

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. ¹H NMR spectra were recorded on a 400 MHz and 300 MHz spectrometers in CDCl₃ with TMS as internal standard. ¹³C 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 CDCl₃ signal at 7.26 ppm for ¹H 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 ¹³C NMR. Data for ¹H 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 °C) was maintained for all the reactions.

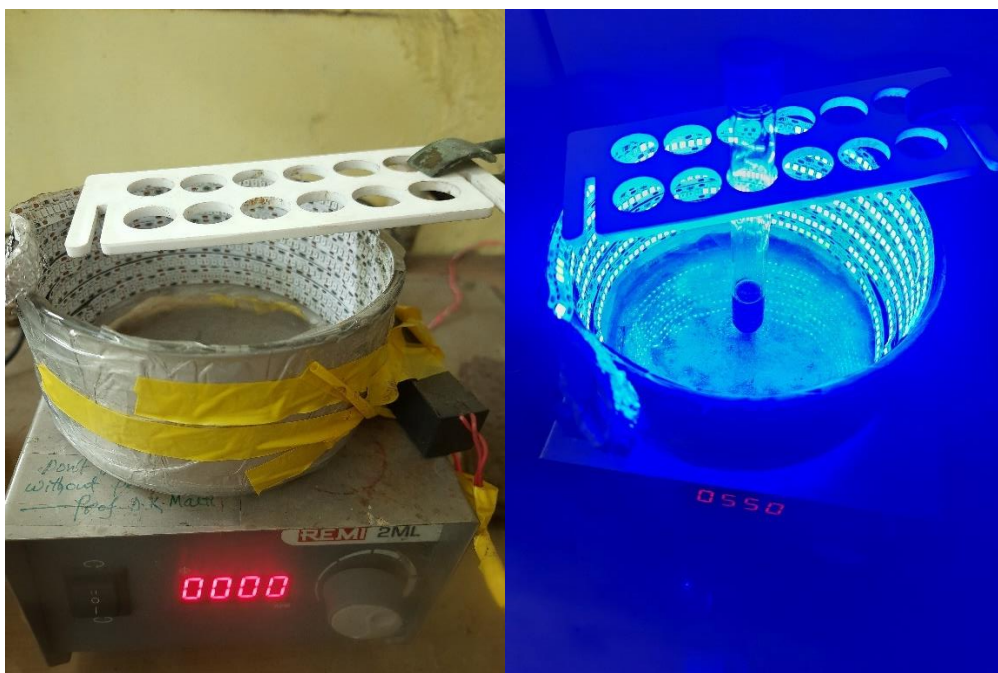


Figure S1. Laboratory Light Setup used for Irradiation of the Reaction.

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).

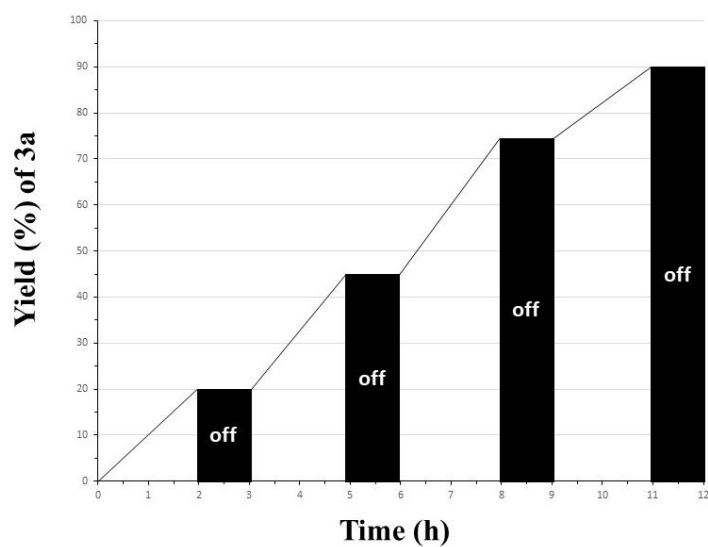


Figure S2. On-Off experiment.

2.3 Fluorescence Quenching Experiments

Formulation of solution: 4-Chlorothiophenol **2a** (7.2 mg) was dissolved in CH₃OH in a 5 mL volumetric flask to obtain a 0.01 M solution. N-phenyl maleimide **1a** (43.3 mg) was dissolved in CH₃OH in a 25 mL volumetric flask to obtain 0.01 M solution. Product **3a** (15.8 mg) was dissolved in CH₃OH in a 5 mL volumetric flask to obtain a 0.01 M solution. The photocatalyst Eosin Y (3.5 mg) was dissolved in CH₃OH 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 CH₃OH, 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 μ L) 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 CH₃OH to prepare a 0.2 μ M solution. 100.0 μ L of a 4-chlorothiophenol solution was added successively and uniformly stirred, and the resulting mixture was irradiated at 525 nm. Fluorescence emission spectra of 0 μ L, 100 μ L, 200 μ L, 300 μ L, 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 $\lambda = 524$ nm.

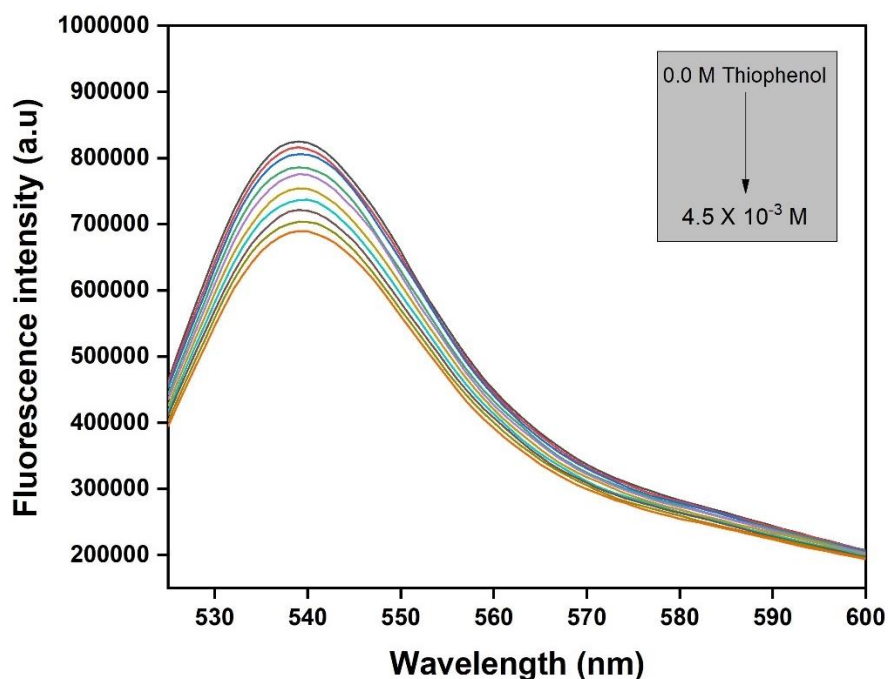


Figure S3. Fluorescence Emission Quenching of Eosin Y in the Presence of *p*-Chlorothiophenol (**2a**).

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

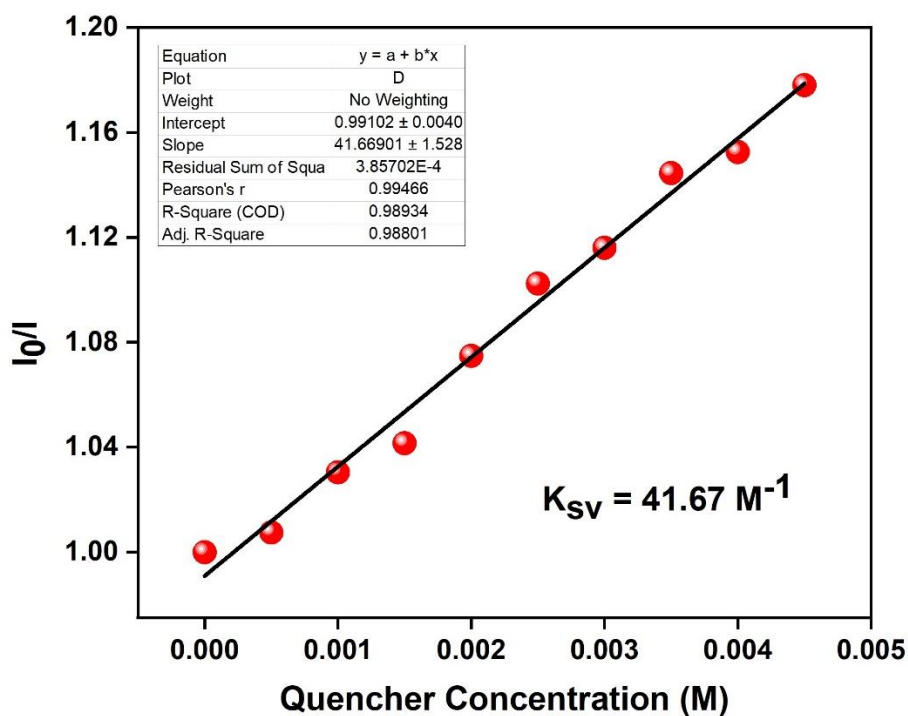


Figure S4. Stern-Volmer Plots for Quenching of Eosin Y Fluorescence Emission in the Presence of *p*-Chlorothiophenol (**2a**).

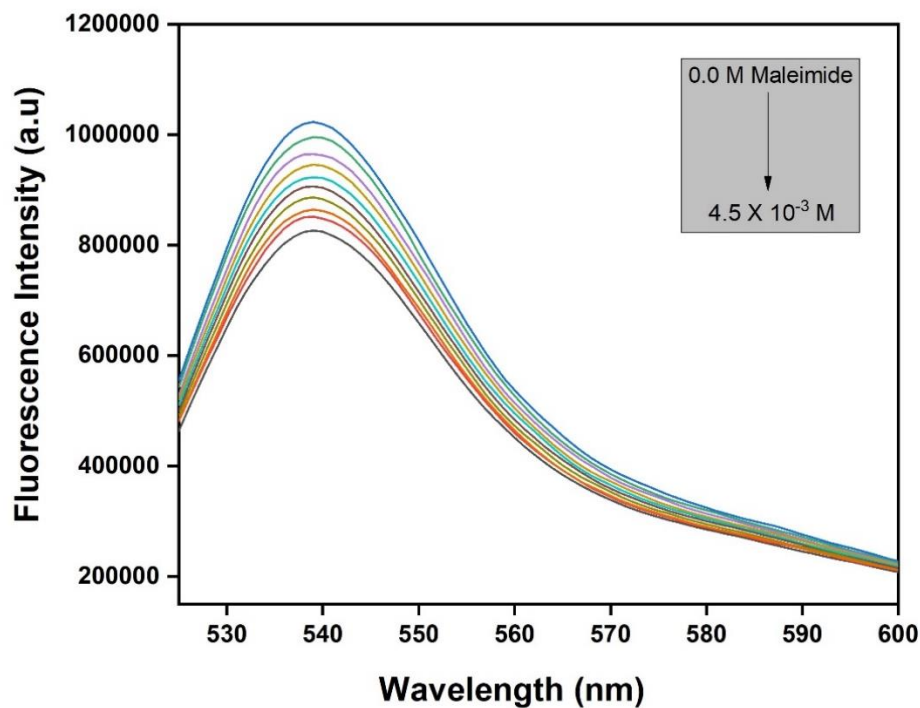


Figure S5. Quenching of Eosin Y Fluorescence Emission in the Presence of Maleimide.

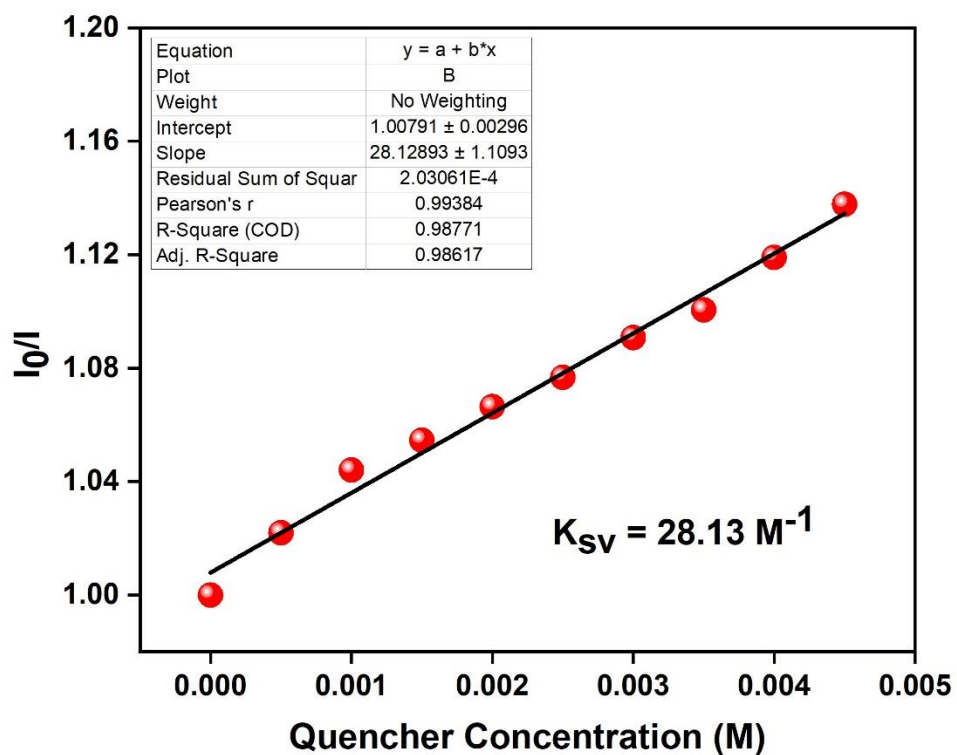


Figure S6. Stern-Volmer Plots for Quenching of Eosin Y Fluorescence Emission in the Presence of Maleimide.

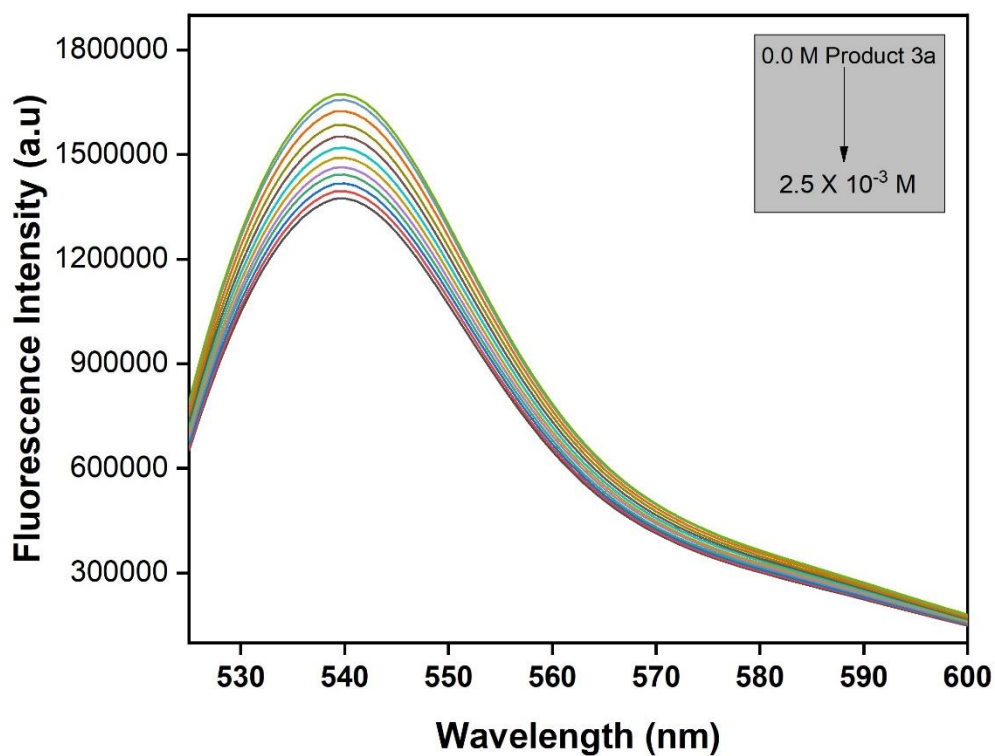


Figure S7. Quenching of Eosin Y Fluorescence Emission in the Presence of Product 3a.

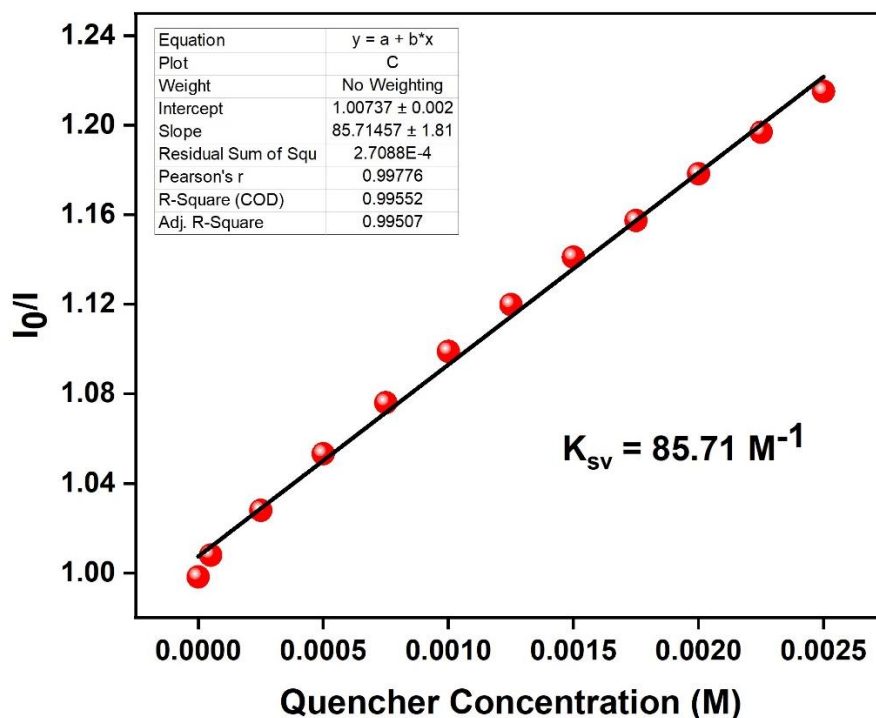


Figure S8. Stern-Volmer Plots for Quenching of Eosin Y Fluorescence Emission in the Presence of Product **3a**.

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 $EY^{\bullet-}$ 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.

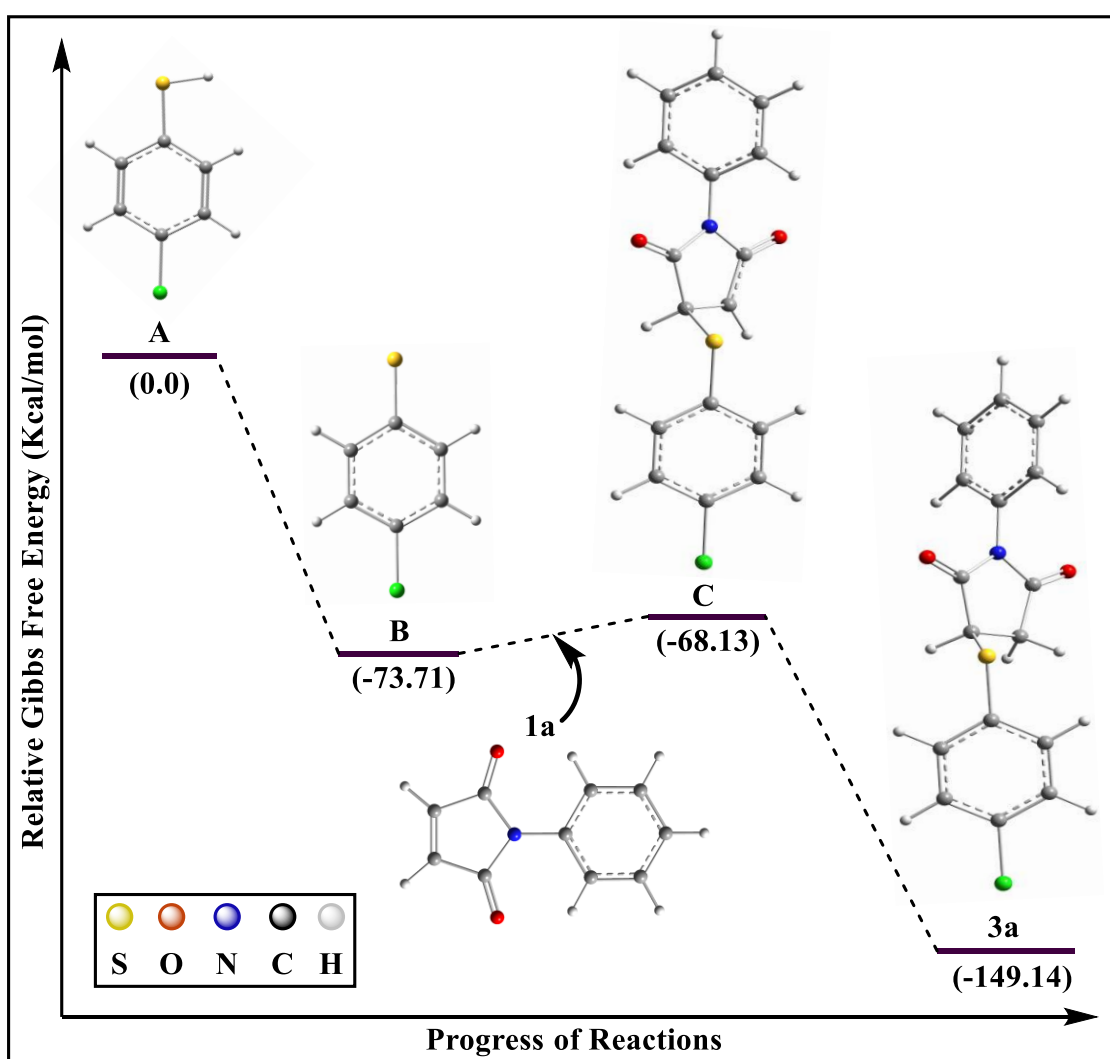
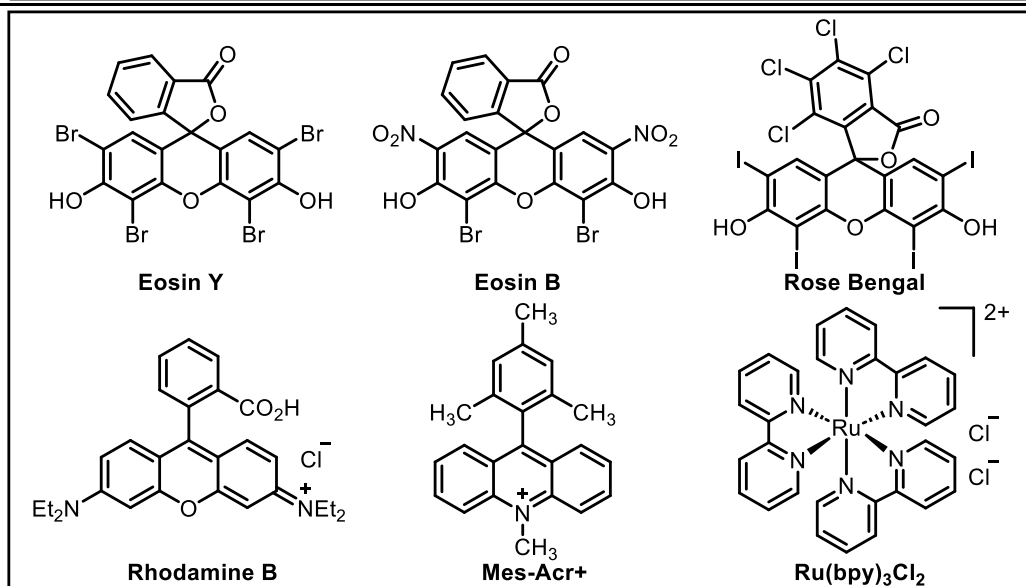
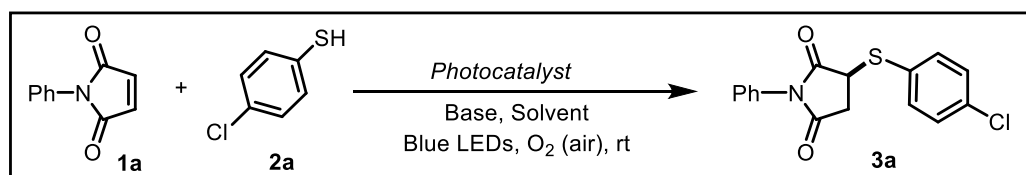


Figure S9. Computed free energy profiles (kcal/mol) of the catalytic cycle for the sulfenylation of maleimides in CH_3CN .

2.5. Table S1. Optimization of the Reaction Conditions^a



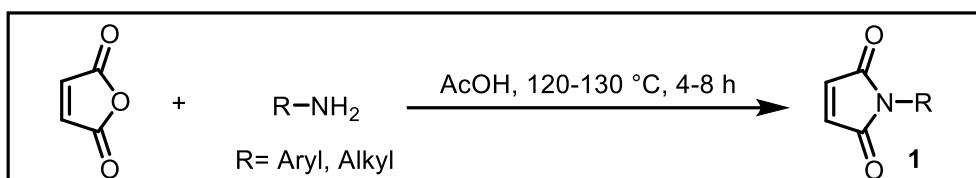
| entry | photocatalyst (mol%) | solvent | base | yield (%) ^b (3a) |
|----------|--|------------|--------------|-----------------------------|
| 1 | Eosin Y (2) | DCE | TMEDA | 76 |
| 2 | Mes-Acr+ (2) | DCE | TMEDA | 25 |
| 3 | Ru(bpy) ₃ Cl ₂ (2) | DCE | TMEDA | 55 |
| 4 | Eosin B (2) | DCE | TMEDA | 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 ₃ N | 26 |
| 18 | Eosin Y (5) | DCE | NaOAc | 27 |
| 19 | Eosin Y (5) | DCE | Na ₂ CO 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 |

^aReaction 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 O₂ (air) at room temperature for 12 h. ^bYield was determined through ¹H NMR analysis using internal standard 1,3,5-trimethoxybenzene. ^cUnder Green LED irradiation. ^dIrradiated under White LEDs. ^eO₂ balloon was used instead of air. ^fN₂ atmosphere. NR= no reaction.

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

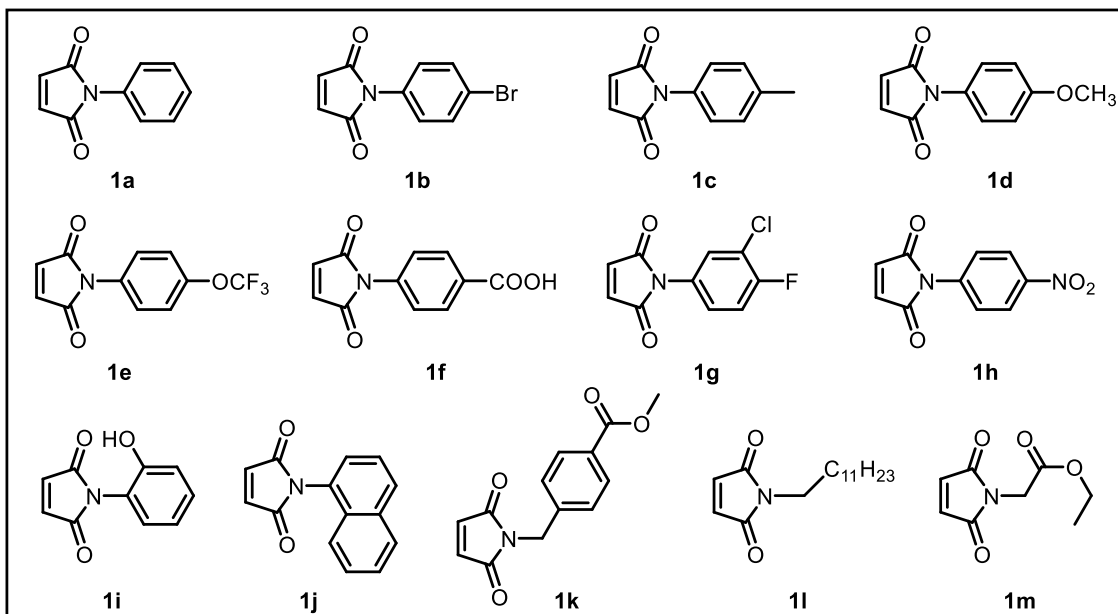
N-Aryl/*N*-Alkyl maleimide were prepared using the modified reported procedure mentioned below from the corresponding primary amine.³



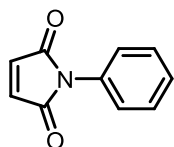
Primary amine (1.0 equiv.) and maleic anhydride (2.0 equiv.) were taken in a round bottomed flask with acetic acid (1.5 mL per mmol of amine). This mixture was stirred until the maleic anhydride dissolved completely and then refluxed for 4-8 h (depending on the type of amine) at 120-130 °C. After completion 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:

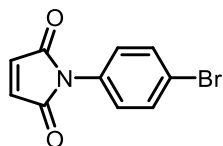


2.7. Characterization Data for Synthesized maleimide derivatives



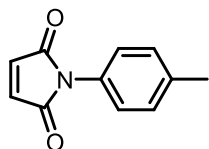
***N*-phenyl-1*H*-pyrrole-2,5-dione (1a)³**

¹H NMR (300 MHz, CDCl₃) δ 7.47 (t, *J* = 7.4 Hz, 2H), 7.39 – 7.32 (m, 3H), 6.83 (s, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 169.57, 134.22, 131.24, 129.18, 128.00, 126.11.



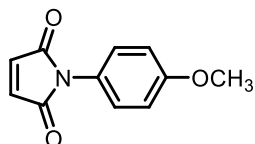
***N*-(4-Bromophenyl)-1*H*-pyrrole-2,5-dione (1b)³**

¹H NMR (400 MHz, CDCl₃) δ 7.60–7.58 (m, 2H), 7.27 (s, 1H), 7.25 (s, 1H), 6.86 (s, 2H). ¹³C NMR (75 MHz, CDCl₃) δ 169.1, 134.3, 132.3, 130.3, 127.4, 121.6.



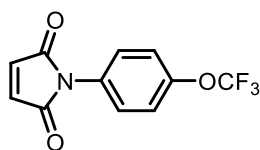
1-(p-Tolyl)-1H-pyrrole-2,5-dione (1c)³

¹H NMR (300 MHz, CDCl₃) 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). **¹³C NMR (75 MHz, CDCl₃)** δ 169.7, 138.0, 134.1, 129.6, 128.5, 126.0, 21.0.



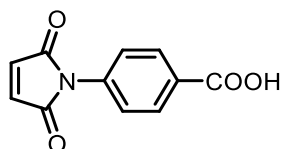
1-(4-Methoxyphenyl)-1H-pyrrole-2,5-dione (1d)³

¹H NMR (300 MHz, CDCl₃) 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). **¹³C NMR (75 MHz, CDCl₃)** δ 169.8, 159.1, 134.1, 127.6, 123.7, 114.5, 55.5.



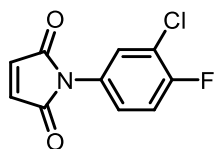
1-(4-(Trifluoromethoxy)phenyl)-1H-pyrrole-2,5-dione (1e)³

¹H NMR (300 MHz, CDCl₃) 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). **¹³C NMR (75 MHz, CDCl₃)** δ 169.1, 148.2, 134.3, 129.7, 127.2, 125.5, 122.1, 121.6, 118.6, 115.2.



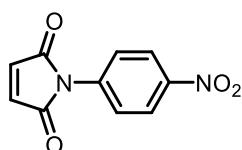
4-(2,5-Dioxo-2,5-dihydro-1H-pyrrol-1-yl)benzoic acid (1f)³

¹H 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). **¹³C NMR (75 MHz, DMSO)** δ 170.0, 167.1, 135.9, 135.3, 130.3, 130.0, 126.6.



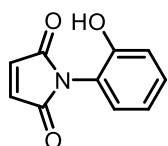
1-(3-Chloro-4-fluorophenyl)-1H-pyrrole-2,5-dione (1g)³

¹H NMR (300 MHz, CDCl₃) δ 7.46 (dd, *J* = 6.5, 2.3 Hz, 1H), 7.27 – 7.22 (m, 2H), 6.86 (s, 2H). **¹³C NMR (75 MHz, CDCl₃)** 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.



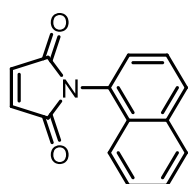
1-(4-Nitrophenyl)-1H-pyrrole-2,5-dione (1h)³

¹H NMR (300 MHz, CDCl₃) δ 8.33 (d, *J* = 9.3 Hz, 2H), 7.68 (d, *J* = 9.3 Hz, 2H), 6.93 (s, 2H). **¹³C NMR (75 MHz, CDCl₃)** δ 168.5, 146.1, 137.1, 134.6, 125.5, 124.4.



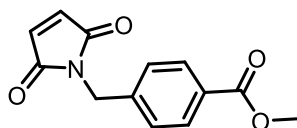
1-(2-Hydroxyphenyl)-1H-pyrrole-2,5-dione (1i)³

¹H NMR (300 MHz, CDCl₃) 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). **¹³C NMR (75 MHz, CDCl₃)** δ 170.1, 134.6, 134.5, 130.5, 129.5, 120.4, 118.4, 117.4.



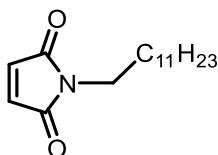
1-(Naphthalen-1-yl)-1H-pyrrole-2,5-dione (1j)³

¹H NMR (300 MHz, CDCl₃) 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). **¹³C NMR (75 MHz, CDCl₃)** 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.



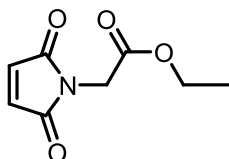
Methyl 4-((2,5-dioxo-2,5-dihydro-1H-pyrrol-1-yl)methyl)benzoate (1k)³

¹H NMR (300 MHz, CDCl₃) 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). ¹³C NMR (75 MHz, CDCl₃) 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

¹H NMR (300 MHz, CDCl₃) 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). ¹³C NMR (75 MHz, CDCl₃) 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.

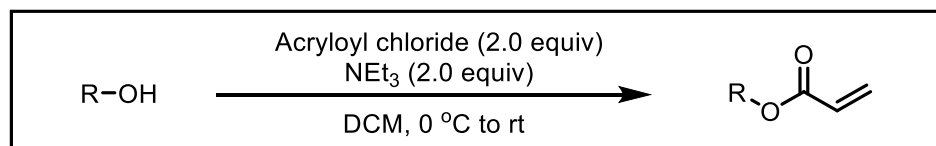


Ethyl 2-((2,5-dioxo-2,5-dihydro-1H-pyrrol-1-yl)acetate (1m)³

¹H NMR (300 MHz, CDCl₃) 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). ¹³C NMR (75 MHz, CDCl₃) 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



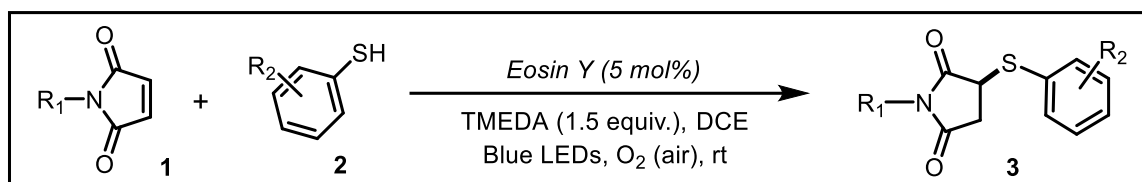
To a solution of R-OH and Et₃N (20 mmol, 2.0 equiv) in CH₂Cl₂ (25 mL) at 0 °C acryloyl chloride (20 mmol, 2.0 equiv) was added dropwise. The reaction mixture was warmed to rt and

stirred for 12 h. The reaction mixture was quenched with water (15 mL) and the mixture was extracted with CH₂Cl₂ (15 mL × 3). The combined organic layer was dried over MgSO₄, 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):

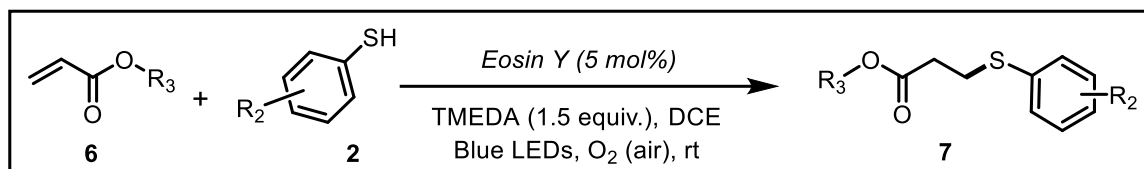
To an oven dried 15 mL borosilicate reaction tube, *N*-substituted maleimides **1** (0.2 mmol, 1 equiv), thiophenols **2** (0.24 mmol, 1.2 equiv), 1.5 equiv of base TMEDA and 5.0 mol% photocatalyst Eosin Y were taken in solvent DCE (1.5 mL), then the system was stirred under the irradiation of blue LEDs 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 Na₂SO₄ 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):

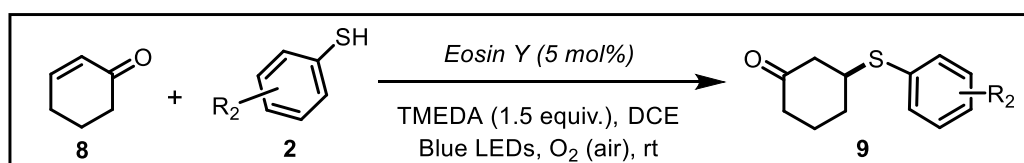
To an oven dried 15 mL borosilicate reaction tube, alkyl acrylates **6** (0.2 mmol, 1 equiv), thiophenols **2** (0.24 mmol, 1.2 equiv), 1.5 equiv of base TMEDA and 5.0 mol% photocatalyst Eosin Y were taken in solvent DCE (1.5 mL), then the system was stirred under the irradiation of blue LEDs at room 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 Na₂SO₄ 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**).

