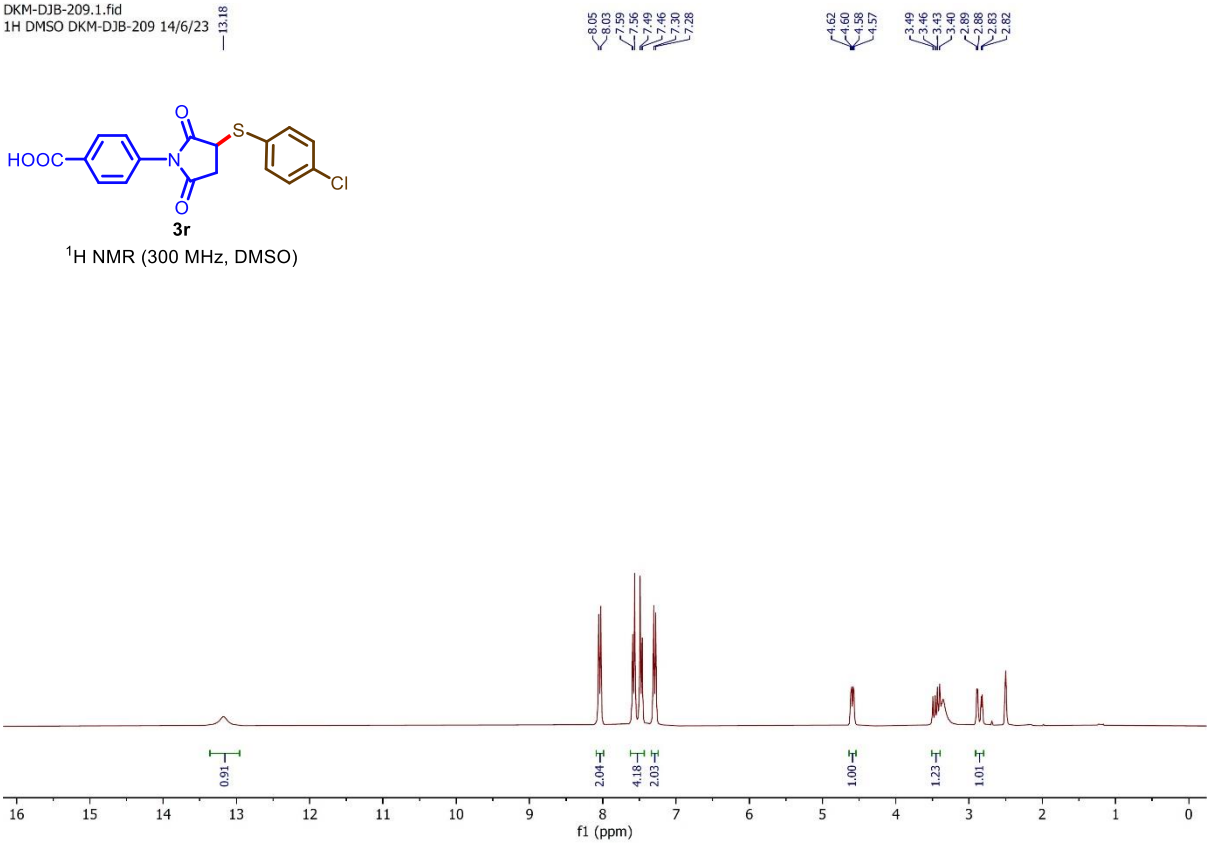


DKM-DJB-182-19F.1.fid
19F CDCl3 DKM-DJB-182-19F 06/11/23



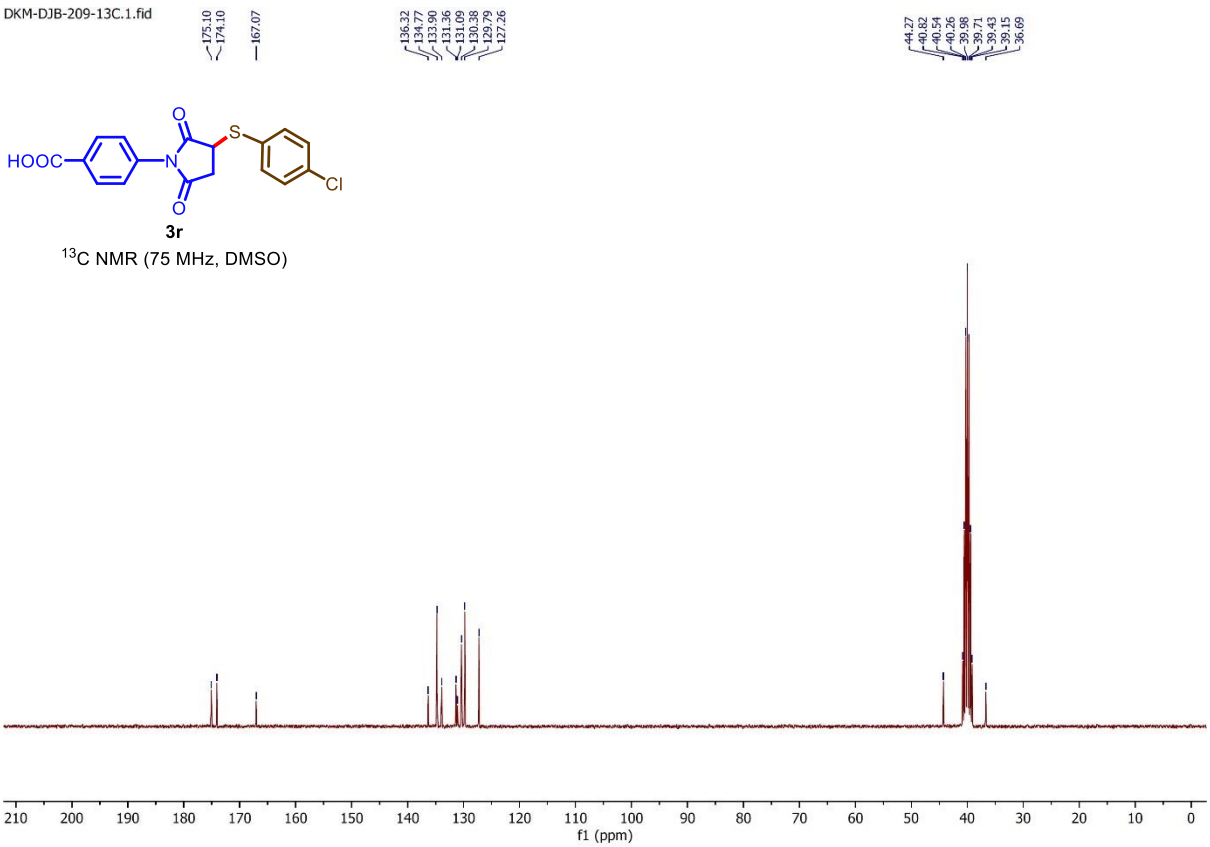
3q

^{19}F NMR (377 MHz, $CDCl_3$)



3r

^{1}H NMR (300 MHz, DMSO)



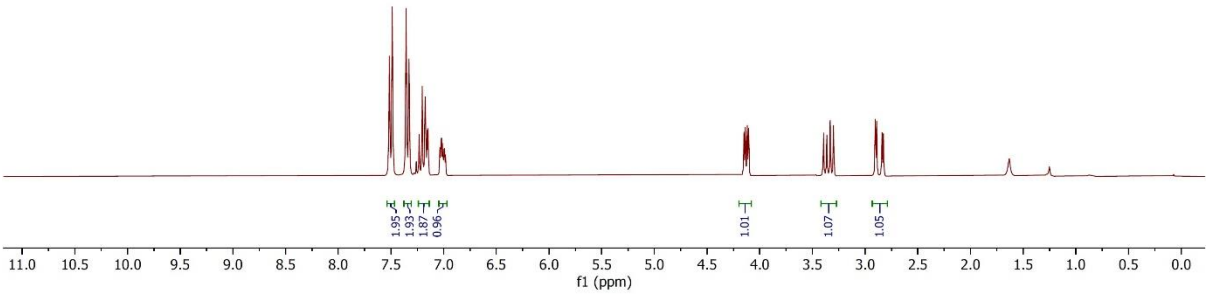
3r

^{13}C NMR (75 MHz, DMSO)

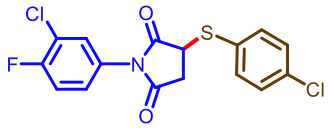
DKM-DJB-184

3s

^{1}H NMR (300 MHz, $CDCl_3$)

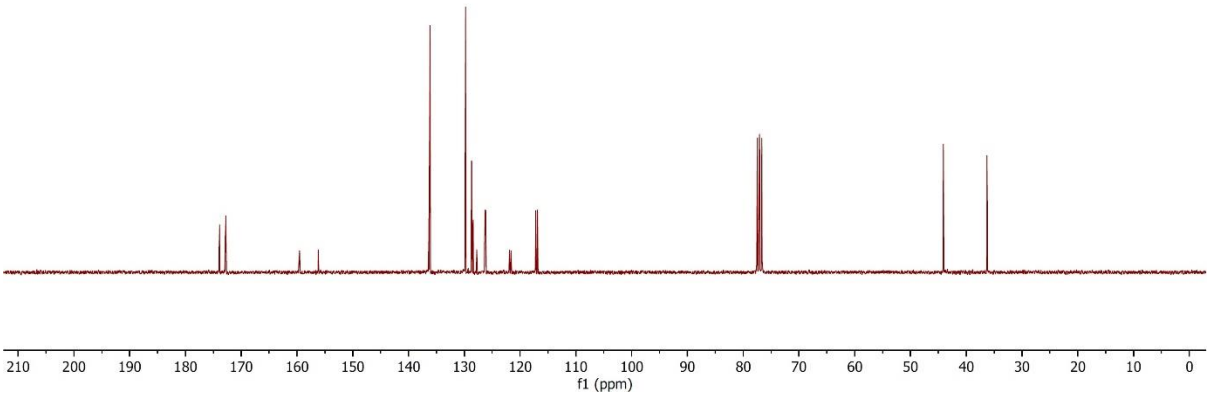


DKM-DJB-184-13C

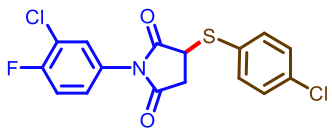


3s

^{13}C NMR (75 MHz, $CDCl_3$)



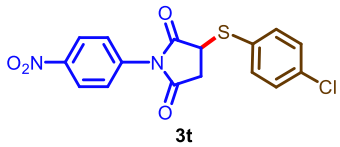
DKM-DJB-184 19F.12.fid
SPD-1-19F



3s

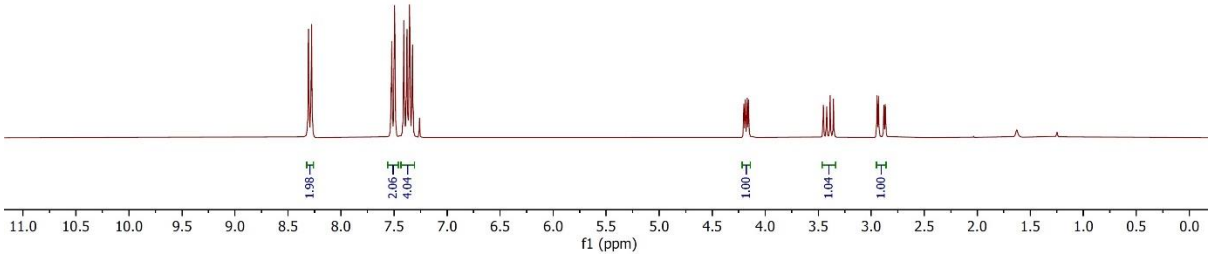
^{19}F NMR (377 MHz, $CDCl_3$)

DKM-DJB-172

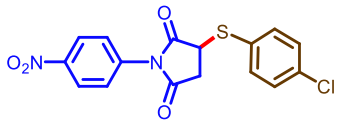


3t

^{1}H NMR (300 MHz, $CDCl_3$)

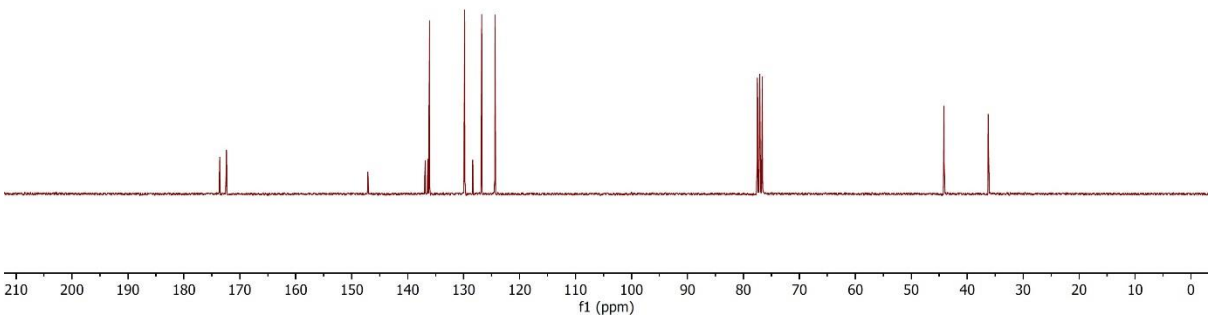


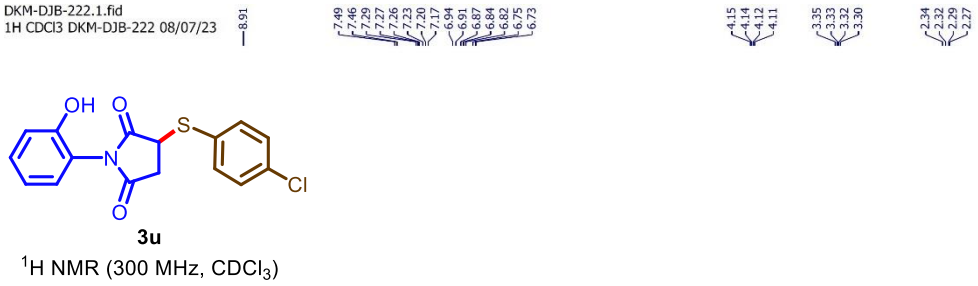
DKM-DJB-172-13C



3t

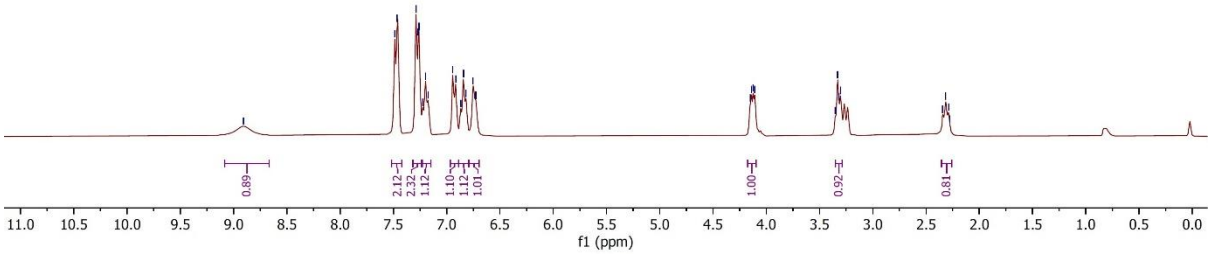
^{13}C NMR (75 MHz, $CDCl_3$)

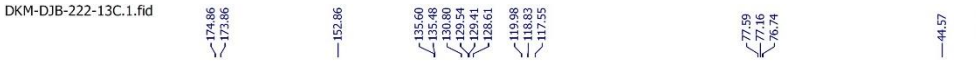


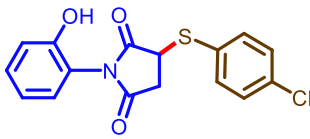


3u

^{1}H NMR (300 MHz, $CDCl_3$)

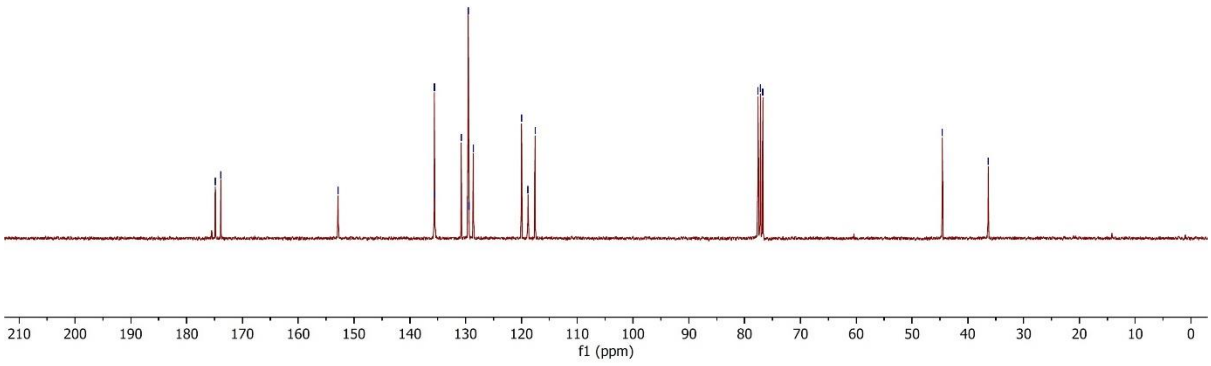


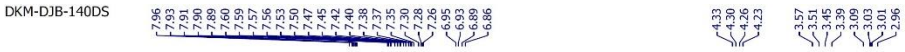


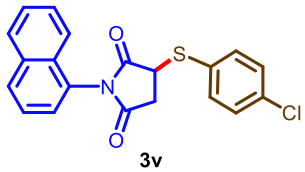


3u

^{13}C NMR (75 MHz, $CDCl_3$)

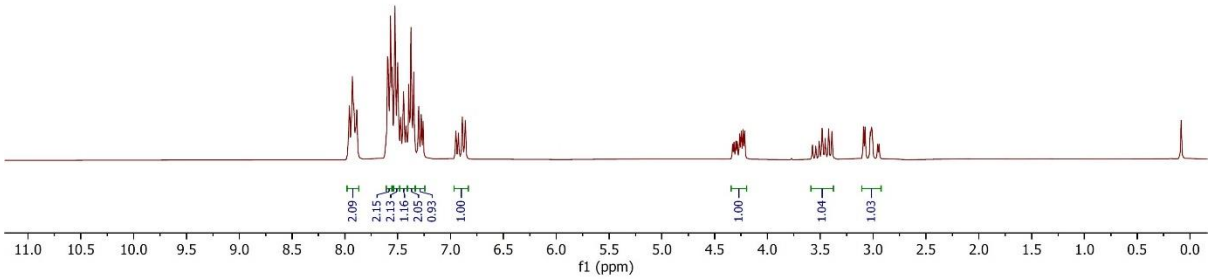


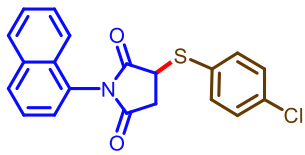




3v

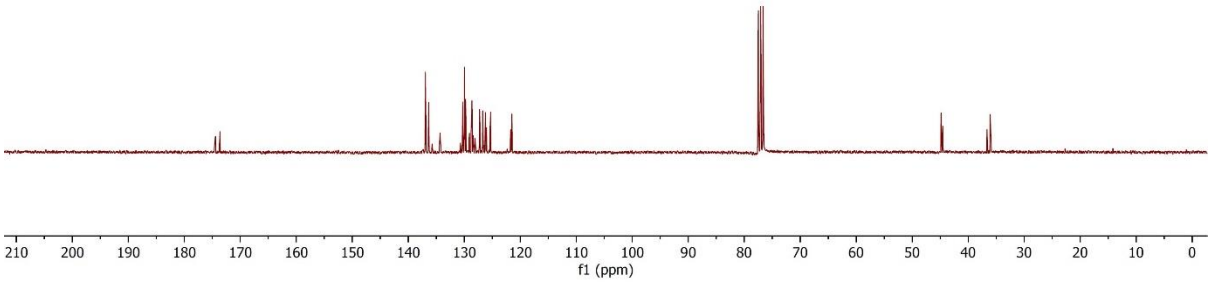
^{1}H NMR (300 MHz, $CDCl_3$)