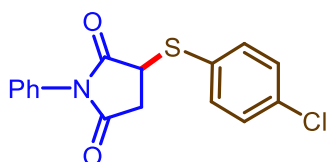


Method C. General procedure for the synthesis of sulfenylated cyclohexenone (9**):**

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 Na₂SO₄ 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**).



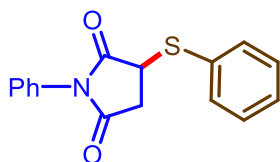
3. Characterization Data for all the Synthesized Compounds



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

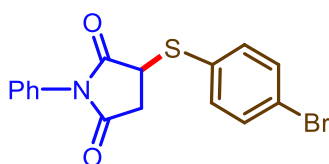
Yield 57.2 mg, 90%; White solid; $R_f = 0.2$ (20% EtOAc in petroleum ether, TLC). ¹H NMR (300 MHz, CDCl₃) δ 7.35 (d, $J = 8.5$ Hz, 2H), 7.31 – 7.22 (m, 3H), 7.17 (d, $J = 8.5$ Hz, 2H),

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 $\text{C}_{16}\text{H}_{13}\text{ClNO}_2\text{S}$ $[\text{M}+\text{H}]^+$: Calculated 318.0350, found 318.0345.



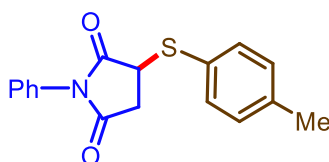
1-Phenyl-3-(phenylthio)pyrrolidine-2,5-dione (3b).

Yield 51.0 mg, 90%; White solid; $R_f = 0.2$ (15% EtOAc in petroleum ether, TLC). ^1H 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 $\text{C}_{16}\text{H}_{14}\text{NO}_2\text{S}$ $[\text{M}+\text{H}]^+$: Calculated 284.0740, found 284.0736.



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

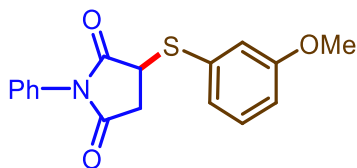
Yield 64.5 mg, 89%; White solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). ^1H NMR (300 MHz, CDCl_3) δ 7.51 – 7.39 (m, 7H), 7.09 (d, $J = 6.8$ Hz, 2H), 4.14 (dd, $J = 9.3, 4.0$ Hz, 1H), 3.38 – 3.29 (m, 1H), 2.89 – 2.81 (m, 1H). ^{13}C NMR (75 MHz, CDCl_3) δ 174.3, 173.2, 136.2, 132.6, 131.4, 129.3, 129.2, 128.9, 126.2, 124.2, 44.0, 36.2. ESI-MS (m/z) for $\text{C}_{16}\text{H}_{13}\text{BrNO}_2\text{S}$ $[\text{M}+\text{H}]^+$: Calculated 361.9845, found 361.9841.



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

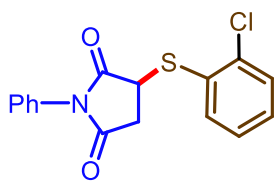
Yield 54.1 mg, 91%; White solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). ^1H NMR (300 MHz, CDCl_3) δ 7.47 – 7.36 (m, 5H), 7.17 (d, $J = 7.7$ Hz, 2H), 7.04 (d, $J = 7.4$ Hz, 2H), 4.07 (dd, $J = 9.5, 3.8$ Hz, 1H), 3.33 – 3.24 (m, 1H), 2.92 – 2.84 (m, 1H), 2.36 (s, 3H). ^{13}C NMR

(75 MHz, CDCl₃) 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 C₁₇H₁₆NO₂S [M+H]⁺: Calculated 298.0896, found 298.0891.



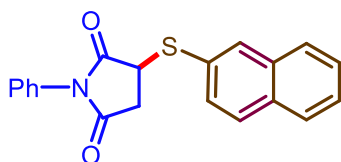
3-((3-Methoxyphenyl)thio)-1-phenylpyrrolidine-2,5-dione (3e).

Yield 57.6 mg, 92%; Yellowish solid; R_f = 0.2 (15% EtOAc in petroleum ether, TLC). ¹H NMR (300 MHz, CDCl₃) 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). ¹³C NMR (75 MHz, CDCl₃) 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 C₁₇H₁₆NO₃S [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). ¹H NMR (300 MHz, CDCl₃) 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). ¹³C NMR (75 MHz, CDCl₃) 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 C₁₆H₁₃ClNO₂S [M+H]⁺: Calculated 318.0350, found 318.0347.

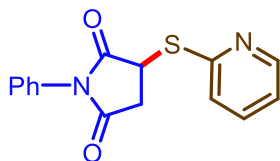


3-(Naphthalen-2-ylthio)-1-phenylpyrrolidine-2,5-dione (3g).

Yield 58.0 mg, 87%; White crystalline solid; R_f = 0.2 (15% EtOAc in petroleum ether, TLC). ¹H NMR (300 MHz, CDCl₃) 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). ¹³C NMR (75 MHz, CDCl₃) δ 174.6, 173.4, 134.7, 133.5,

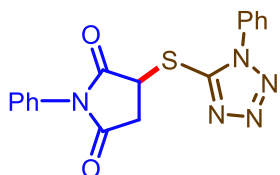
133.3, 131.5, 130.9, 129.2, 129.1, 128.8, 127.8, 127.8, 127.3, 127.2, 127.0, 126.3, 44.2, 36.3.

ESI-MS (m/z) for C₂₀H₁₆NO₂S [M+H]⁺: Calculated 334.0896, found 334.0890.



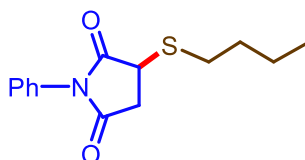
1-Phenyl-3-(pyridin-2-ylthio)pyrrolidine-2,5-dione (3h).

Yield 46.6 mg, 82%; Yellow solid; R_f = 0.2 (15% EtOAc in petroleum ether, TLC). **¹H NMR (300 MHz, CDCl₃)** 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). **¹³C NMR (75 MHz, CDCl₃)** 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 C₁₅H₁₃N₂O₂S [M+H]⁺:** Calculated 285.0692, found 285.0687.



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

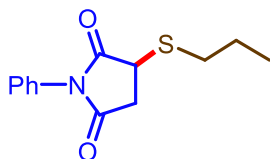
Yield 54.8 mg, 78%; Yellow solid; R_f = 0.2 (20% EtOAc in petroleum ether, TLC). **¹H NMR (300 MHz, CDCl₃)** δ 7.58 (s, 5H), 7.55 – 7.37 (m, 5H), 4.62 (dd, *J* = 9.6, 5.7 Hz, 1H), 3.61 – 3.51 (m, 1H), 3.34 – 3.26 (m, 1H). **¹³C NMR (75 MHz, CDCl₃)** δ 172.8, 172.5, 151.2, 133.1, 131.8, 130.6, 130.0, 129.3, 129.1, 126.6, 123.7, 42.8, 36.4. **ESI-MS (m/z) for C₁₇H₁₄N₅O₂S [M+H]⁺:** Calculated 352.0863, found 352.0867.



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

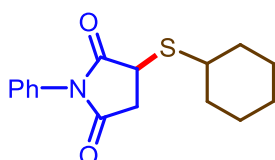
Yield 37.9 mg, 72%; Yellowish sticky solid; R_f = 0.2 (8% EtOAc in petroleum ether, TLC). **¹H NMR (300 MHz, CDCl₃)** δ 7.50 – 7.39 (m, 3H), 7.29 (d, *J* = 7.8 Hz, 2H), 3.88 – 3.84 (m, 1H), 3.34 – 3.25 (m, 1H), 3.00 – 2.91 (m, 1H), 2.86 – 2.76 (m, 1H), 2.71 – 2.63 (m, 1H), 1.65 (q, *J* = 6.9 Hz, 2H), 1.44 (q, *J* = 7.3 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). **¹³C NMR (75 MHz, CDCl₃)**

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.



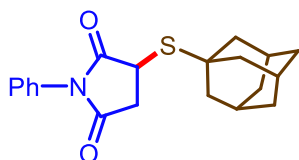
1-Phenyl-3-(propylthio)pyrrolidine-2,5-dione (3k).

Yield 34.9 mg, 70%; Brown liquid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). **1H 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).

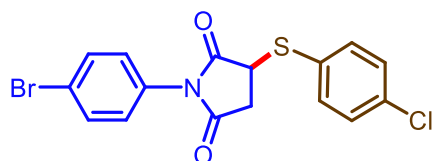
Yield 39.9 mg, 69%; Pale white solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). **1H NMR (300 MHz, $CDCl_3$)** δ 7.48 (t, $J = 7.4$ Hz, 2H), 7.41 (d, $J = 7.2$ Hz, 1H), 7.32 – 7.27 (m, 2H), 3.96 (dd, $J = 9.1, 3.6$ Hz, 1H), 3.35 – 3.25 (m, 1H), 3.25 (d, $J = 3.3$ Hz, 1H), 2.71 – 2.63 (m, 1H), 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).

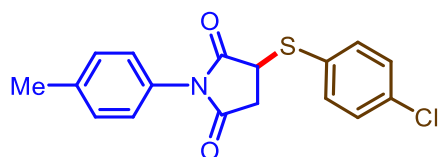
Yield 44.3 mg, 65%; White solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). **1H NMR (300 MHz, $CDCl_3$)** δ 7.46 (dd, $J = 8.3, 6.5$ Hz, 2H), 7.41 – 7.35 (m, 1H), 7.28 (dd, $J = 7.3, 2.1$ Hz, 2H), 4.01 (dd, $J = 9.4, 4.5$ Hz, 1H), 3.41 – 3.31 (m, 1H), 2.85 – 2.77 (m, 1H), 2.11 – 2.09 (m, 3H), 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 C₂₀H₂₄NO₂S [M+H]⁺**: Calculated 342.1522, found 342.1515.



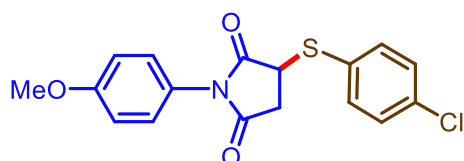
1-(4-Bromophenyl)-3-((4-chlorophenyl)thio)pyrrolidine-2,5-dione (3n).

Yield 61.9 mg, 78%; Off-white solid; $R_f = 0.2$ (20% EtOAc in petroleum ether, TLC). **¹H NMR (300 MHz, CDCl₃)** 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). **¹³C NMR (75 MHz, CDCl₃)** 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 C₁₆H₁₂BrClNO₂S [M+H]⁺**: Calculated 395.9455, found 395.9448.



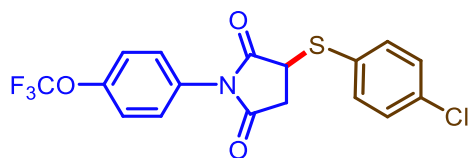
3-((4-Chlorophenyl)thio)-1-(p-tolyl)pyrrolidine-2,5-dione (3o).

Yield 52.4 mg, 79%; White solid; $R_f = 0.2$ (20% EtOAc in petroleum ether, TLC). **¹H NMR (300 MHz, CDCl₃)** 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). **¹³C NMR (75 MHz, CDCl₃)** 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 C₁₇H₁₅ClNO₂S [M+H]⁺**: Calculated 332.0507, found 332.0501.



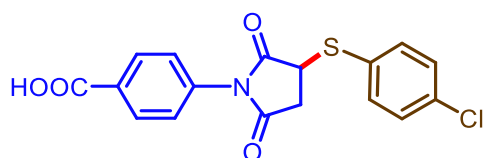
3-((4-Chlorophenyl)thio)-1-(4-methoxyphenyl)pyrrolidine-2,5-dione (3p).

Yield 59.8 mg, 86%; White solid; $R_f = 0.2$ (20% EtOAc in petroleum ether, TLC). **¹H NMR (300 MHz, CDCl₃)** 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). **¹³C NMR (75 MHz, CDCl₃)** 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 C₁₇H₁₅ClNO₃S [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). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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. $^{19}\text{F NMR}$ (377 MHz, CDCl_3) δ -57.87. ESI-MS (m/z) for $\text{C}_{17}\text{H}_{12}\text{ClF}_3\text{NO}_3\text{S}$ $[\text{M}+\text{H}]^+$: Calculated 402.0173, found 402.0168.



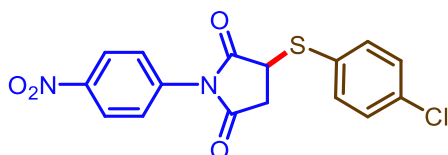
4-(3-((4-Chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoic acid (3r).

Yield 55.7 mg, 77%; Pinkish solid; $R_f = 0.2$ (30% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{17}\text{H}_{13}\text{ClNO}_4\text{S}$ $[\text{M}+\text{H}]^+$: Calculated 362.0248, found 362.0242.



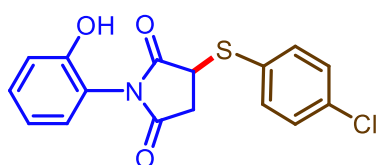
1-(3-Chloro-4-fluorophenyl)-3-((4-chlorophenyl)thio)pyrrolidine-2,5-dione (3s).

Yield 57.8 mg, 78%; Pale yellow solid; $R_f = 0.25$ (20% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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. $^{19}\text{F NMR}$ (377 MHz, CDCl_3) δ -113.73. ESI-MS (m/z) for $\text{C}_{16}\text{H}_{11}\text{Cl}_2\text{FNO}_2\text{S}$ $[\text{M}+\text{H}]^+$: Calculated 369.9866, found 369.9860.



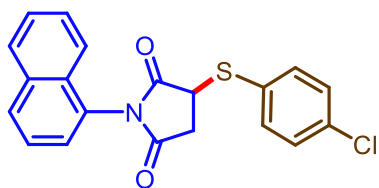
3-((4-Chlorophenyl)thio)-1-(4-nitrophenyl)pyrrolidine-2,5-dione (3t).

Yield 53.7 mg, 74%; Yellow solid; $R_f = 0.2$ (15% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{16}\text{H}_{12}\text{ClN}_2\text{O}_4\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 363.0201, found 363.0194.



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

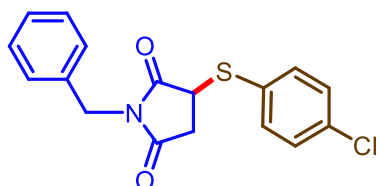
Yield 51.4 mg, 77%; Brownish semi-solid; $R_f = 0.2$ (30% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.91 (s, 1H), 7.48 (d, $J = 6.4$ Hz, 2H), 7.29 – 7.26 (m, 2H), 7.20 (t, $J = 7.8$ Hz, 1H), 6.93 (d, $J = 8.3$ Hz, 1H), 6.84 (t, $J = 7.6$ Hz, 1H), 6.74 (d, $J = 8.1$ Hz, 1H), 4.13 (dd, $J = 8.8, 4.1$ Hz, 1H), 3.32 (d, $J = 8.4$ Hz, 1H), 2.34 – 2.27 (m, 1H). $^{13}\text{C NMR}$ (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 $\text{C}_{16}\text{H}_{13}\text{ClNO}_3\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 334.0299, found 334.0292.



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

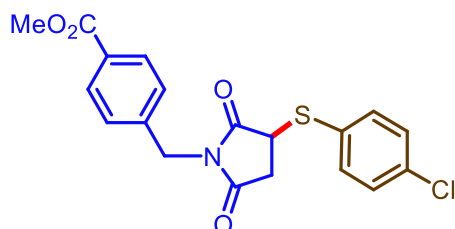
Yield 59.6 mg, 81%; Light brownish liquid; $R_f = 0.2$ (20% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.96 – 7.89 (m, 2H, isomers mixture), 7.58 (dd, $J = 8.5, 2.1$ Hz, 2H, isomers mixture), 7.51 (d, $J = 7.9$ Hz, 2H, isomers mixture), 7.45 (t, $J = 7.6$ Hz, 1H, isomers mixture), 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). $^{13}\text{C NMR}$ (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 $\text{C}_{20}\text{H}_{15}\text{ClNO}_2\text{S}$ $[\text{M}+\text{H}]^+$** : Calculated 368.0507, found 368.0502.



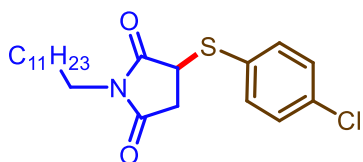
1-Benzyl-3-((4-chlorophenyl)thio)pyrrolidine-2,5-dione (3w).

Yield 48.4 mg, 73%; Pale brownish solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). **^1H 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 $\text{C}_{17}\text{H}_{15}\text{ClNO}_2\text{S}$ $[\text{M}+\text{H}]^+$** : Calculated 332.0507, found 332.0509.



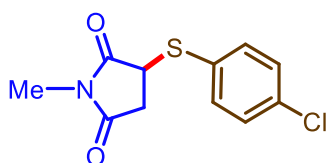
Methyl 4-((3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)methyl)benzoate (3x).

Yield 56.1 mg, 72%; White solid; $R_f = 0.2$ (20% EtOAc in petroleum ether, TLC). **^1H 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 $\text{C}_{19}\text{H}_{17}\text{ClNO}_4\text{S}$ $[\text{M}+\text{H}]^+$** : Calculated 390.0561, found 390.0554.



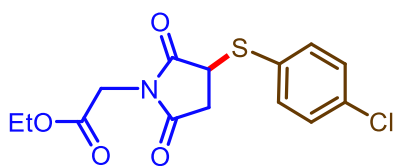
4-((4-Chlorophenyl)thio)-1-dodecylpyrrolidin-2-one (3y).

Yield 56.6 mg, 69%; Off-white solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{22}\text{H}_{33}\text{ClNO}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 410.1915, found 410.1921.



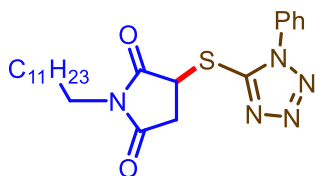
3-((4-Chlorophenyl)thio)-1-methylpyrrolidine-2,5-dione (3z).

Yield 35.8 mg, 70%; White solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{11}\text{H}_{11}\text{ClNO}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 256.0194, found 256.0199.



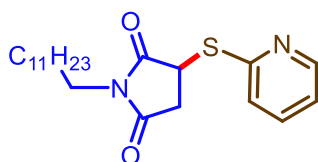
Ethyl 2-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)acetate (3aa).

Yield 48.5 mg, 74%; Pale yellowish solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{14}\text{H}_{15}\text{ClNO}_4\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 328.0405, found 328.0401.



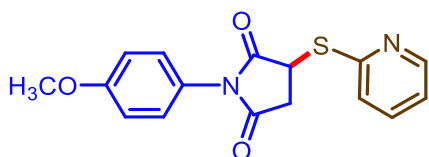
1-Dodecyl-3-((1-phenyl-1H-tetrazol-5-yl)thio)pyrrolidine-2,5-dione (3ab).

Yield 64.8 mg, 73%; Yellow solid; $R_f = 0.2$ (20% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{23}\text{H}_{34}\text{N}_5\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 444.2428, found 444.2434.



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

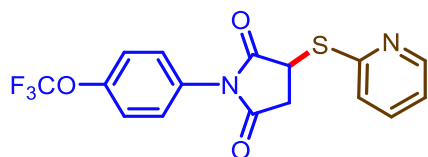
Yield 57.2 mg, 76%; Reddish brown liquid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.20 (d, $J = 5.0$ Hz, 1H), 7.52 – 7.46 (m, 1H), 7.17 (d, $J = 8.1$ Hz, 1H), 6.98 (dd, $J = 7.6, 4.7$ Hz, 1H), 4.20 (dd, $J = 9.4, 5.5$ Hz, 1H), 3.58 – 3.53 (m, 2H), 3.24 – 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). $^{13}\text{C NMR}$ (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 $\text{C}_{21}\text{H}_{33}\text{N}_2\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 377.2257, found 377.2253.



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

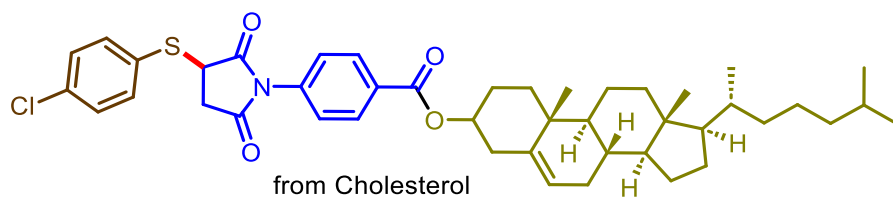
Yield 49.7 mg, 79%; Off-white solid; $R_f = 0.2$ (15% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.31 (d, $J = 4.4$ Hz, 1H), 7.55 (t, $J = 7.7$ Hz, 1H), 7.29 – 7.27 (m, 2H), 7.23 (d, $J = 8.1$ Hz, 1H), 7.02 (t, $J = 9.0$ Hz, 3H), 4.28 (dd, $J = 9.6, 5.5$ Hz, 1H), 3.84 (s, 3H), 3.43 – 3.34 (m, 1H), 3.16 – 3.08 (m, 1H). $^{13}\text{C NMR}$ (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 C₁₆H₁₅N₂O₃S [M+H]⁺**: Calculated 315.0798, found 315.0791.



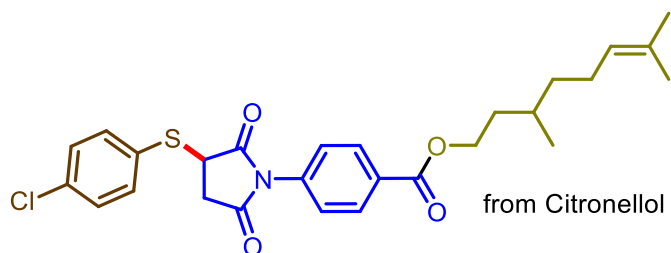
3-(Pyridin-2-ylthio)-1-(4-(trifluoromethoxy)phenyl)pyrrolidine-2,5-dione (3ae).

Yield 57.4 mg, 78%; Off-white solid; $R_f = 0.2$ (15% EtOAc in petroleum ether, TLC). **¹H NMR (300 MHz, CDCl₃)** 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). **¹³C NMR (75 MHz, CDCl₃)** 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. **¹⁹F NMR (377 MHz, CDCl₃)** δ -57.83. **ESI-MS (m/z) for C₁₆H₁₂F₃N₂O₃S [M+H]⁺**: Calculated 369.0515, found 369.0522.



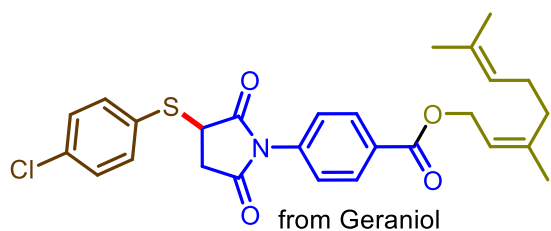
(8S,9S,10R,13R,14S,17R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl 4-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoate (5a).

Yield 105.2 mg, 72%; Yellow ochre solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). **¹H NMR (300 MHz, CDCl₃)** 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). **¹³C NMR (75 MHz, CDCl₃)** 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 C₄₄H₅₇ClNO₄S [M+H]⁺**: Calculated 730.3691, found 730.3682.



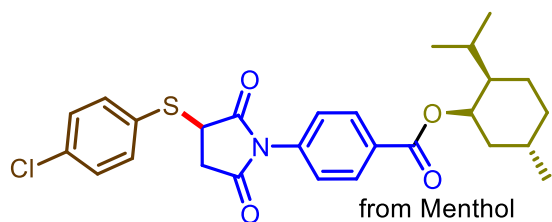
3,7-Dimethyloct-6-en-1-yl 4-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoate (5b).

Yield 70.0 mg, 70%; Pale white solid; $R_f = 0.2$ (15% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{27}\text{H}_{31}\text{ClNO}_4\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 500.1657, found 500.1649.



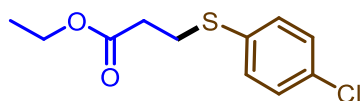
(Z)-3,7-dimethylocta-2,6-dien-1-yl 4-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoate (5c).

Yield 67.7 mg, 68%; Pale yellow solid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{14}\text{H}_{15}\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 500.1657, found 247.0798. **ESI-MS (m/z) for $\text{C}_{27}\text{H}_{29}\text{ClNO}_4\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 498.1500, found 498.1492.



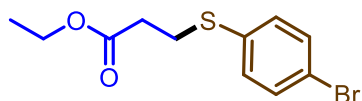
2-Isopropyl-5-methylcyclohexyl 4-(3-((4-chlorophenyl)thio)-2,5-dioxopyrrolidin-1-yl)benzoate (5d).

Yield 73.0 mg, 73%; Pale yellow solid; $R_f = 0.2$ (12% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{27}\text{H}_{31}\text{ClNO}_4\text{S}$ $[\text{M}+\text{H}]^+$:** 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). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{11}\text{H}_{14}\text{ClO}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 245.0398, found 245.0393.

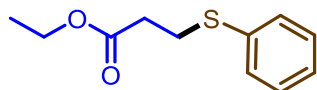


Ethyl 3-((4-bromophenyl)thio)propanoate (7b).

Yield 46.8 mg, 81%; Pale yellow solid; $R_f = 0.2$ (3% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$

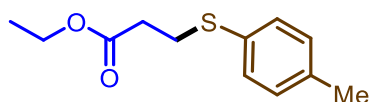
NMR (101 MHz, CDCl₃) δ 171.49, 134.61, 132.06, 131.48, 120.41, 60.80, 34.26, 29.06, 14.2.

ESI-MS (m/z) for C₁₁H₁₄BrO₂S [M+H]⁺: Calculated 288.9892, found 288.9897.



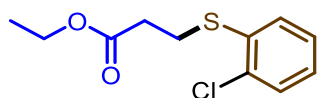
Ethyl 3-(phenylthio)propanoate (7c).

Yield 33.6 mg, 80%; Yellow liquid; R_f = 0.2 (2% EtOAc in petroleum ether, TLC). ¹H NMR (300 MHz, CDCl₃) 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). ¹³C NMR (75 MHz, CDCl₃) δ 171.4, 135.4, 129.8, 128.9, 126.4, 60.5, 34.3, 28.9, 14.1. ESI-MS (m/z) for C₁₁H₁₅O₂S [M+H]⁺: Calculated 211.0787, found 211.0783.



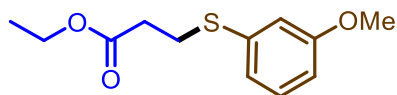
Ethyl 3-(p-tolylthio)propanoate (7d).

Yield 38.1 mg, 85%; Light yellow liquid; R_f = 0.2 (3% EtOAc in petroleum ether, TLC). ¹H NMR (300 MHz, CDCl₃) 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). ¹³C NMR (75 MHz, CDCl₃) 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 C₁₂H₁₇O₂S [M+H]⁺: Calculated 225.0944, found 225.0949.



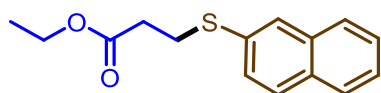
Ethyl 3-((2-chlorophenyl)thio)propanoate (7e).

Yield 36.7 mg, 75%; Whitish liquid; R_f = 0.2 (2% EtOAc in petroleum ether, TLC). ¹H NMR (300 MHz, CDCl₃) 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). ¹³C NMR (75 MHz, CDCl₃) 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 C₁₁H₁₄ClO₂S [M+H]⁺: Calculated 245.0398, found 245.0392.



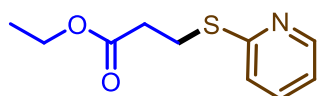
Ethyl 3-((3-methoxyphenyl)thio)propanoate (7f).

Yield 39.9 mg, 83%; Light yellowish liquid; $R_f = 0.2$ (2% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{12}\text{H}_{17}\text{O}_3\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 241.0893, found 241.0897.



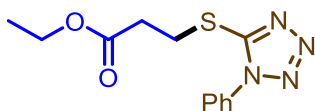
Ethyl 3-(naphthalen-2-ylthio)propanoate (7g).

Yield 42.2 mg, 81%; Light pink liquid; $R_f = 0.2$ (2% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{15}\text{H}_{17}\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 261.0944, found 261.0941.



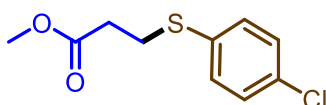
Ethyl 3-(pyridin-2-ylthio)propanoate (7h).

Yield 30.4 mg, 72%; Deep yellowish liquid; $R_f = 0.2$ (2% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{10}\text{H}_{14}\text{NO}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 212.0740, found 212.0735.



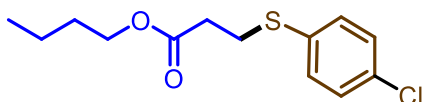
Ethyl 3-((1-phenyl-1H-tetrazol-5-yl)thio)propanoate (7i).

Yield 39.5 mg, 71%; Deep yellowish liquid; $R_f = 0.2$ (15% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{12}\text{H}_{15}\text{N}_4\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 279.0910, found 279.0915.



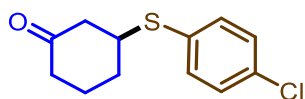
Methyl 3-((4-chlorophenyl)thio)propanoate (7j).

Yield 31.8 mg, 69%; Yellow liquid; $R_f = 0.2$ (2% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{10}\text{H}_{12}\text{ClO}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 231.0241, found 231.0236.



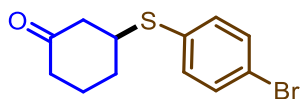
Butyl 3-((4-chlorophenyl)thio)propanoate (7k).

Yield 41.5 mg, 76%; Yellow liquid; $R_f = 0.2$ (2% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{13}\text{H}_{18}\text{ClO}_2\text{S}$ $[\text{M}+\text{H}]^+$:** Calculated 273.0711, found 273.0703.



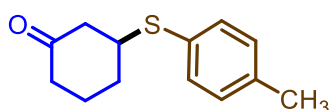
3-((4-Chlorophenyl)thio)cyclohexan-1-one (9a).

Yield 36.6 mg, 76%; Yellow sticky liquid; $R_f = 0.2$ (5% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{12}\text{H}_{14}\text{ClOS}$ $[\text{M}+\text{H}]^+$** : Calculated 241.0448, found 241.0443.



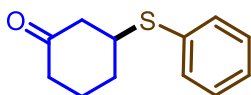
3-((4-Bromophenyl)thio)cyclohexan-1-one (9b).

Yield 42.8 mg, 75%; Brownish yellow liquid; $R_f = 0.2$ (6% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{12}\text{H}_{14}\text{BrOS}$ $[\text{M}+\text{H}]^+$** : Calculated 284.9943, found 284.9949.



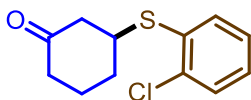
3-((p-Tolyl)thio)cyclohexan-1-one (9c).

Yield 34.8 mg, 79%; Light brownish liquid; $R_f = 0.2$ (5% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{13}\text{H}_{17}\text{OS}$ $[\text{M}+\text{H}]^+$** : Calculated 221.0995, found 221.0990.



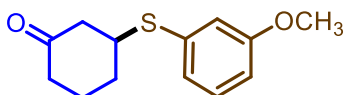
3-(Phenylthio)cyclohexan-1-one (9d).

Yield 31.4 mg, 76%; Yellow liquid; $R_f = 0.2$ (5% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{12}\text{H}_{15}\text{OS}$ $[\text{M}+\text{H}]^+$** : Calculated 207.0838, found 207.0834.



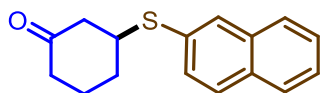
3-((2-Chlorophenyl)thio)cyclohexan-1-one (9e).

Yield 33.2 mg, 69%; Yellow-brownish liquid; $R_f = 0.2$ (10% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{12}\text{H}_{14}\text{ClOS}$ $[\text{M}+\text{H}]^+$** : Calculated 241.0448, found 241.0445.



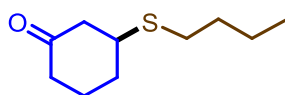
3-((3-Methoxyphenyl)thio)cyclohexan-1-one (9f).

Yield 36.4 mg, 77%; Light-yellowish liquid; $R_f = 0.2$ (75.15% EtOAc in petroleum ether, TLC). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{13}\text{H}_{17}\text{O}_2\text{S}$ $[\text{M}+\text{H}]^+$** : 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). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{16}\text{H}_{17}\text{OS}$ $[\text{M}+\text{H}]^+$: 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). $^1\text{H NMR}$ (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). $^{13}\text{C NMR}$ (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 $\text{C}_{10}\text{H}_{19}\text{OS}$ $[\text{M}+\text{H}]^+$: Calculated 187.1151, found 187.1148.

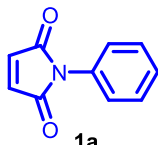
4. References

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 Carbonylation of Alkenes. *Angew. Chem. Int. Ed.* **2019**, *58*, 12286–12290.

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

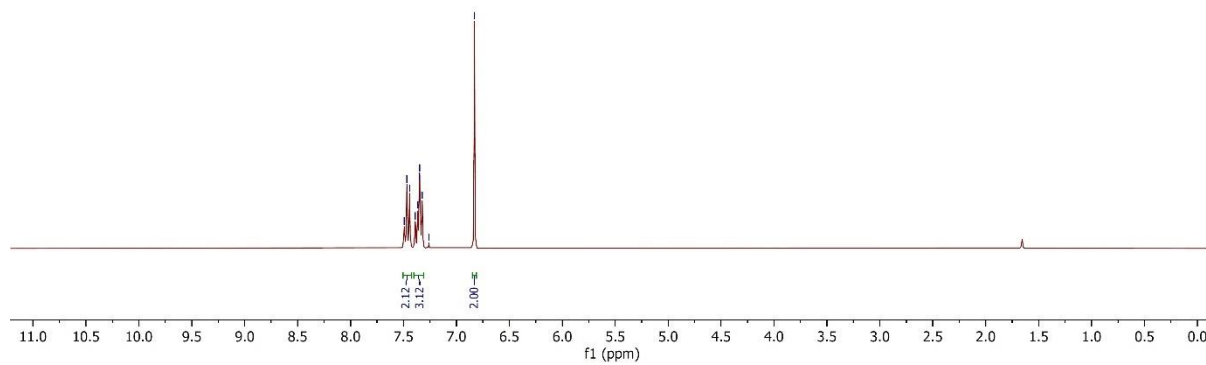
GS-90-DB-1.1.fid

7.49
7.47
7.44
7.36
7.35
7.35
7.32
7.26
6.83



1a

^1H NMR (300 MHz, CDCl_3)

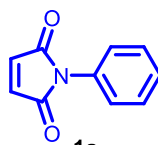


GS-90-DB-1.2.fid

169.57

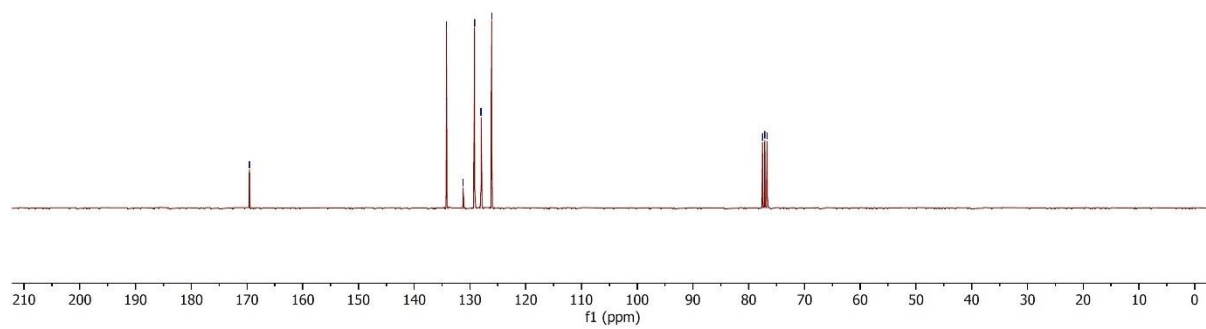
134.22
133.18
128.00
126.11

77.54
77.11
76.69



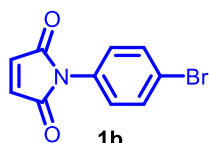
1a

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

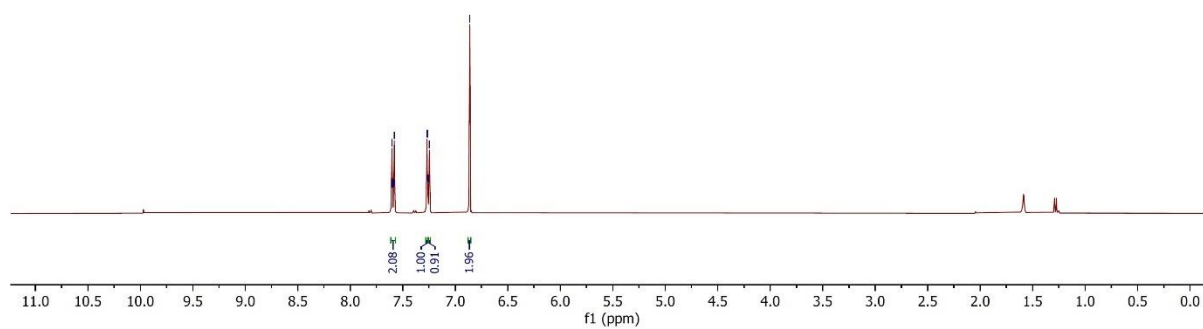


DKM-DJB-162.1.fid

7.60
7.60
7.58
7.37
7.26
7.25
-6.86



¹H NMR (400 MHz, CDCl₃)



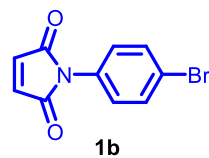
DKM-DJB-162-13C.1.fid

169.11

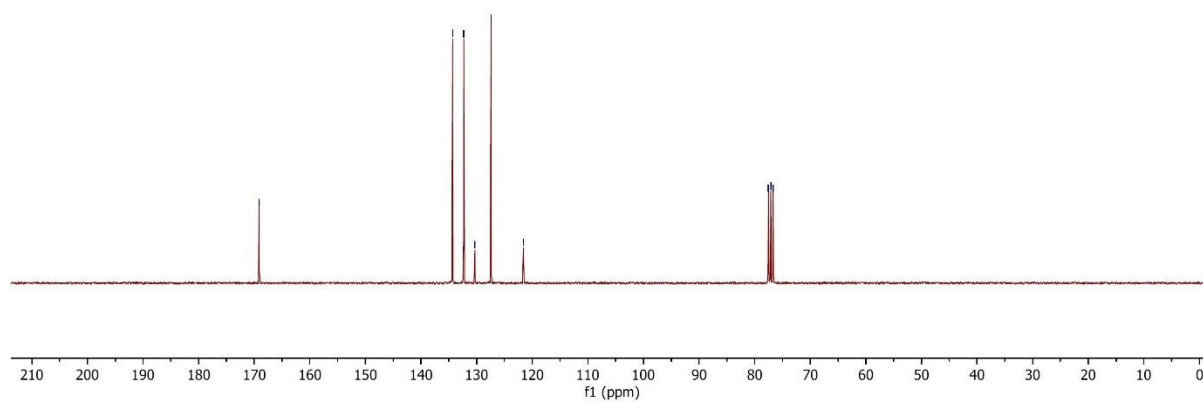
134.33
132.32
130.31
127.40

121.61

77.52
77.10
76.68



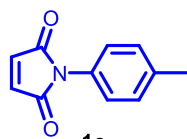
¹³C NMR (75 MHz, CDCl₃)



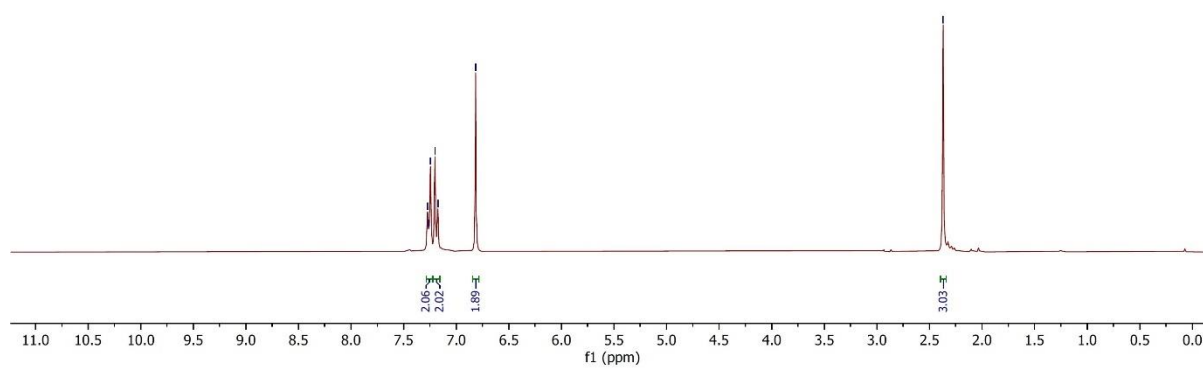
DKM-DIB-216SM.10.fid

7.27
7.26
7.24
7.20
7.17
6.81

2.37



¹H NMR (300 MHz, CDCl₃)



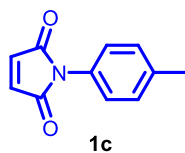
DKM-DIB-216SM.11.fid

168.74

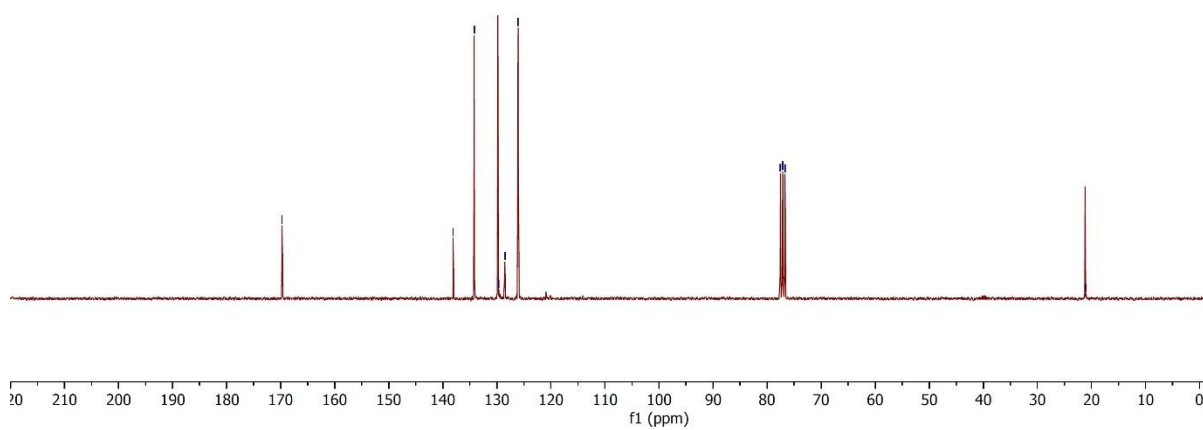
138.09
137.19
136.43
128.51
126.06

77.54
77.12
76.69

21.03



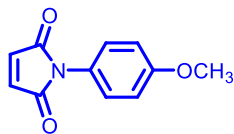
¹³C NMR (75 MHz, CDCl₃)



DKM-DJB-174.1.fid

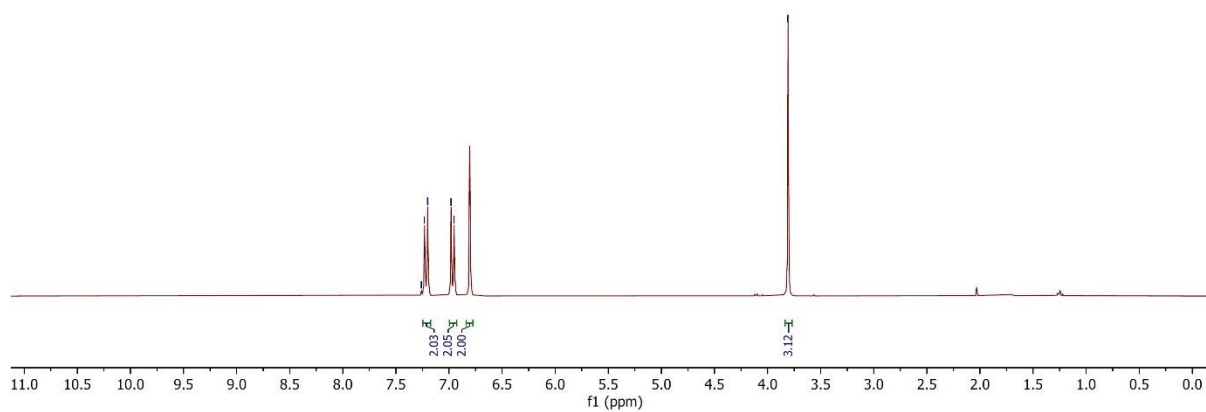
7.26
7.23
7.18
6.98
6.95

3.81



1d

¹H NMR (300 MHz, CDCl₃)



DKM-DJB-174 13C.2.fid

169.88

159.18

134.16

127.63

123.77

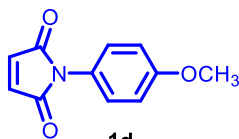
114.51

77.53

77.41

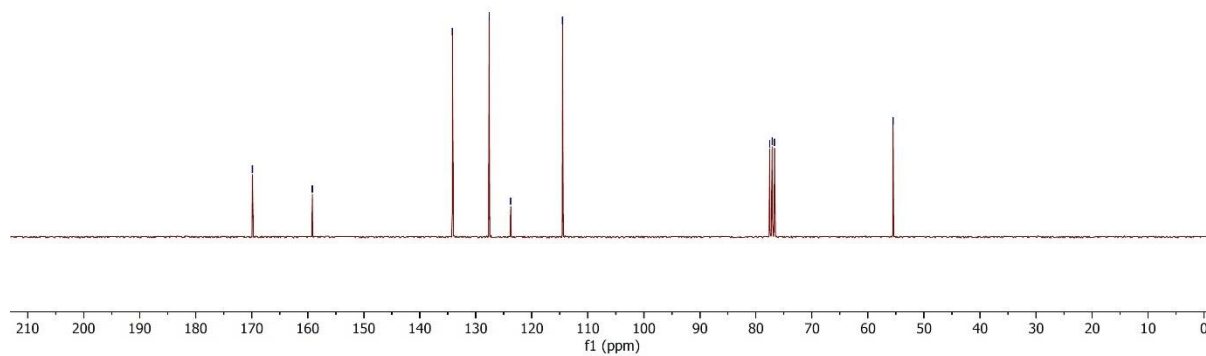
76.68

55.52



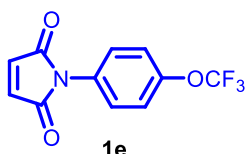
1d

¹³C NMR (75 MHz, CDCl₃)

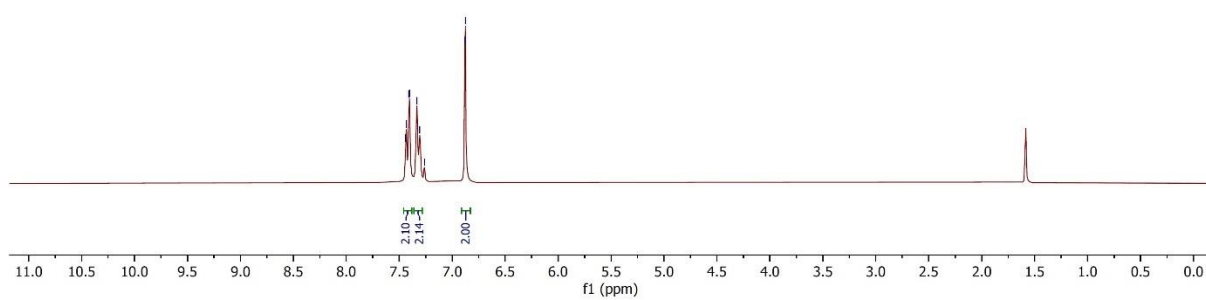


DKM-DJB-177.1.fid

7.44
7.43
7.41
7.40
7.33
7.31
7.26
6.88
6.87

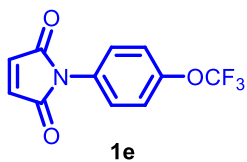


¹H NMR (300 MHz, CDCl₃)

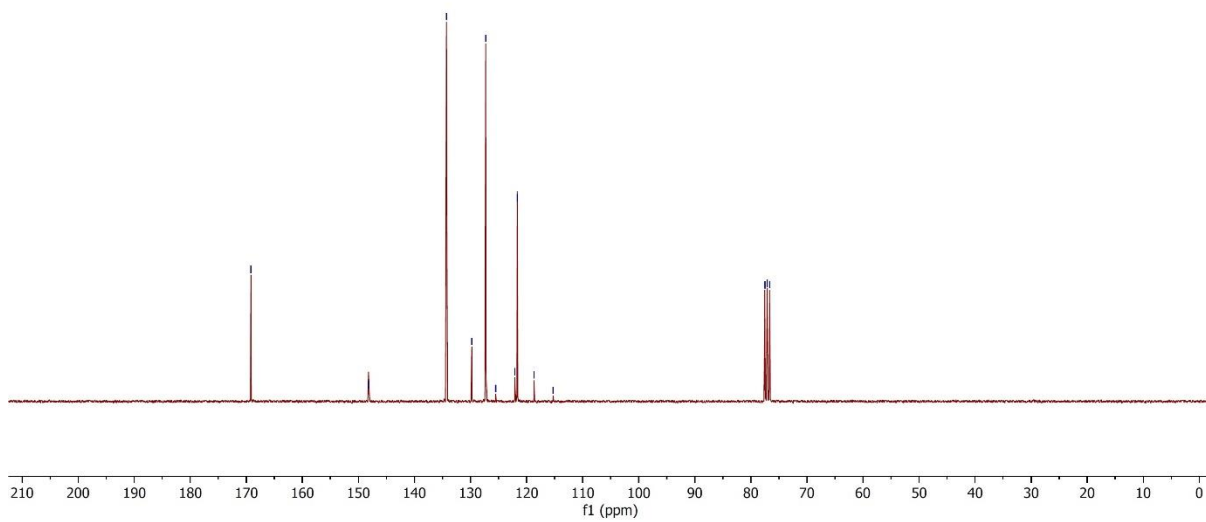


DKM-DJB-177-13C.1.fid

169.17
148.23
134.30
127.77
127.29
125.52
122.11
121.66
118.69
115.27
77.50
77.08
76.66



¹³C NMR (75 MHz, CDCl₃)



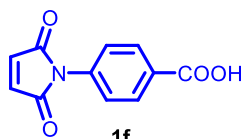
DKM-DJB-211.1.fid

10.60

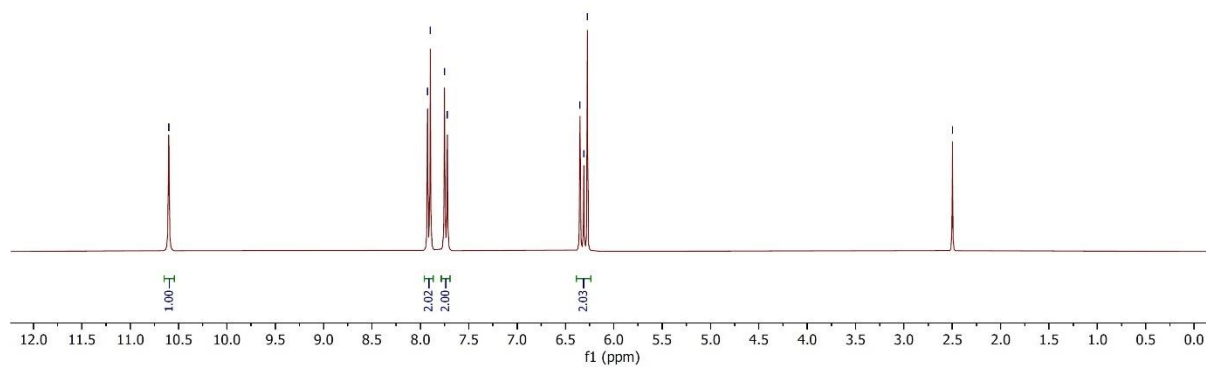
7.93
7.90
7.75
7.72

6.35
6.31
6.27

2.50



¹H NMR (300 MHz, DMSO)

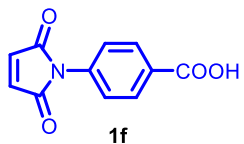


DKM-DJB-211-13C.1.fid

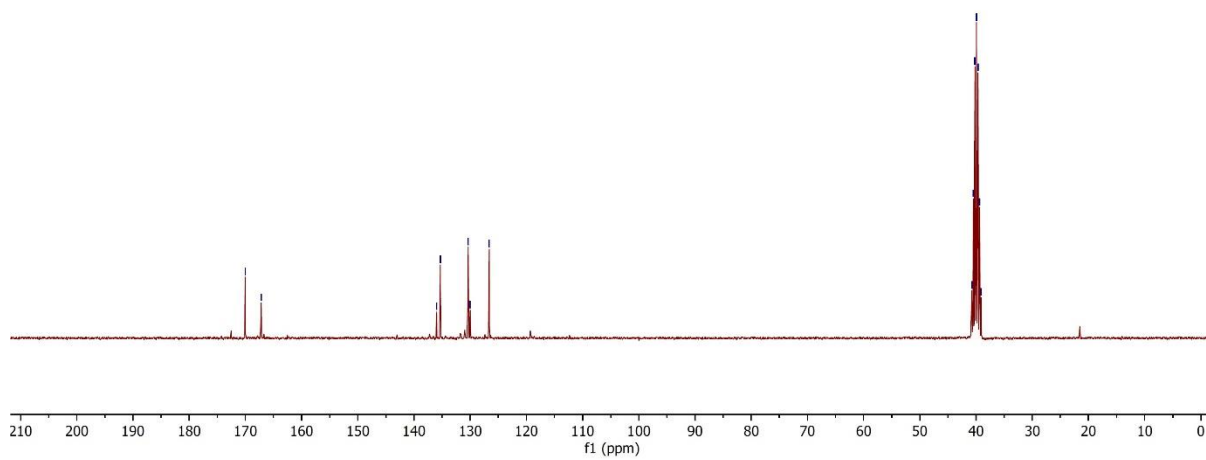
170.02
167.18

135.97
135.32
130.37
130.01
128.62

40.76
40.48
39.81
39.63
39.65
39.37
39.09

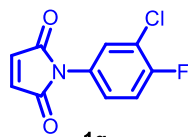


¹³C NMR (75 MHz, DMSO)



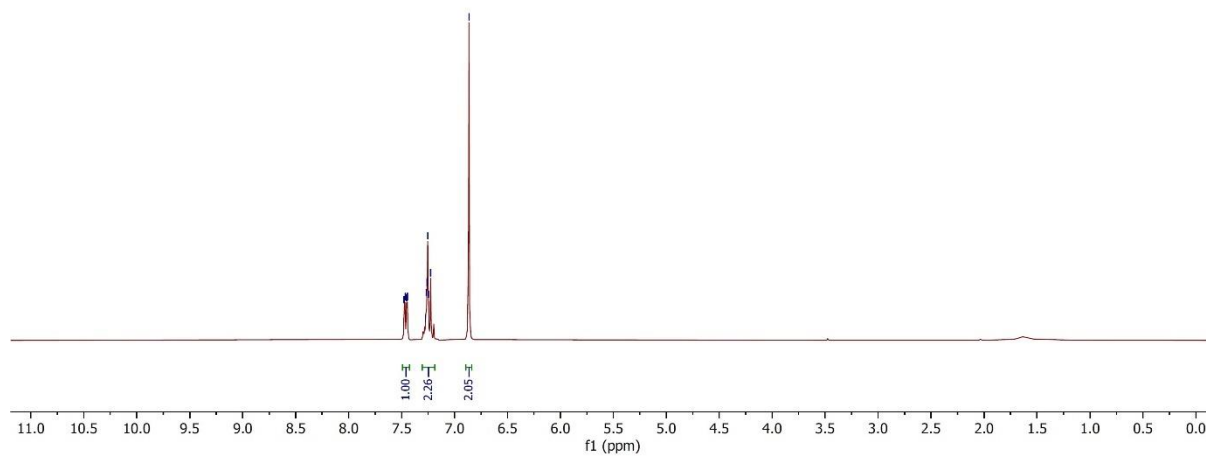
DKM-DJB-180.1.fid

7.47
7.46
7.45
7.27
7.26
7.25
7.24
7.22
-8.86



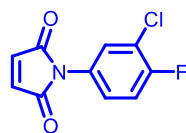
1g

¹H NMR (300 MHz, CDCl₃)



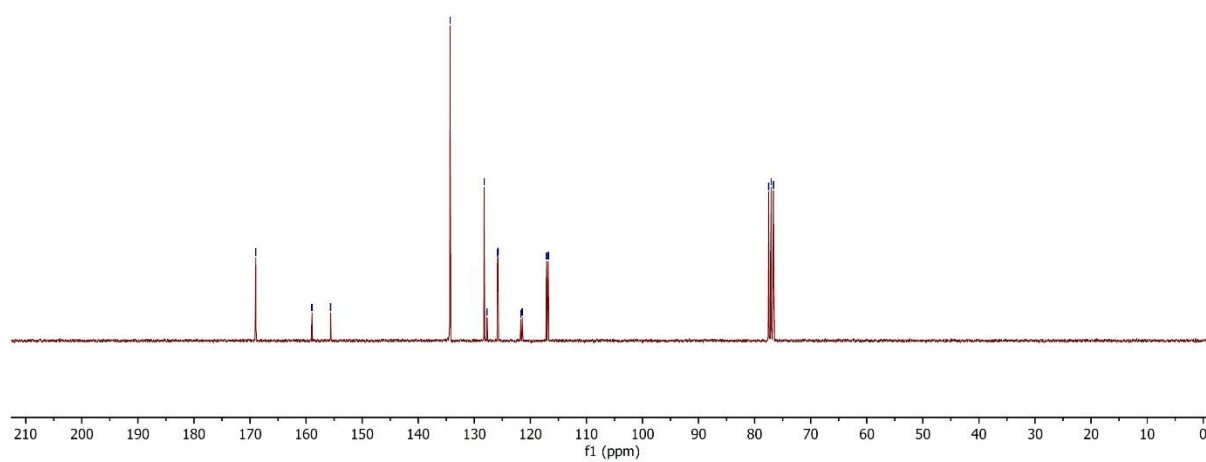
DKM-DJB-180-13C.1.fid

168.96
158.94
155.62
134.33
128.23
127.75
125.86
125.76
121.71
121.46
117.10
116.01
77.69
77.08
76.66



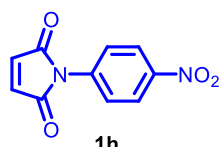
1g

¹³C NMR (75 MHz, CDCl₃)

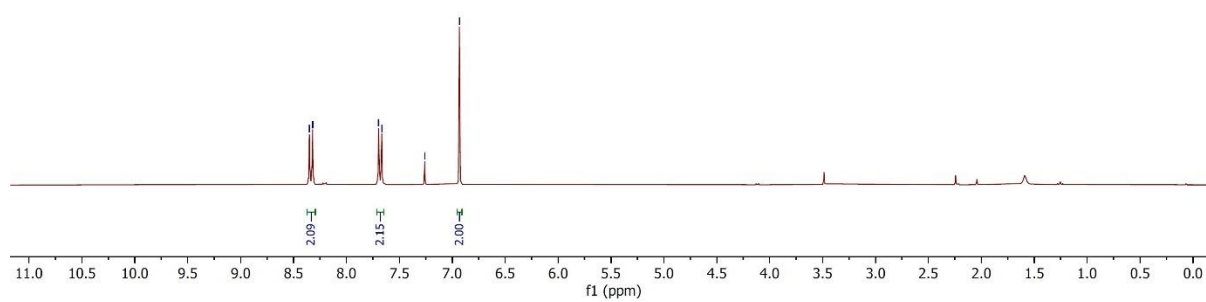


DKM-DJB-170.1.fid

8.35
8.32
7.70
7.67
7.26
6.93

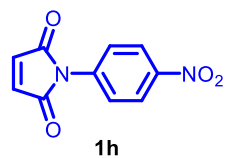


¹H NMR (300 MHz, CDCl₃)

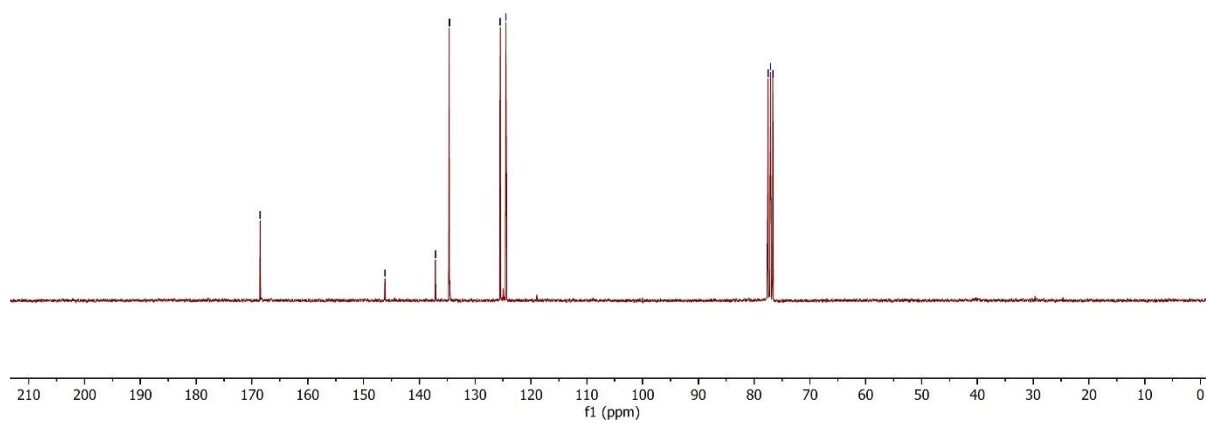


DKM-DJB-170 13C.2.fid

168.53
146.16
137.12
134.64
125.51
124.48
77.51
77.08
76.66

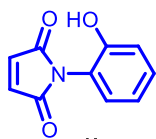


¹³C NMR (75 MHz, CDCl₃)



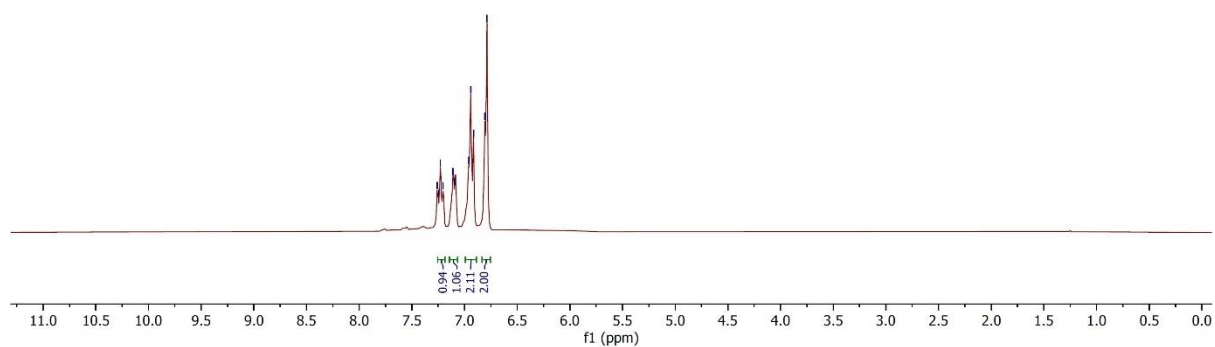
DKM-DJB-213.2.fid

7.26
7.25
7.23
7.20
7.11
7.09
6.96
6.91
6.91
6.79



1i

¹H NMR (300 MHz, CDCl₃)



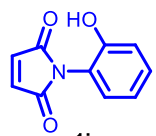
DKM-DJB-213-13C.1.fid

170.15

134.62
130.53
129.54

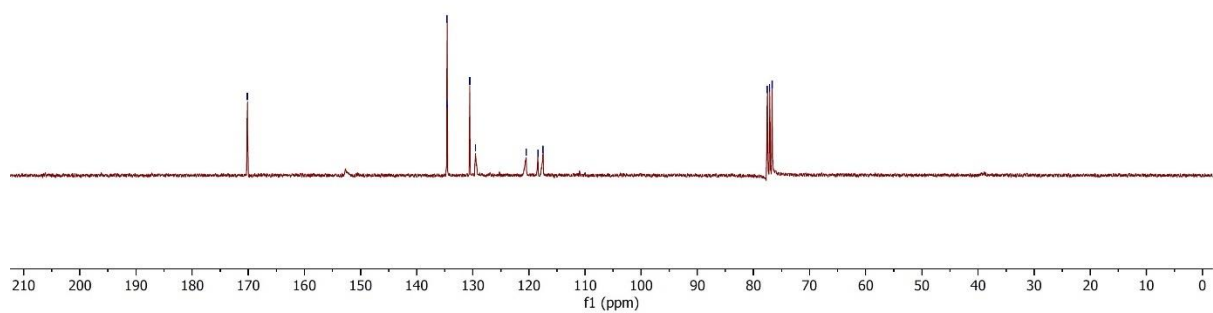
120.48
118.41
117.49

77.53
77.11
76.68



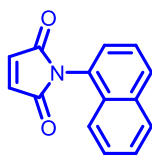
1i

¹³C NMR (75 MHz, CDCl₃)



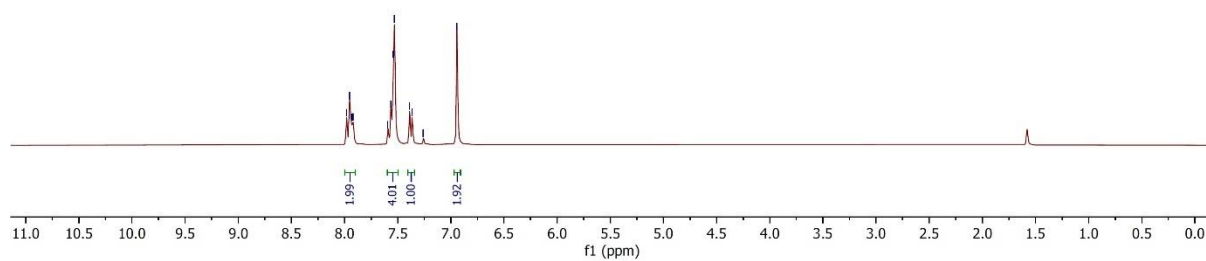
DKM-DJB-137.1.fid

7.98
7.95
7.94
7.92
7.91
7.87
7.84
7.53
7.39
7.26
6.94



1j

¹H NMR (300 MHz, CDCl₃)

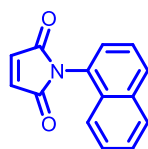


DKM-DJB-137-13C.1.fid

170.08

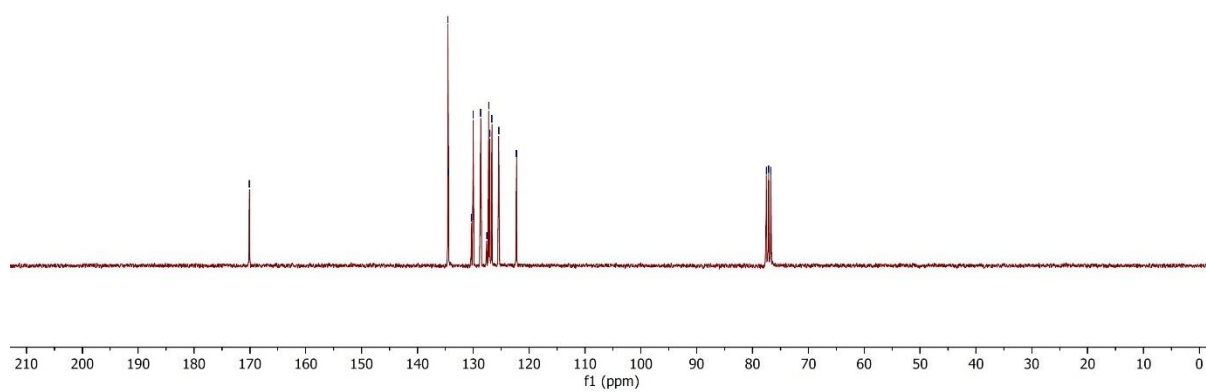
134.53
134.48
130.36
129.60
128.67
127.60
127.25
127.03
126.65
125.43
122.24

77.54
77.12
76.70



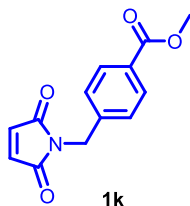
1j

¹³C NMR (75 MHz, CDCl₃)

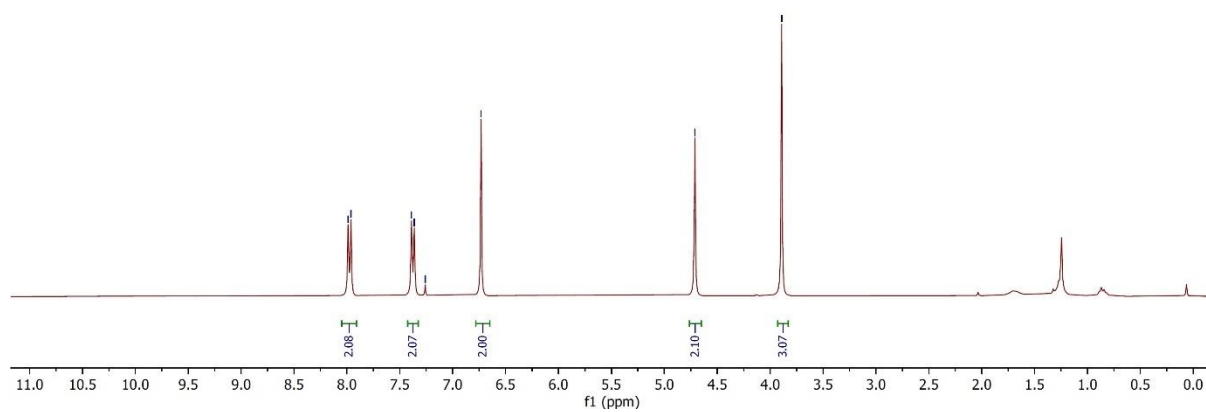


DKM-DJB-179.1.fid

7.69
7.66
7.39
7.36
7.26
6.73
4.71
3.89

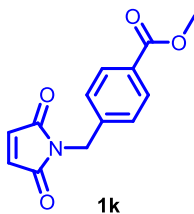


¹H NMR (300 MHz, CDCl₃)

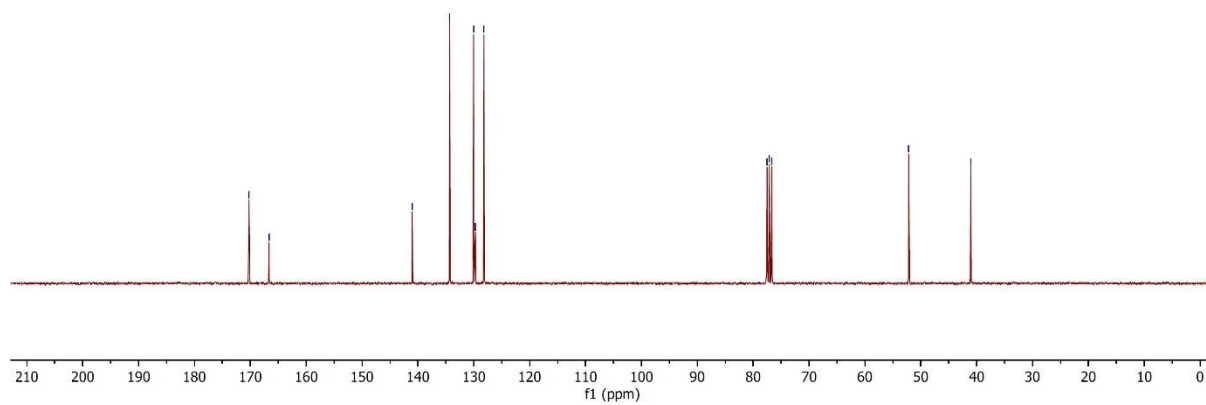


DKM-DJB-179-13C.1.fid

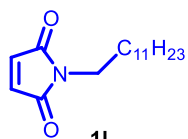
170.24
166.65
141.03
134.31
130.03
129.71
128.19
77.52
77.10
76.67
52.16
41.06