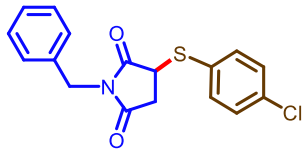


3v

^{13}C NMR (75 MHz, $CDCl_3$)

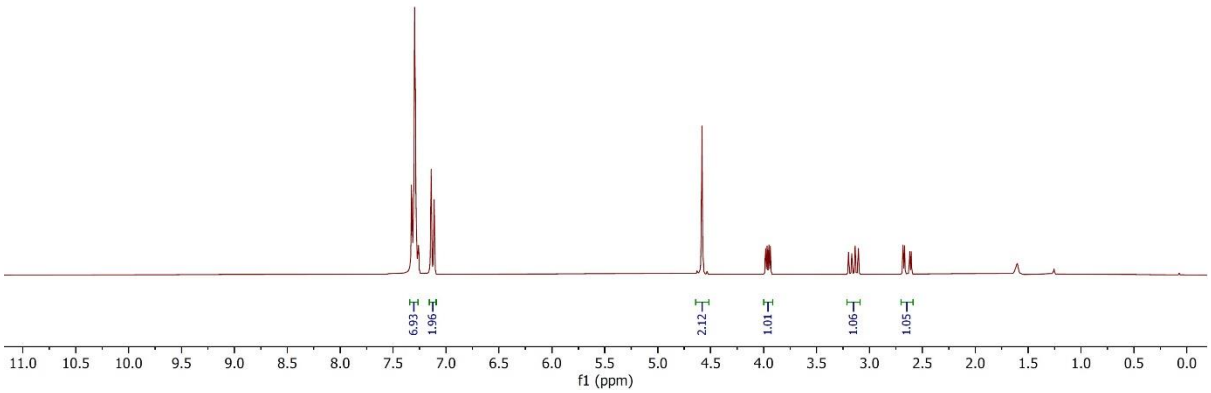


DKM-DJB-139

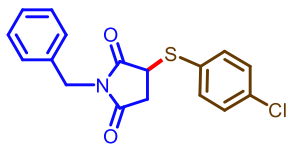


3w

^{1}H NMR (300 MHz, $CDCl_3$)

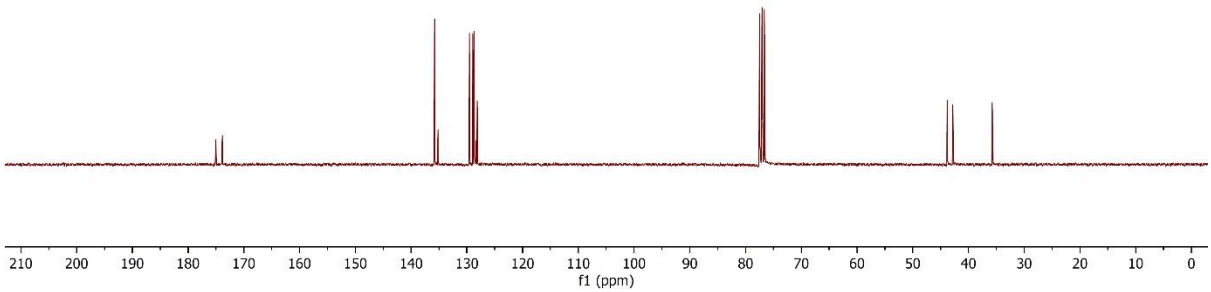


DKM-DJB-139-13C

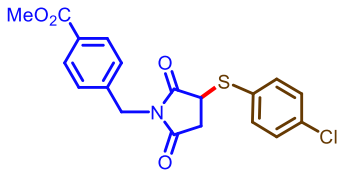


3w

^{13}C NMR (75 MHz, $CDCl_3$)

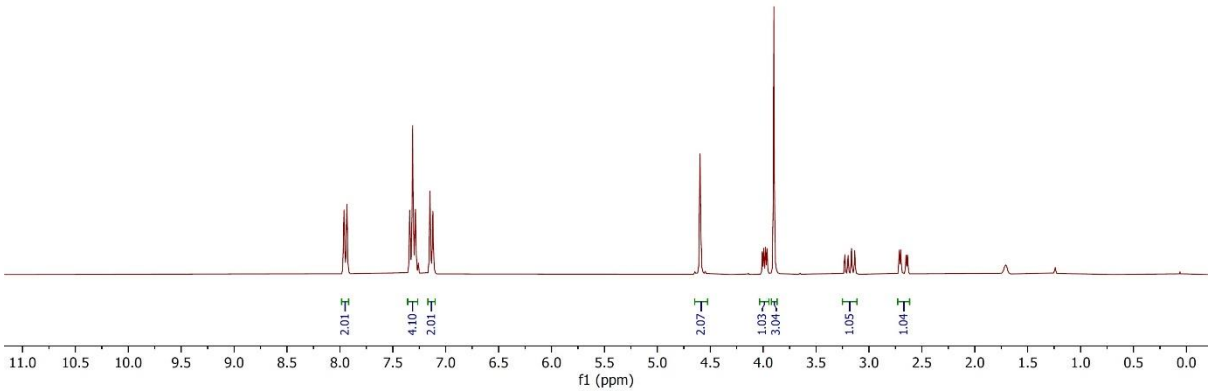


DKM-DJB-183

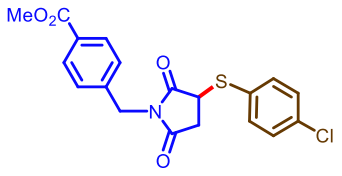


3x

^{1}H NMR (300 MHz, $CDCl_3$)

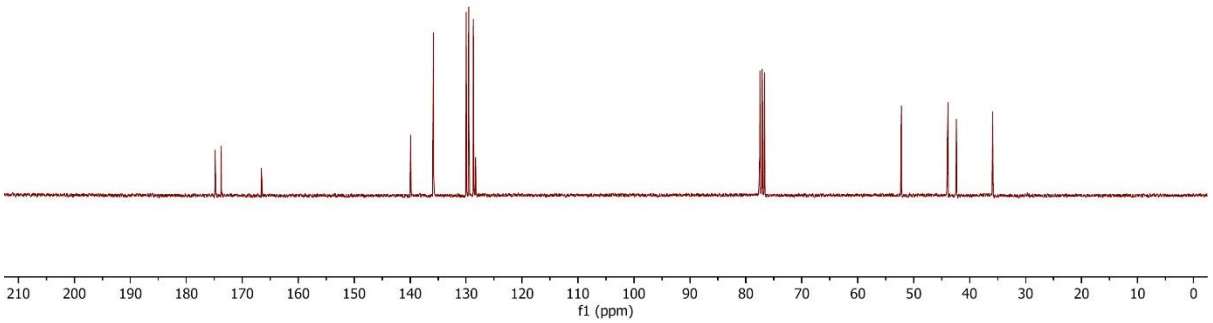


DKM-DJB-183-13C

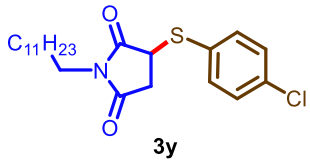


3x

^{13}C NMR (75 MHz, $CDCl_3$)



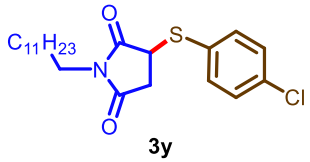
DKM-DJB-151



3y

^{1}H NMR (300 MHz, $CDCl_3$)

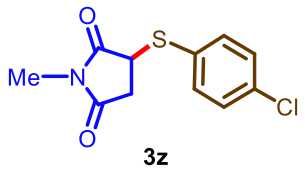
DKM-DJB-151-13C



3y

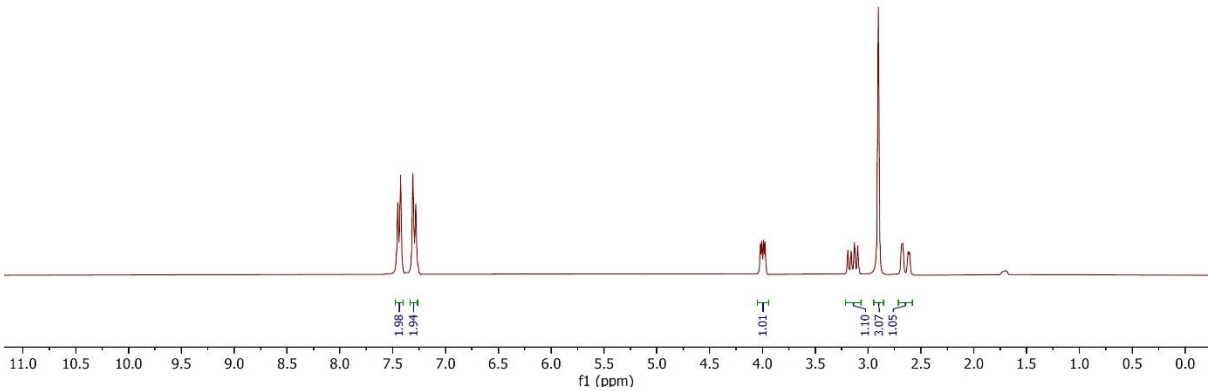
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-127

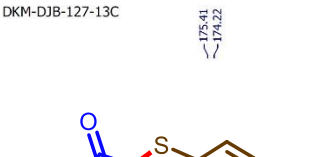


3z

^{1}H NMR (300 MHz, $CDCl_3$)



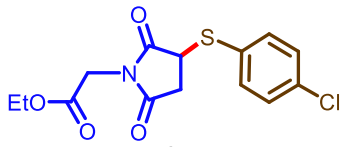
DKM-DJB-127-13C



3z

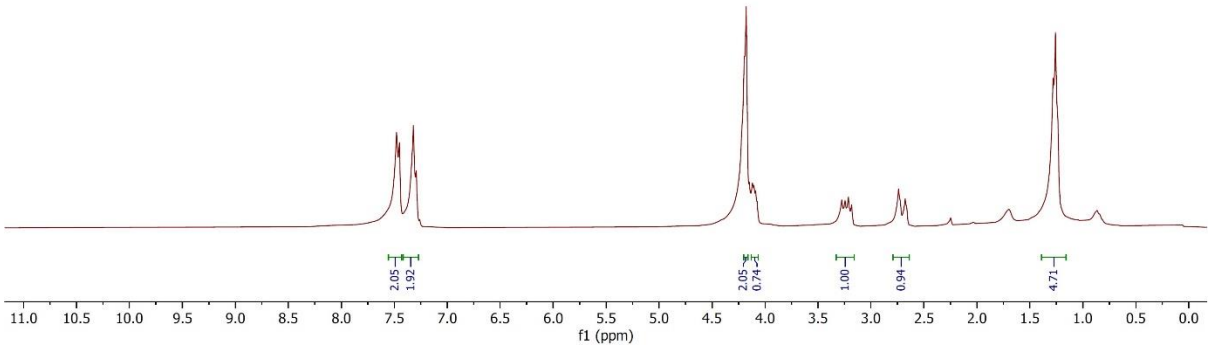
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-155

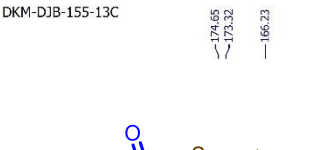


3aa

^{1}H NMR (300 MHz, $CDCl_3$)

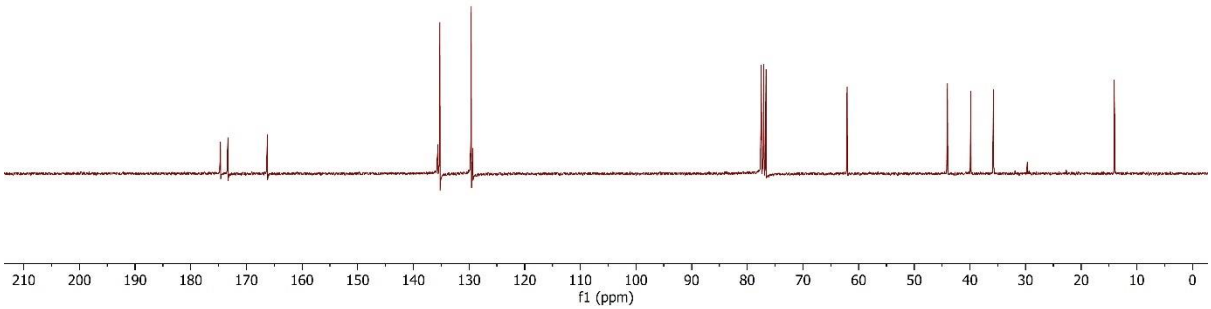


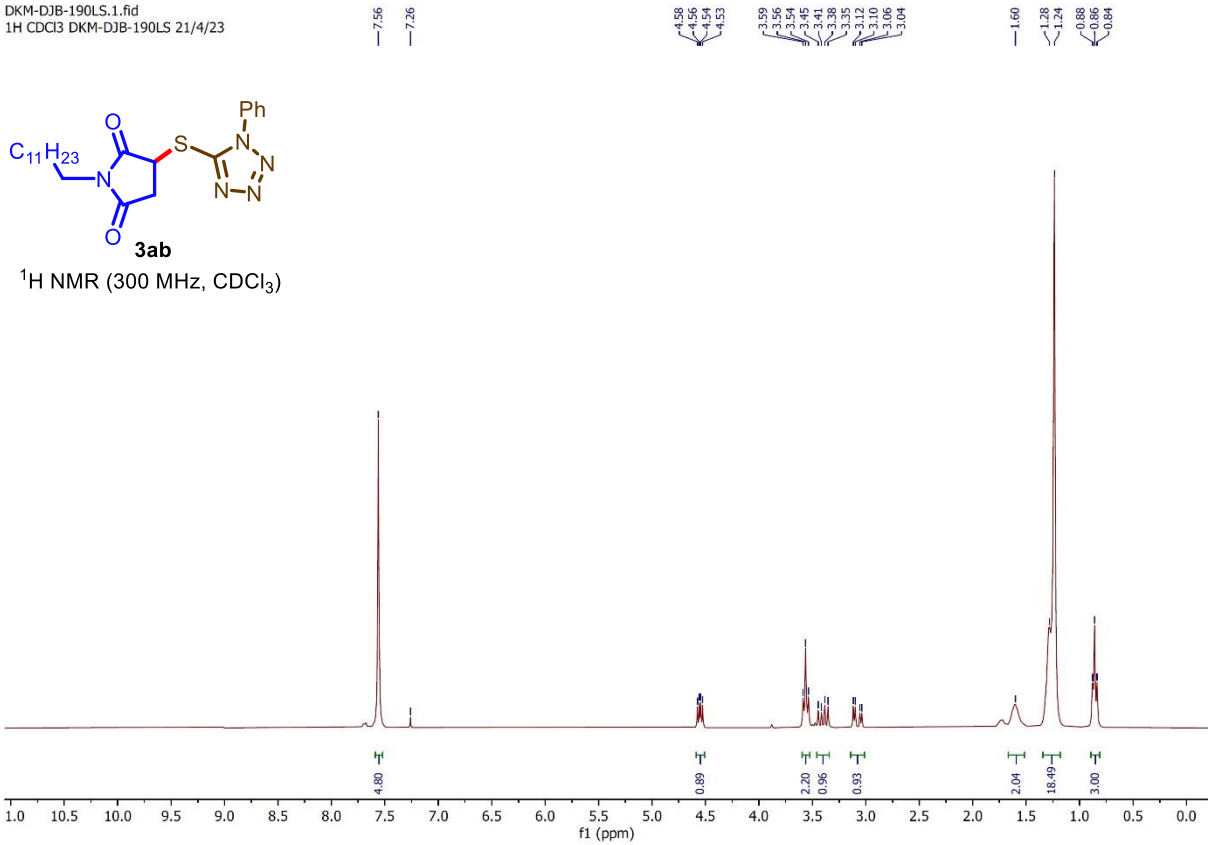
DKM-DJB-155-13C



3aa

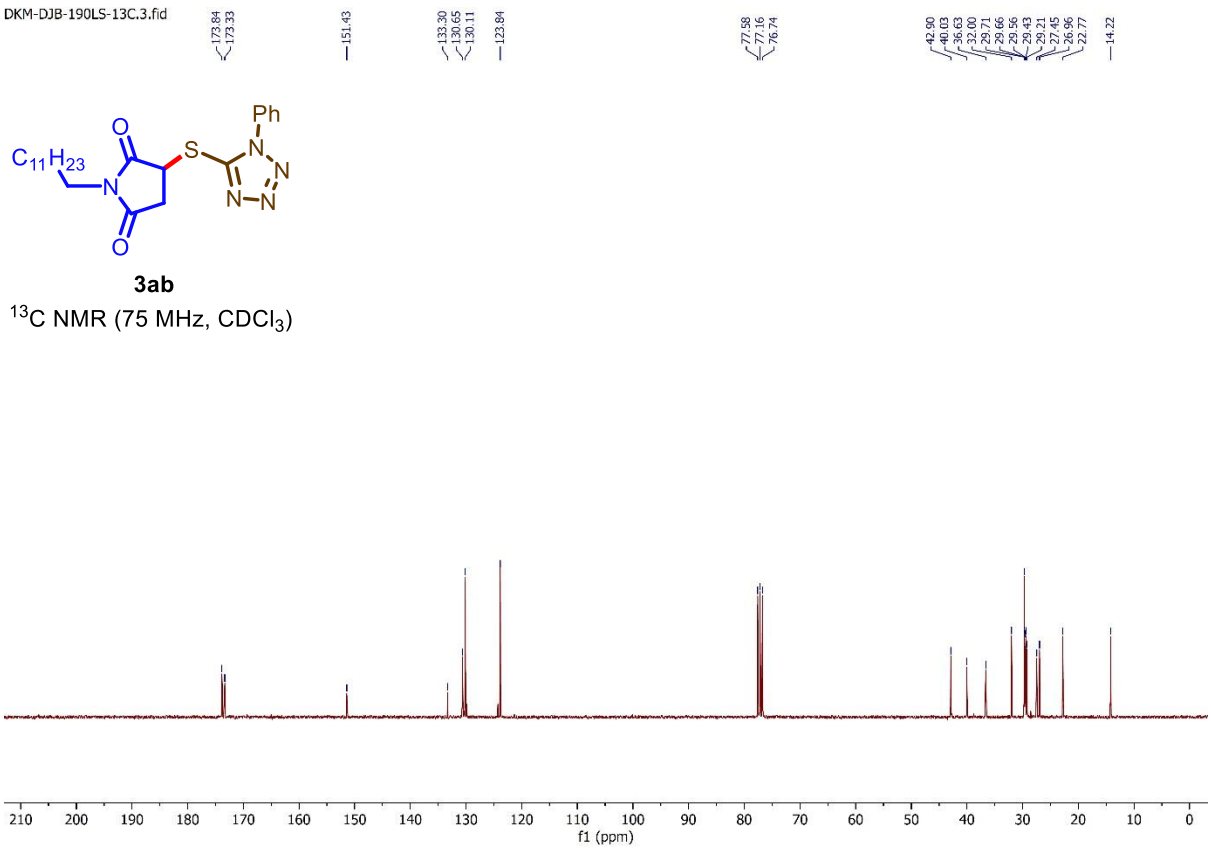
^{13}C NMR (75 MHz, $CDCl_3$)





3ab

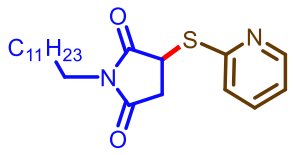
^{1}H NMR (300 MHz, $CDCl_3$)



3ab

^{13}C NMR (75 MHz, $CDCl_3$)

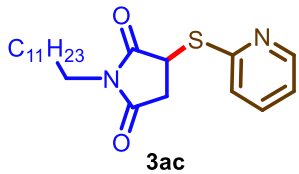
DKM-DJB-189



3ac

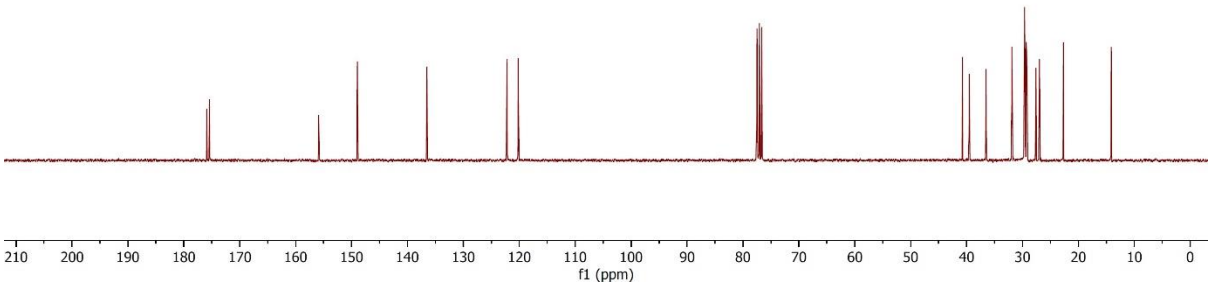
^{1}H NMR (300 MHz, $CDCl_3$)

DKM-DJB-189-13C

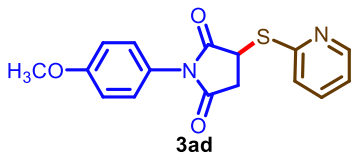


3ac

^{13}C NMR (75 MHz, $CDCl_3$)

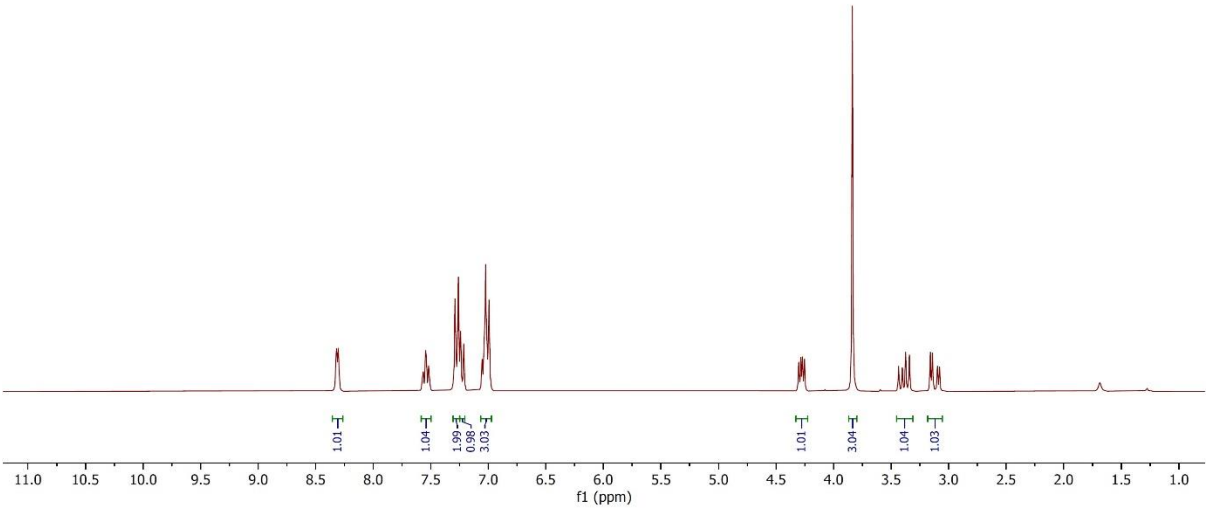


DKM-DJB-191

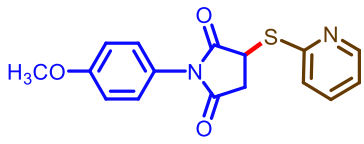


3ad

^{1}H NMR (300 MHz, $CDCl_3$)

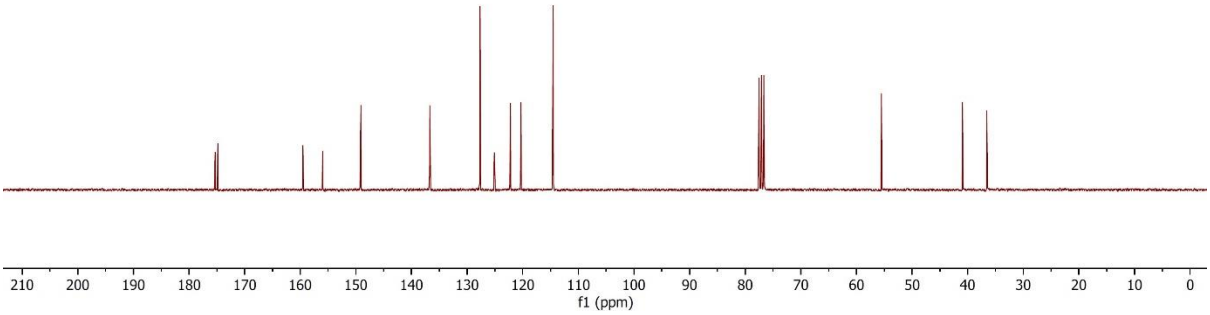


DKM-DJB-191-13C

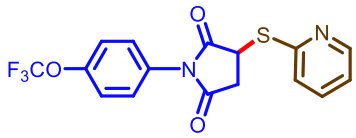


3ad

^{13}C NMR (75 MHz, $CDCl_3$)

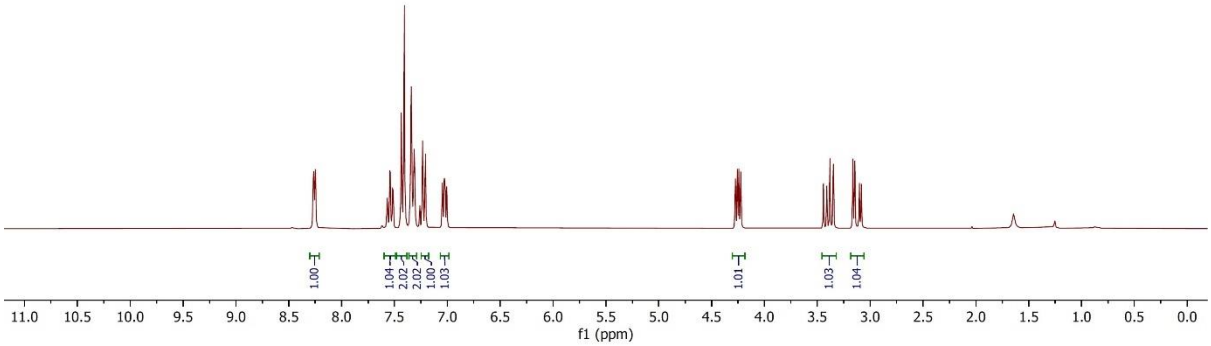


DKM-DJB-192

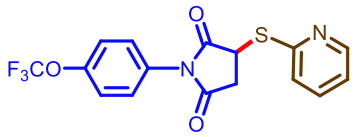


3ae

^{1}H NMR (300 MHz, $CDCl_3$)

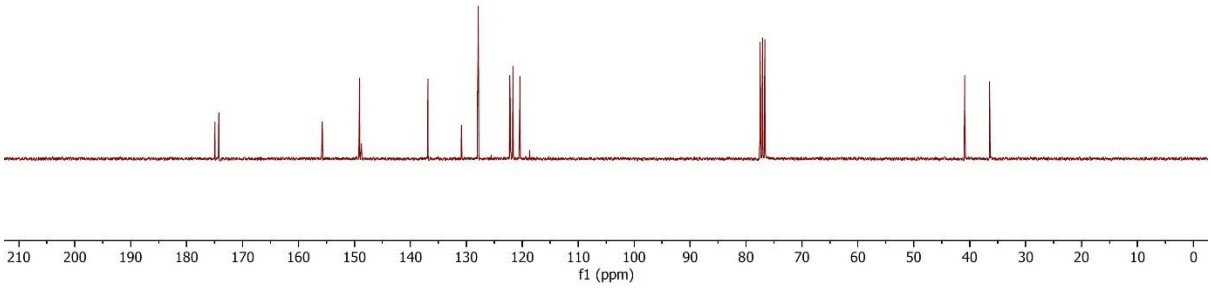


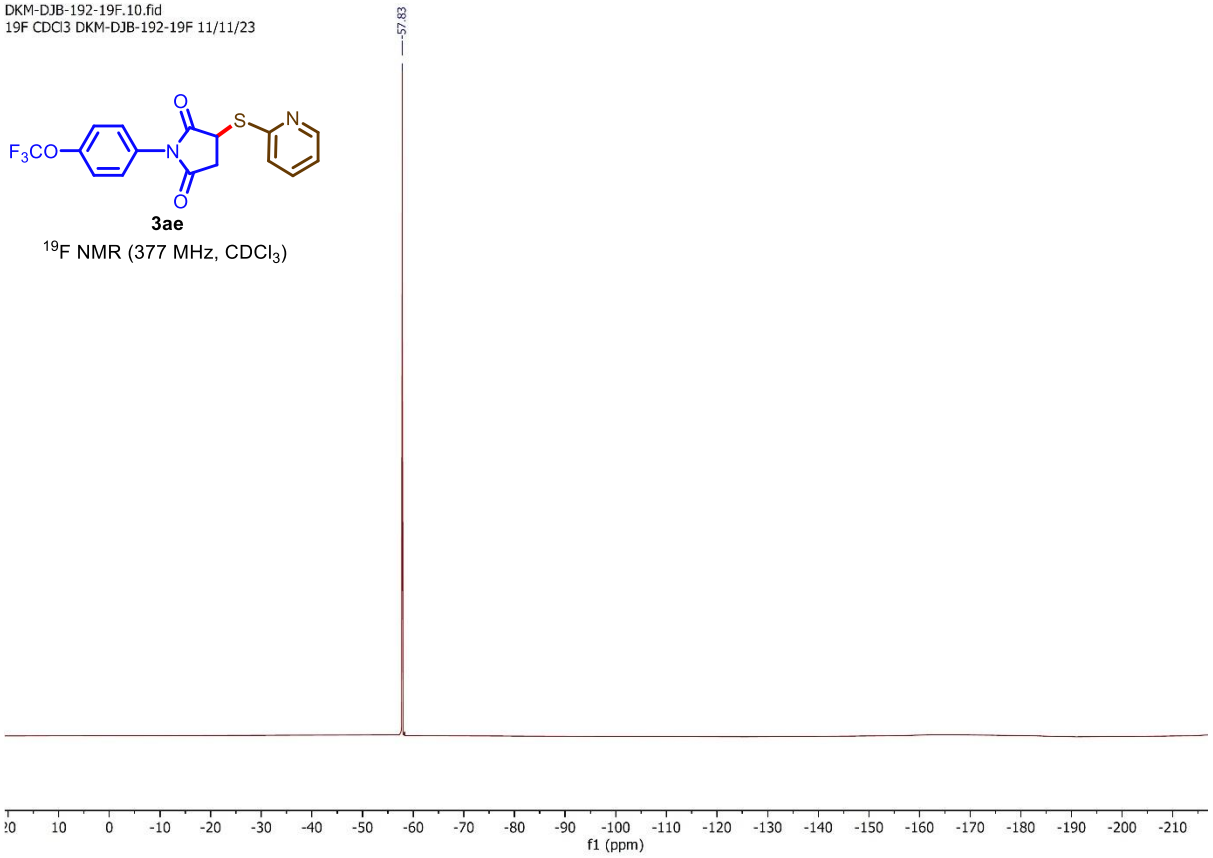
DKM-DJB-192-13C

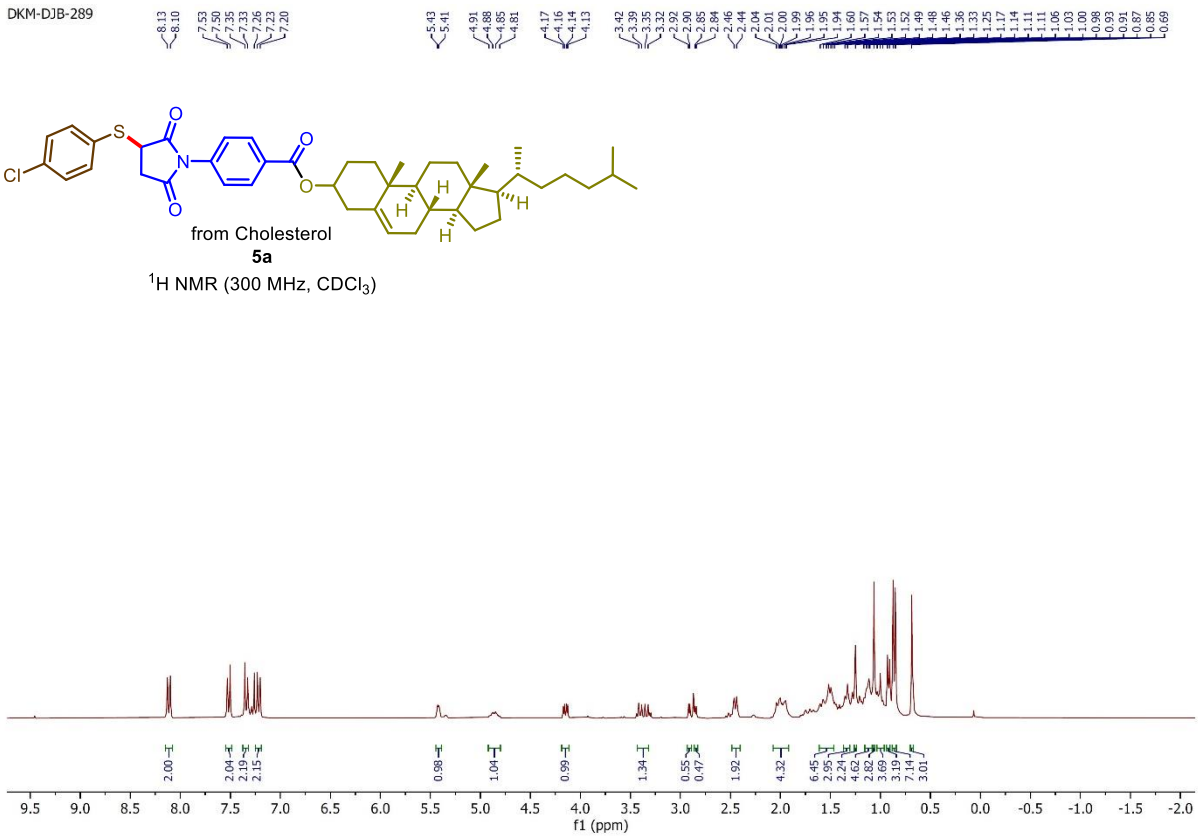


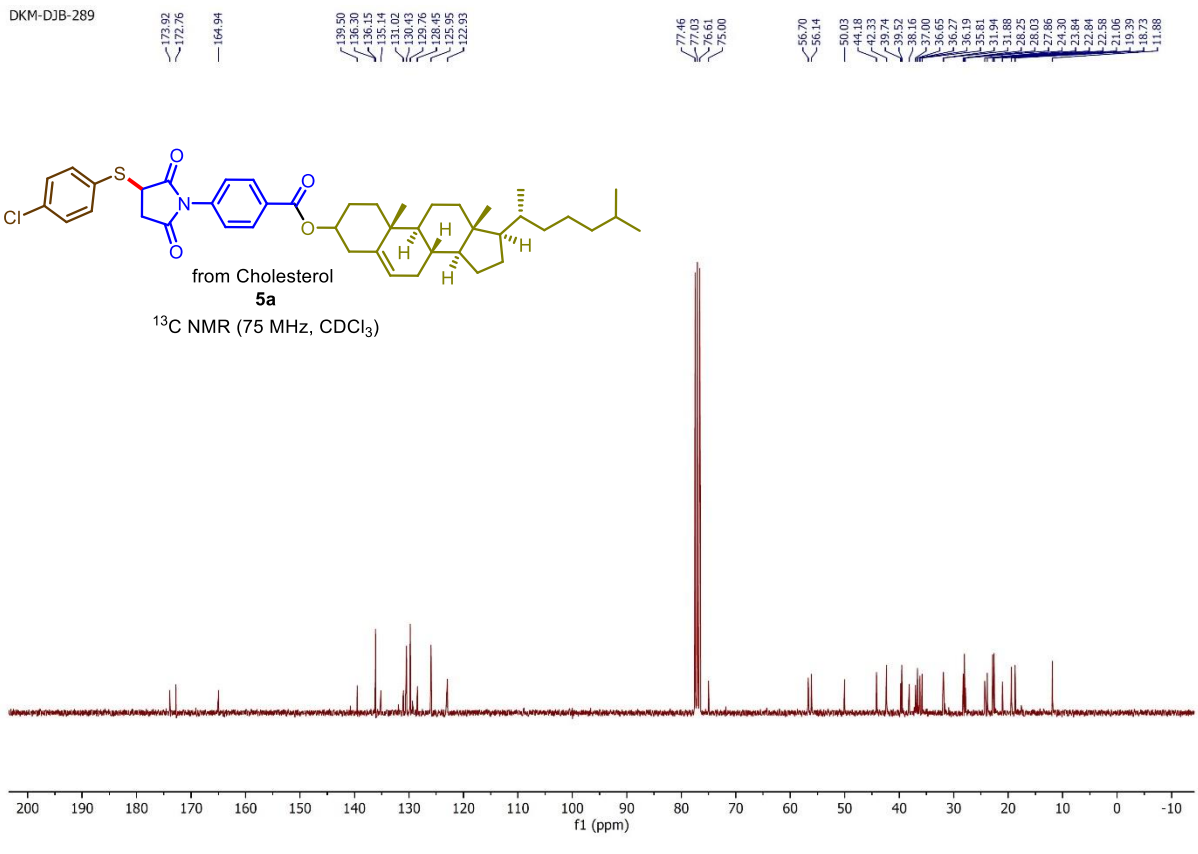
3ae

^{13}C NMR (75 MHz, $CDCl_3$)