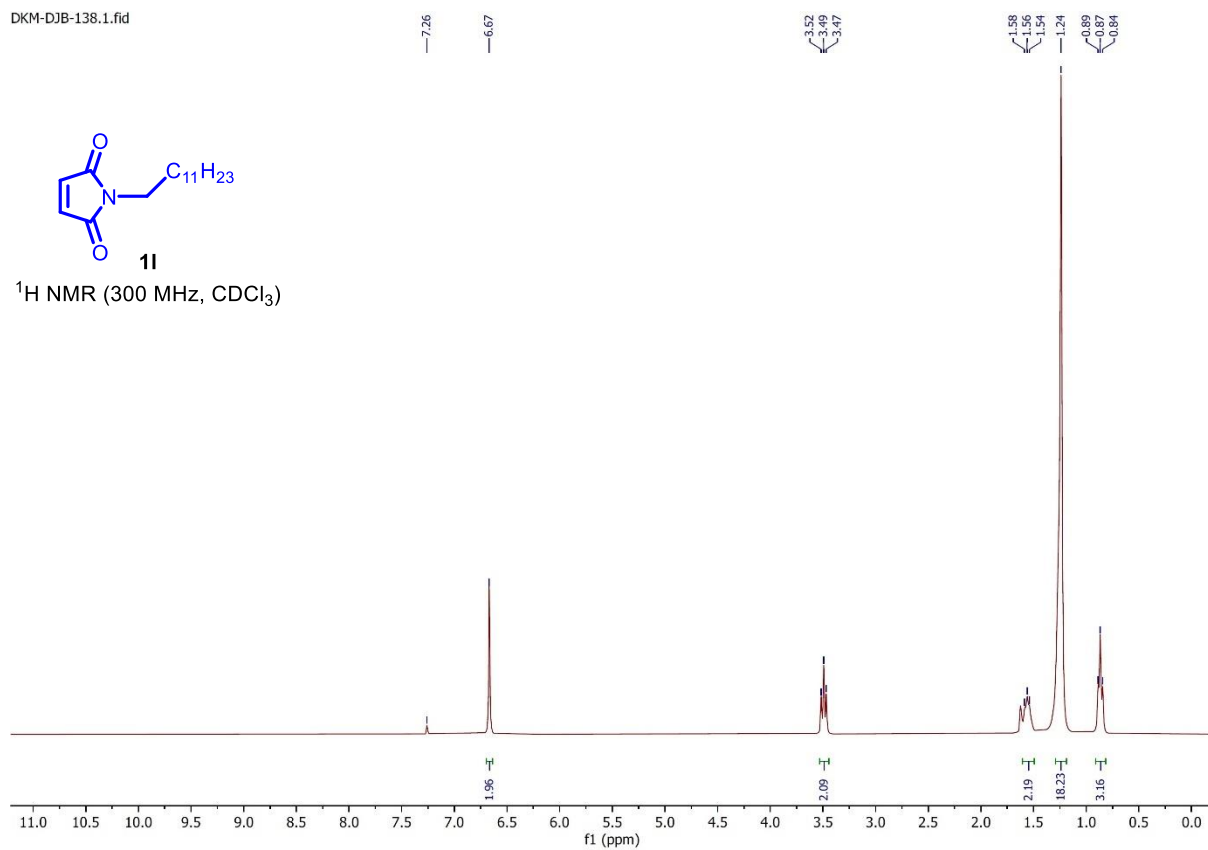
¹³C NMR (75 MHz, CDCl₃)



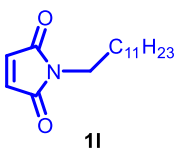
DKM-DJB-138.1.fid



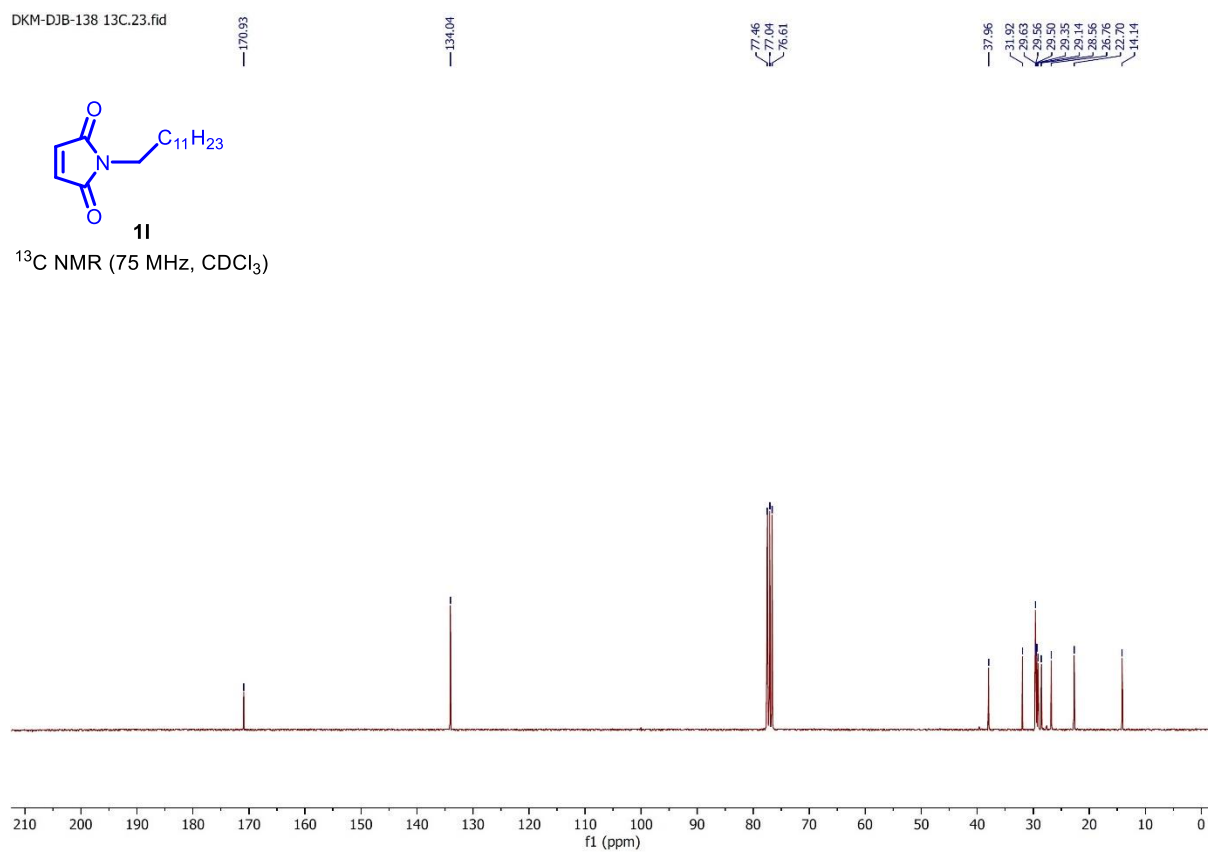
¹H NMR (300 MHz, CDCl₃)



DKM-DJB-138 13C.23.fid



¹³C NMR (75 MHz, CDCl₃)

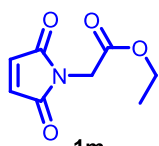


DKM-DJB-153.2.fid

7.26
7.25

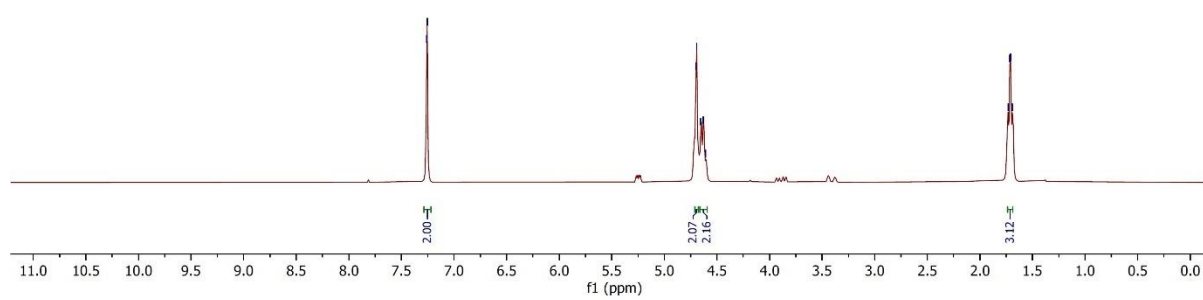
4.70
4.69
4.66
4.63
4.61

1.73
1.72
1.71
1.69



1m

¹H NMR (300 MHz, CDCl₃)



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

169.84
167.20

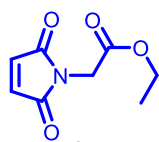
134.48

77.68
77.43
76.83

61.79

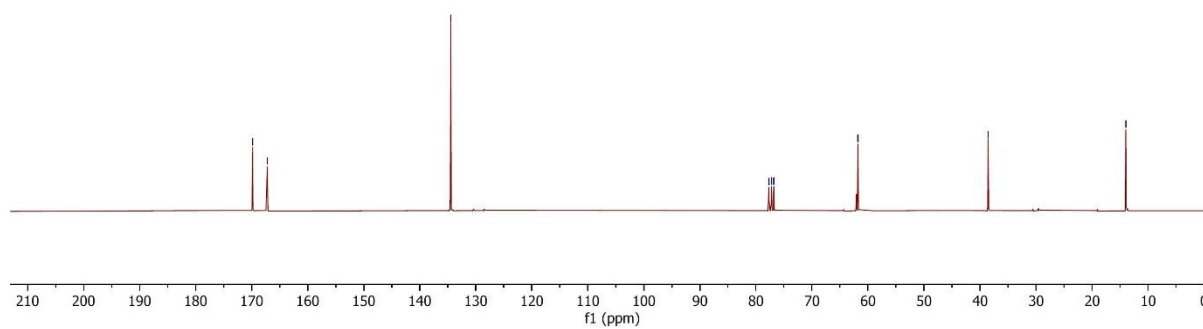
38.56

13.97



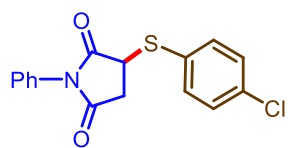
1m

¹³C NMR (75 MHz, CDCl₃)

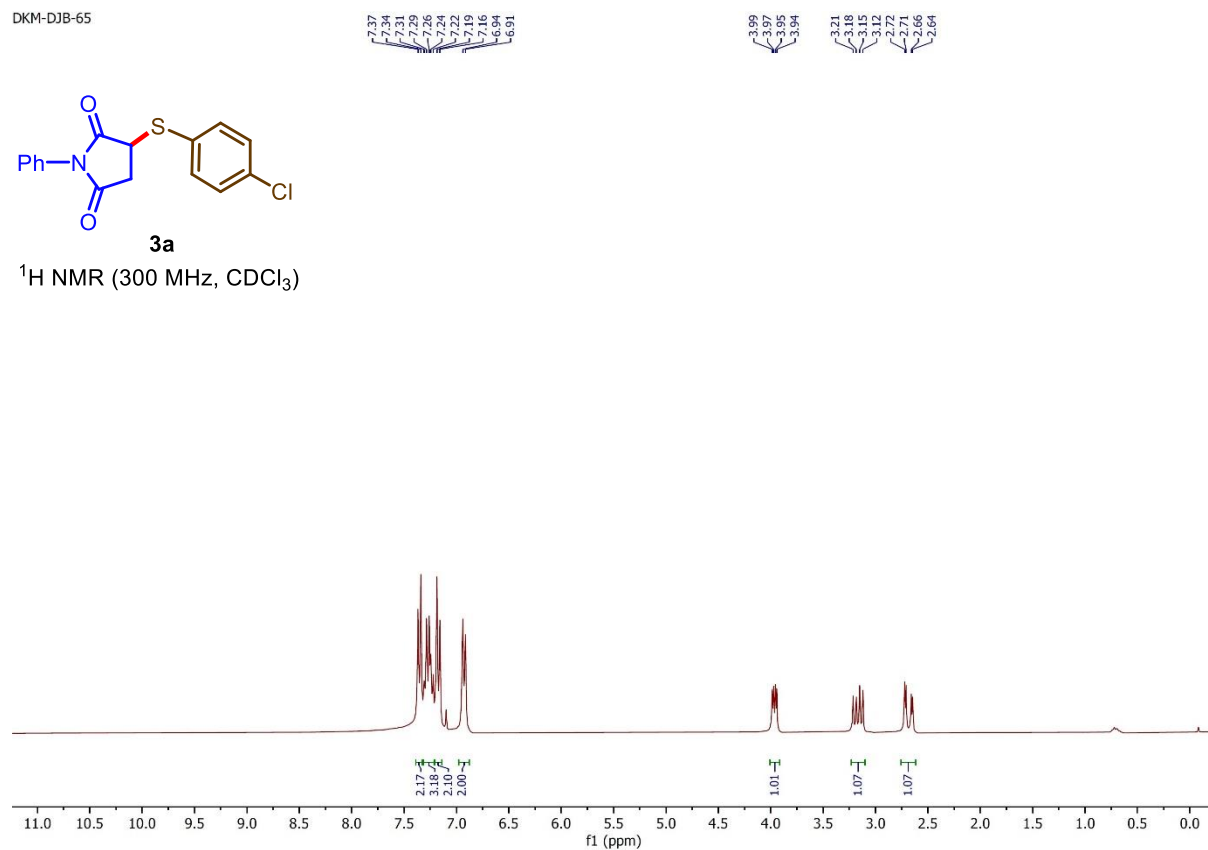


¹H and ¹³C NMR Spectra for all the Synthesized Compounds

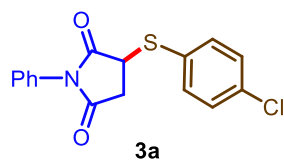
DKM-DJB-65



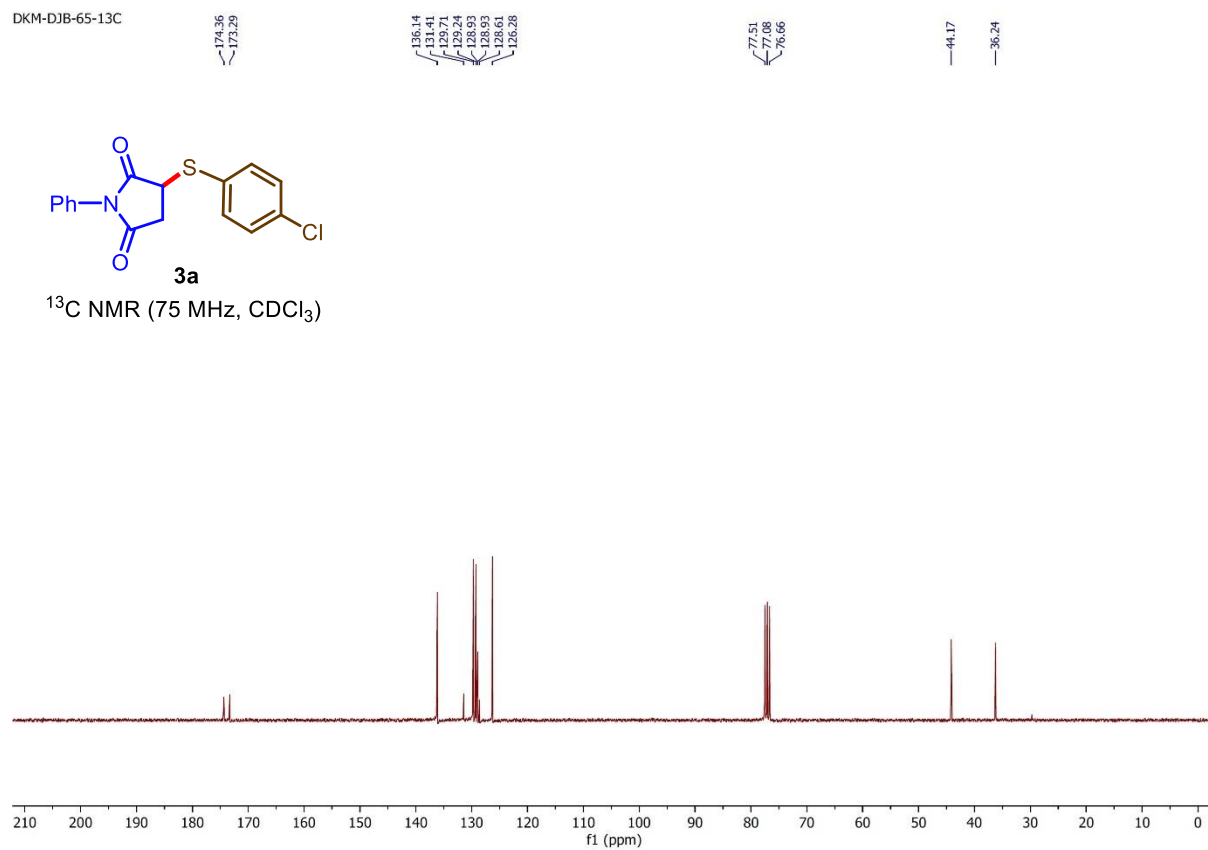
¹H NMR (300 MHz, CDCl₃)



DKM-DJB-65-13C



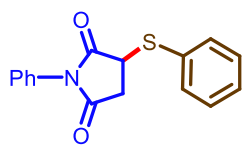
¹³C NMR (75 MHz, CDCl₃)



DKM-DJB-133

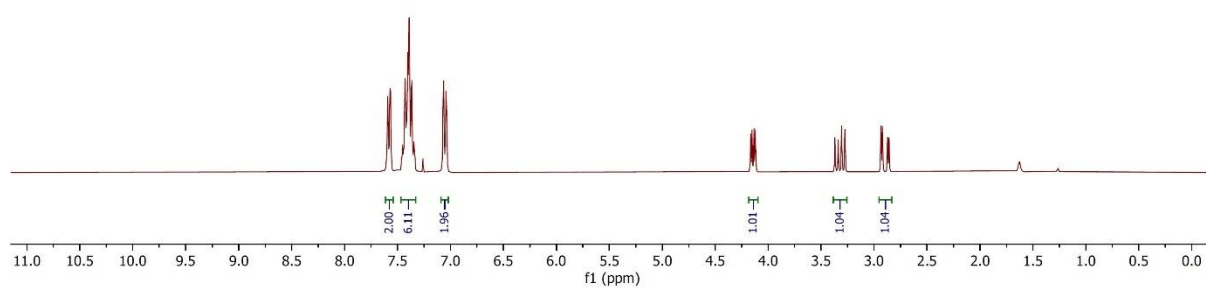
7.60
7.59
7.57
7.57
7.48
7.45
7.43
7.40
7.39
7.39
7.37
7.36
7.35
7.34
7.34
7.26
7.07
7.06
7.04
7.04

4.16
4.15
4.13
4.12
3.37
3.34
3.31
3.28
2.99
2.97
2.86



3b

¹H NMR (300 MHz, CDCl₃)



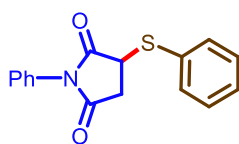
DKM-DJB-133-13C

177.52
173.53

135.11
131.53
129.80
129.70
129.53
129.17
128.82
128.36

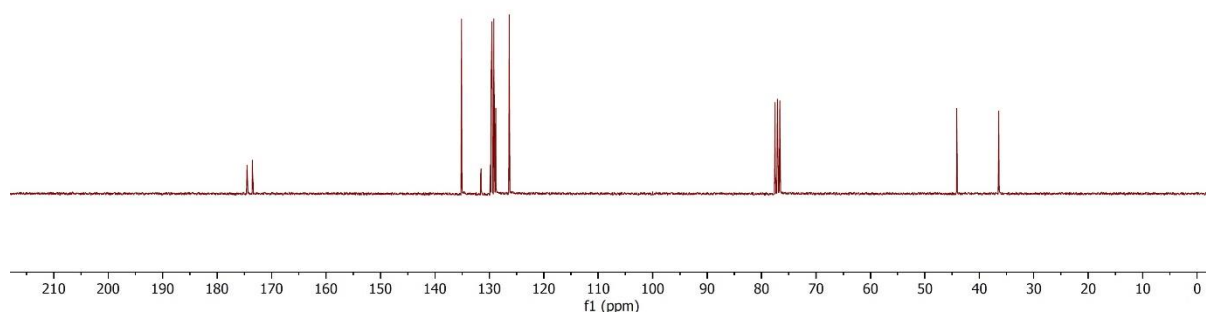
77.51
77.09
76.66

44.15
36.42



3b

¹³C NMR (75 MHz, CDCl₃)

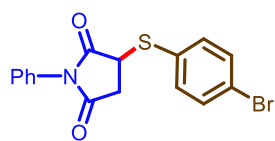


DKM-DJB-131

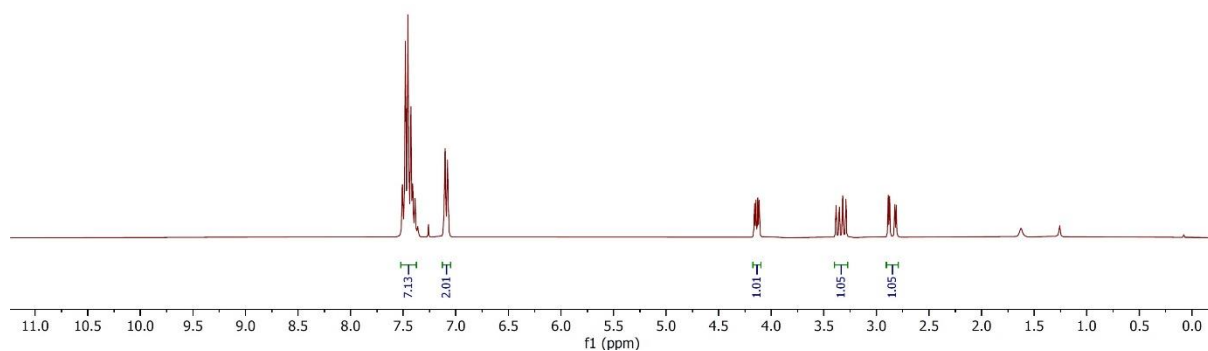
7.51
7.48
7.46
7.43
7.41
7.38
7.36
7.10
7.08

4.16
4.15
4.13
4.11

3.38
3.35
3.32
3.29
2.89
2.87
2.82
2.81



¹H NMR (300 MHz, CDCl₃)



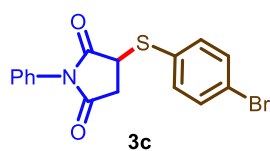
DKM-DJB-131-13C

174.30
173.24

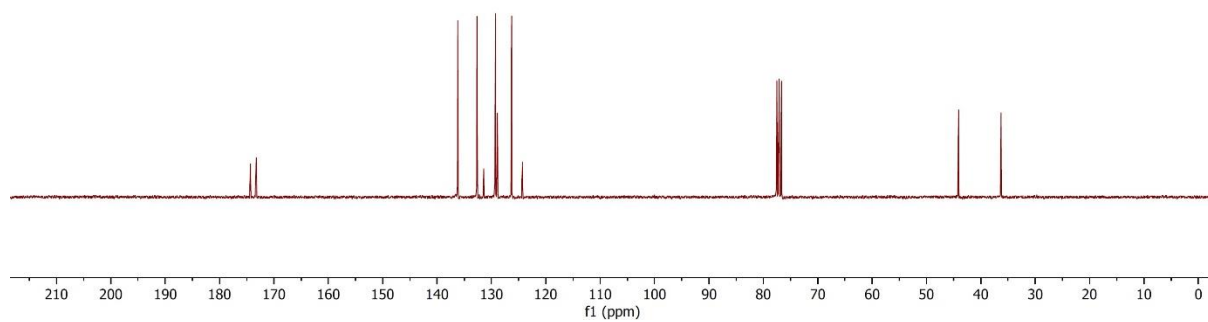
138.20
137.66
137.02
129.37
129.24
128.92
126.28
124.29

77.50
77.08
76.66

44.07
36.28



¹³C NMR (75 MHz, CDCl₃)



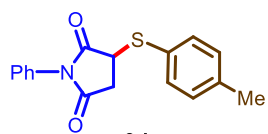
DKM-DJB-71

7.47
7.45
7.42
7.40
7.39
7.36
7.28
7.18
7.16
7.05
7.03

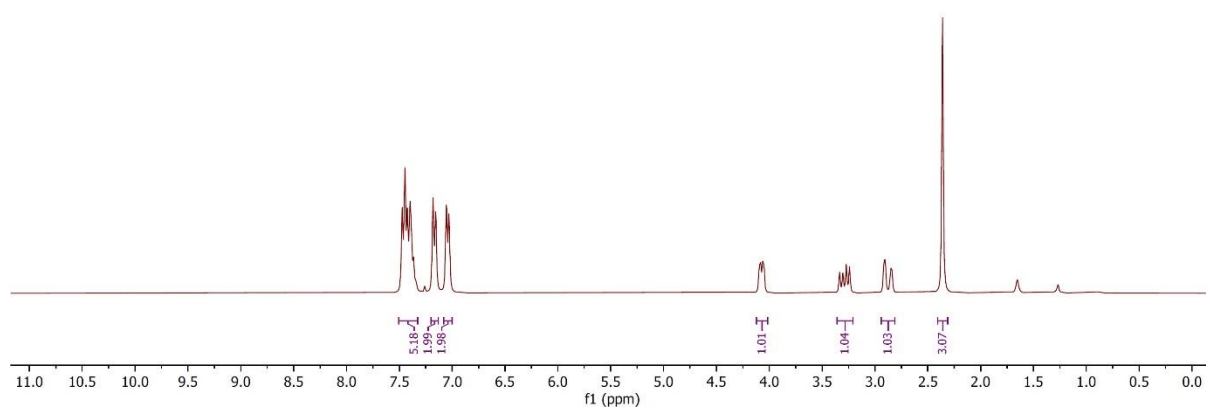
4.10
4.08
4.06
4.05

3.33
3.30
3.27
3.24
2.92
2.90
2.85
2.84

2.36



^1H NMR (300 MHz, CDCl_3)



DKM-DJB-71-13C

174.65
172.68

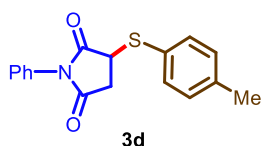
140.26
135.54
131.60
130.31
129.13
128.78
126.38
125.89

77.54
77.12
76.69

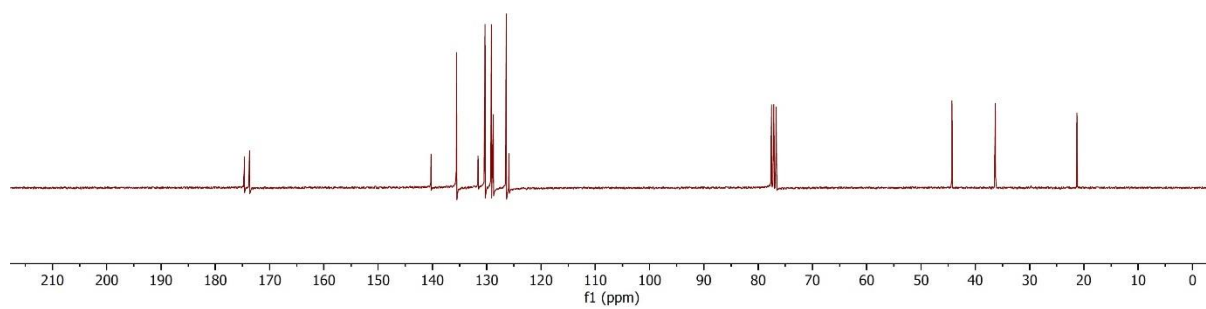
44.30

36.31

21.28



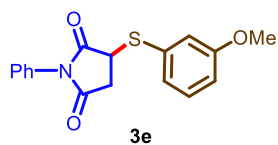
^{13}C NMR (75 MHz, CDCl_3)



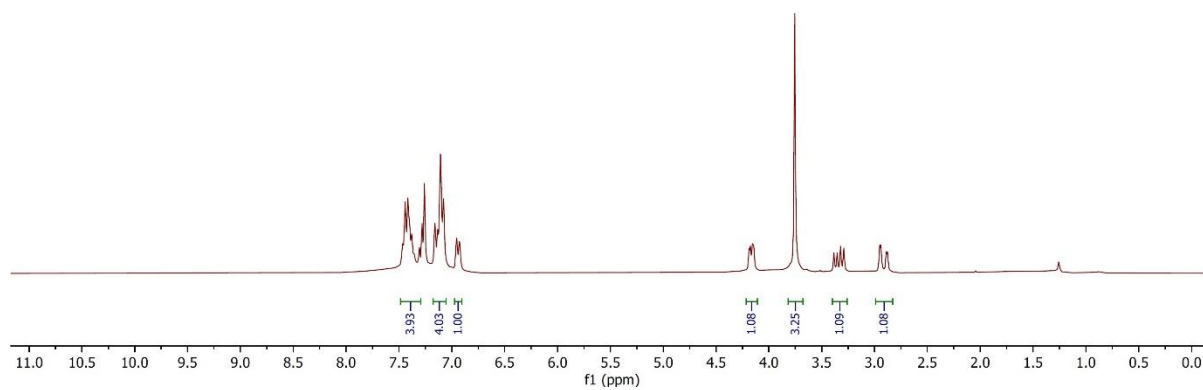
DKM-DJB-70

7.46
7.44
7.43
7.38
7.26
7.16
7.13
7.11
6.98
6.92

4.19
4.17
4.16
4.14
3.75
3.38
3.35
3.32
3.29
2.95
2.89
2.88



¹H NMR (300 MHz, CDCl₃)



DKM-DJB-70-13C

174.60
173.56

160.03

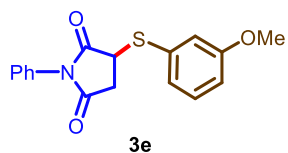
131.58
130.92
130.29
129.16
128.82
128.86
118.77
115.65

77.52
77.09
76.67

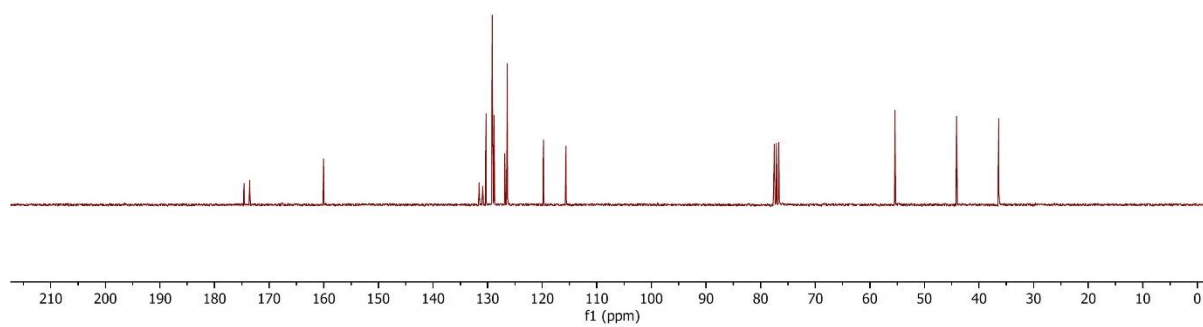
55.38

44.13

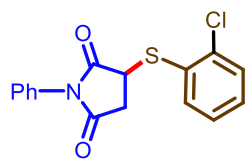
36.41



¹³C NMR (75 MHz, CDCl₃)

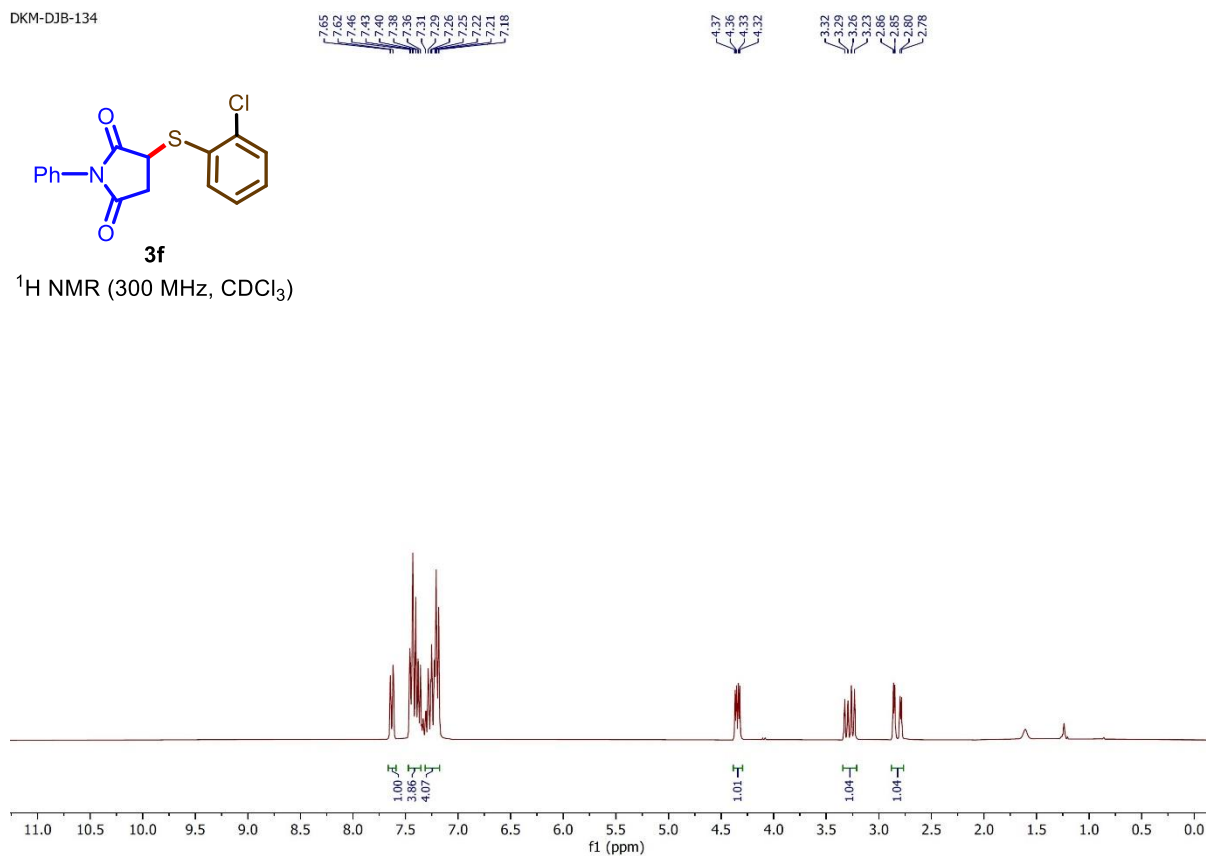


DKM-DJB-134

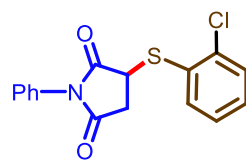


3f

^1H NMR (300 MHz, CDCl_3)

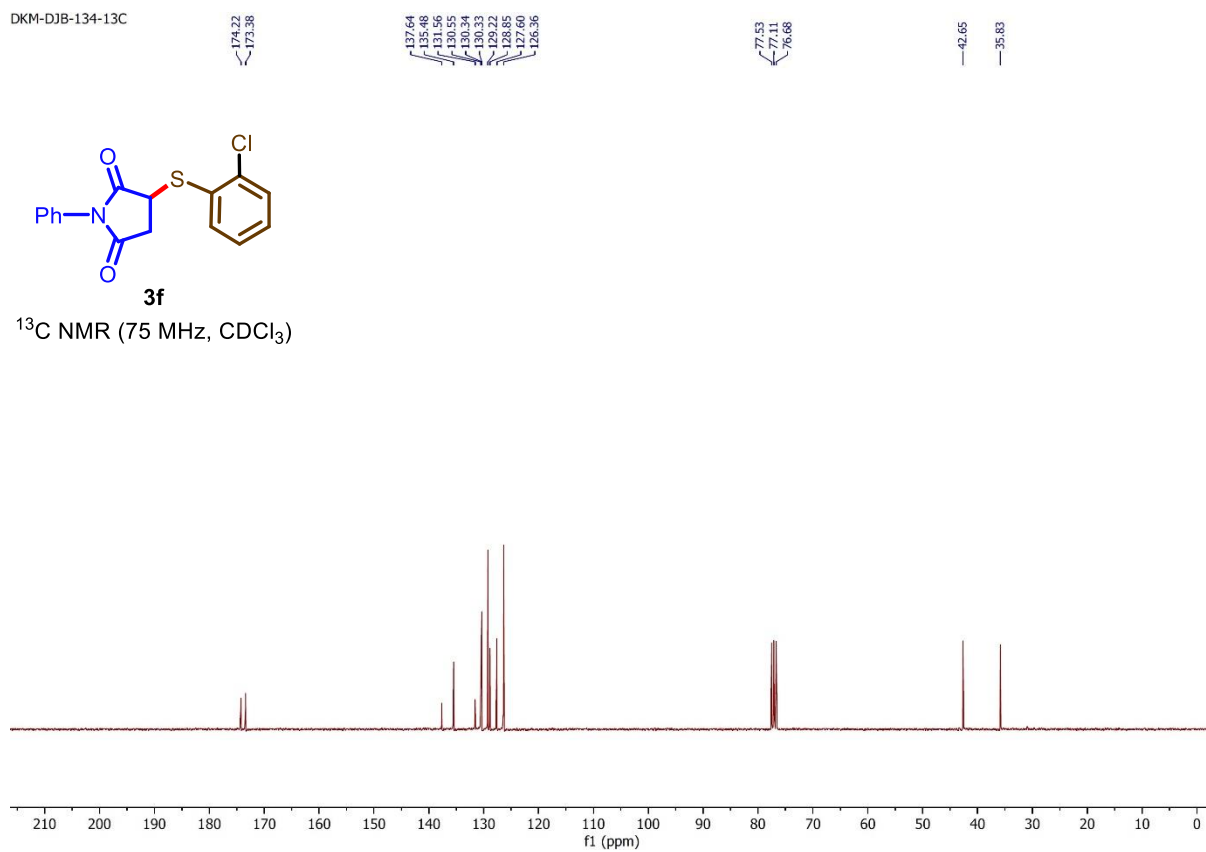


DKM-DJB-134-13C



3f

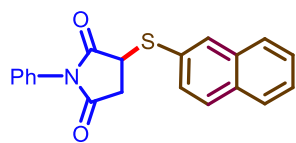
^{13}C NMR (75 MHz, CDCl_3)