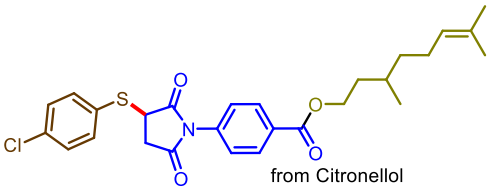



DKM-DJB-192-19F.10.fid
19F CDCl3 DKM-DJB-192-19F 11/11/23
-57.83
F3CO
O
N
S
N
O
3ae
19F NMR (377 MHz, CDCl3)
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)


DKM-DJB-289
from Cholesterol
5a
1H NMR (300 MHz, CDCl3)
f1 (ppm)


DKM-DJB-289
from Cholesterol
5a
13C NMR (75 MHz, CDCl3)
f1 (ppm)

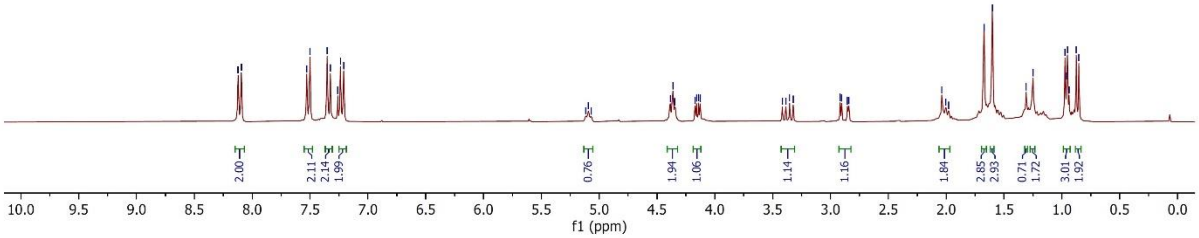
DKM-DJB-284R.1.fid



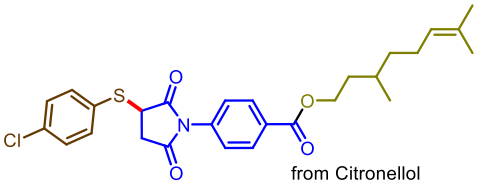
from Citronellol

5b

^{1}H NMR (300 MHz, $CDCl_3$)



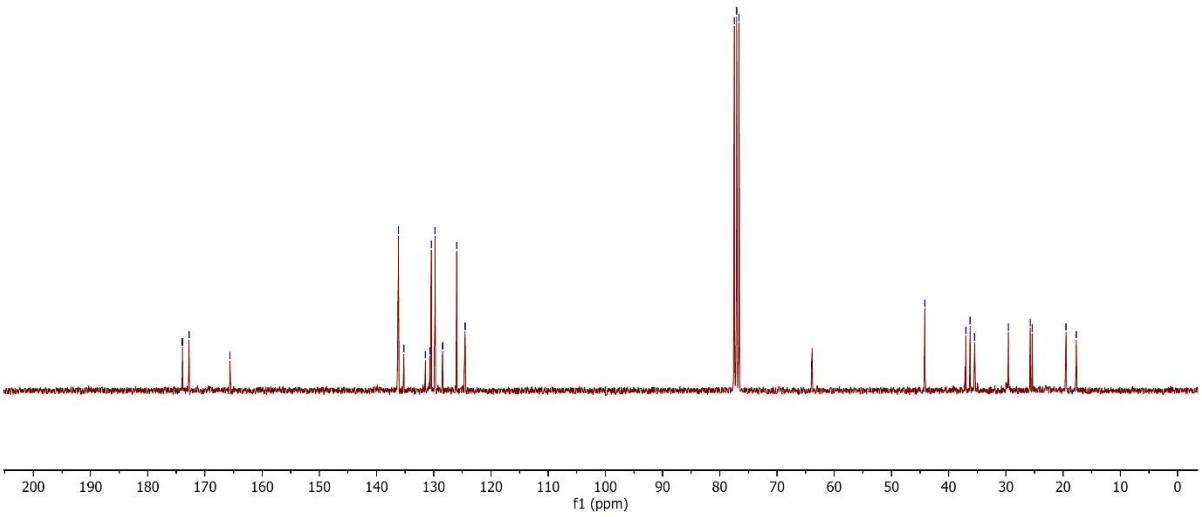
DKM-DJB-284R-13C.3.fid



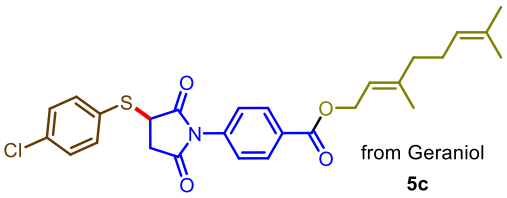
from Citronellol

5b

^{13}C NMR (75 MHz, $CDCl_3$)



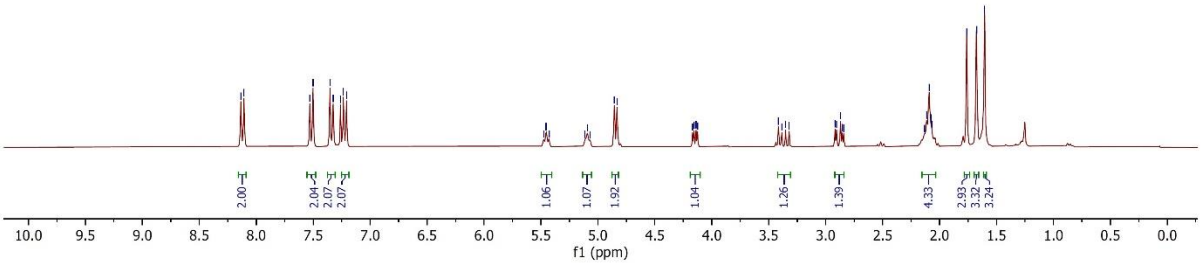
DKM-DJB-315.1.fid



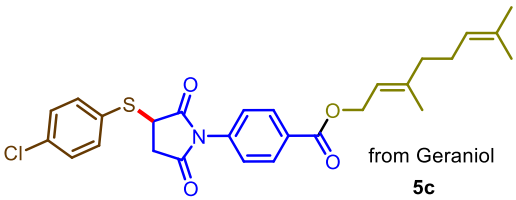
from Geraniol

5c

^{1}H NMR (300 MHz, $CDCl_3$)



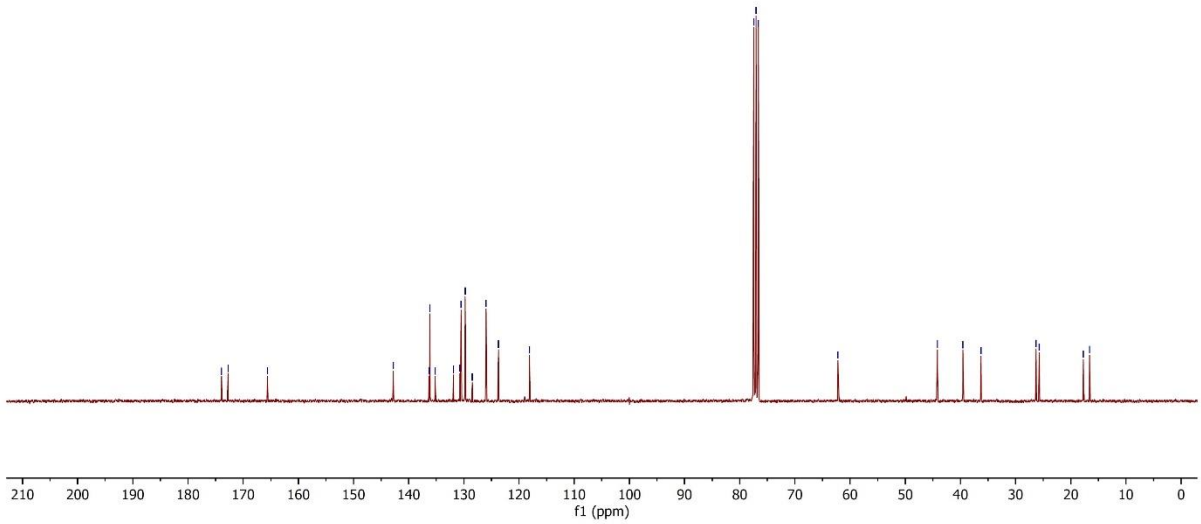
DKM-DJB-315.2.fid



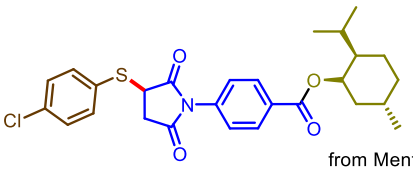
from Geraniol

5c

^{13}C NMR (75 MHz, $CDCl_3$)



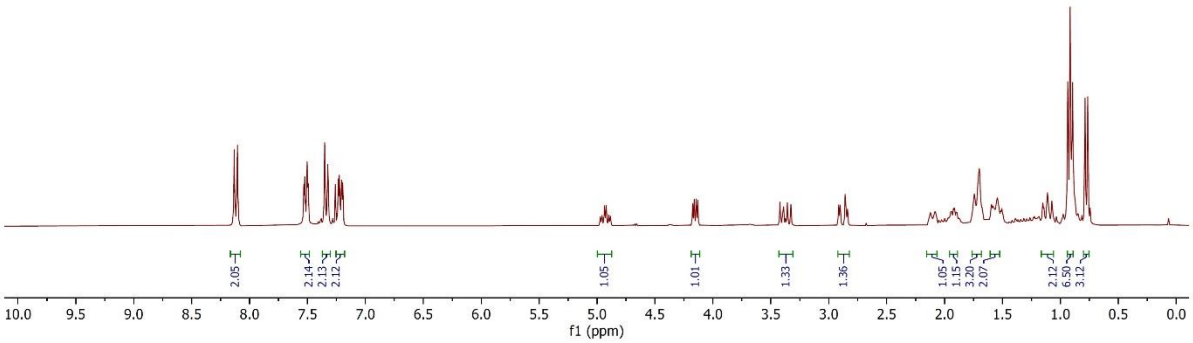
DKM-DJB-285



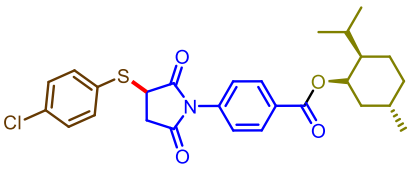
from Menthol

5d

^{1}H NMR (300 MHz, $CDCl_3$)



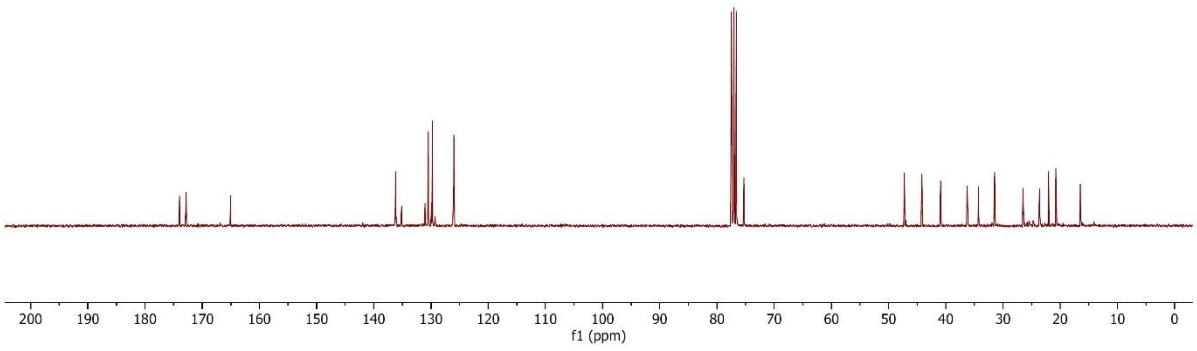
DKM-DJB-285-13C



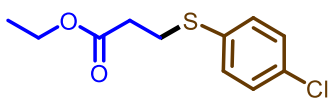
from Menthol

5d

^{13}C NMR (75 MHz, $CDCl_3$)

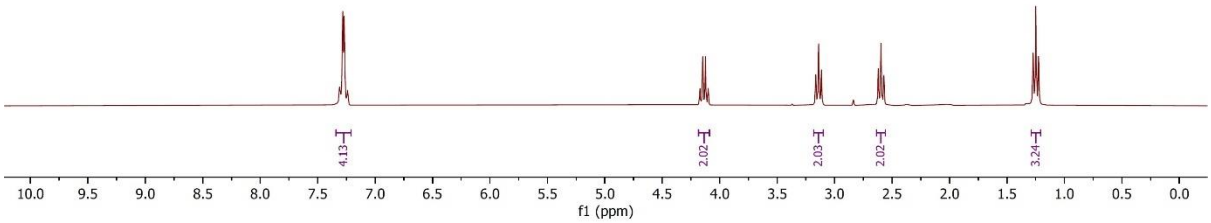


DKM-DJB-226US



7a

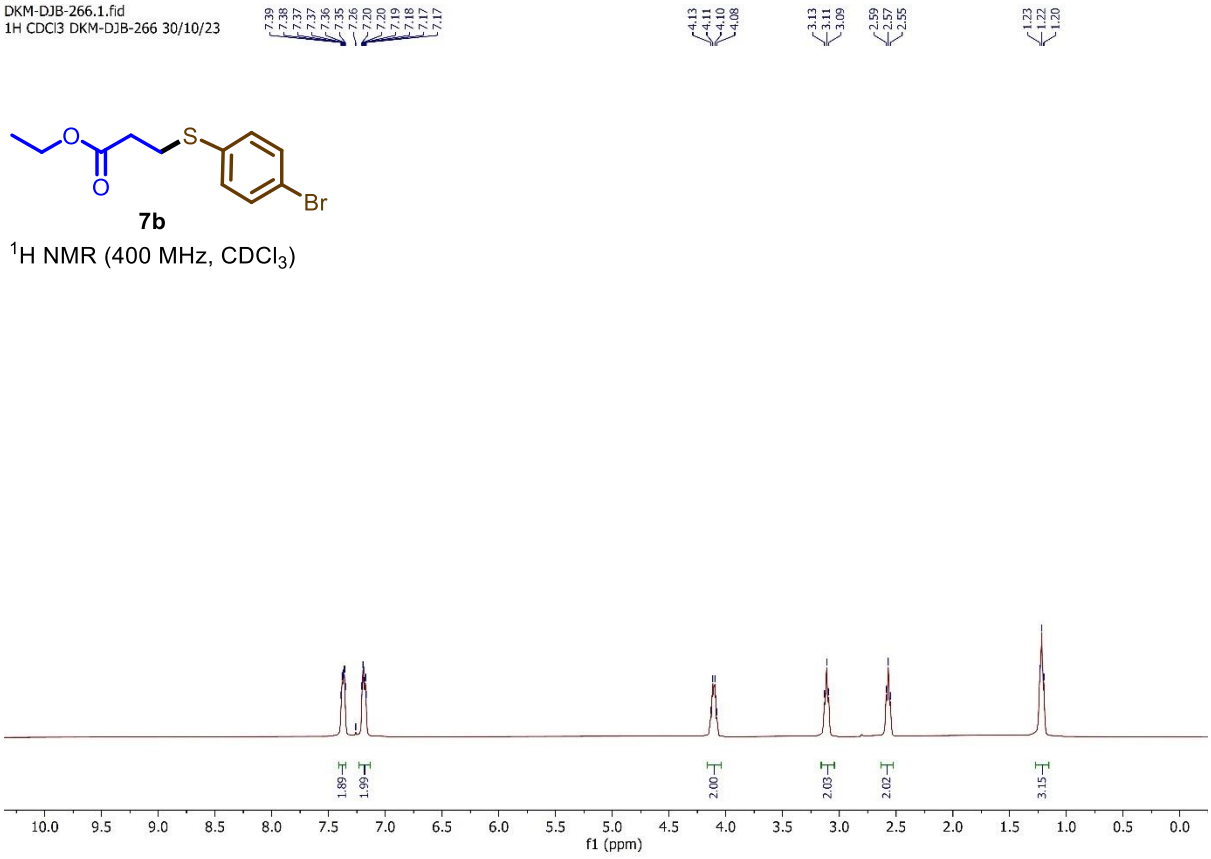
^{1}H NMR (300 MHz, $CDCl_3$)



DKM-DJB-226US-13C

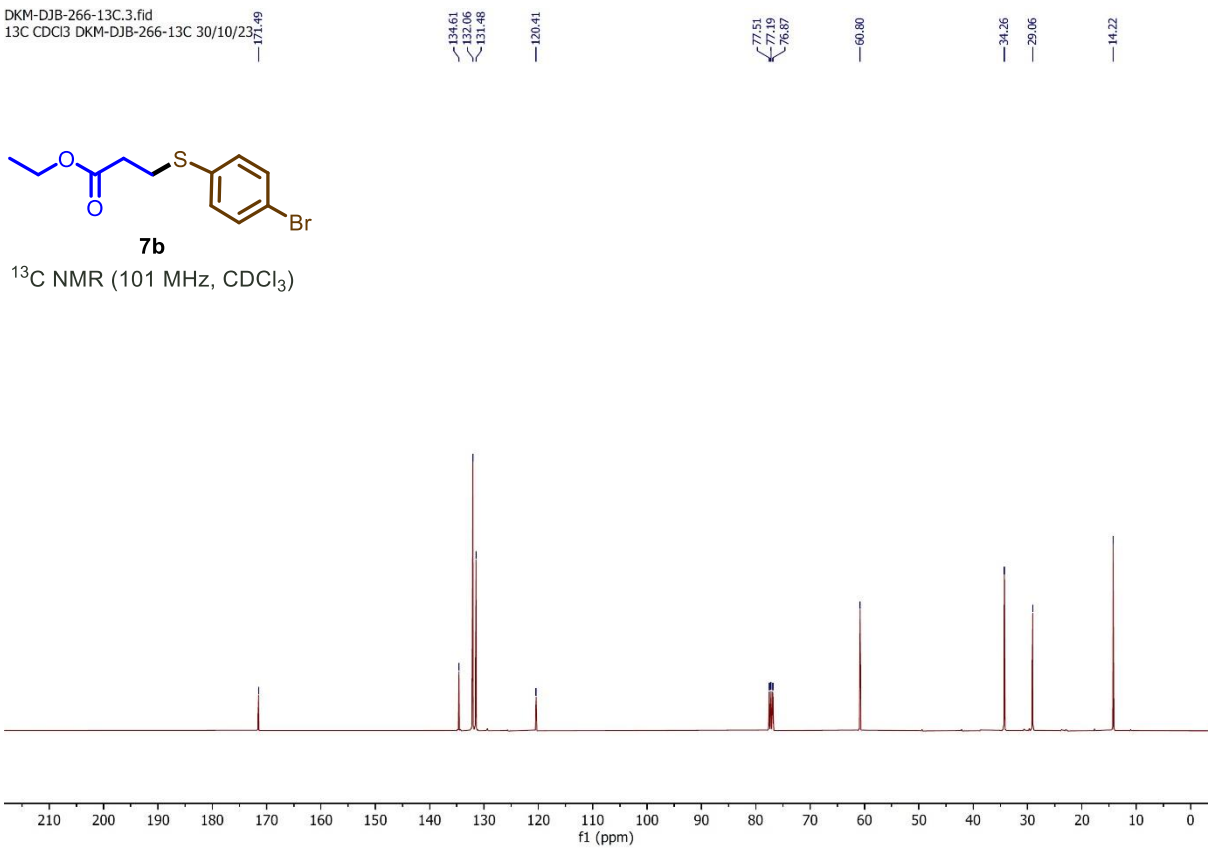
7a

^{13}C NMR (75 MHz, $CDCl_3$)



7b

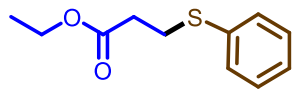
^{1}H NMR (400 MHz, $CDCl_3$)



7b

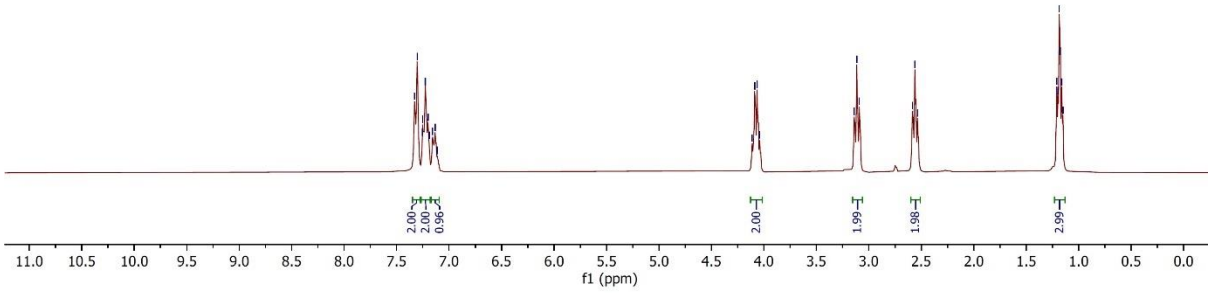
^{13}C NMR (101 MHz, $CDCl_3$)

DKM-DJB-269.1.fid
1H CDCl3 DKM-DJB-269 4/11/23

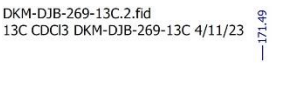


7c

^{1}H NMR (300 MHz, $CDCl_3$)

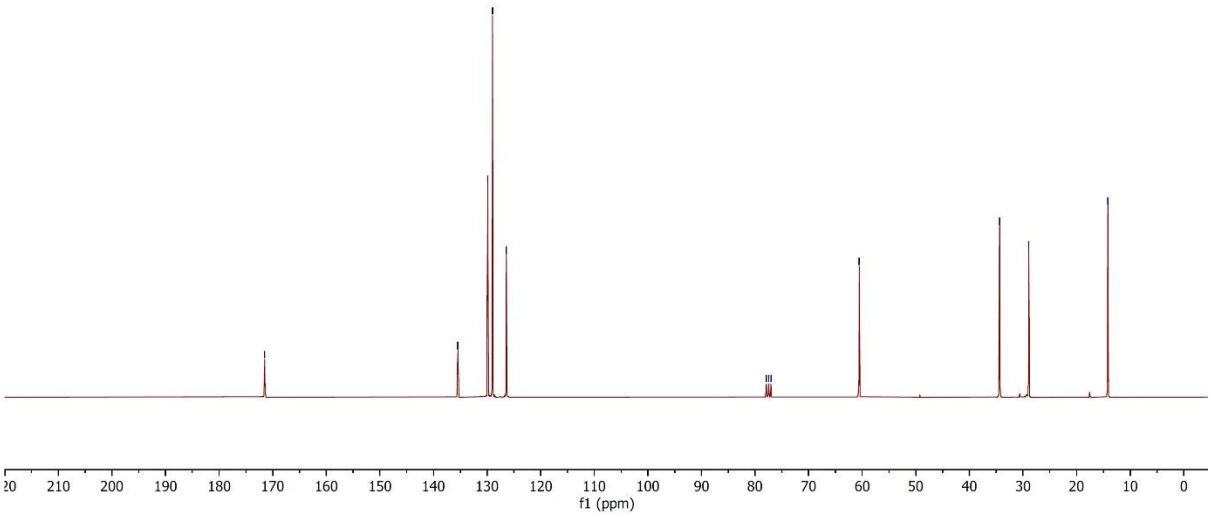


DKM-DJB-269-13C.2.fid
13C CDCl3 DKM-DJB-269-13C 4/11/23

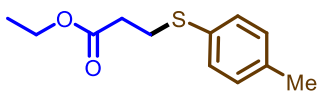


7c

^{13}C NMR (75 MHz, $CDCl_3$)

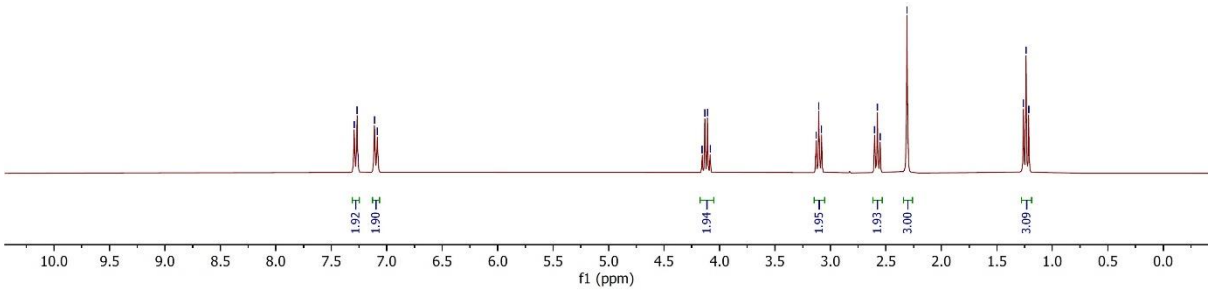


DKM-DJB-261.22.fid
1H-CDCL3-DKM-DJB-261-01-11-2023

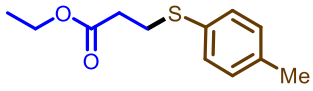


7d

^{1}H NMR (300 MHz, $CDCl_3$)

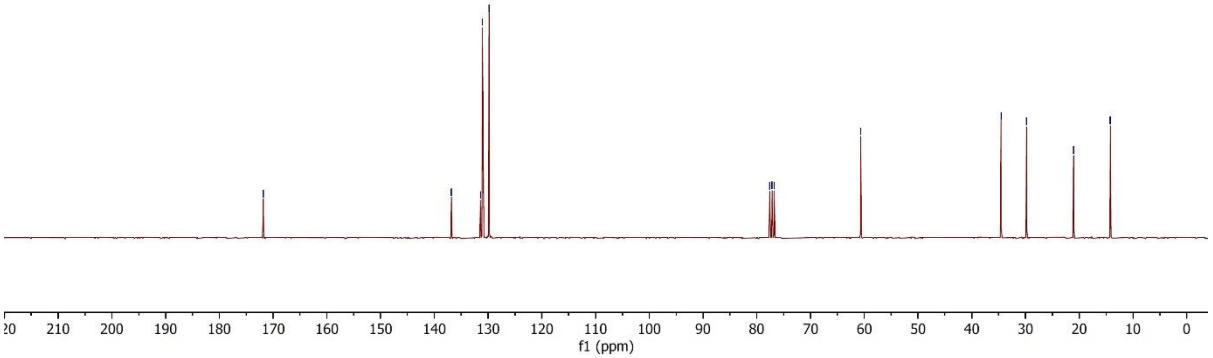


DKM-DJB-261.23.fid
13C-CDCL3-DKM-DJB-261-01-11-2023

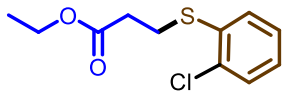


7d

^{13}C NMR (75 MHz, $CDCl_3$)

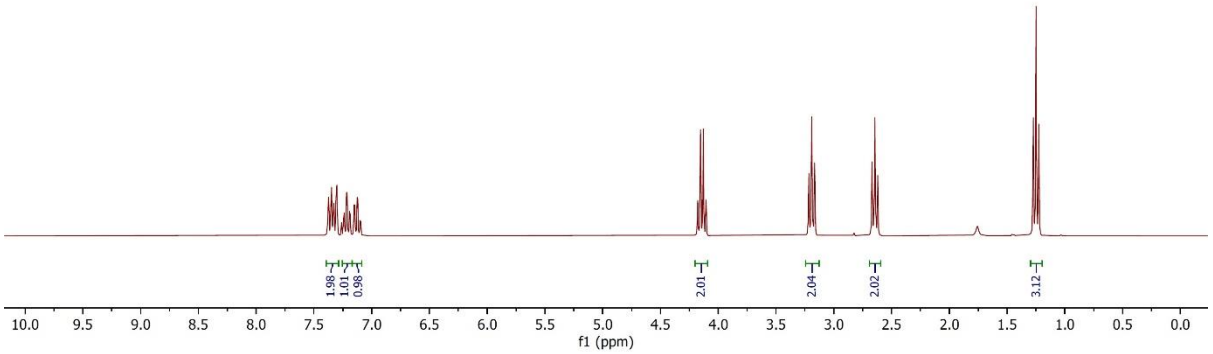


DKM-DJB-237

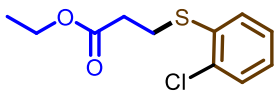


7e

^{1}H NMR (300 MHz, $CDCl_3$)

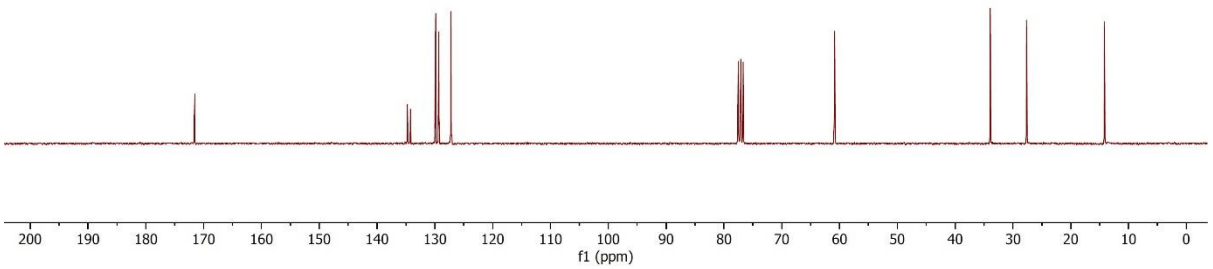


DKM-DJB-237-13C

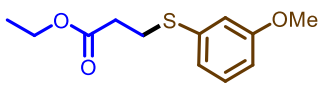


7e

^{13}C NMR (75 MHz, $CDCl_3$)

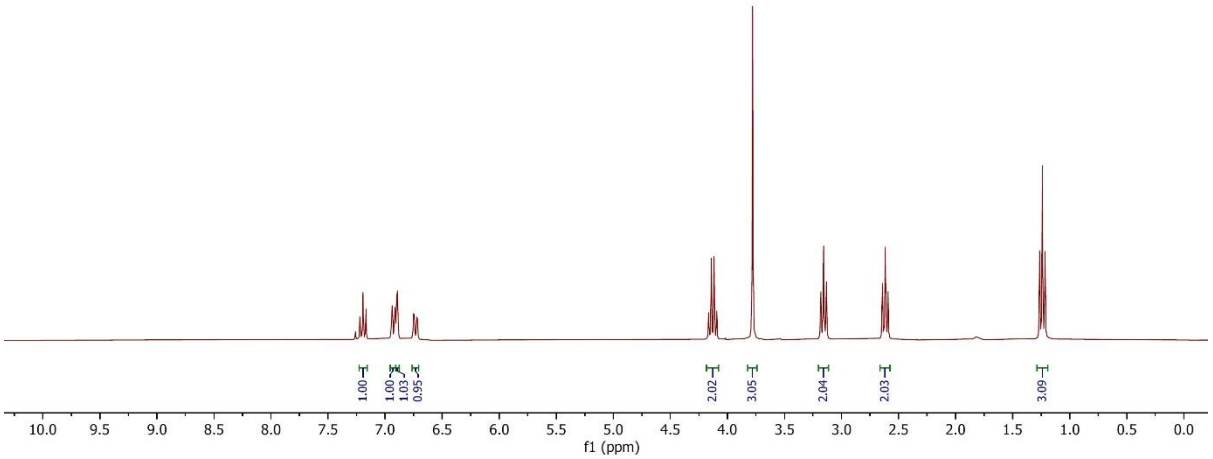


DKM-DJB-236

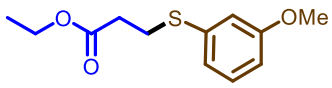


7f

^{1}H NMR (300 MHz, $CDCl_3$)

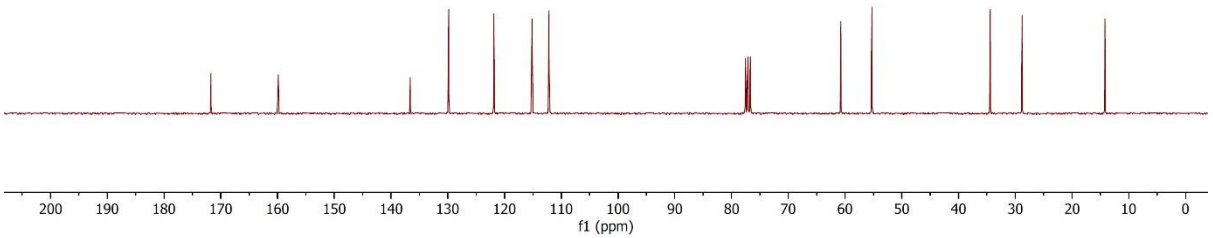


DKM-DJB-236-13C

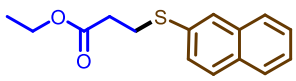


7f

^{13}C NMR (75 MHz, $CDCl_3$)

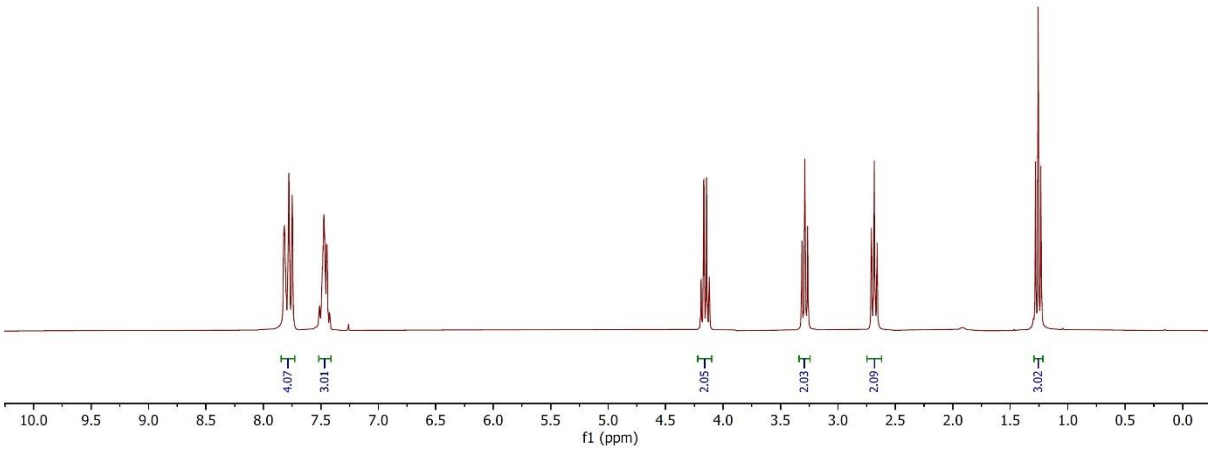


DKM-DJB-238

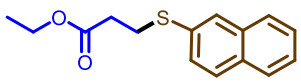


7g

^{1}H NMR (300 MHz, $CDCl_3$)