DKM-DJB-130

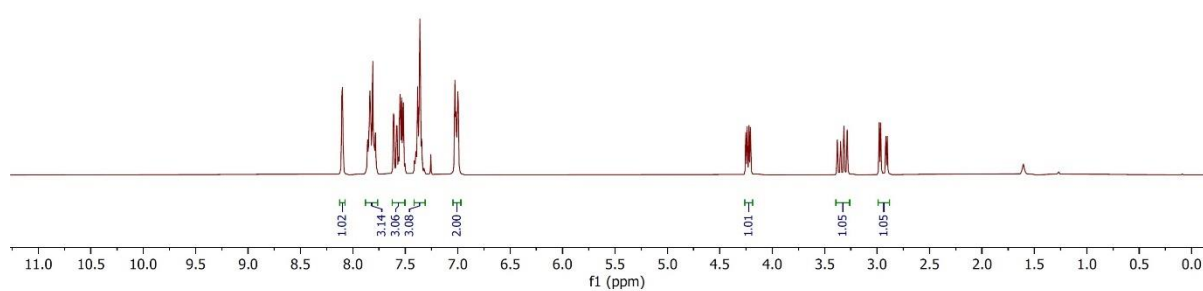
8.10
7.86
7.84
7.81
7.78
7.76
7.55
7.53
7.52
7.38
7.36
7.35
7.35
7.03
7.00
6.99

4.25
4.24
4.22
4.21
3.38
3.35
3.32
3.29
2.98
2.97
2.90



3g

¹H NMR (300 MHz, CDCl₃)



DKM-DJB-130-13C

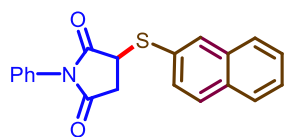
174.63
173.49

134.78
133.56
133.32
131.51
129.77
129.22
129.14
128.80
127.87
127.80
127.36
127.01
127.00
126.35

77.51
77.08
76.66

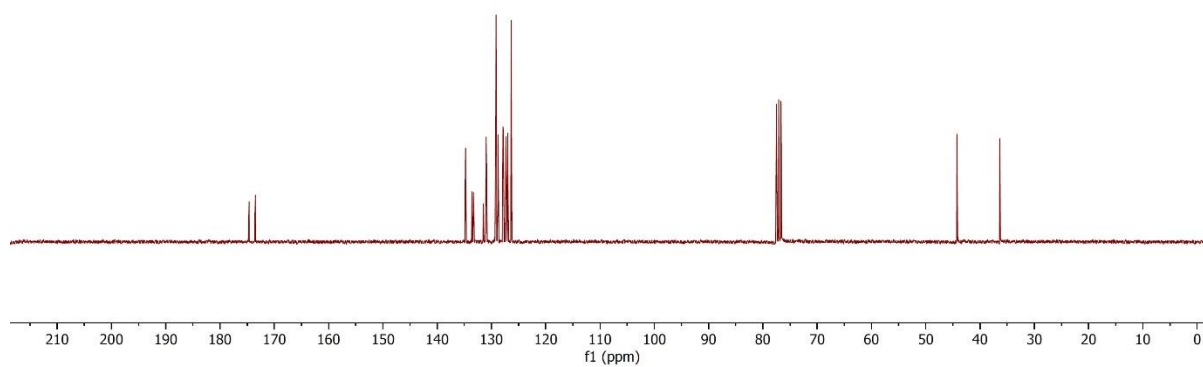
44.25

36.37



3g

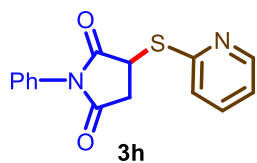
¹³C NMR (75 MHz, CDCl₃)



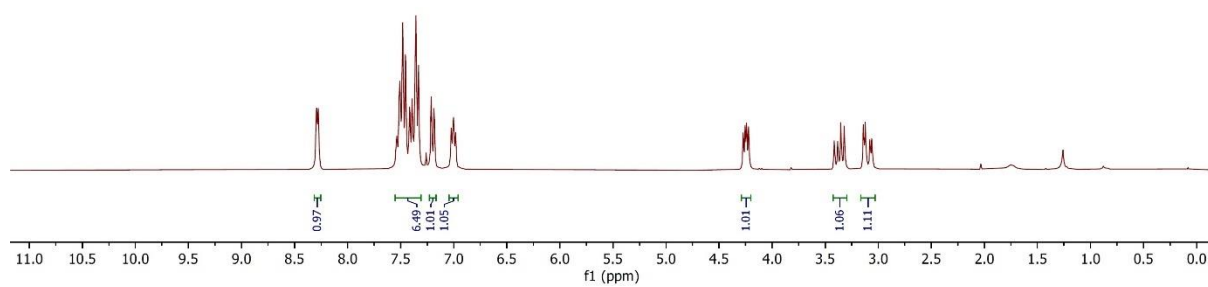
DKM-DJB-185

8.39
8.27
7.54
7.51
7.48
7.45
7.42
7.38
7.33
7.26
7.21
7.19
7.02
6.98

4.37
4.25
4.24
4.22
3.42
3.38
3.35
3.32
3.14
3.12
3.08
3.06



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

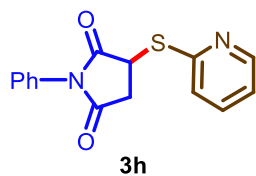


DKM-DJB-185-13C

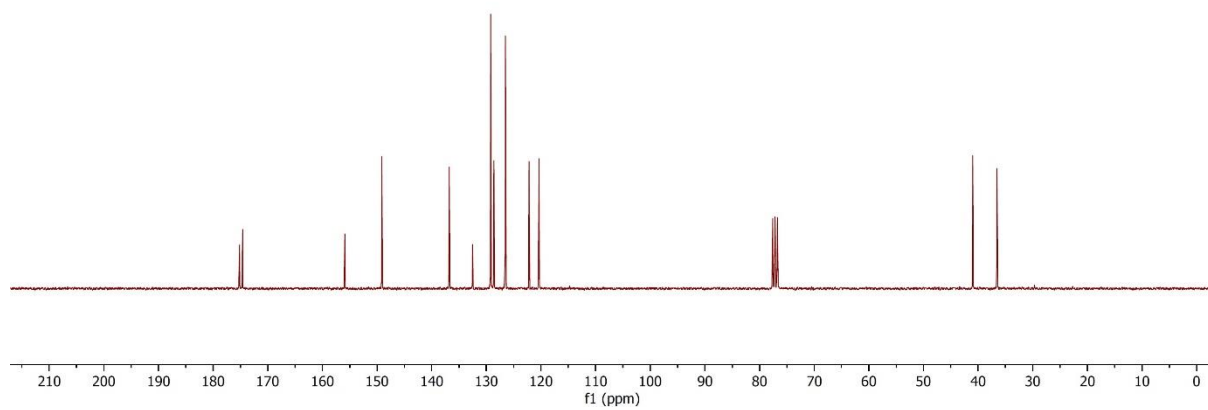
175.16
174.59
155.89
149.12
136.78
132.52
129.20
128.62
126.51
122.19
120.36

77.99
77.16
76.74

40.98
36.55



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

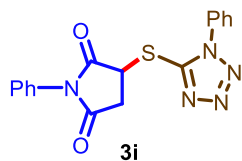


DKM-DJB-169LS

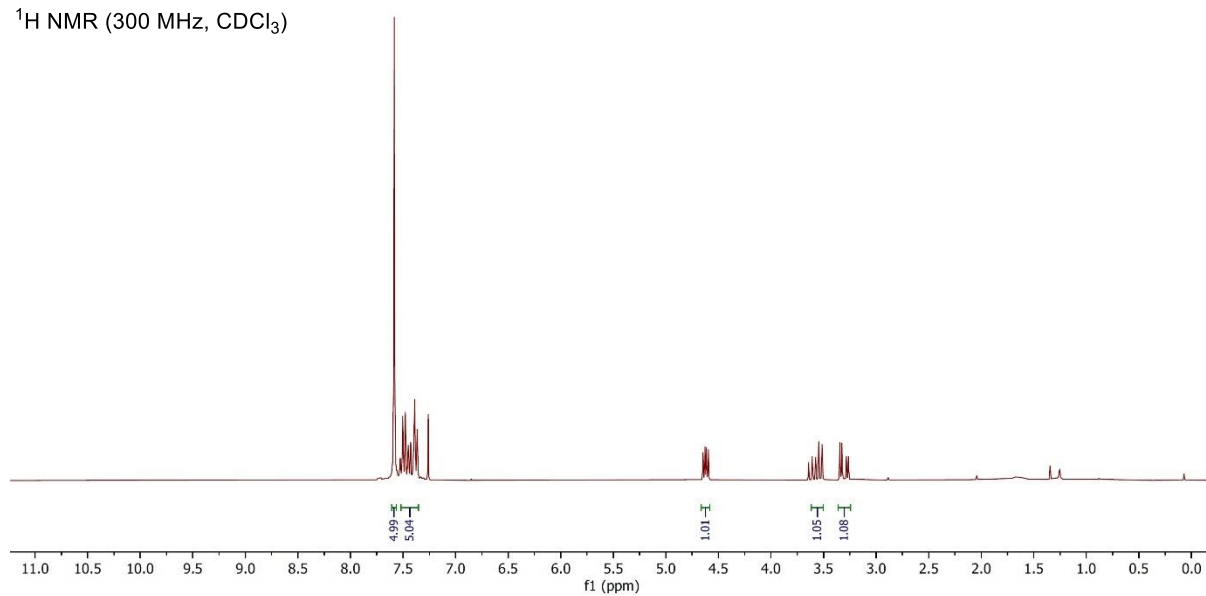
7.58
7.50
7.48
7.45
7.43
7.39
7.37
7.26

4.65
4.63
4.60

3.61
3.58
3.55
3.51
3.48
3.28
3.26



¹H NMR (300 MHz, CDCl₃)



DKM-DJB-169LS-13C

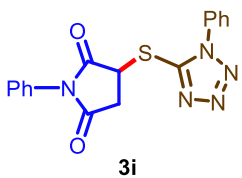
172.85
172.56

151.29

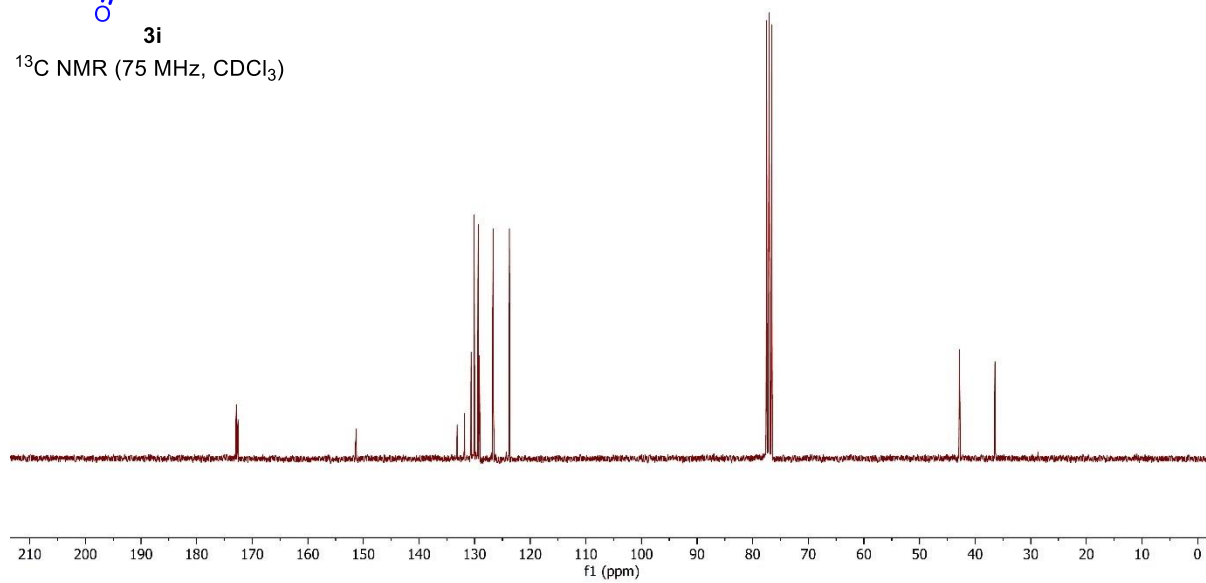
133.15
131.83
130.64
130.07
129.36
128.14
127.98
127.75

77.46
77.04
76.62

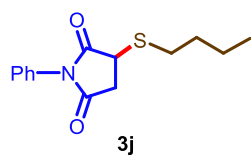
42.82
36.44



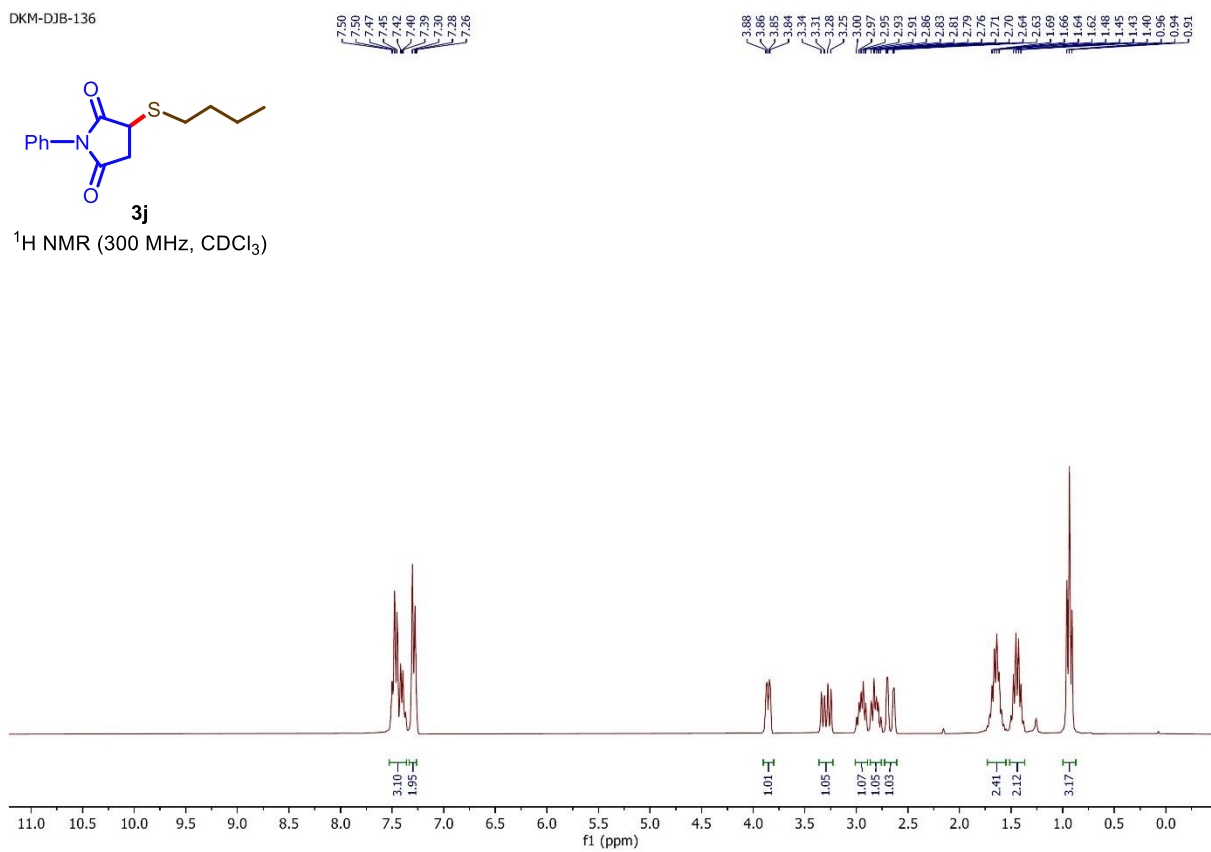
¹³C NMR (75 MHz, CDCl₃)



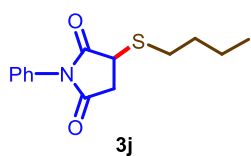
DKM-DJB-136



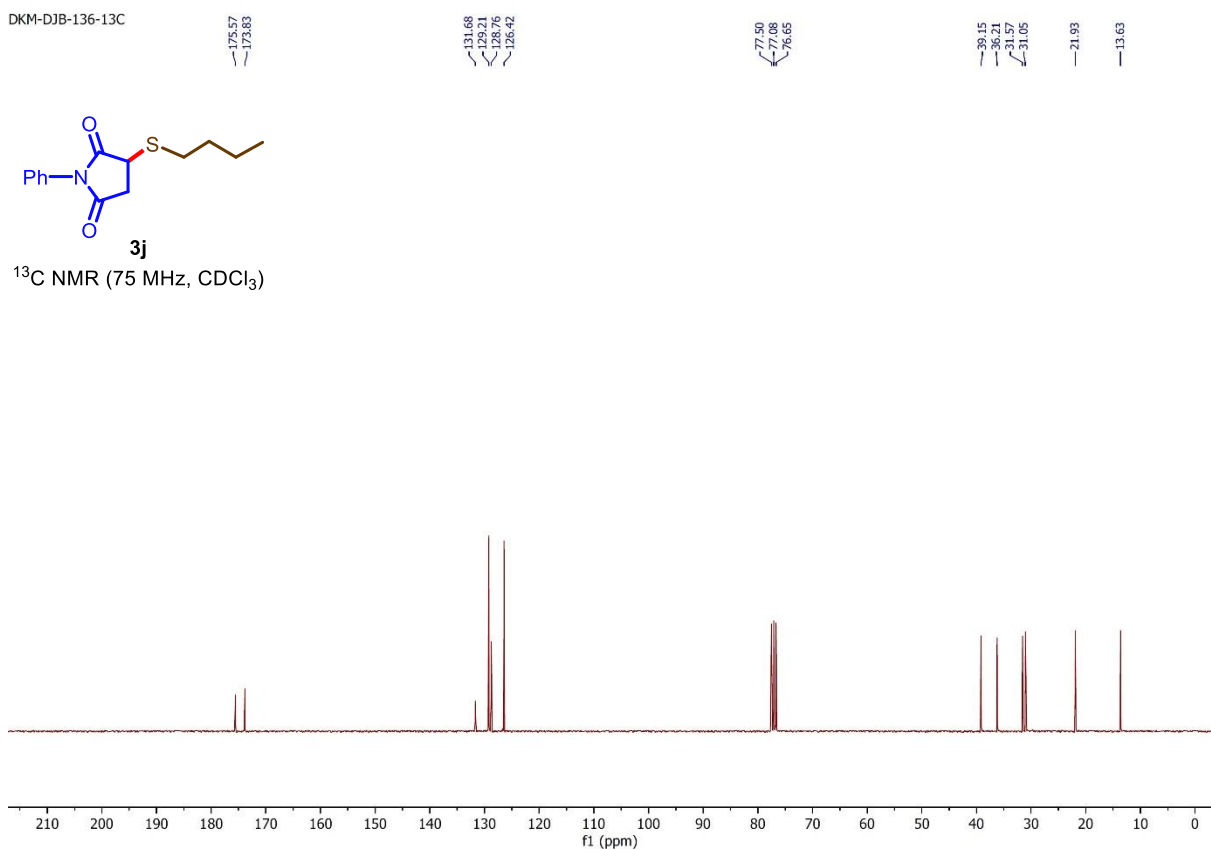
¹H NMR (300 MHz, CDCl₃)



DKM-DJB-136-13C



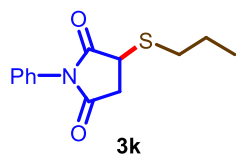
¹³C NMR (75 MHz, CDCl₃)



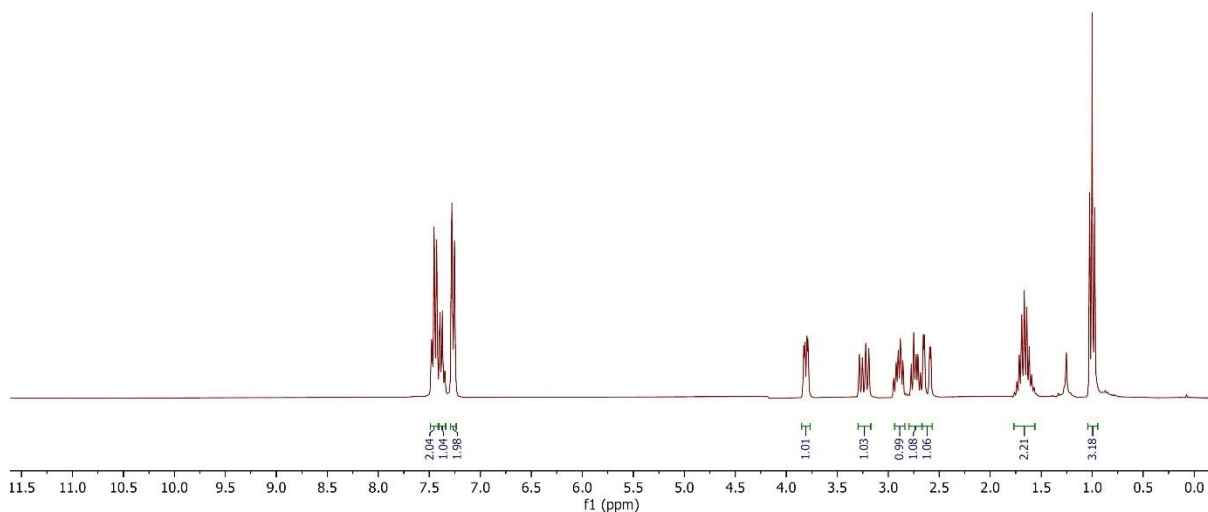
DKM-DJB-343
1H CDCl3 DKM-DJB-343 30/08/2024

7.477
7.454
7.452
7.375
7.372
7.362
7.346
7.277
7.260
7.253

3.838
3.816
3.797
3.786
3.282
3.252
3.220
3.189
2.993
2.982
2.881
2.855
2.774
2.751
2.735
2.714
2.709
2.686
2.656
2.644
2.594
2.581
1.761
1.752
1.715
1.690
1.666
1.642
1.618
1.595
1.572
1.025
1.001
0.976



¹H NMR (300 MHz, CDCl₃)



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

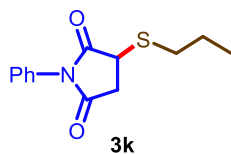
175.66
173.94

131.74
128.20
126.47

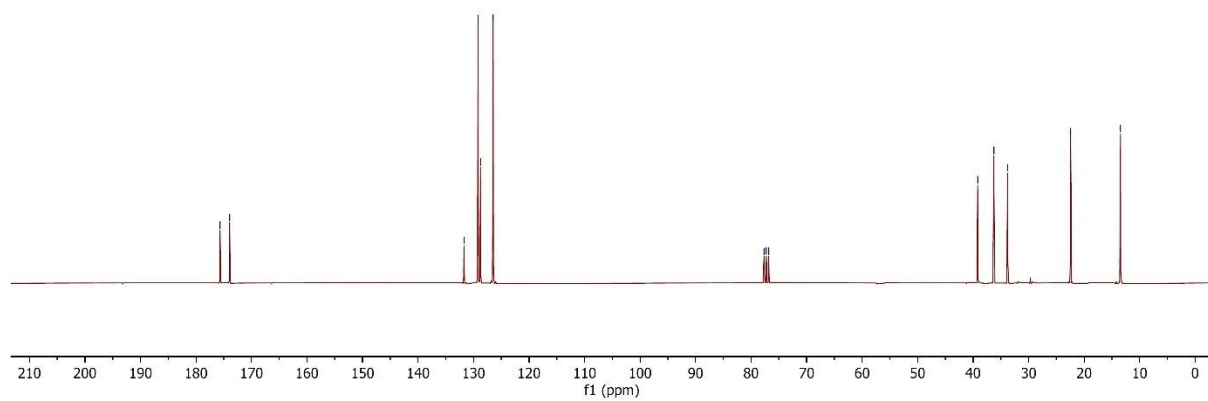
77.69
77.25
76.83

39.17
36.25
33.78

22.42
13.44



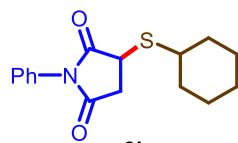
¹³C NMR (75 MHz, CDCl₃)



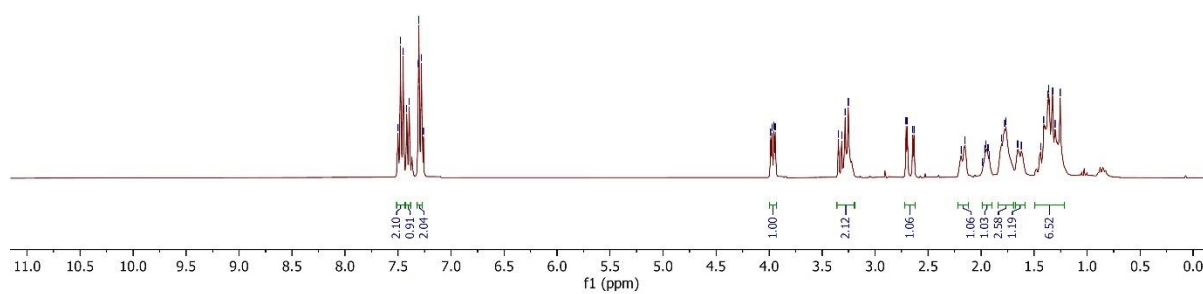
DKM-DJB-344.1.fid

7.50
7.48
7.45
7.42
7.39
7.31
7.29
7.28
7.26

3.98
3.97
3.94
3.35
3.32
3.28
3.25
2.71
2.70
2.64
2.62
2.19
2.15
1.98
1.97
1.95
1.94
1.82
1.78
1.77
1.65
1.62
1.44
1.41
1.37
1.36
1.32
1.30
1.25



¹H NMR (300 MHz, CDCl₃)



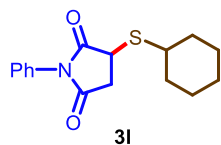
DKM-DJB-344-13C.2.fid

175.81
173.96

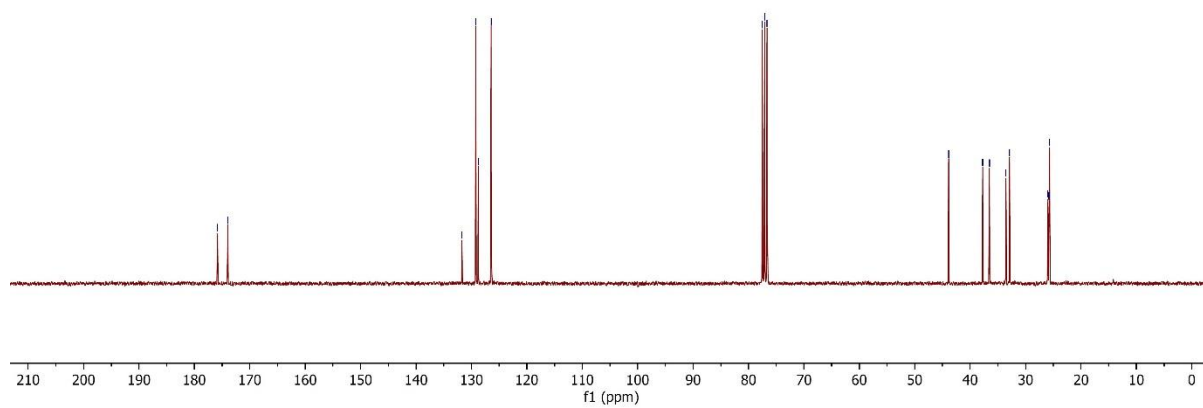
131.72
129.71
128.75
126.44

77.51
77.09
76.66

43.88
37.74
36.52
33.54
32.90
25.97
25.76
25.68



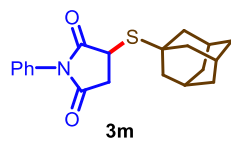
¹³C NMR (75 MHz, CDCl₃)



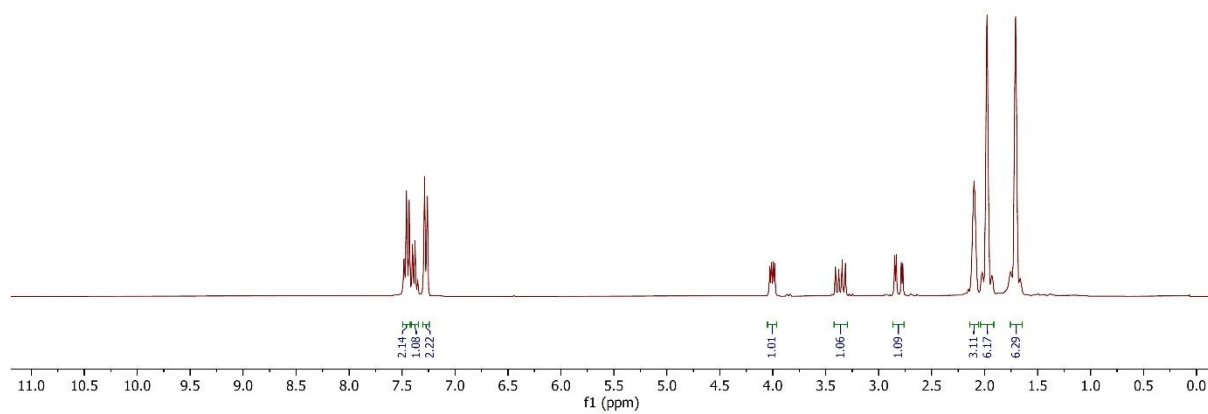
DKM-DJB-345
1H CDCl3 DKM-DJB-345 30/08/2024

7.487
7.486
7.485
7.458
7.452
7.432
7.406
7.401
7.396
7.386
7.385
7.377
7.367
7.357
7.352
7.287
7.270
7.268

4.028
4.014
3.997
3.982
3.408
3.397
3.385
3.314
2.849
2.834
2.787
2.772
2.110
2.082
2.080
2.024
2.015
1.977
1.930
1.922
1.754
1.738
1.683



¹H NMR (300 MHz, CDCl₃)



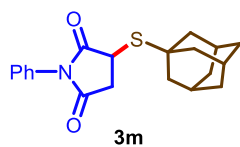
DKM-DJB-345-13C.1.fid

176.35
174.10

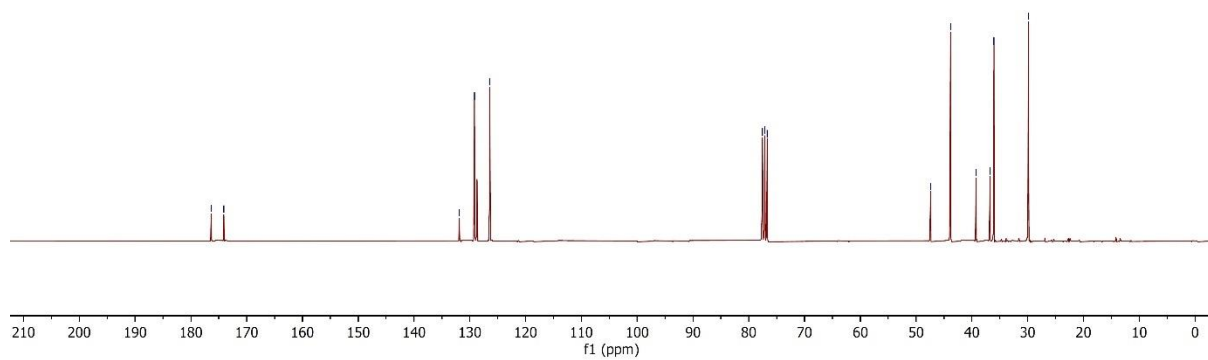
131.90
129.17
126.44

77.55
77.12
76.70

47.40
45.82
36.74
36.04
29.84



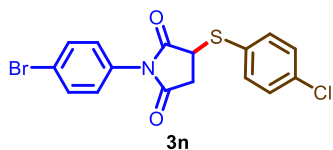
¹³C NMR (75 MHz, CDCl₃)



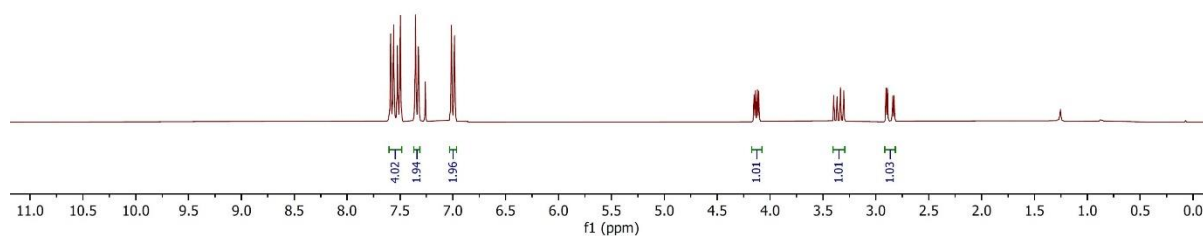
DKM-DJB-163

7.59
7.56
7.52
7.50
7.33
7.26
7.01
6.98

4.15
4.14
4.12
4.11
3.40
3.37
3.33
3.30
2.90
2.89
2.84
2.83



¹H NMR (300 MHz, CDCl₃)



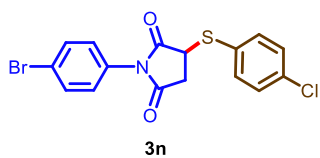
DKM-DJB-163-13C

173.95
172.83

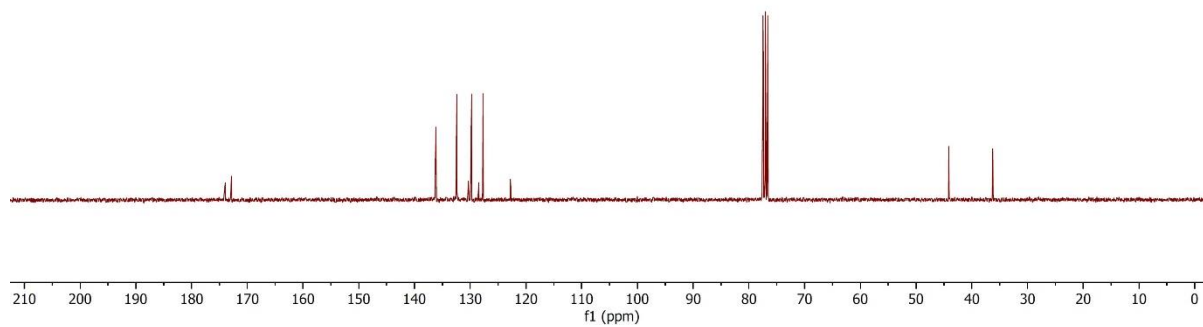
136.29
134.06
132.40
130.35
129.74
128.47
127.72
122.79

77.46
77.03
76.61

44.14
36.25



¹³C NMR (75 MHz, CDCl₃)



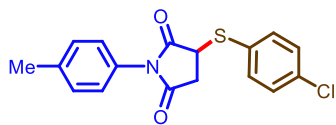
DKM-DJB-216.1.fid

7.55
7.52
7.36
7.34
7.28
7.28
7.25
6.96
6.96

4.16
4.14
4.13
4.11

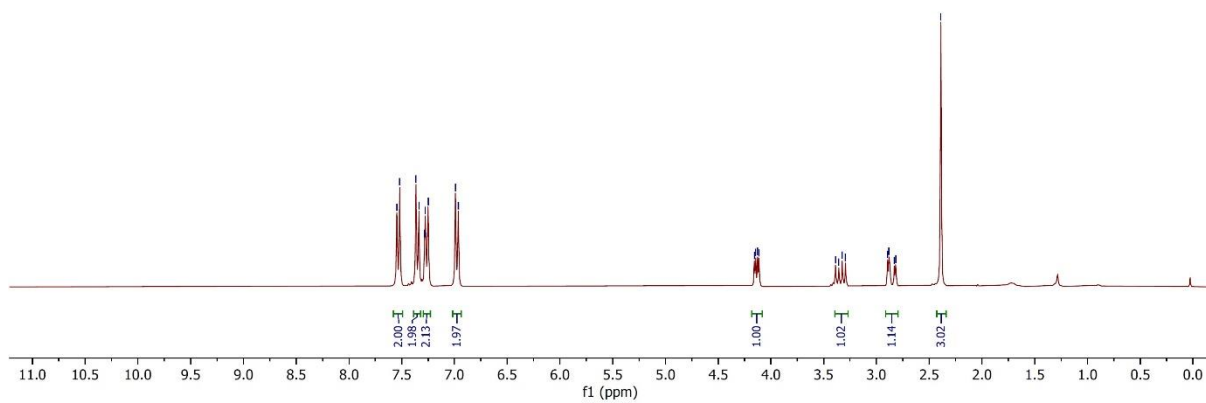
3.39
3.36
3.33
3.29
2.89
2.83
2.82

— 2.39



3o

¹H NMR (300 MHz, CDCl₃)



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

174.48
173.45

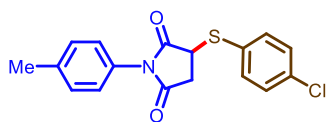
139.06
138.72
138.68
129.69
129.36
128.79
128.69
126.08

77.53
77.10
76.68

— 44.18

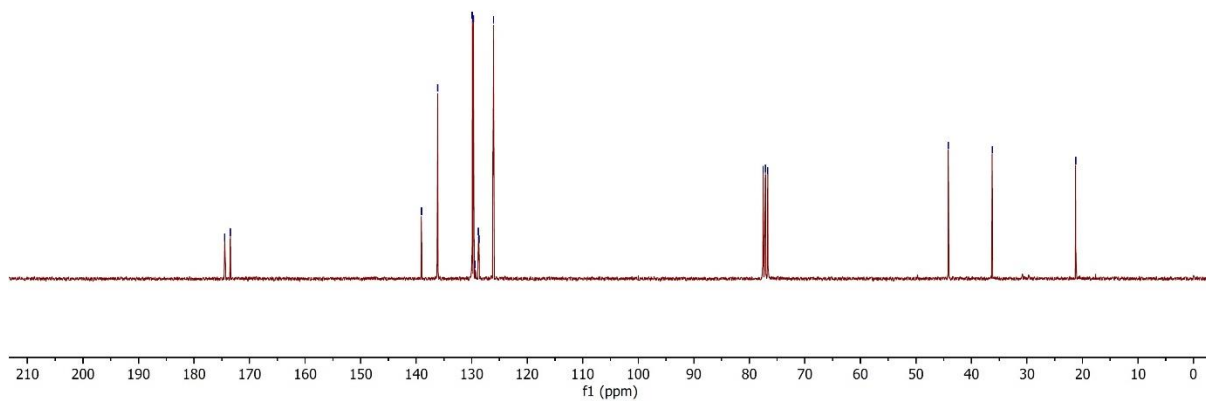
— 36.27

— 21.23



3o

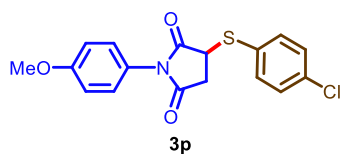
¹³C NMR (75 MHz, CDCl₃)



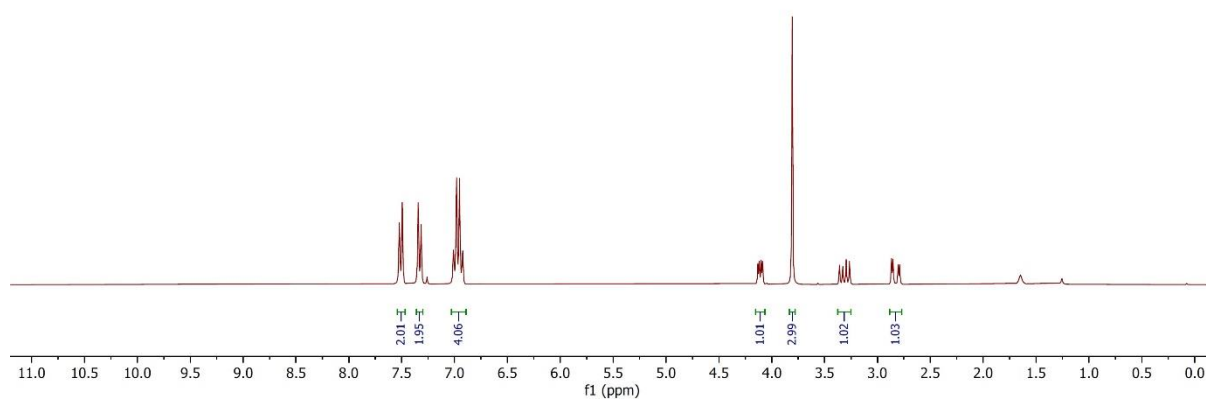
DKM-DJB-181

7.52
7.49
7.34
7.32
7.26
7.01
6.98
6.95
6.92

4.13
4.12
4.10
4.09
3.81
3.96
3.93
3.30
3.26
2.87
2.85
2.80
2.79



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



DKM-DJB-181-13C

174.57
173.54

159.69

136.09

129.68

128.70

127.50

123.99

114.53

77.50
77.08
76.66

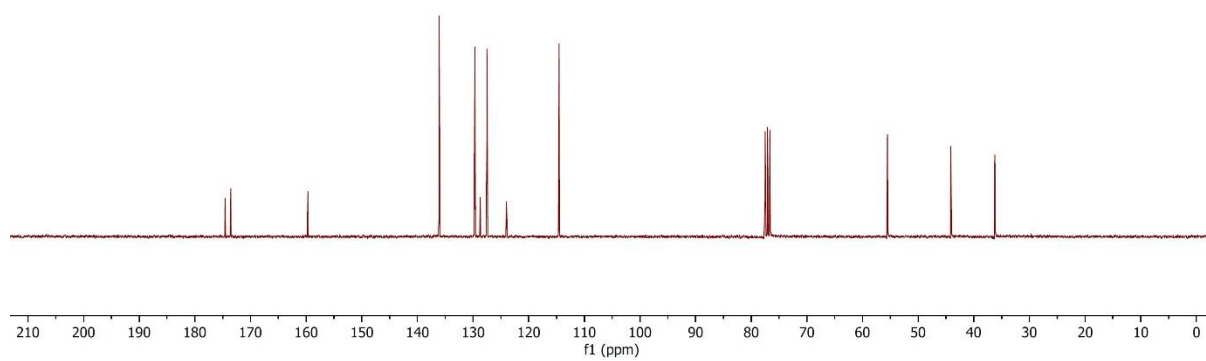
55.51

44.13

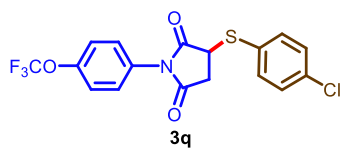
36.22



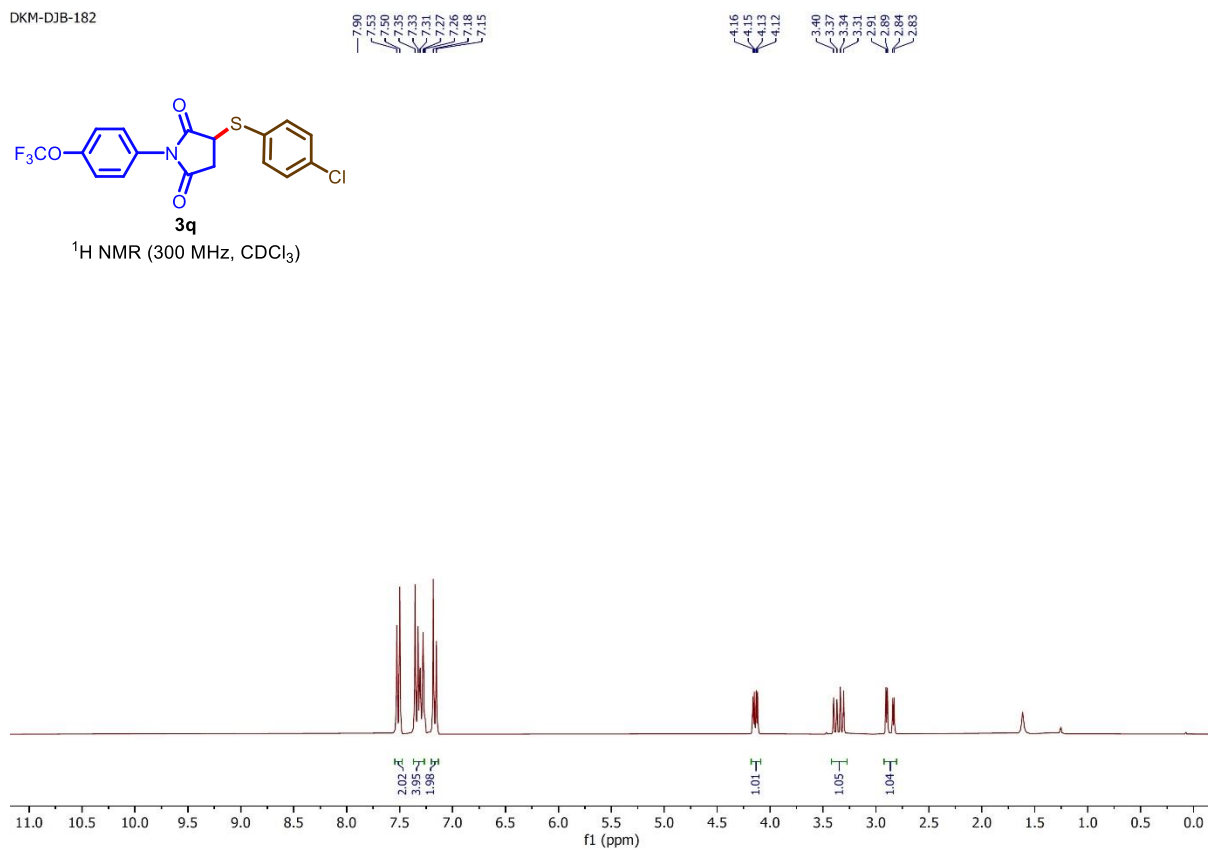
$^{13}\text{C NMR}$ (75 MHz, CDCl_3)



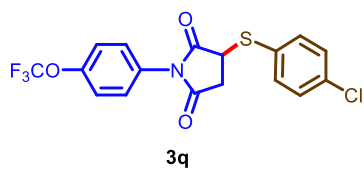
DKM-DJB-182



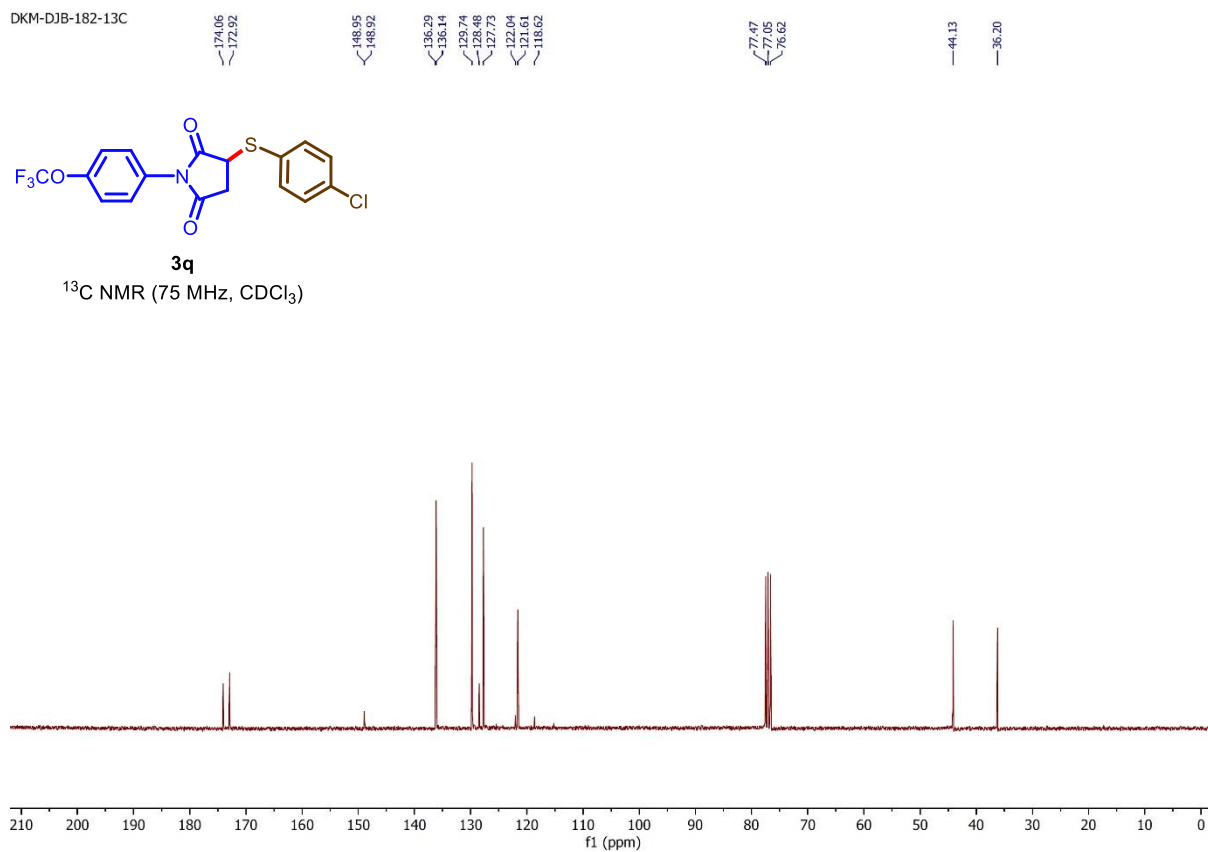
¹H NMR (300 MHz, CDCl₃)



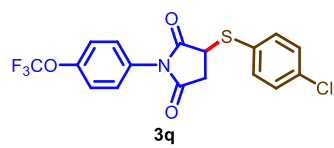
DKM-DJB-182-13C



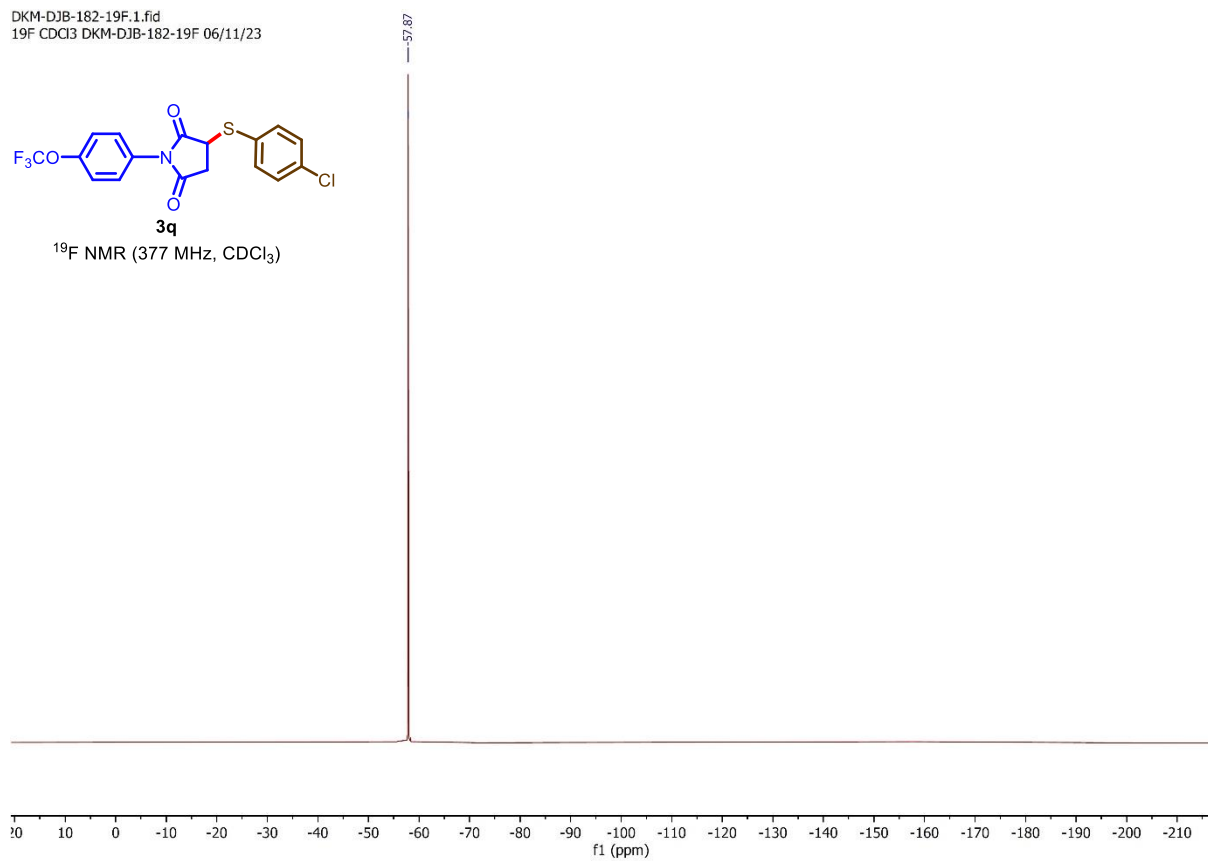
¹³C NMR (75 MHz, CDCl₃)



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



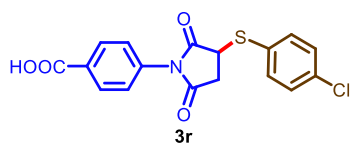
3q
¹⁹F NMR (377 MHz, CDCl₃)



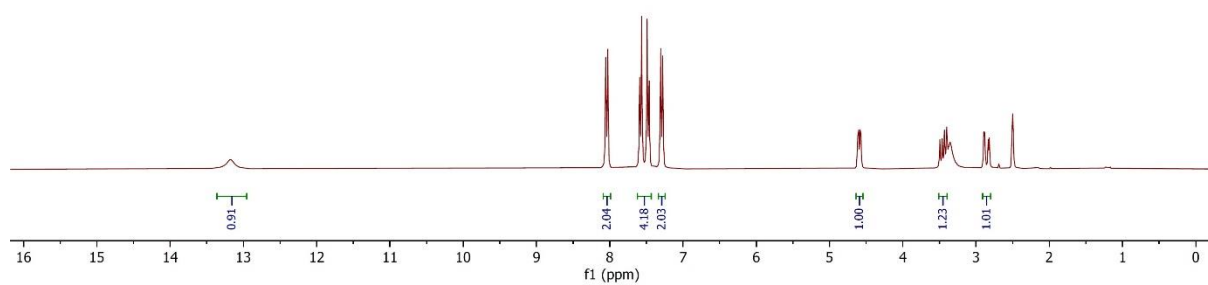
DKM-DJB-209.1.fid
1H DMSO DKM-DJB-209 14/6/23

8.05
7.99
7.96
7.49
7.46
7.30
7.28

4.62
4.60
4.57
3.49
3.43
3.40
2.89
2.88
2.83
2.82



¹H NMR (300 MHz, DMSO)

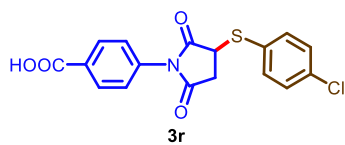


DKM-DJB-209-13C.1.fid

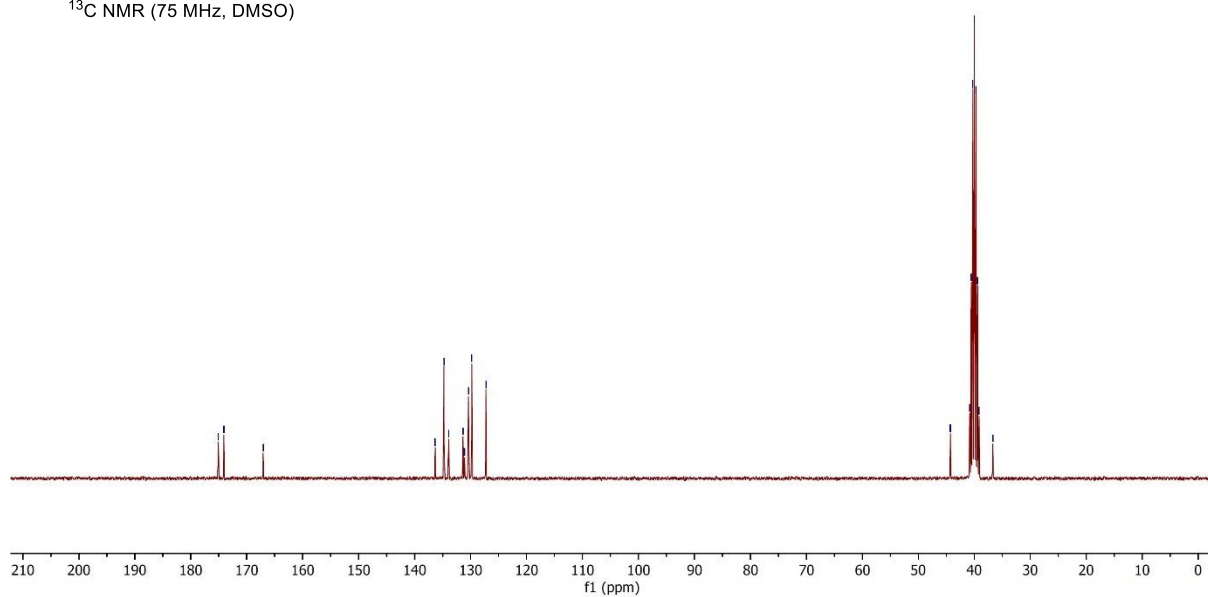
175.10
174.10
167.07

136.32
134.77
131.90
131.56
131.00
130.38
129.79
127.26

44.27
44.26
40.54
40.26
39.98
39.71
39.43
39.15
36.09



¹³C NMR (75 MHz, DMSO)

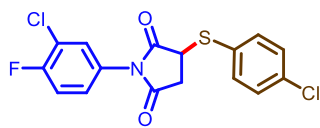


DKM-DJB-184

7.52
7.51
7.36
7.33
7.26
7.23
7.18
7.15
7.12
7.03
7.00
6.99

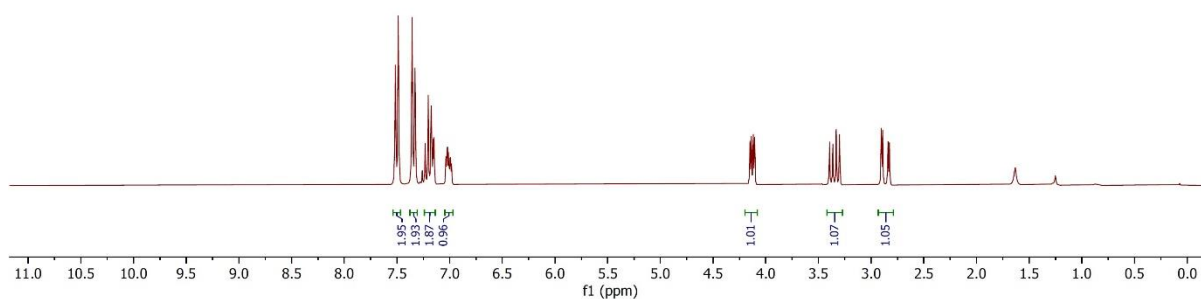
4.15
4.14
4.12
4.11

3.39
3.36
3.33
3.30
2.90
2.88
2.83



3s

^1H NMR (300 MHz, CDCl_3)



DKM-DJB-184-13C

173.88
172.76

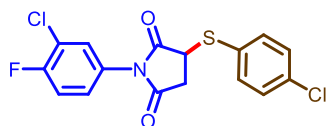
159.53
156.19

136.36
136.15
129.78
128.71
128.42
127.77
126.28
126.17
121.90
121.65
117.79
116.89

77.50
77.08
76.65

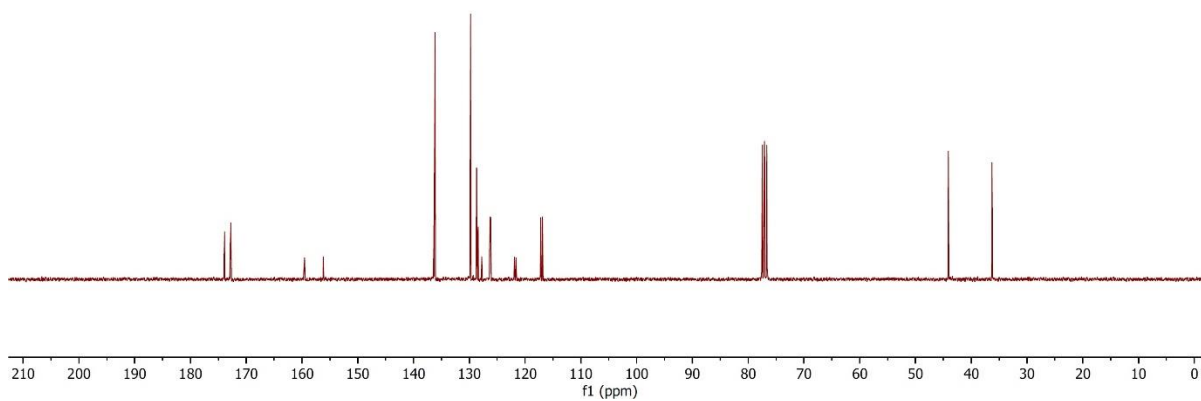
41.11

36.28

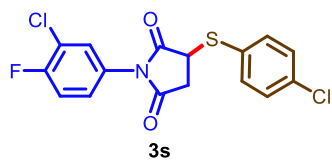


3s

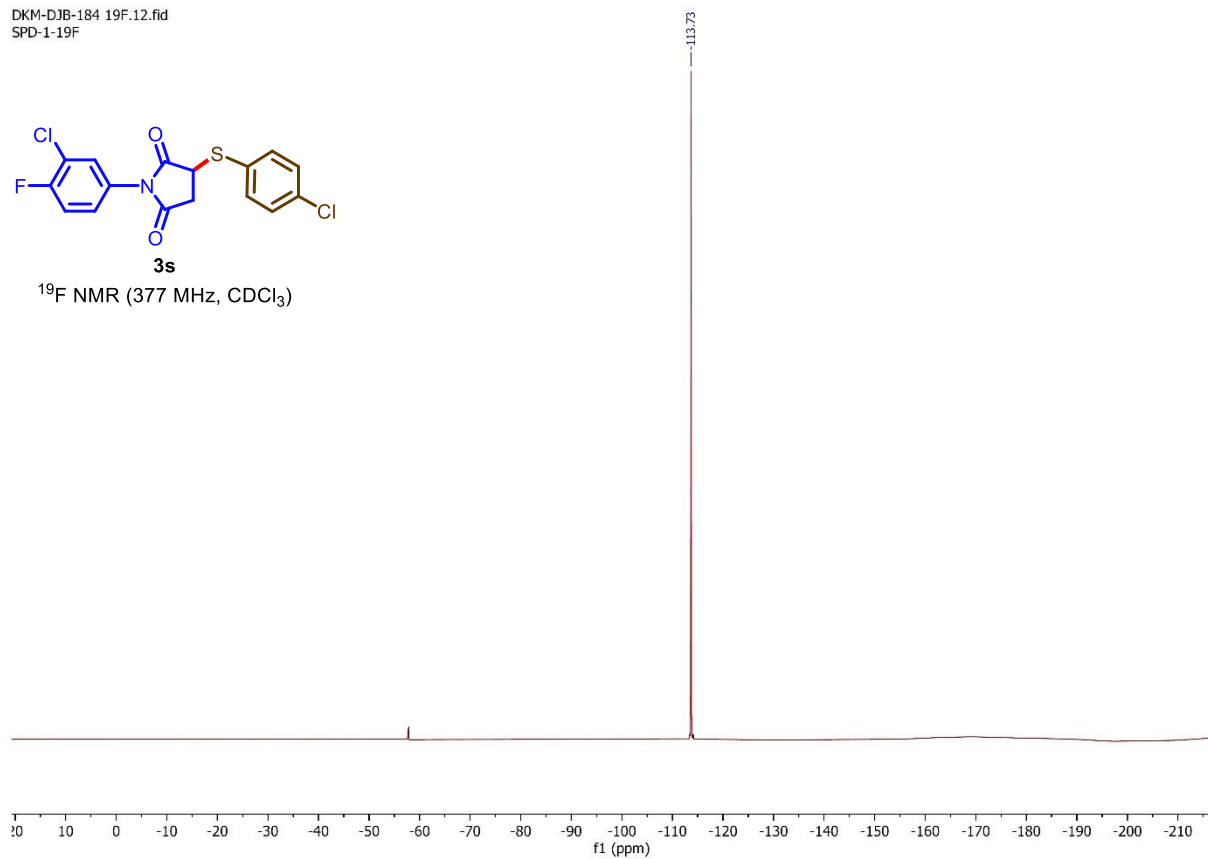
^{13}C NMR (75 MHz, CDCl_3)



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



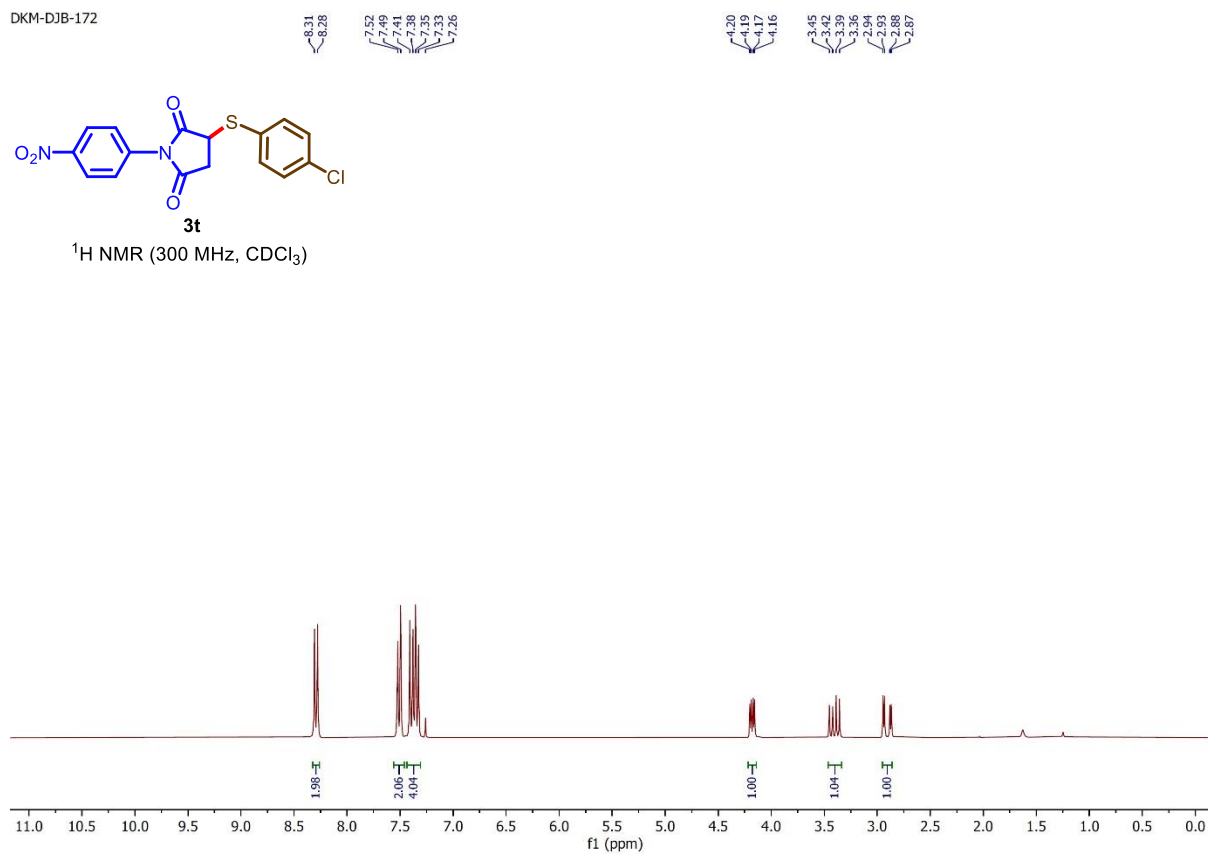
3s
¹⁹F NMR (377 MHz, CDCl₃)



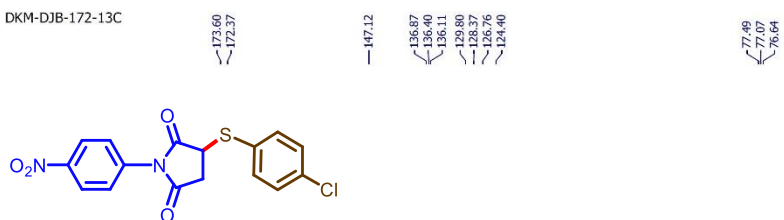
DKM-DJB-172



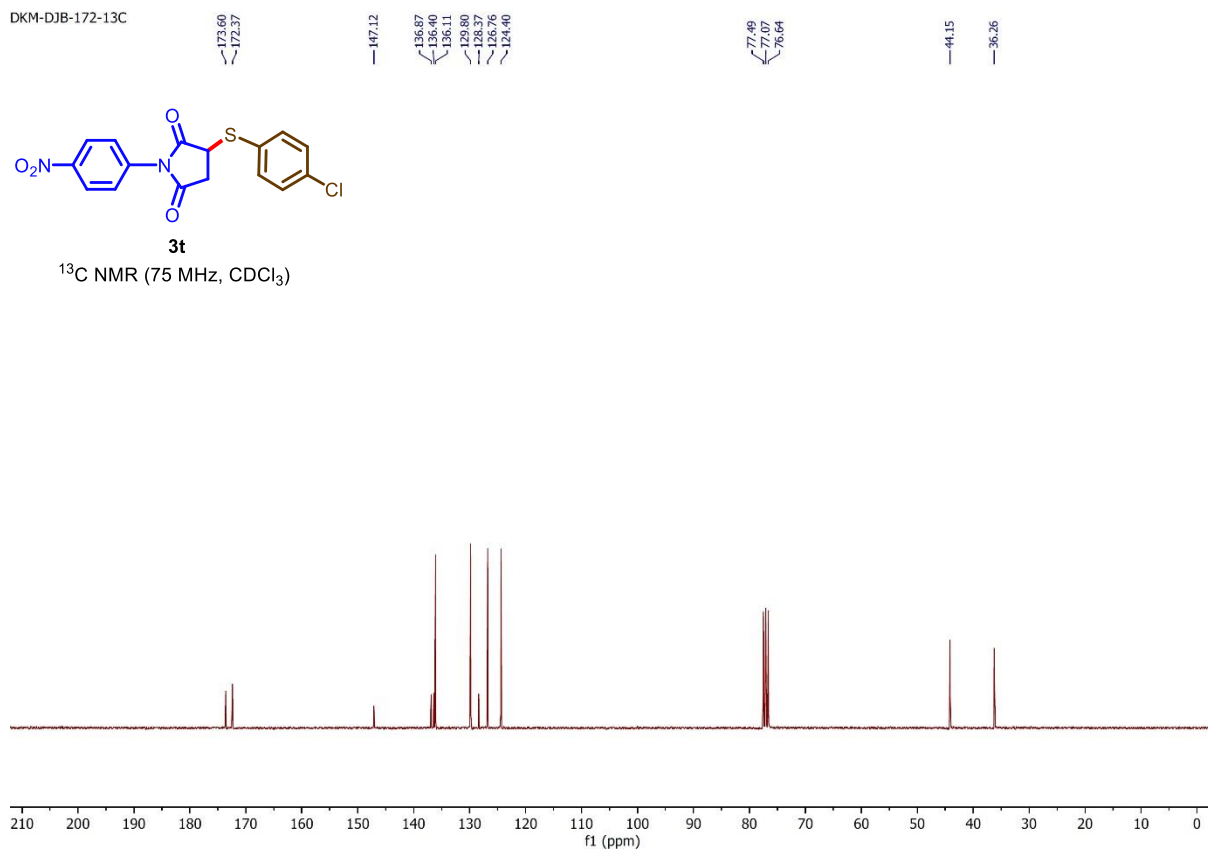
^1H NMR (300 MHz, CDCl_3)



DKM-DJB-172-13C



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



DKM-DJB-222.1.fid
1H CDCl3 DKM-DJB-222 08/07/23

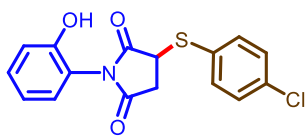
8.91

7.48
7.46
7.29
7.27
7.26
7.23
7.20
7.17
6.84
6.81
6.87
6.84
6.82
6.75
6.73

4.15
4.14
4.12
4.11

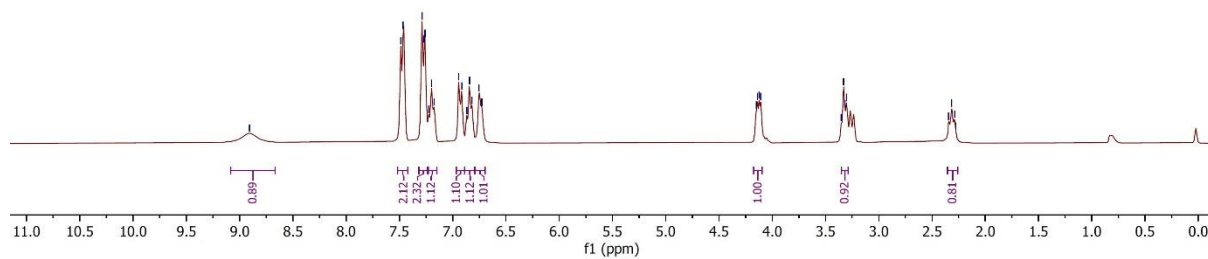
3.35
3.33
3.32
3.30

2.34
2.32
2.29
2.27



3u

¹H NMR (300 MHz, CDCl₃)



DKM-DJB-222-13C.1.fid

174.86
173.86

152.86

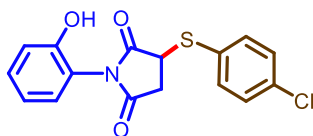
135.60
135.06
130.88
129.54
129.41
128.61