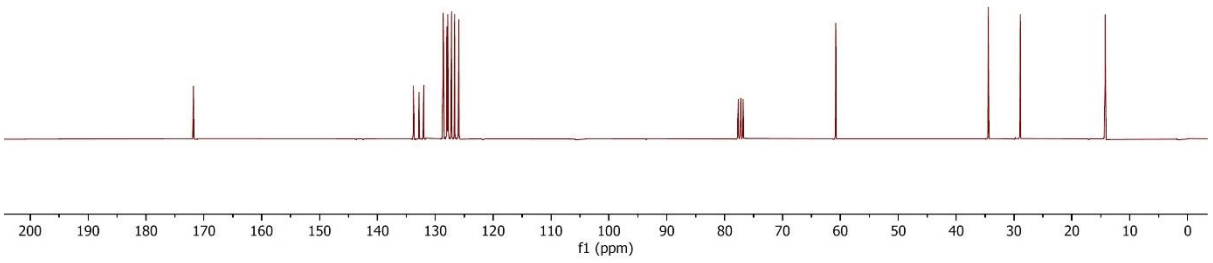


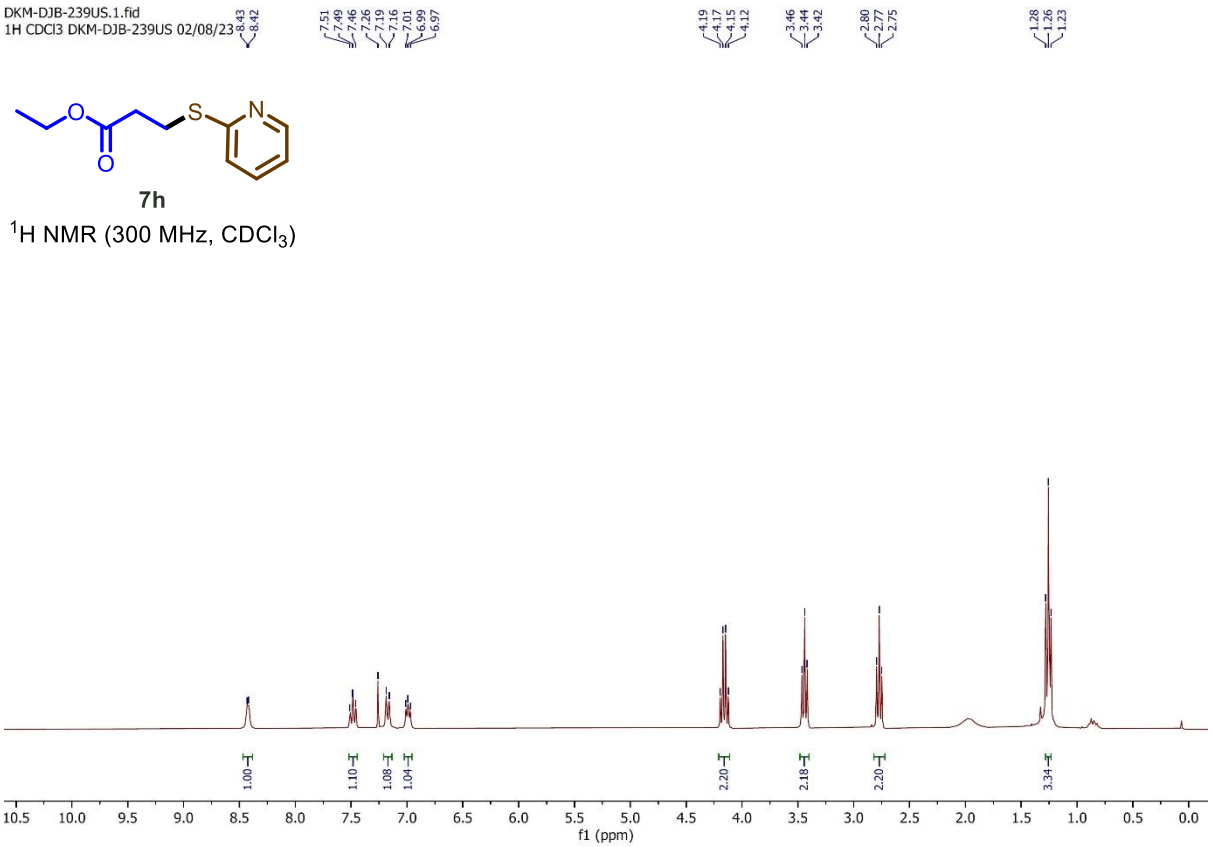
DKM-DJB-238-13C



7g

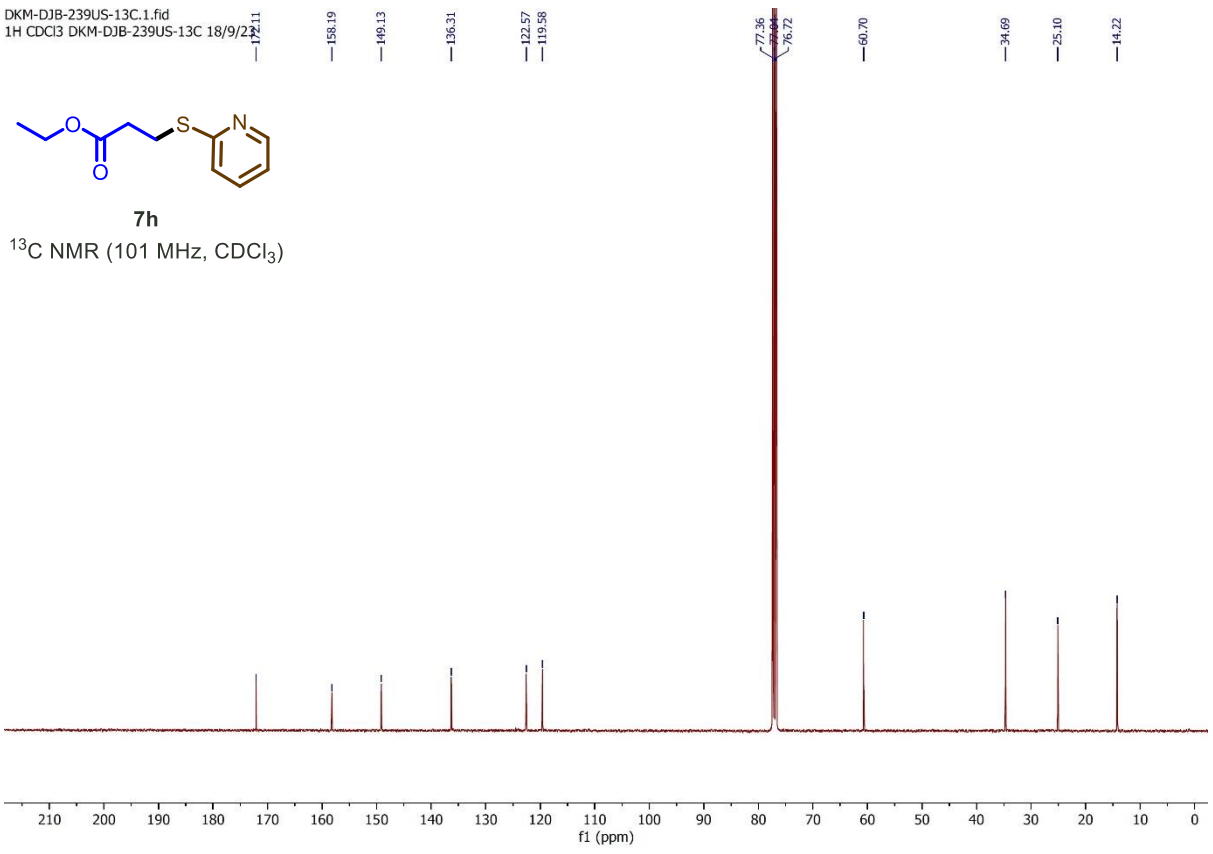
^{13}C NMR (75 MHz, $CDCl_3$)





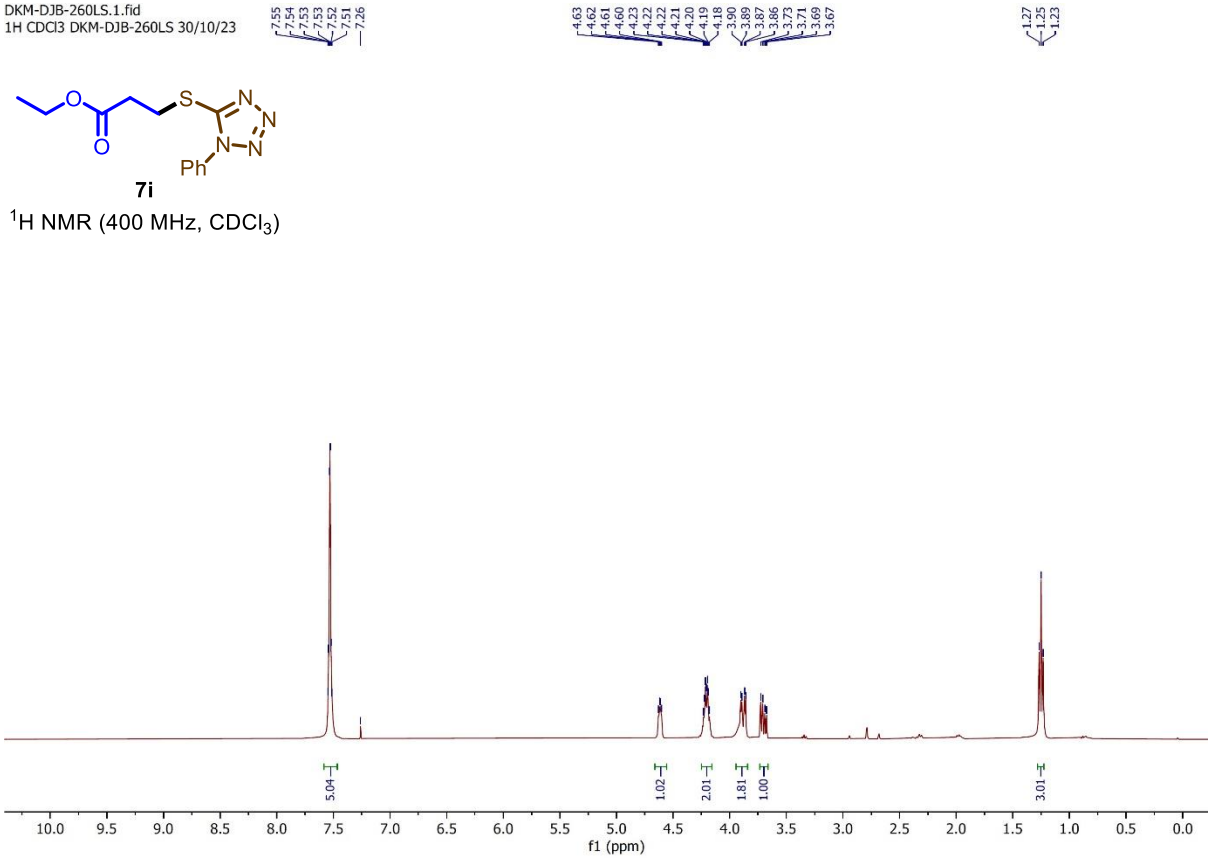
7h

^{1}H NMR (300 MHz, $CDCl_3$)



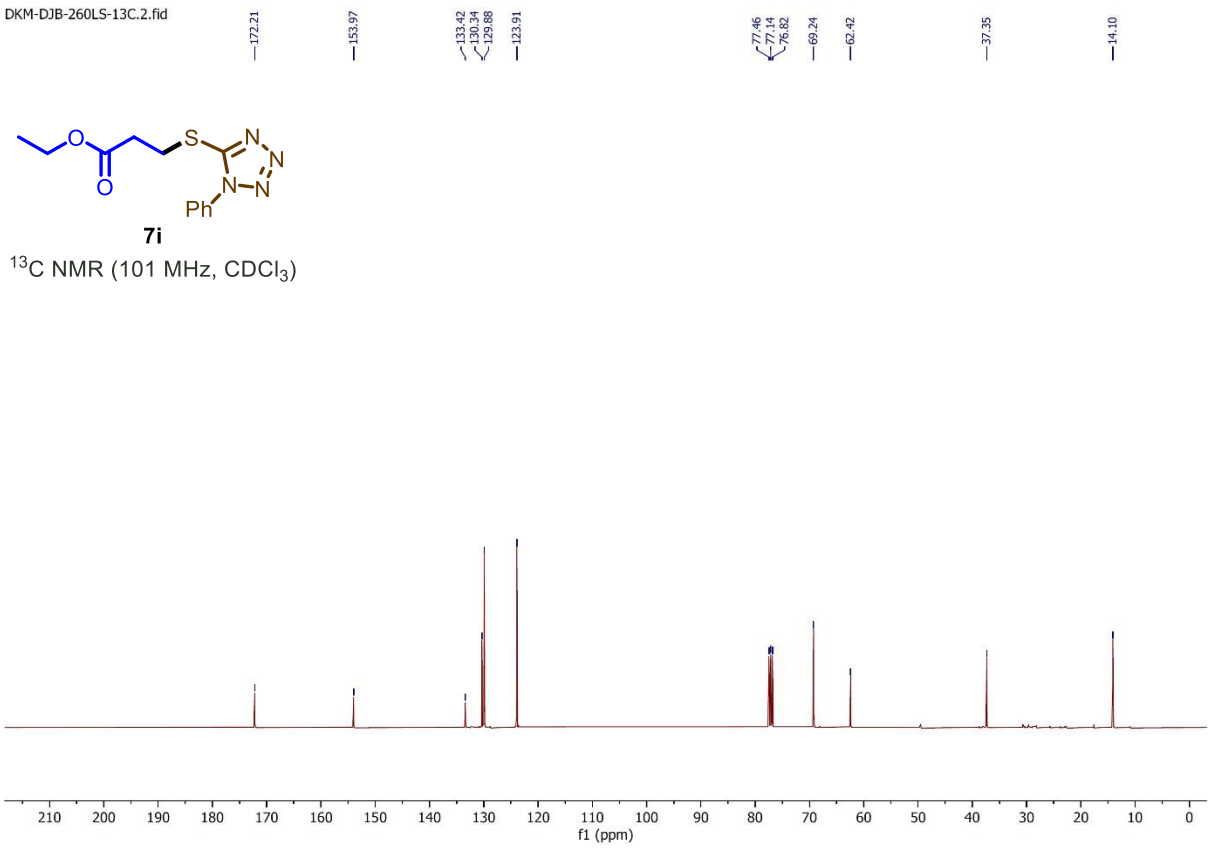
7h

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



7i

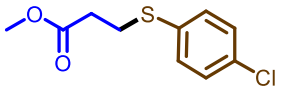
^{1}H NMR (400 MHz, $CDCl_3$)



7i

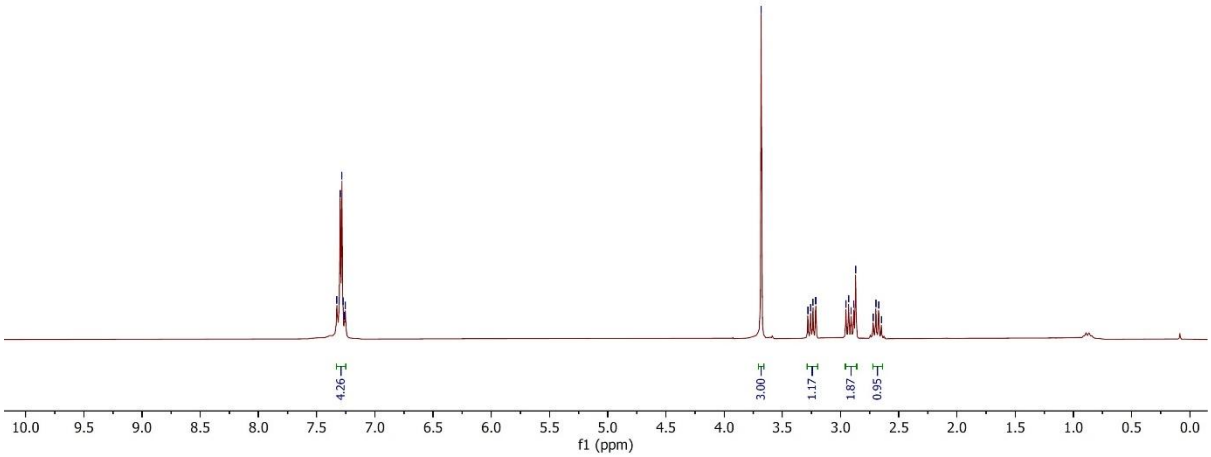
^{13}C NMR (101 MHz, $CDCl_3$)

DKM-DJB-270.1.fid
DKM-DJB-270,1H,CDCL3,03/11/23

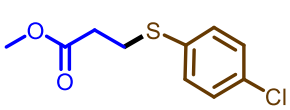


7j

^{1}H NMR (300 MHz, $CDCl_3$)

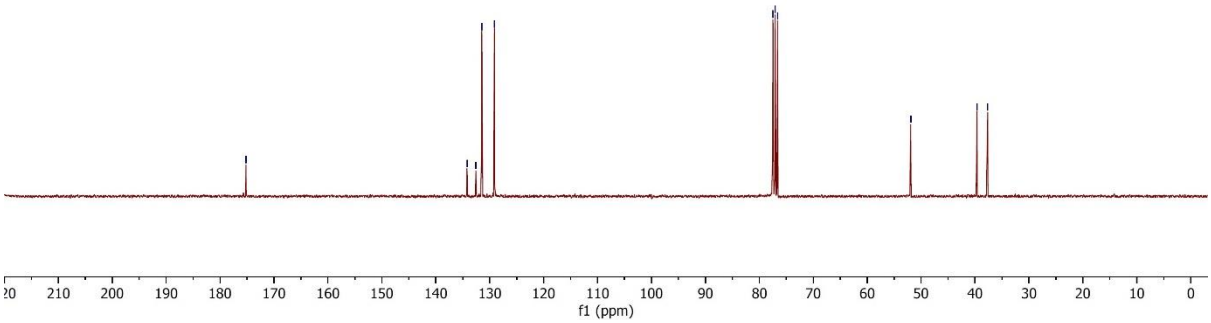


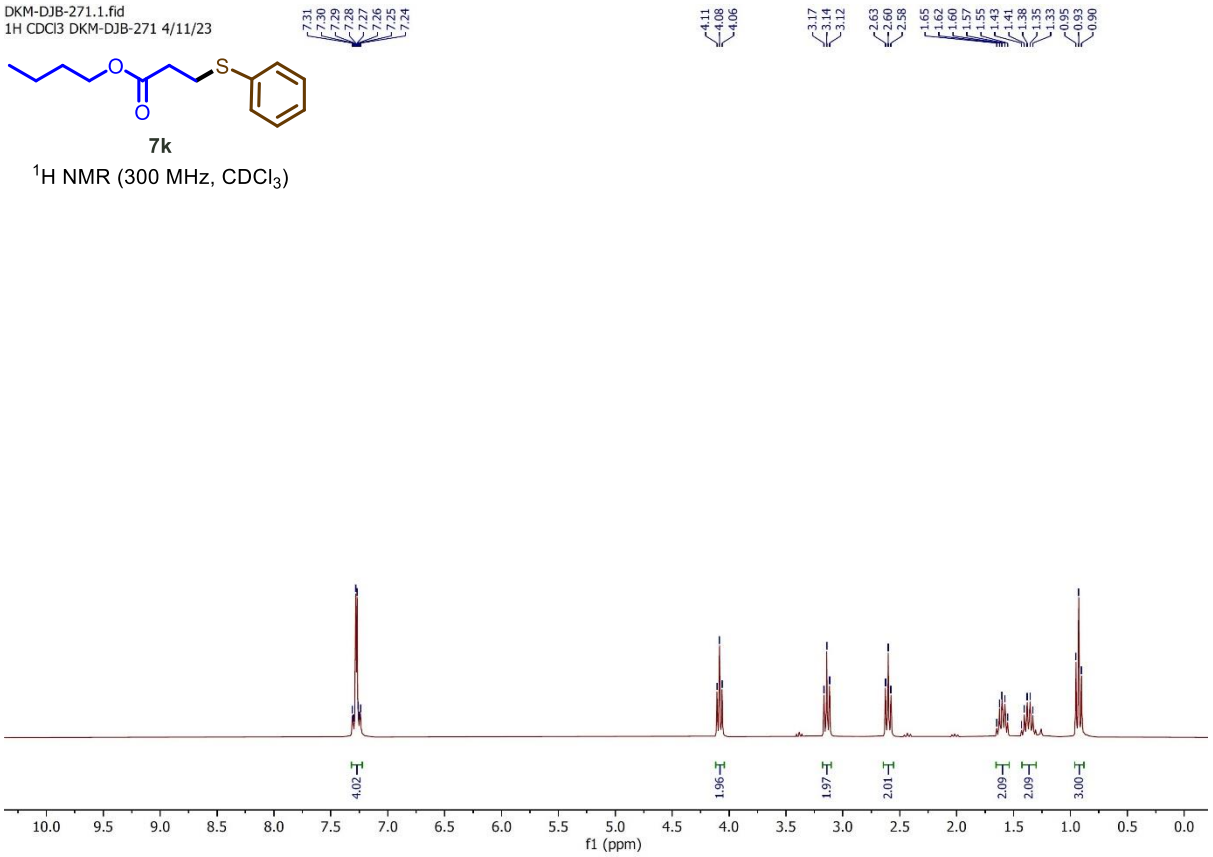
DKM-DJB-270.2.fid
13C_CDCl3_DKM-DJB-270_08.11.2023