119.98
118.83
117.55

77.59
77.16
76.74

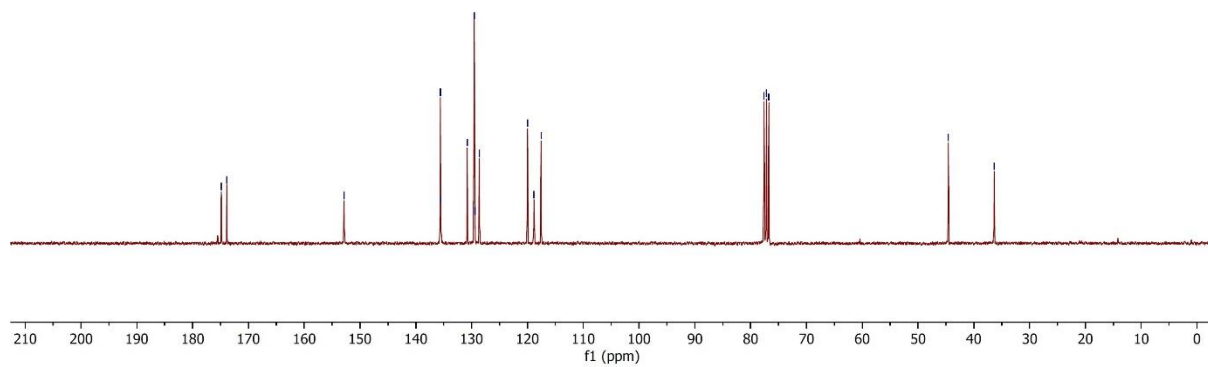
44.57

36.35

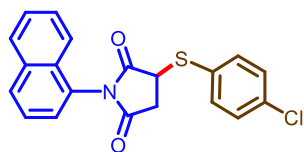


3u

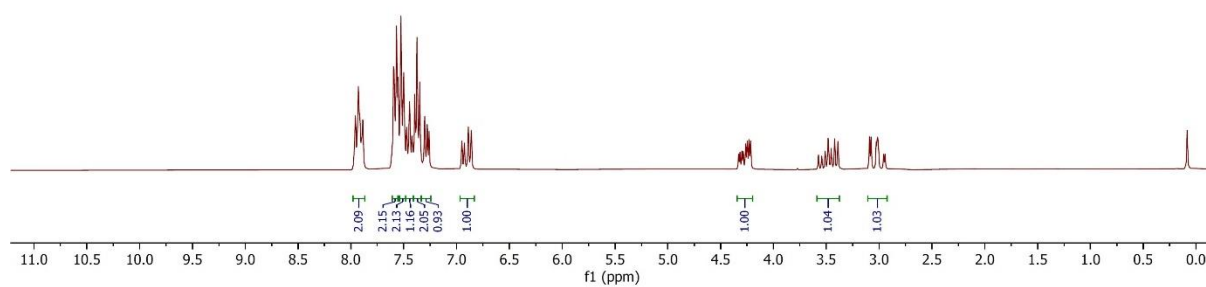
¹³C NMR (75 MHz, CDCl₃)



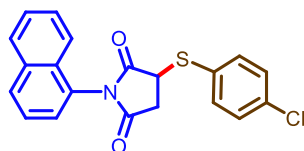
DKM-DJB-140DS



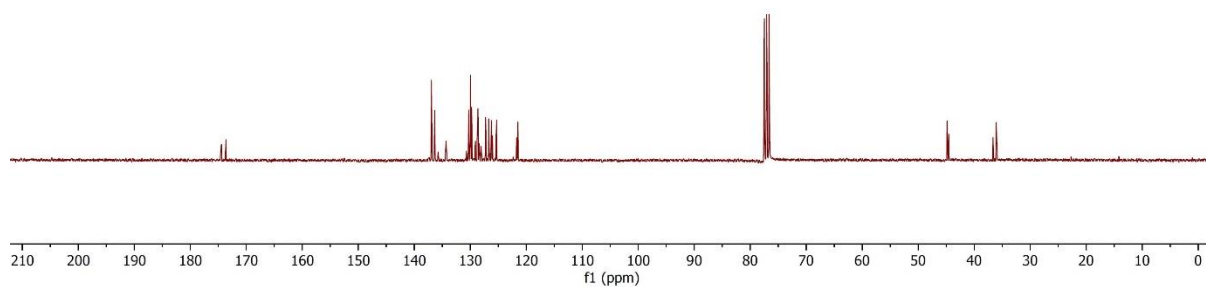
¹H NMR (300 MHz, CDCl₃)



DKM-DJB-140DS-13C



¹³C NMR (75 MHz, CDCl₃)



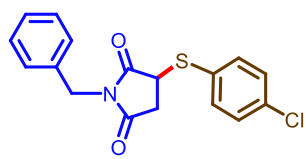
DKM-DJB-139

7.34
7.33
7.32
7.31
7.30
7.29
7.28
7.26
7.14
7.11

4.58

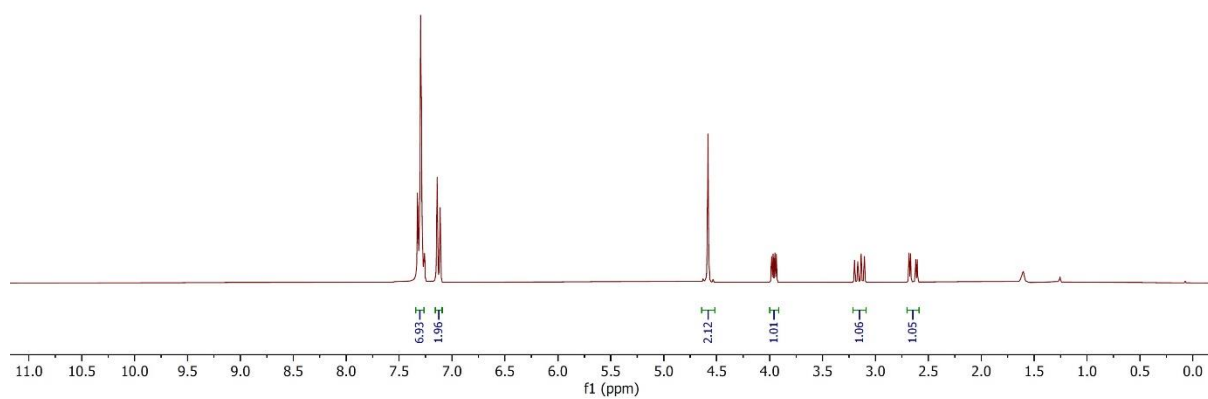
3.98
3.97
3.95
3.94

3.20
3.17
3.13
3.10
2.68
2.67
2.62
2.60



3w

^1H NMR (300 MHz, CDCl_3)



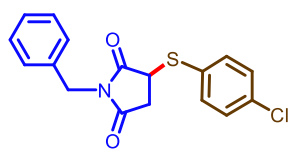
DKM-DJB-139-13C

175.02
173.85

135.79
135.78
135.77
129.53
128.92
128.69
128.32
128.13

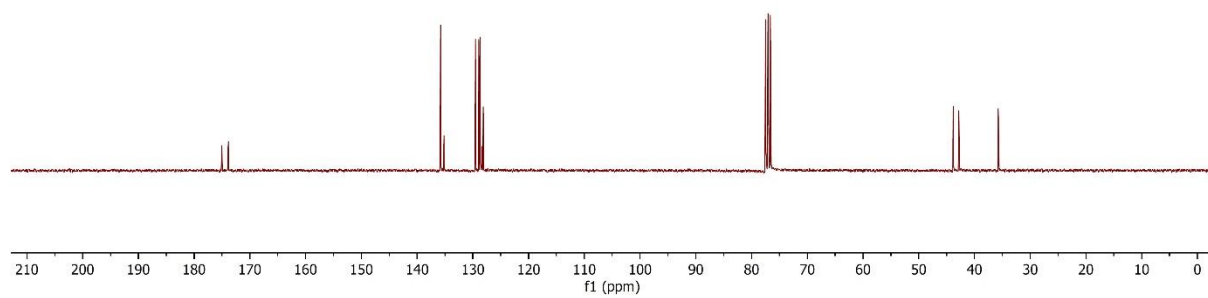
77.46
77.04
76.62

43.82
42.81
35.73



3w

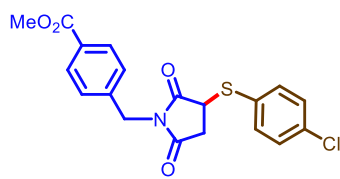
^{13}C NMR (75 MHz, CDCl_3)



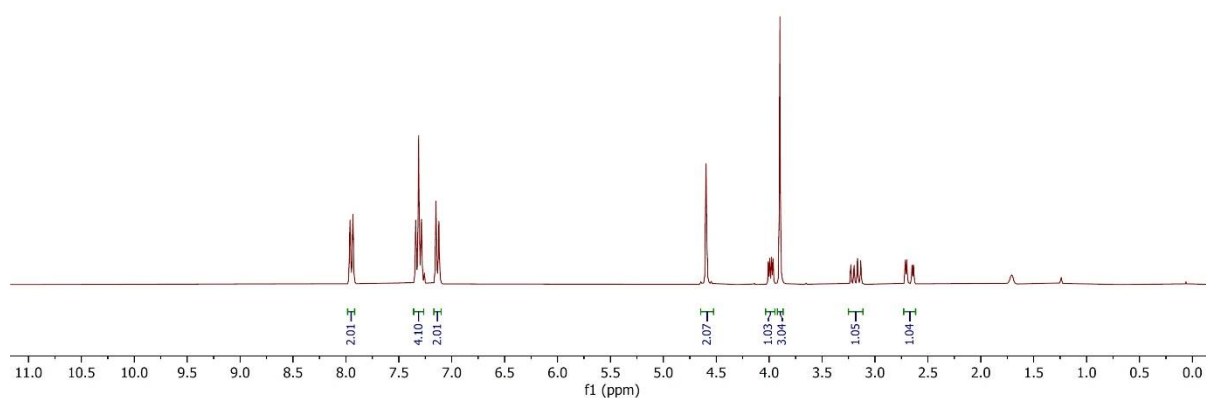
DKM-DJB-183

7.96
7.95
7.34
7.29
7.26
7.15
7.12

4.60
4.01
3.99
3.98
3.90
3.32
3.20
3.17
3.13
2.71
2.70
2.65
2.64



^1H NMR (300 MHz, CDCl_3)



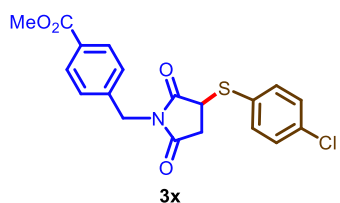
DKM-DJB-183-13C

174.64
173.75
166.60

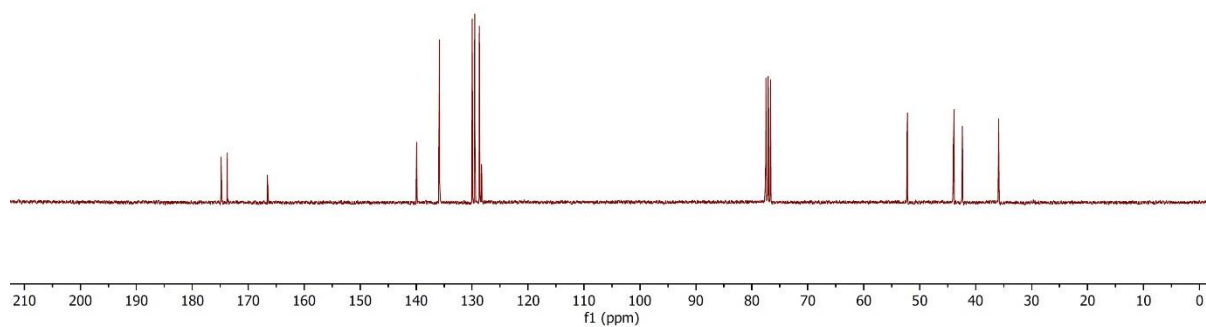
139.91
135.91
135.84
135.84
130.94
129.53
128.71
128.30

77.69
77.07
76.64

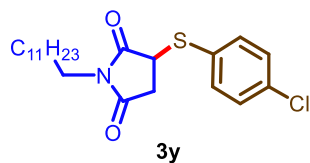
52.20
43.88
42.38
35.88



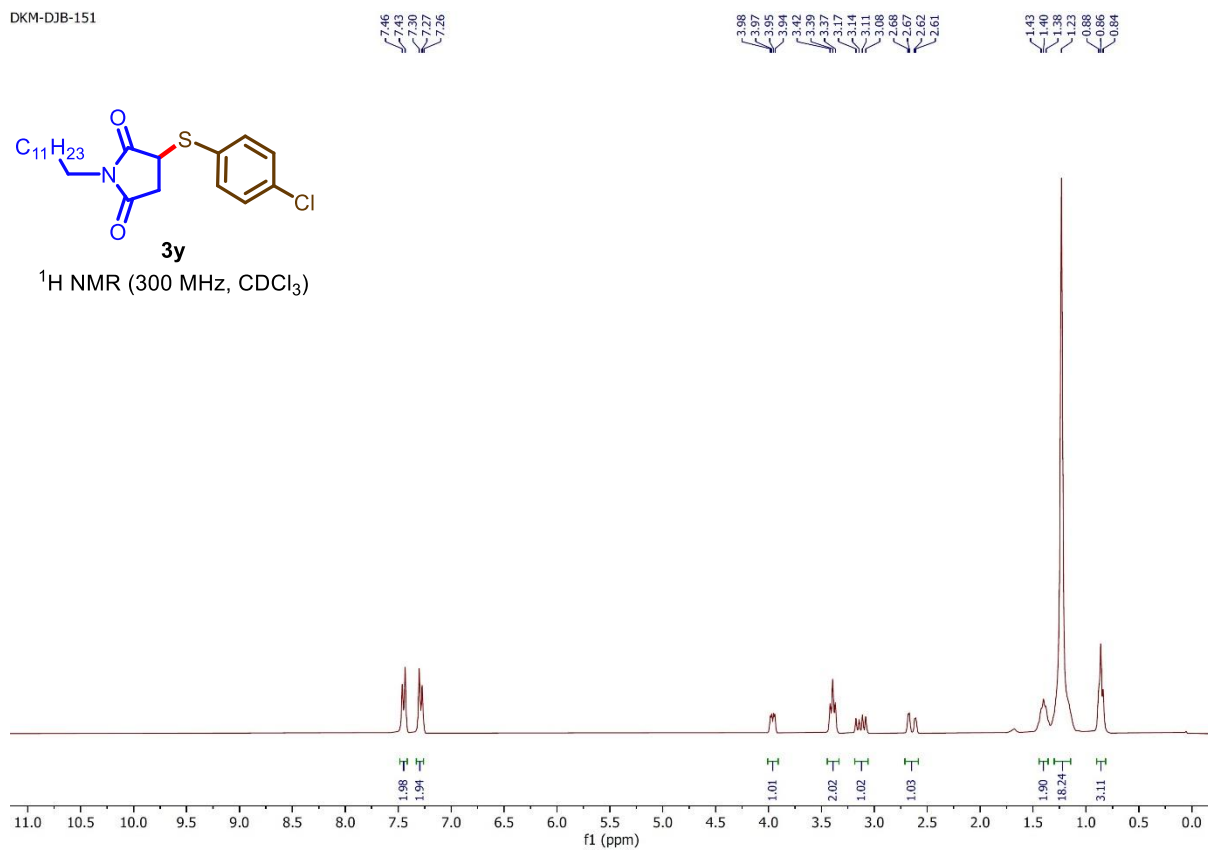
^{13}C NMR (75 MHz, CDCl_3)



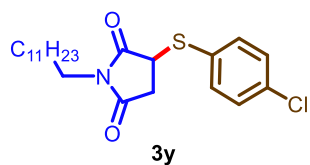
DKM-DJB-151



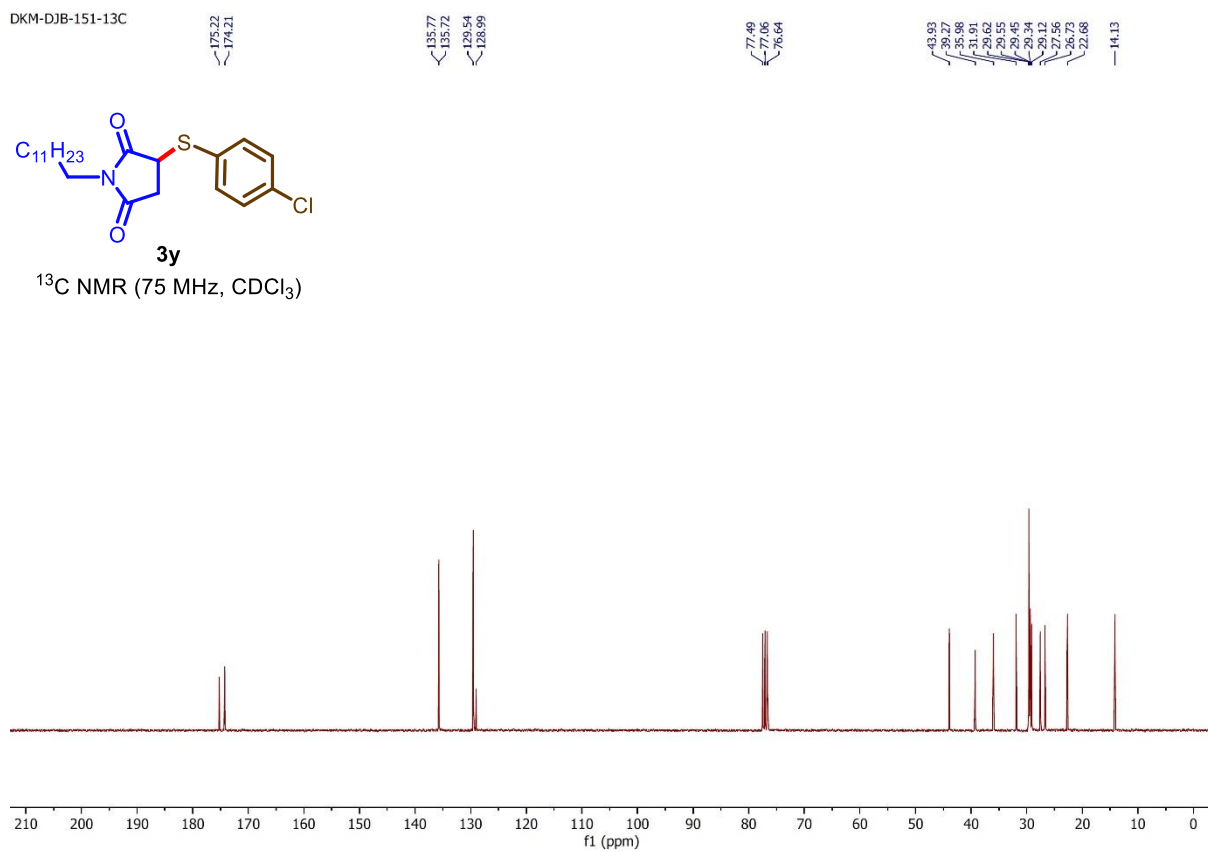
^1H NMR (300 MHz, CDCl_3)



DKM-DJB-151-13C



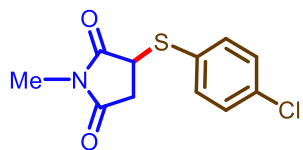
^{13}C NMR (75 MHz, CDCl_3)



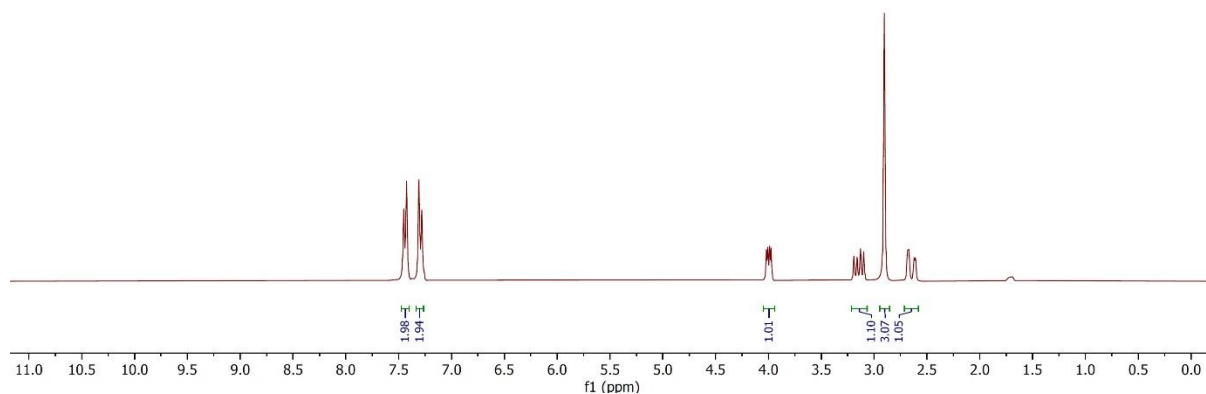
DKM-DJB-127

7.45
7.43
7.31
7.28
7.26

4.02
4.01
3.99
3.97
3.19
3.16
3.13
3.10
2.91
2.88
2.67
2.61



¹H NMR (300 MHz, CDCl₃)



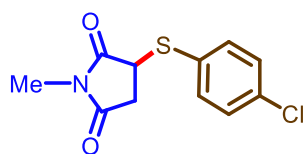
DKM-DJB-127-13C

175.41
174.22

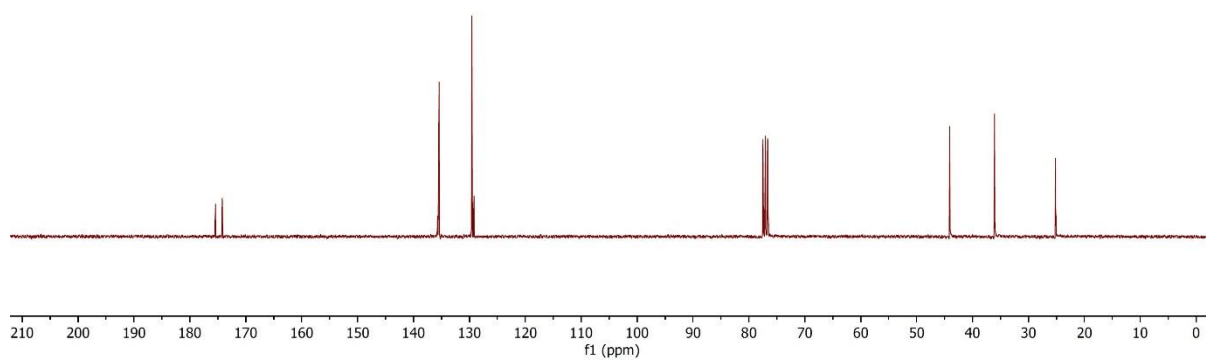
135.70
135.41
129.56
129.17

77.50
77.08
76.65

44.10
36.07
25.16



¹³C NMR (75 MHz, CDCl₃)

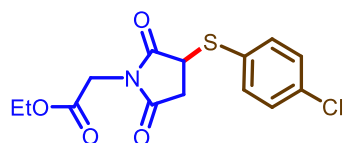


DKM-DJB-155

7.48
7.45
7.32
7.29
7.26

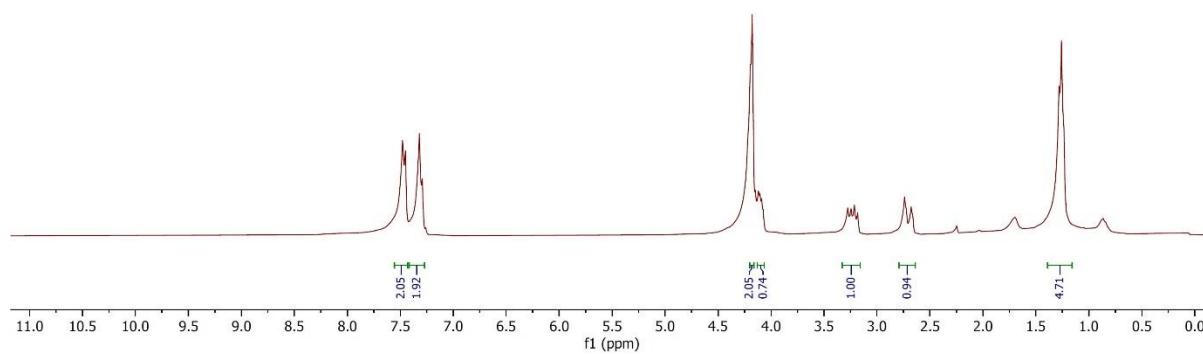
4.22
4.20
4.18
4.15
4.12
4.10
4.09
4.07
3.27
3.24
3.21
3.18
2.74
2.72
2.68
2.66

1.36
1.30
1.26
1.23



3aa

^1H NMR (300 MHz, CDCl_3)



DKM-DJB-155-13C

174.65
173.32
166.23

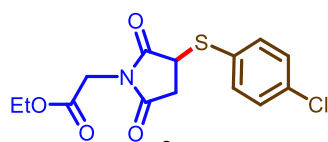
135.57
135.20
129.69
129.30

77.49
77.07
76.64

62.06

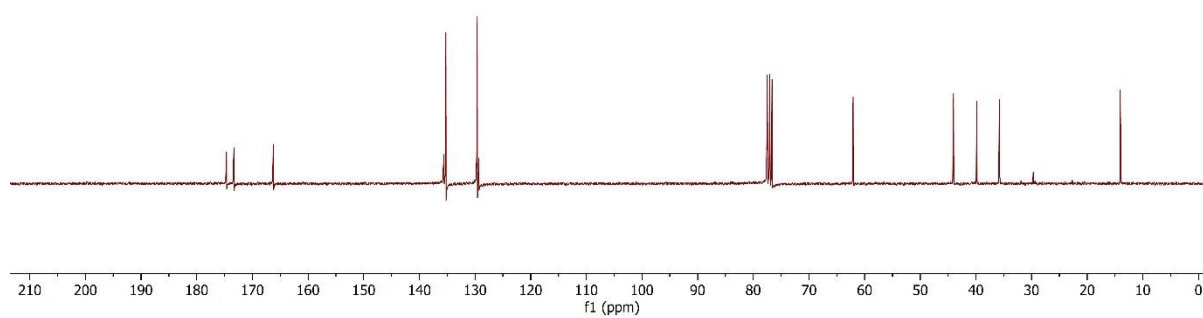
44.03
39.83
35.78

14.06



3aa

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

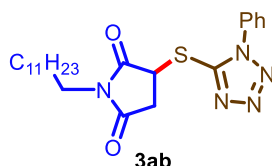


DKM-DJB-190LS.1.fid
1H CDCl3 DKM-DJB-190LS 21/4/23

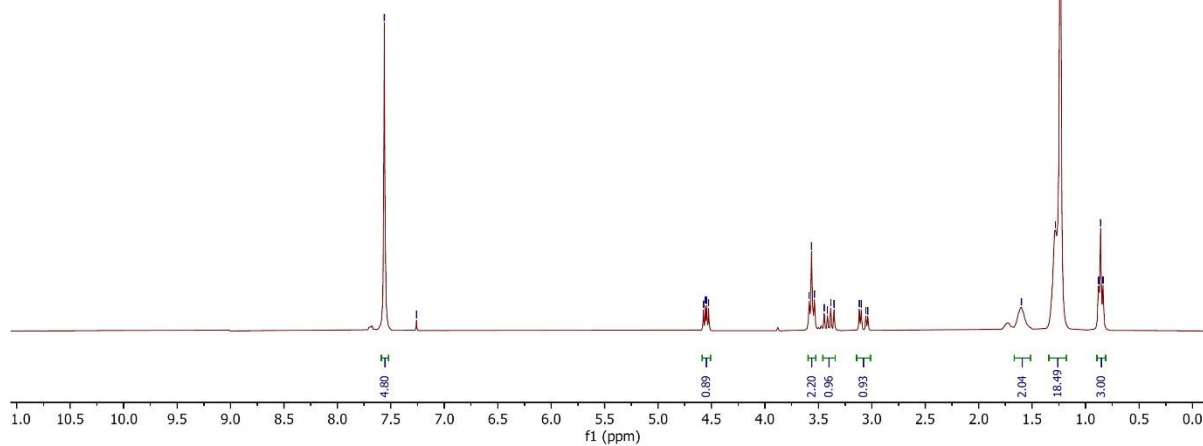
7.56
7.26

4.58
4.56
4.54
4.53
3.59
3.56
3.54
3.45
3.41
3.38
3.35
3.32
3.10
3.04

1.60
1.28
1.24
0.88
0.84



¹H NMR (300 MHz, CDCl₃)



DKM-DJB-190LS-13C.3.fid

173.81
173.33

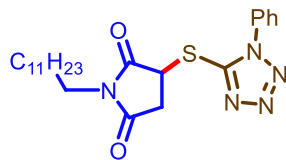
151.43

133.30
130.65
130.11

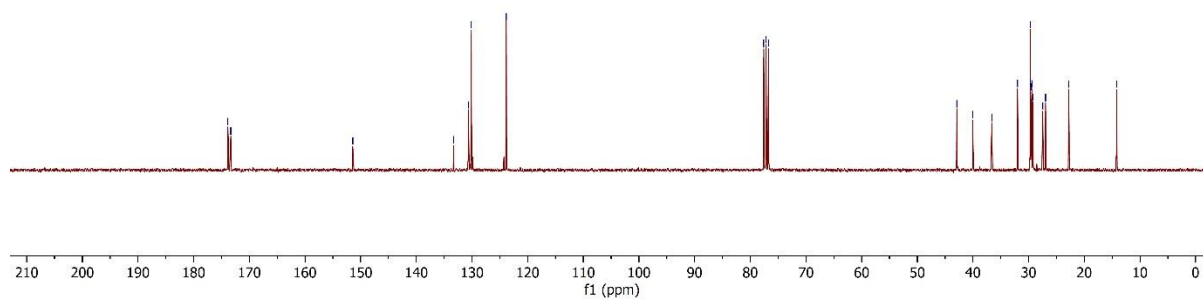
123.81

77.58
77.16
76.74

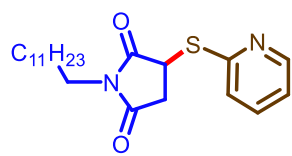
42.50
40.03
38.03
36.03
34.03
32.01
29.66
29.56
29.43
29.21
27.45
26.96
22.77
14.22



¹³C NMR (75 MHz, CDCl₃)

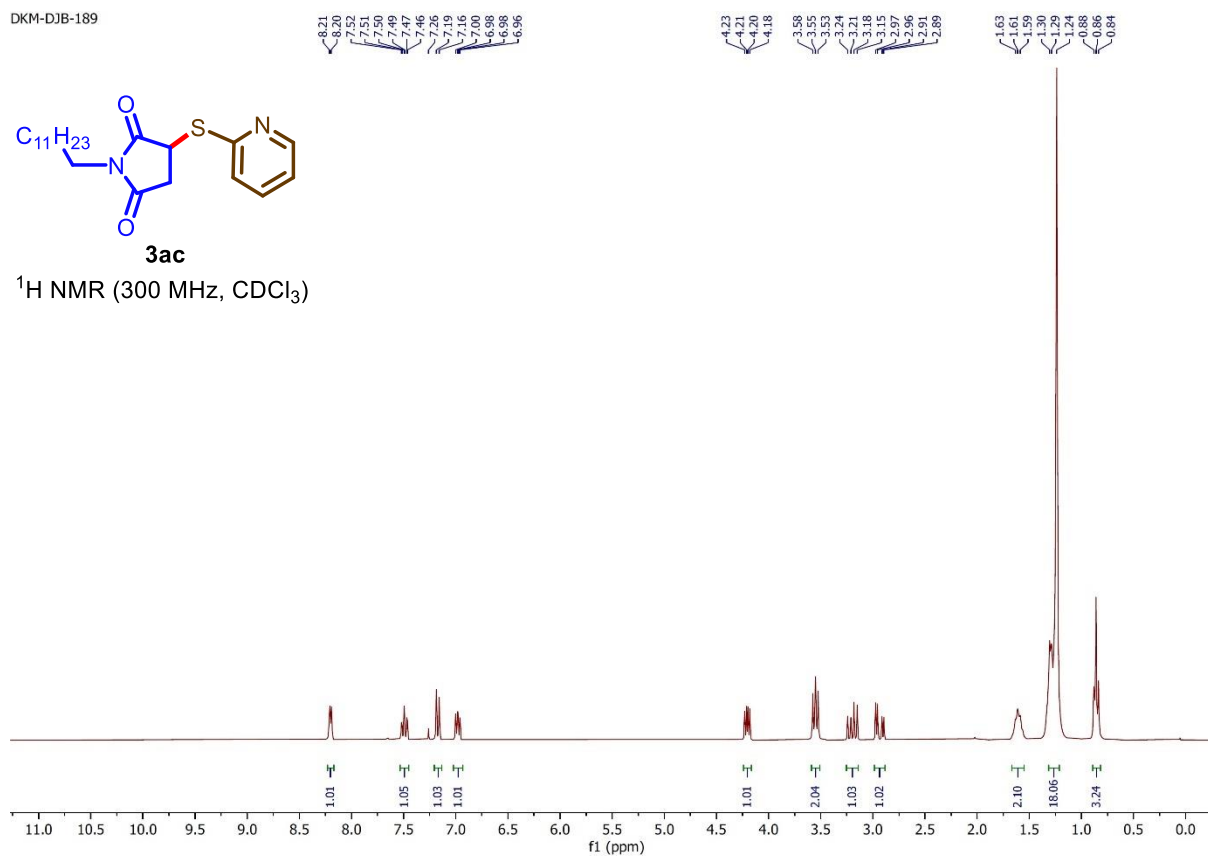


DKM-DJB-189

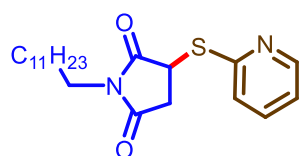


3ac

¹H NMR (300 MHz, CDCl₃)

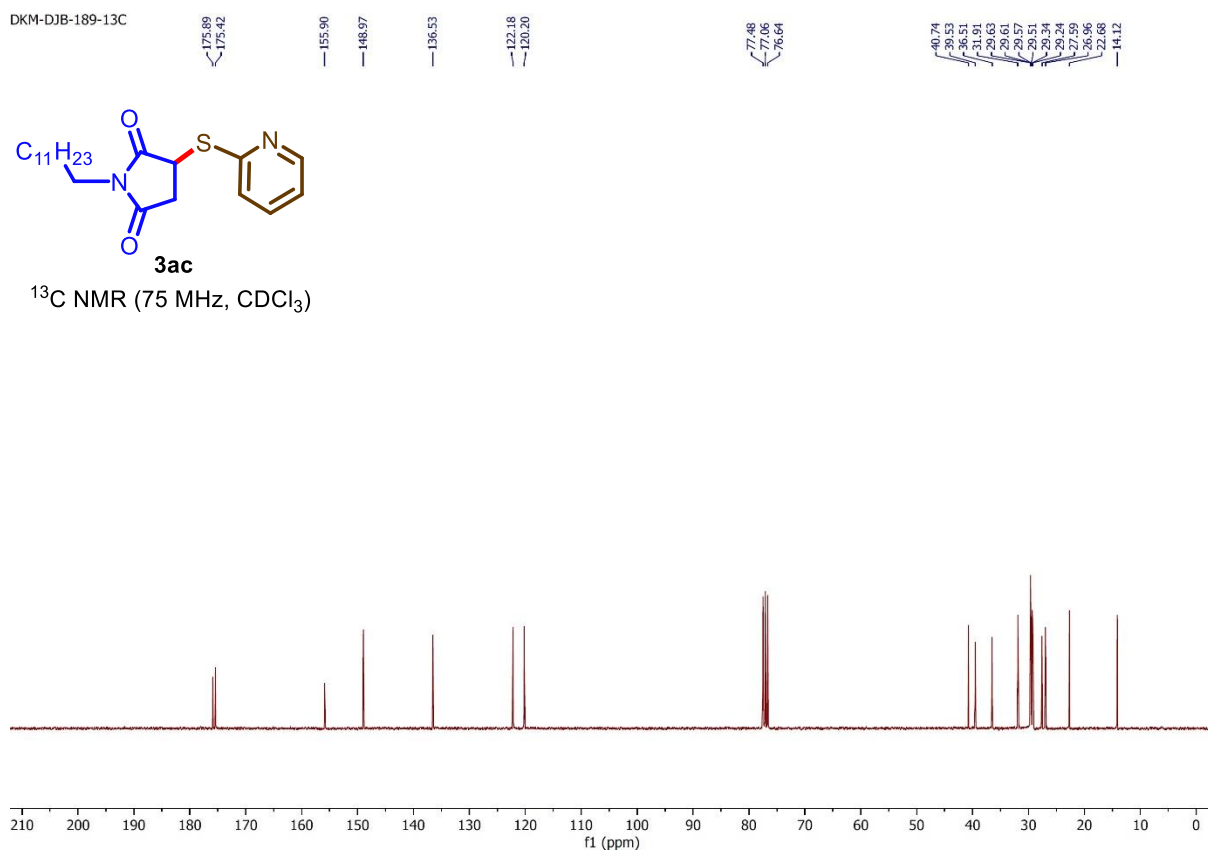


DKM-DJB-189-13C



3ac

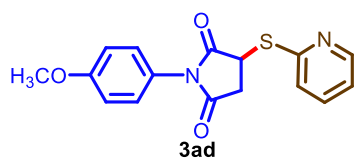
¹³C NMR (75 MHz, CDCl₃)