7j

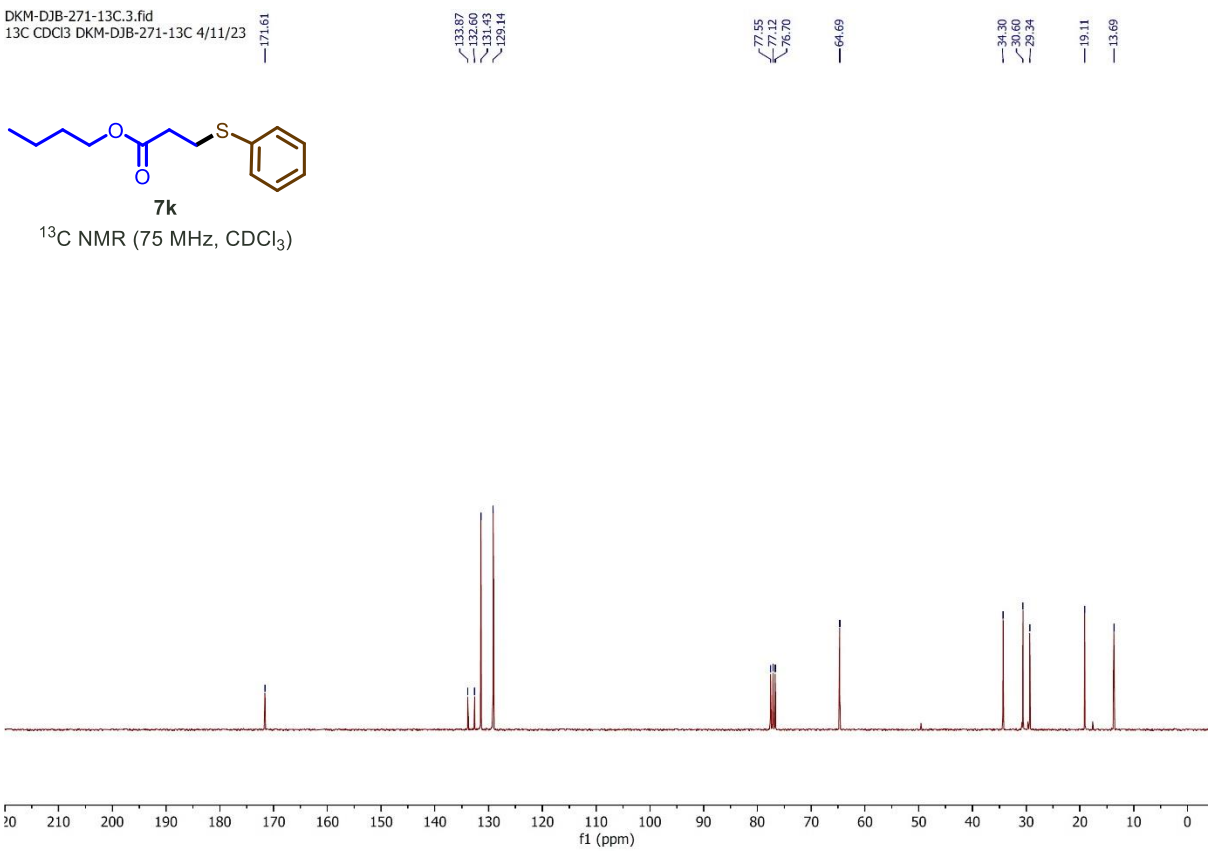
^{13}C NMR (75 MHz, $CDCl_3$)





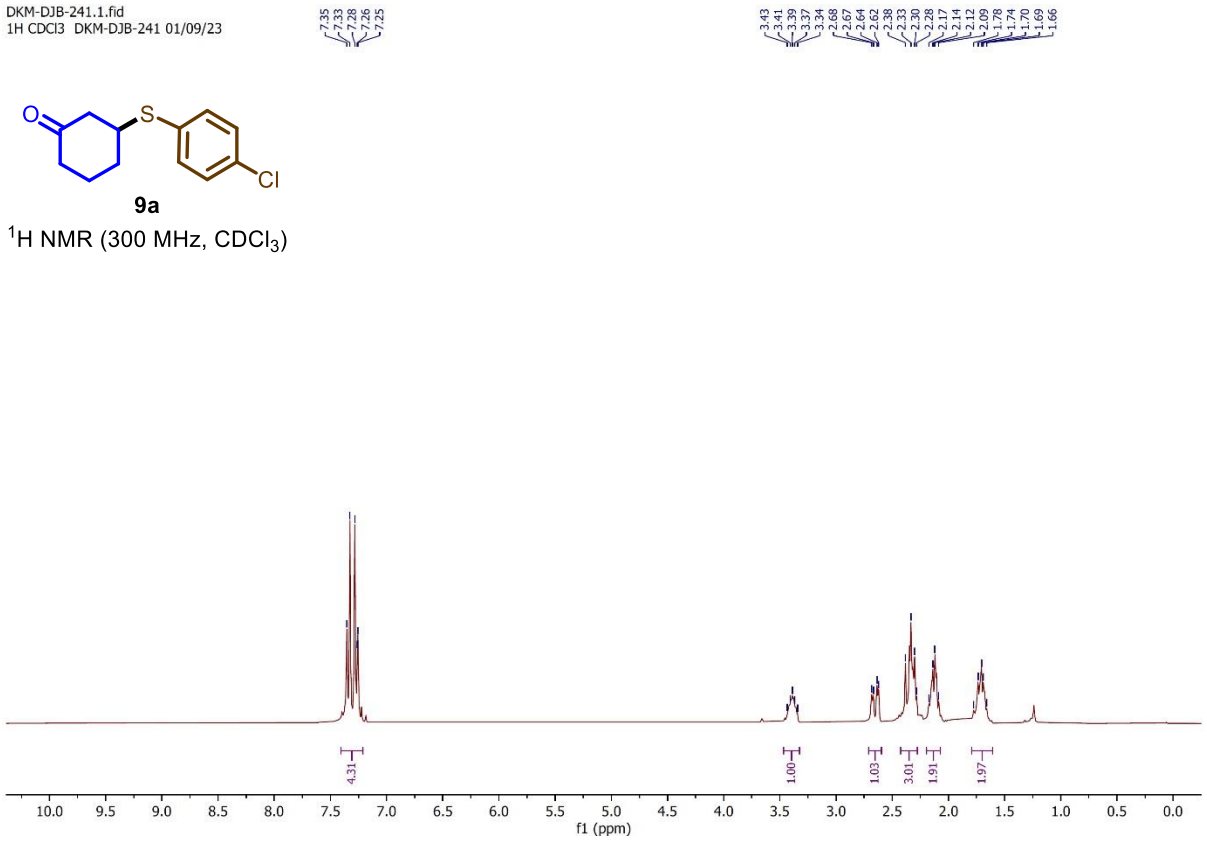
7k

^{1}H NMR (300 MHz, $CDCl_3$)



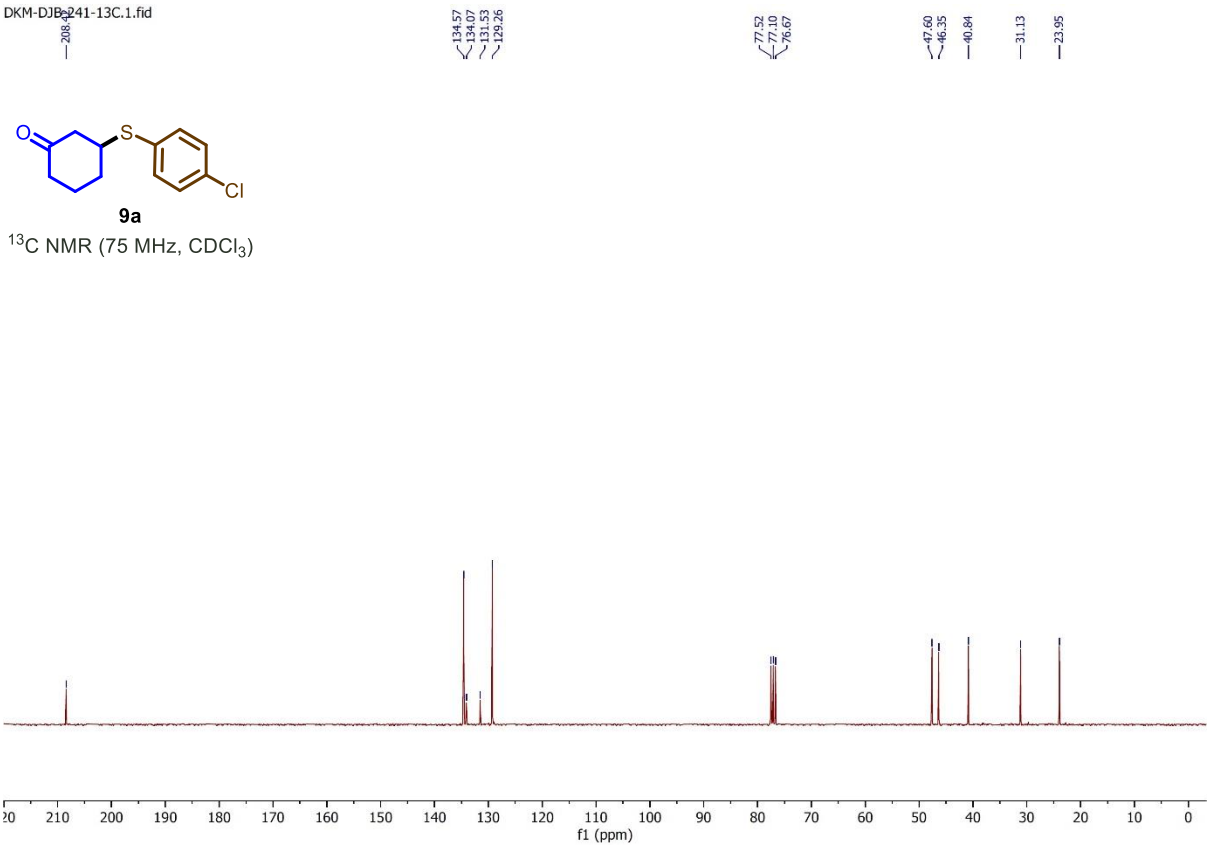
7k

^{13}C NMR (75 MHz, $CDCl_3$)



9a

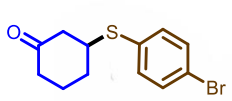
^{1}H NMR (300 MHz, $CDCl_3$)



9a

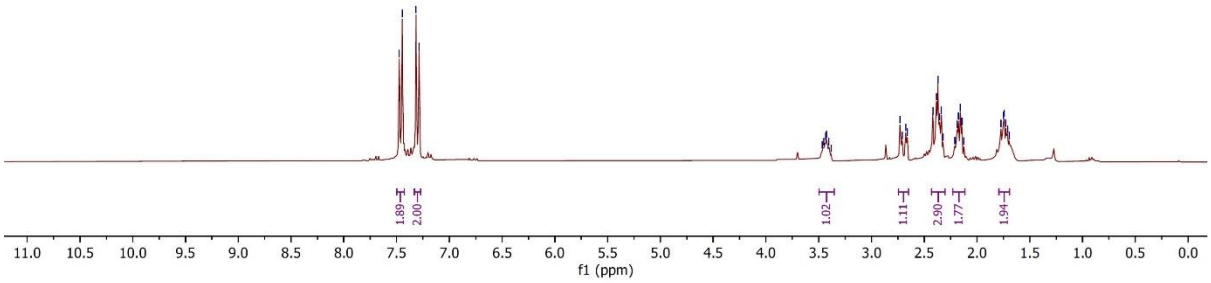
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-263.10.fid
1H-CDCL3-DKM-DJB-263-01-11-2023

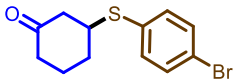


9b

^{1}H NMR (300 MHz, $CDCl_3$)



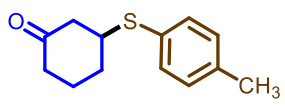
DKM-DJB-263-13C.1.fid



9b

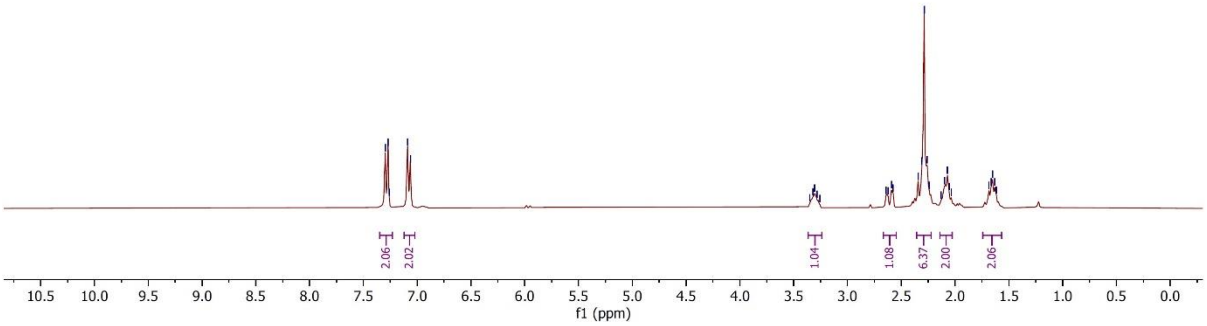
^{13}C NMR (75 MHz, $CDCl_3$)

DKM-DJB-246.1.fid
1H CDCl3 DKM-DJB-246 13/9/23

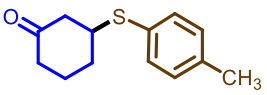


9c

^{1}H NMR (300 MHz, $CDCl_3$)

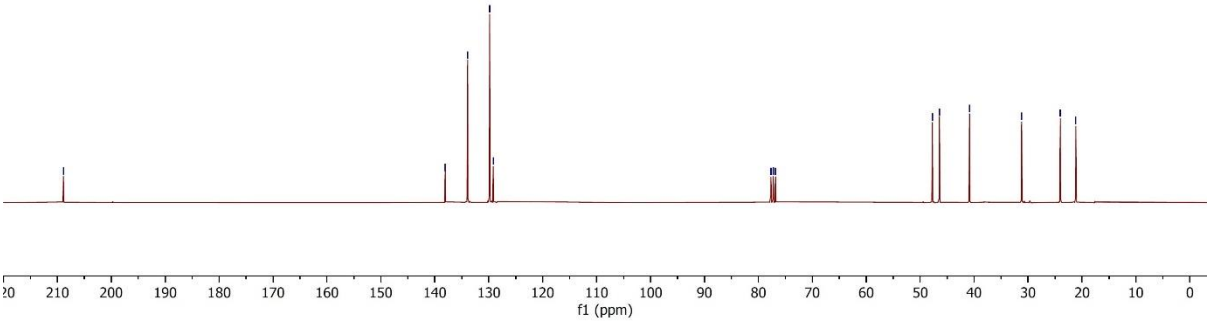


DKM-DJB-246-13C.3.fid



9c

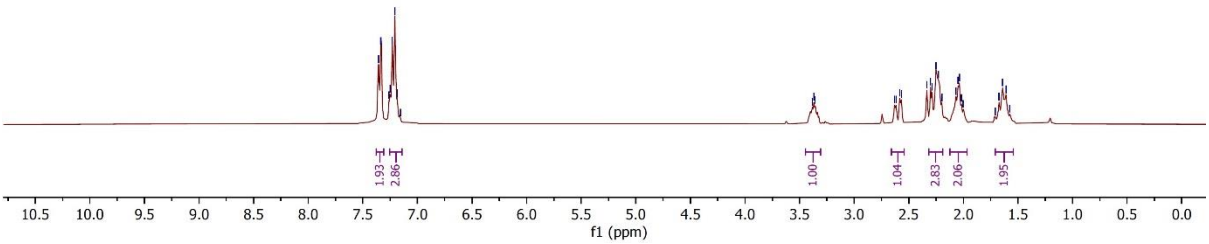
^{13}C NMR (75 MHz, $CDCl_3$)



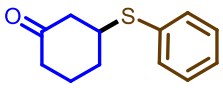
DKM-DJB-268.1.fid
1H CDCl3 DKM-DJB-268 4/11/23

9d

^{1}H NMR (300 MHz, $CDCl_3$)

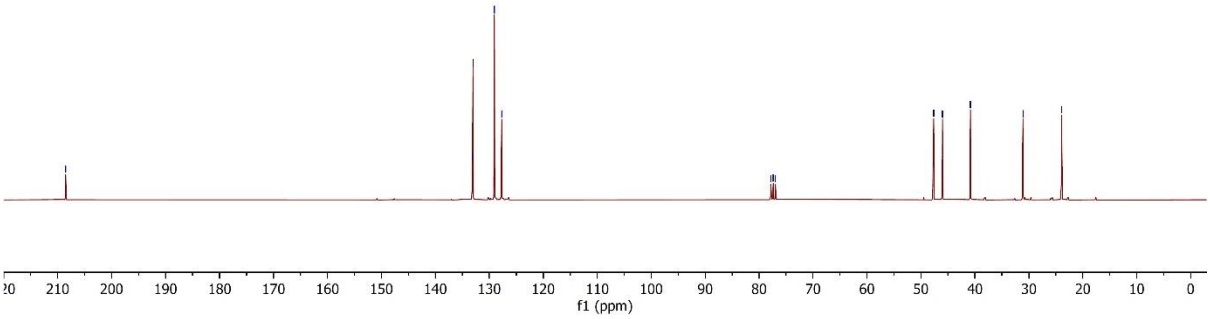


DKM-DJB-268-13C.2.fid

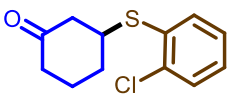


9d

^{13}C NMR (75 MHz, $CDCl_3$)

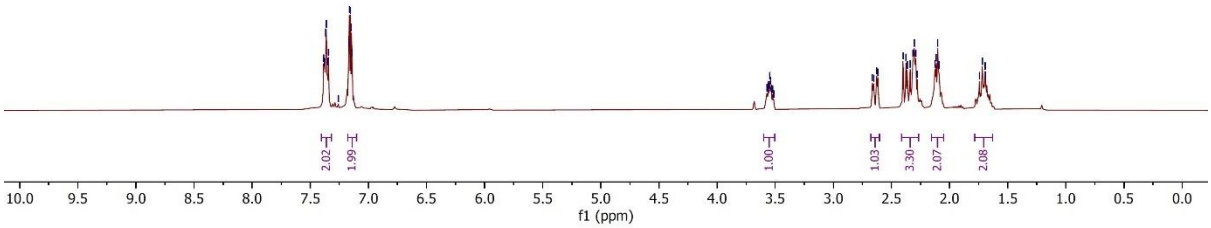


DKM-DJB-256.1.fid
1H CDCl3 DKM-DJB-256 30/10/23



9e

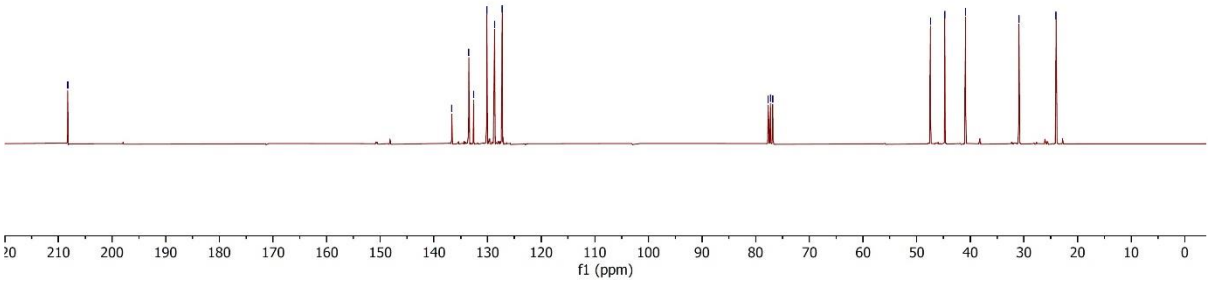
^{1}H NMR (400 MHz, $CDCl_3$)



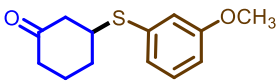
DKM-DJB-256.10.fid

9e

^{13}C NMR (75 MHz, $CDCl_3$)

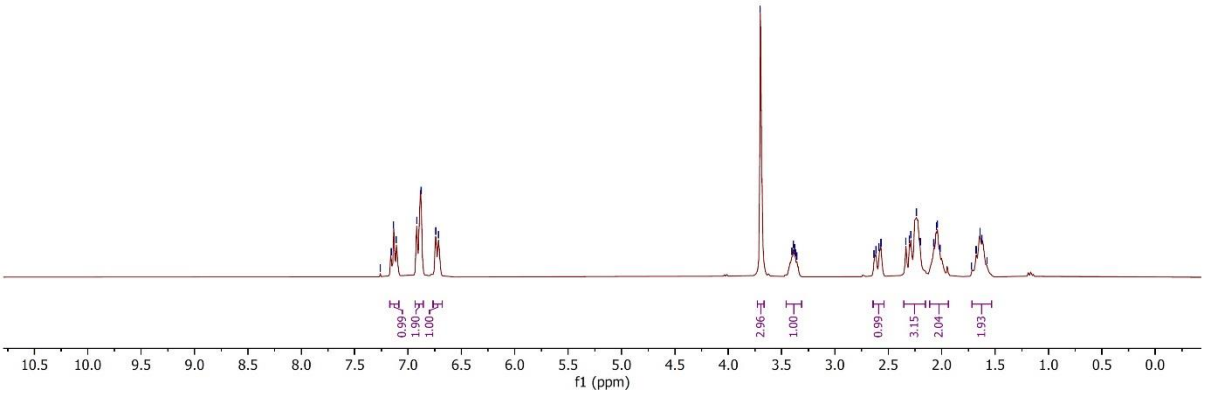


DKM-DJB-245.1.fid
1H CDCl3 DKM-DJB-245 13/9/23

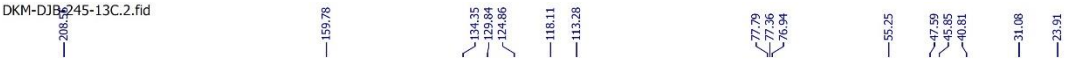


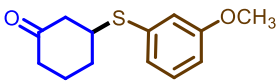
9f

^{1}H NMR (300 MHz, $CDCl_3$)



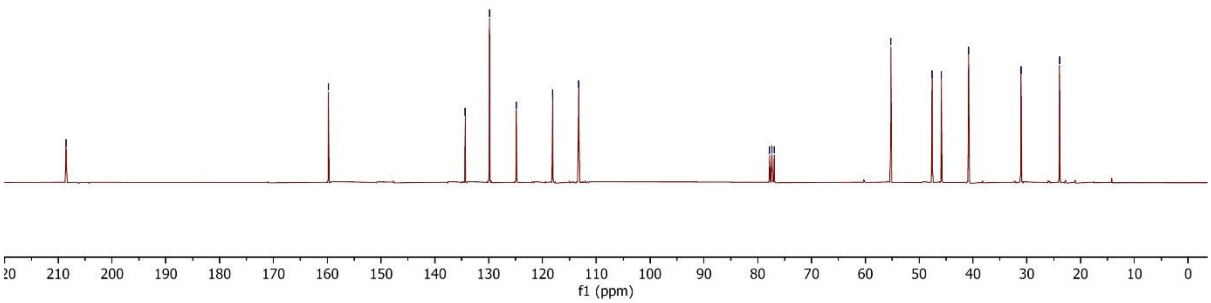
DKM-DJB-245-13C.2.fid



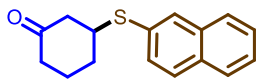


9f

^{13}C NMR (75 MHz, $CDCl_3$)

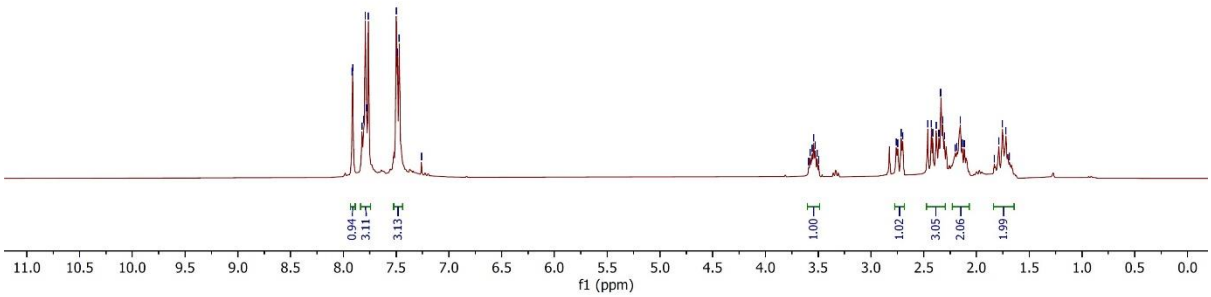


DKM-DJB-262UK.10.fid
1H-CDCL3-DKM-DJB-262UK-01-11-2023

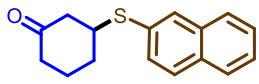


9g

^{1}H NMR (300 MHz, $CDCl_3$)

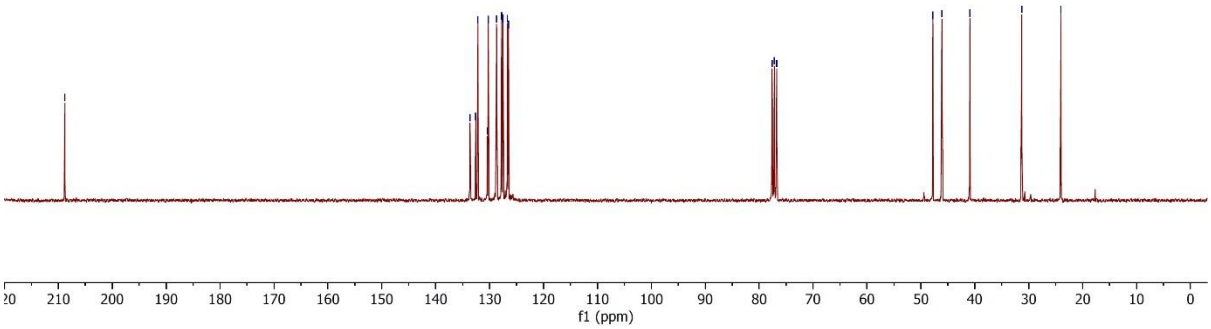


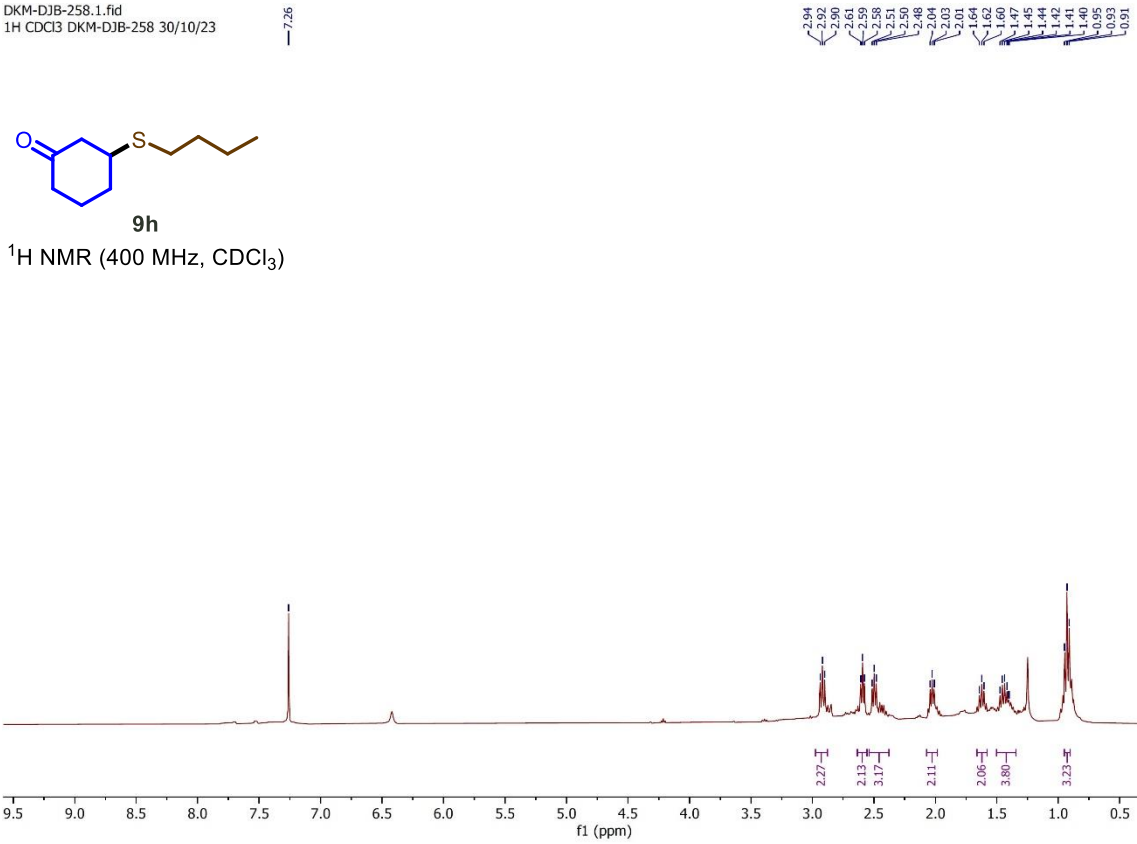
DKM-DJB-262UK-13C.1.fid



9g

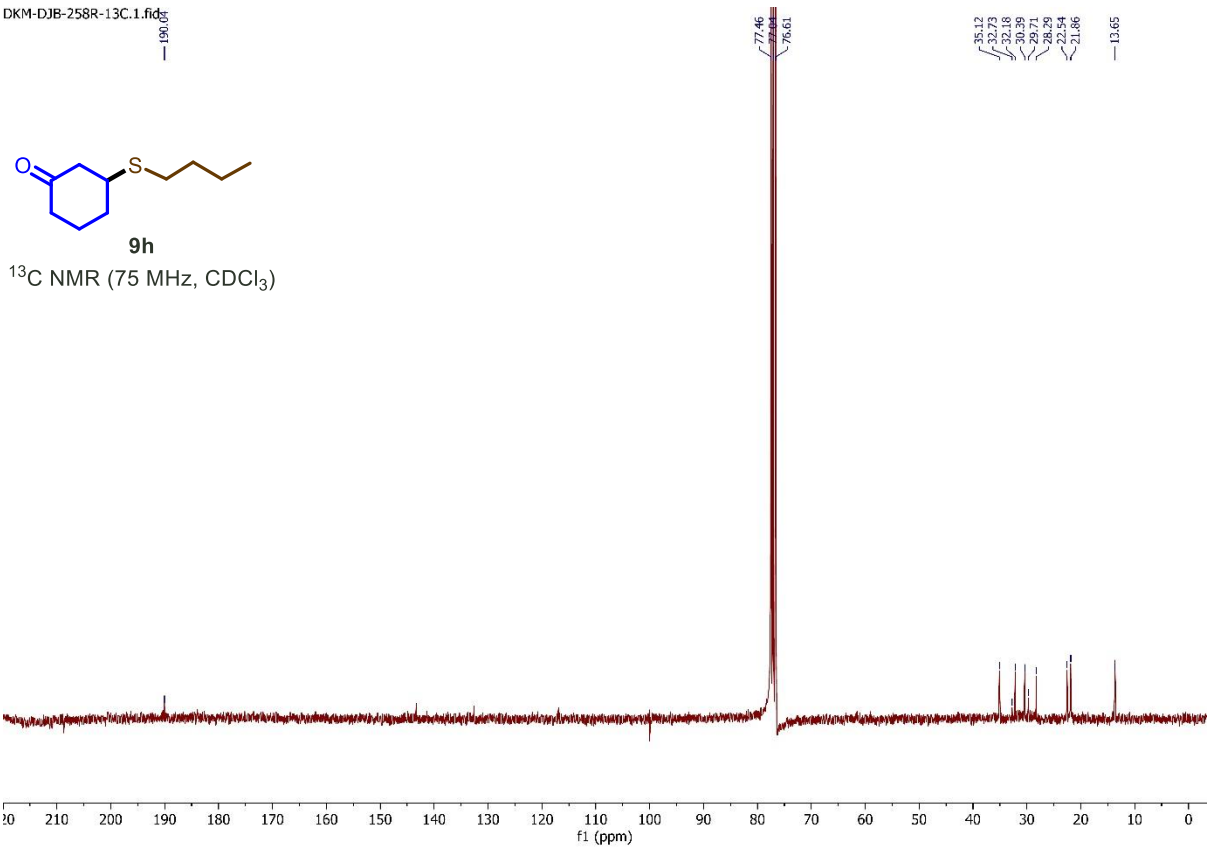
^{13}C NMR (75 MHz, $CDCl_3$)





9h

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



9h

^{13}C NMR (75 MHz, $CDCl_3$)

6. X-ray Crystallography data for the compound of 3h

Crystal of the compound 5a was obtained after slow evaporation of chloroform solvent. Molecular structure of 5a with 50% ellipsoid probability.

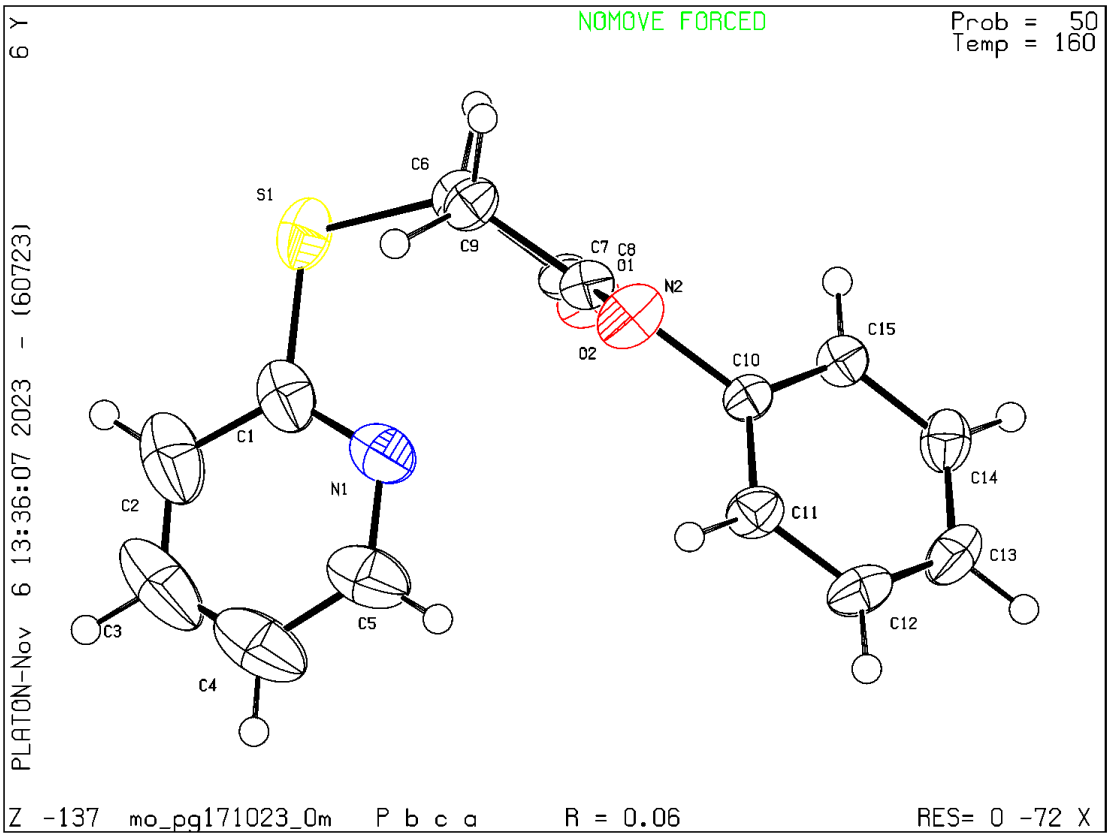


Table 1 Crystal data and structure refinement for mo_PG171023_0m.	
Identification code	mo_PG171023_0m
Empirical formula	$C_{15}H_{12}N_2O_2S$
Formula weight	284.33
Temperature/K	160.15
Crystal system	orthorhombic
Space group	Pbca
a/Å	10.087(2)
b/Å	10.871(2)
c/Å	25.038(6)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	2745.5(10)
Z	8
ρ_{calc}g/cm^3	1.376

μ/mm^{-1}	0.238
F(000)	1184.0
Crystal size/mm^3	0.6 × 0.57 × 0.2
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.4 to 54.212
Index ranges	$-12 \leq h \leq 12$, $-13 \leq k \leq 13$, $-29 \leq l \leq 31$
Reflections collected	25125
Independent reflections	2989 [R_{int} = 0.0738, R_{sigma} = 0.0407]
Data/restraints/parameters	2989/0/181
Goodness-of-fit on F^2	1.108
Final R indexes [I>=2σ (I)]	R_1 = 0.0575, wR_2 = 0.1336
Final R indexes [all data]	R_1 = 0.0643, wR_2 = 0.1379
Largest diff. peak/hole / e $Å^{-3}$	0.34/-0.31