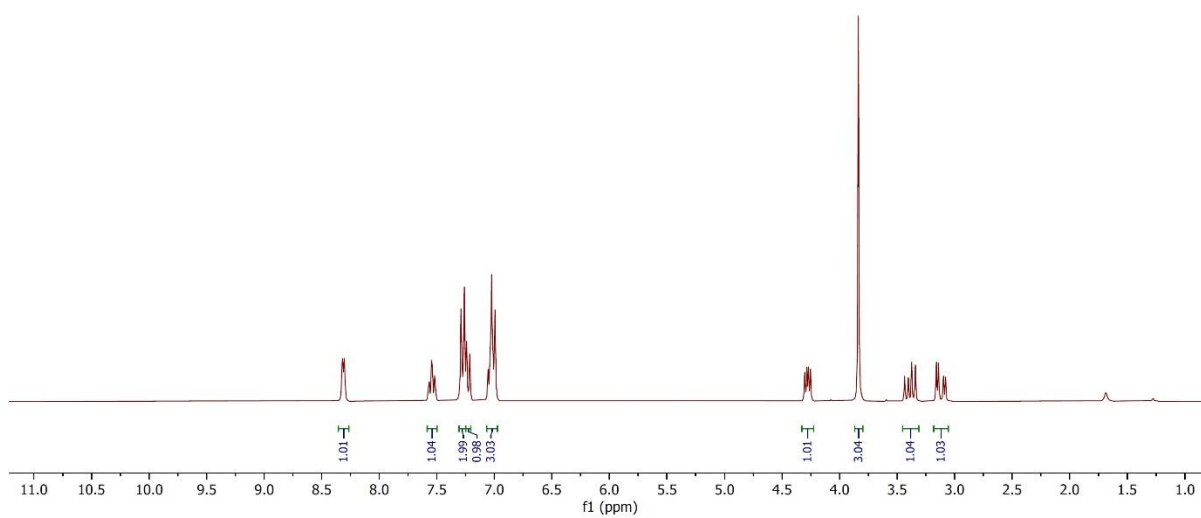
DKM-DJB-191

8.32
8.30
7.57
7.55
7.52
7.29
7.28
7.27
7.26
7.24
7.21
7.05
7.02
6.99

4.30
4.28
4.27
4.25
3.84
3.43
3.40
3.37
3.34
3.16
3.14
3.08



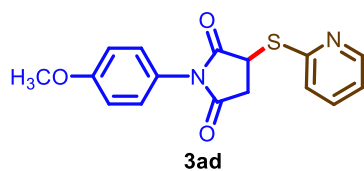
^1H NMR (300 MHz, CDCl_3)



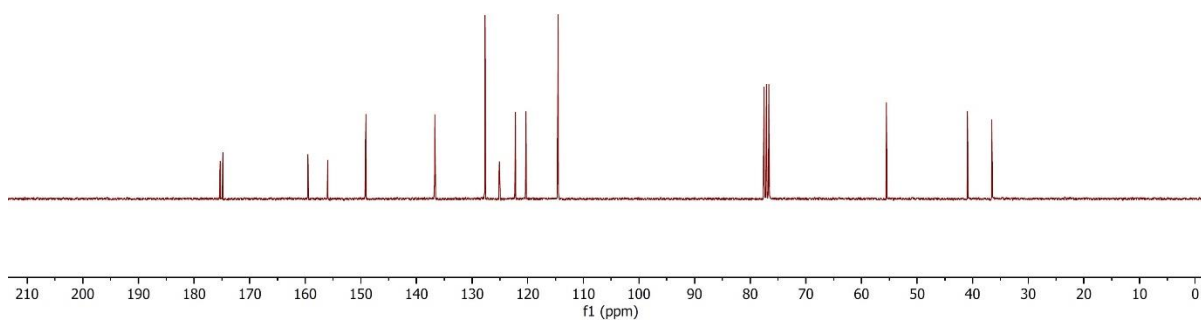
DKM-DJB-191-13C

175.31
174.81
159.53
155.95
149.10
136.72
127.67
125.11
122.21
120.32
114.54

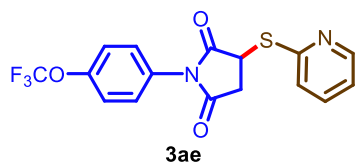
77.51
77.08
76.65
55.51
40.94
36.56



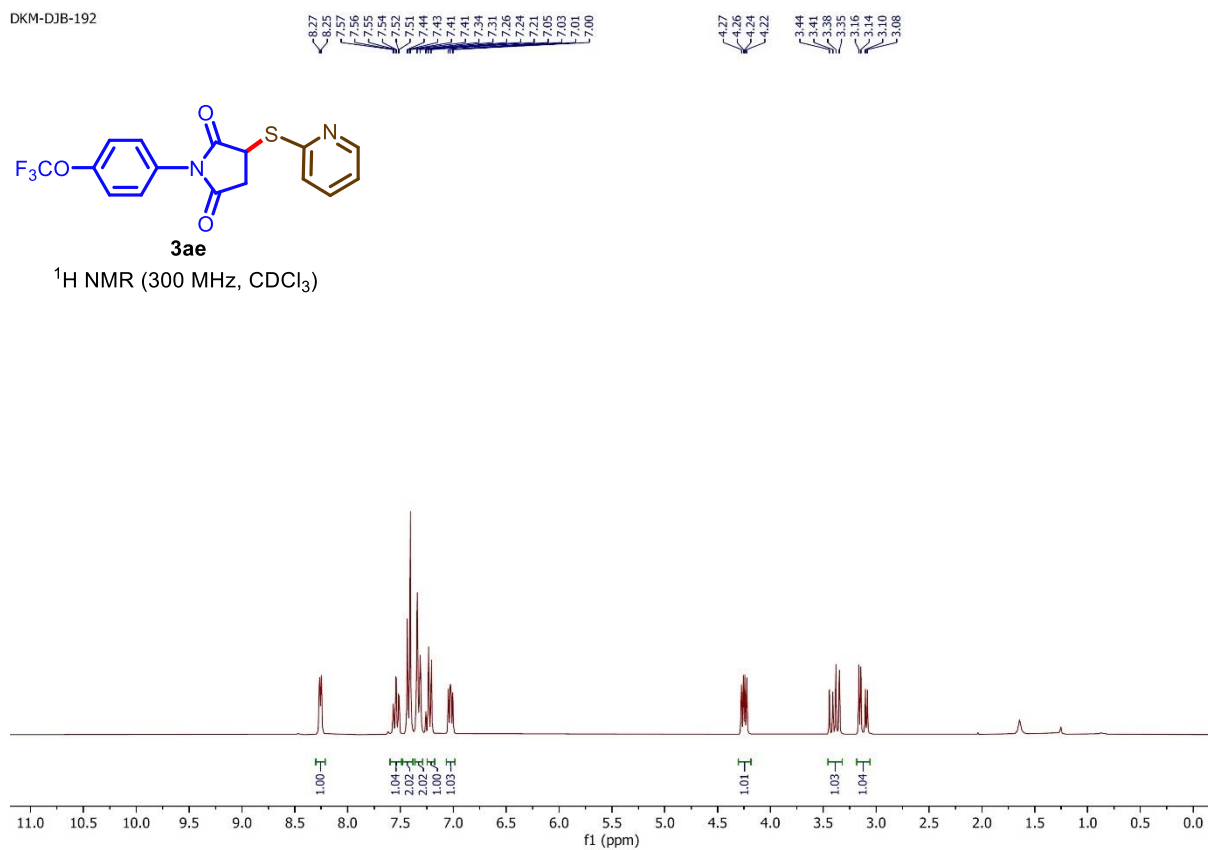
^{13}C NMR (75 MHz, CDCl_3)



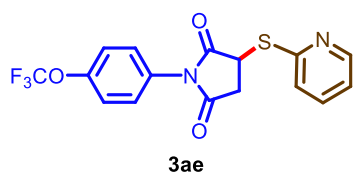
DKM-DJB-192



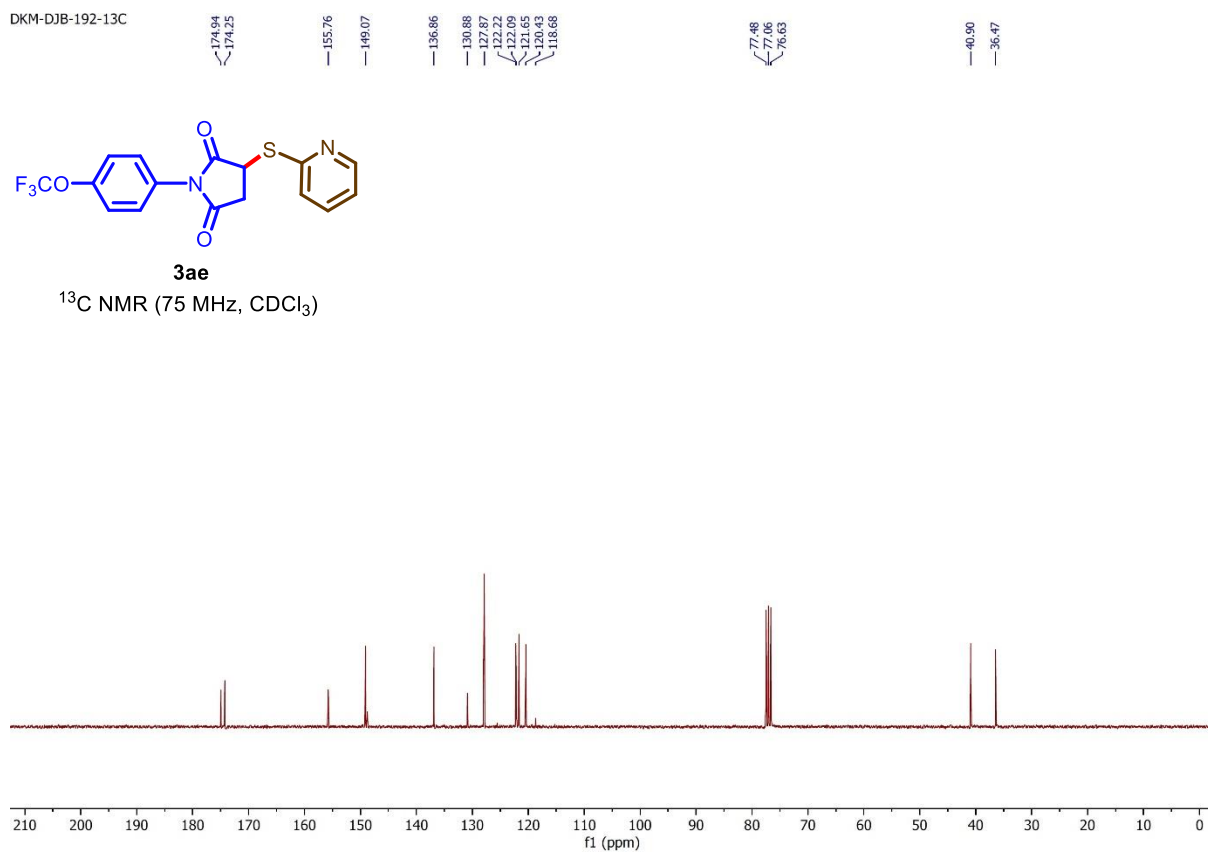
^1H NMR (300 MHz, CDCl_3)



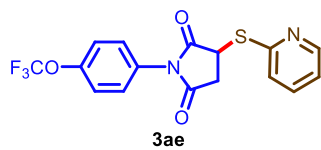
DKM-DJB-192-13C



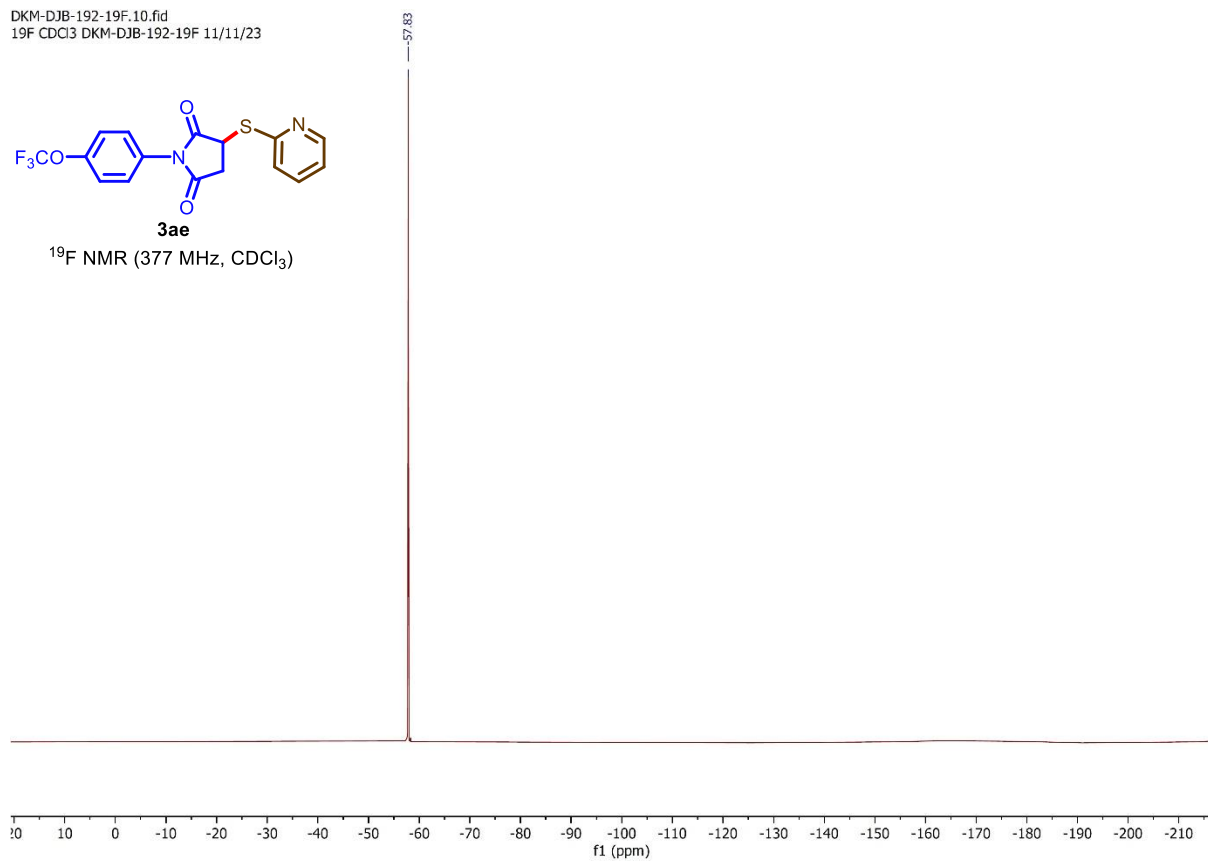
^{13}C NMR (75 MHz, CDCl_3)



DKM-DJB-192-19F.10.fid
19F CDCl3 DKM-DJB-192-19F 11/11/23



¹⁹F NMR (377 MHz, CDCl₃)



DKM-DJB-289

8.13
8.10
7.53
7.50
7.35
7.33
7.26
7.23
7.20

5.13
5.11
4.91
4.88
4.85
4.81

4.17
4.16
4.14
4.13

3.52
3.35
3.33
3.32

2.92
2.90
2.85
2.84

2.44
2.04
2.01

2.00
1.96
1.99
1.94

1.60
1.57
1.54

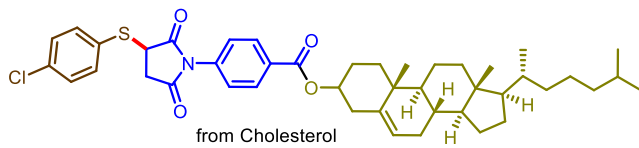
1.53
1.52
1.48
1.46

1.36
1.33
1.25
1.17

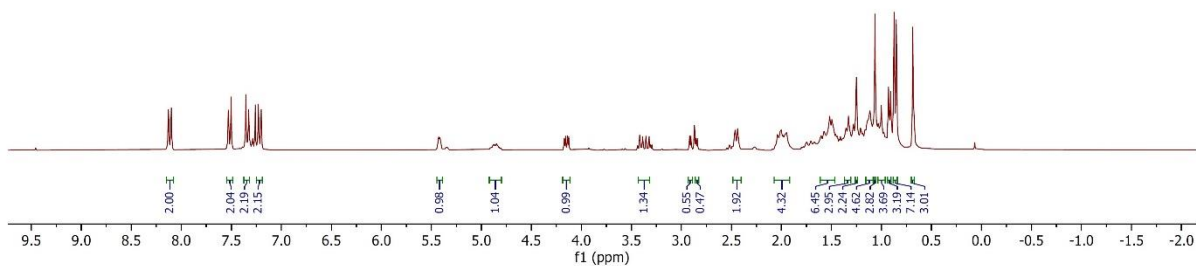
1.14
1.11
1.11

1.06
1.00
1.00

0.98
0.93
0.91
0.87
0.85
0.69



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



DKM-DJB-289

173.92
172.76
164.94

139.50
136.30
136.15
135.14
132.62
130.43
129.76
128.45
125.95
122.93

77.46
77.03
76.61
75.00

55.70
55.14

50.03
44.18
43.72
39.74

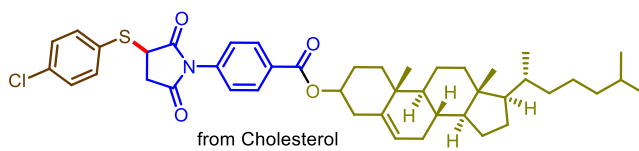
39.52
38.16
37.00
36.65

36.27
35.79
35.81

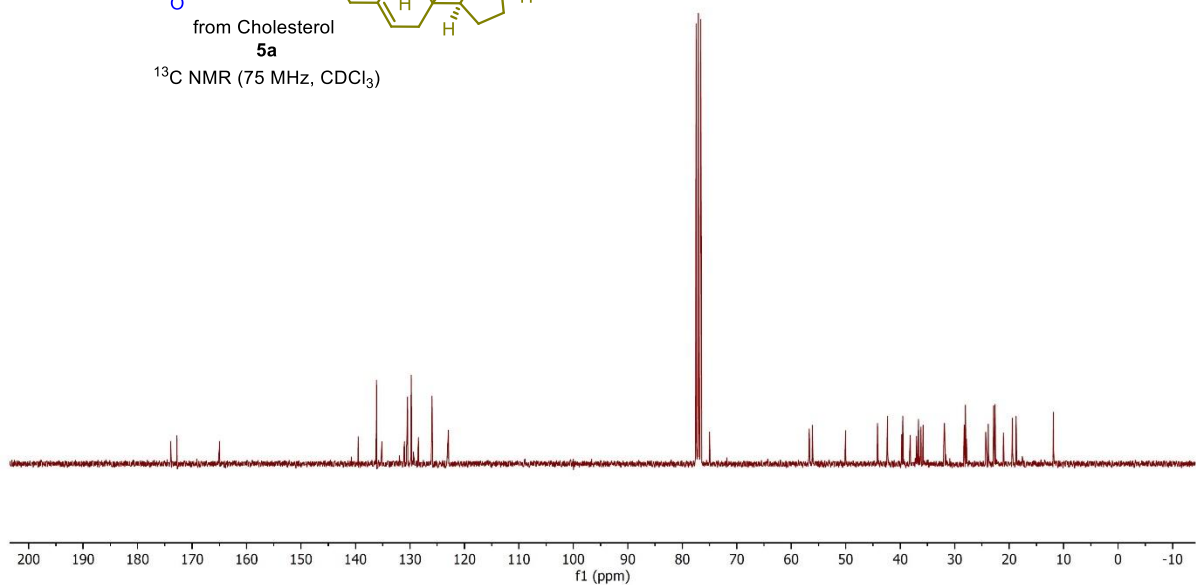
31.94
31.88
28.25
28.03

27.96
27.92
23.84
22.84

22.58
21.06
19.39
18.73
11.88



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



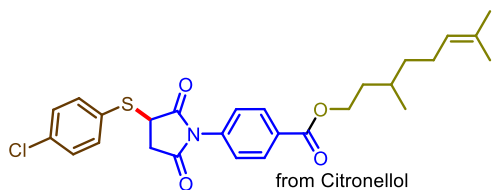
DKM-DJB-284R.1.fid

8.12
8.09
7.53
7.35
7.32
7.26
7.24
7.21

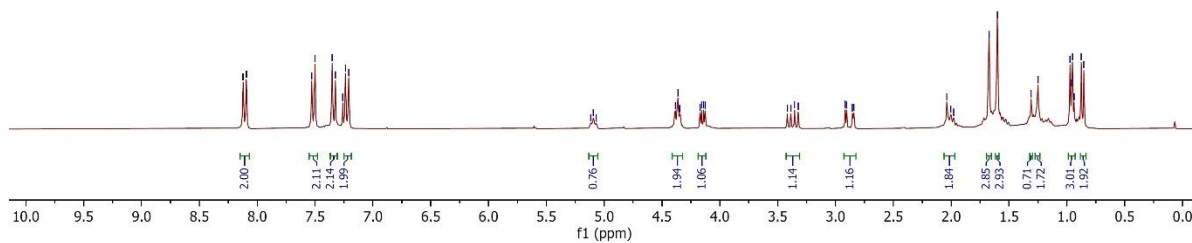
5.12
5.09
5.07
4.38
4.36
4.35
4.17
4.16
4.14
4.13

3.42
3.39
3.35
3.32
2.92
2.89
2.85
2.84

2.04
2.00
1.98
1.67
1.60
1.31
1.25
0.96
0.94
0.88
0.85



¹H NMR (300 MHz, CDCl₃)



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

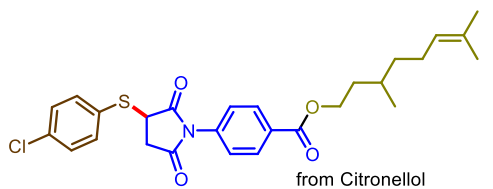
173.94
172.78
165.63

136.32
136.17
135.23
134.66
130.69
130.44
129.77
128.45
128.00
124.53

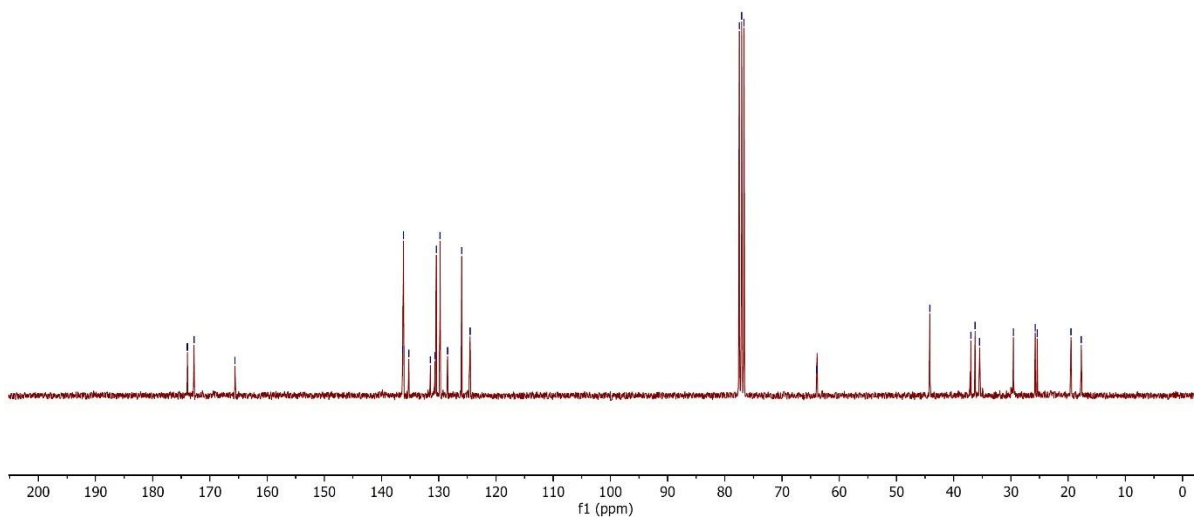
77.48
77.06
76.63

63.95

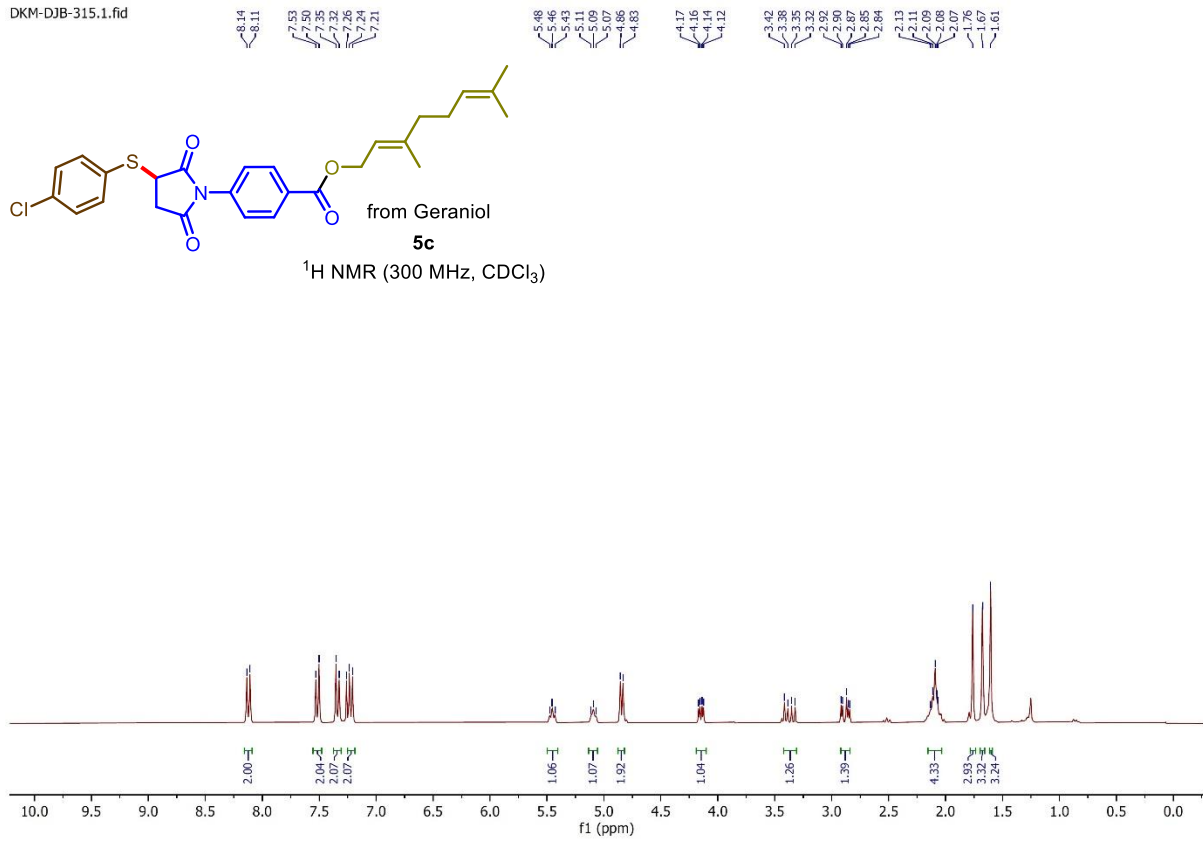
44.17
36.98
36.26
35.46
29.56
25.75
25.41
19.49
17.70



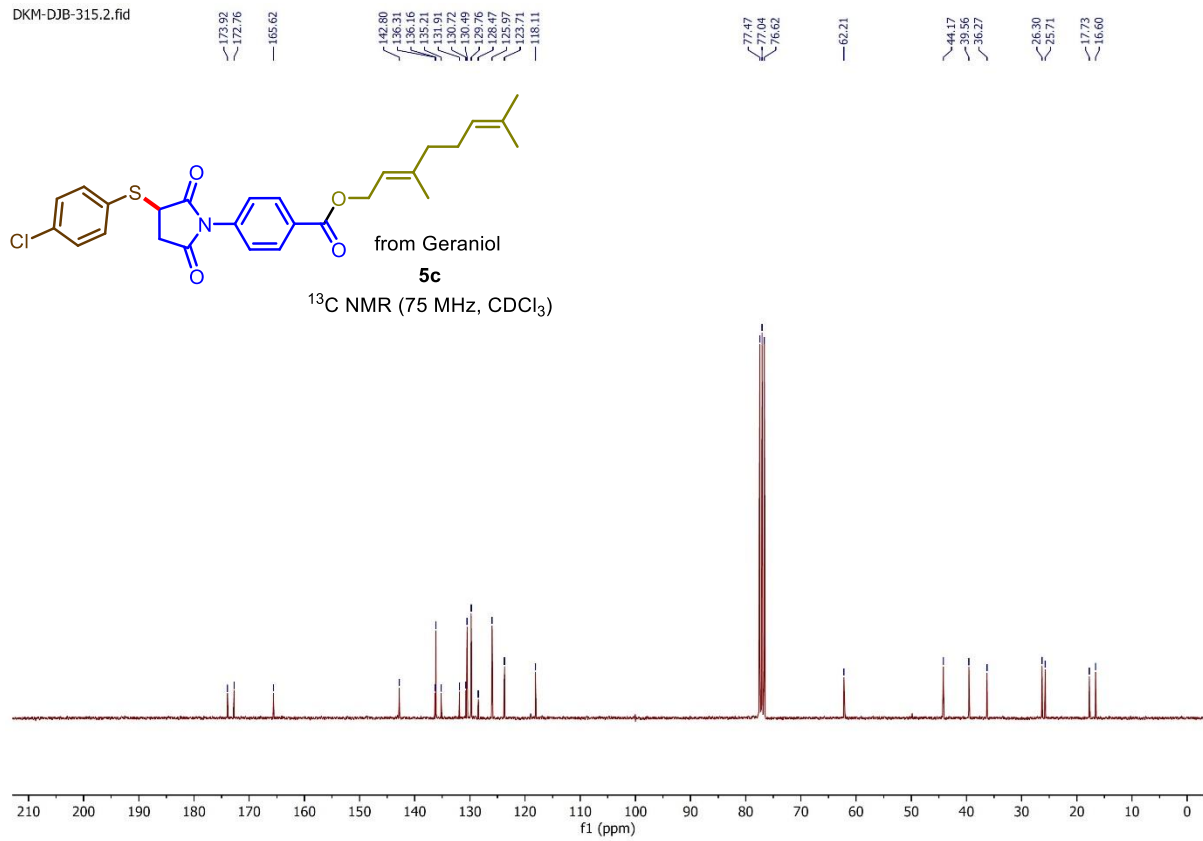
¹³C NMR (75 MHz, CDCl₃)



DKM-DJB-315.1.fid



DKM-DJB-315.2.fid



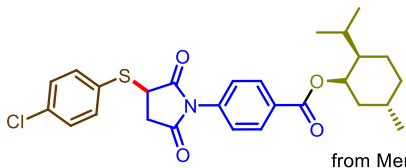
DKM-DJB-285

8.12
7.53
7.50
7.35
7.32
7.26
7.23
7.20

4.97
4.96
4.94
4.92
4.89
4.88
4.17
4.16
4.14
4.13

3.42
3.39
3.36
3.33
2.91
2.90
2.86
2.84

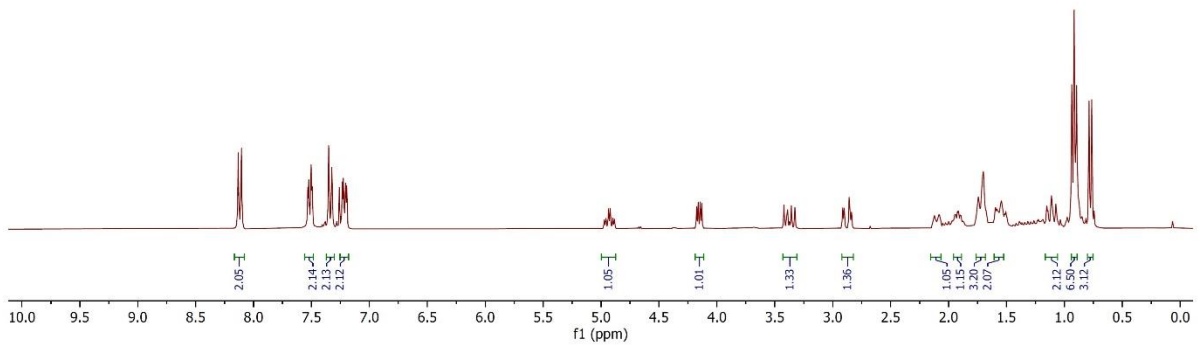
2.12
2.08
1.95
1.92
1.90
1.79
1.71
1.59
1.58
1.55
1.15
1.11
1.07
0.94
0.92
0.89
0.79
0.76



from Menthol

5d

¹H NMR (300 MHz, CDCl₃)



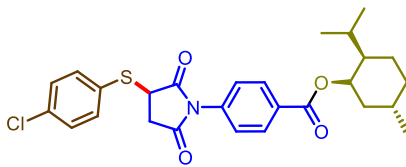
DKM-DJB-285-13C

173.95
172.80
165.03

136.20
136.15
135.14
131.04
130.47
129.89
129.75
126.01

77.47
77.05
76.62
75.29

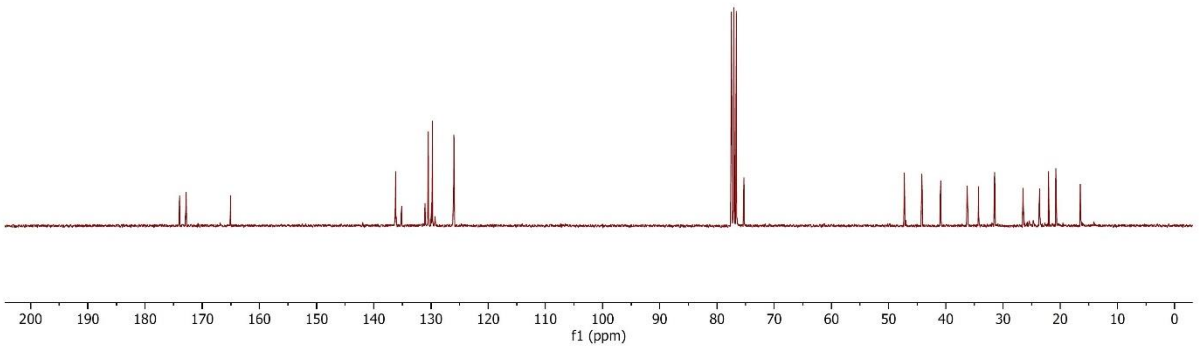
47.75
44.18
40.90
36.27
34.28
31.45
26.51
23.60
22.05
20.76
16.49



from Menthol

5d

¹³C NMR (75 MHz, CDCl₃)



DKM-DJB-226US

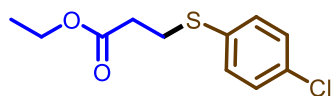
7.31
7.28
7.27
7.26
7.24

4.17
4.15
4.12
4.10

3.16
3.14
3.11

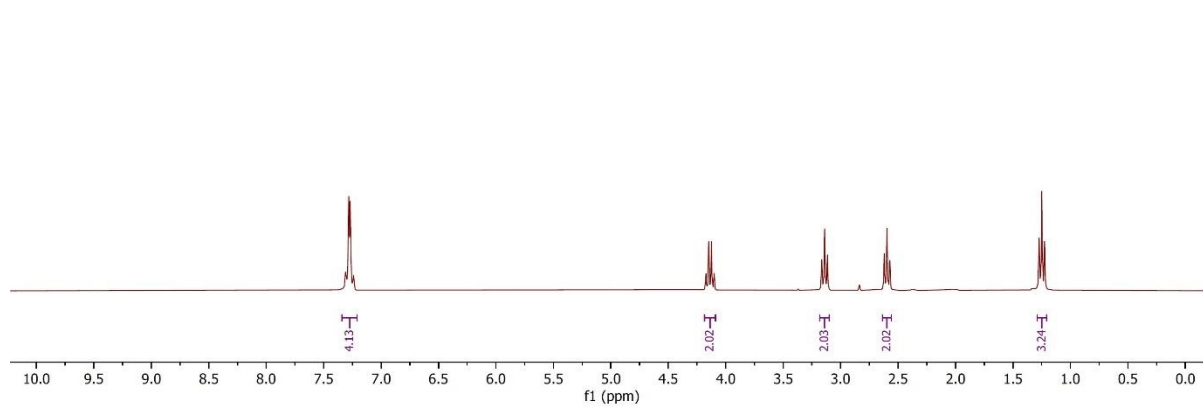
2.62
2.60
2.57

1.37
1.35
1.22



7a

^1H NMR (300 MHz, CDCl_3)



DKM-DJB-226US-13C

171.54

133.84
132.57
131.41
129.13

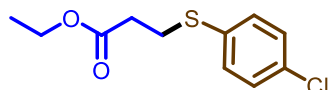
77.54
77.12
76.70

60.79

34.29

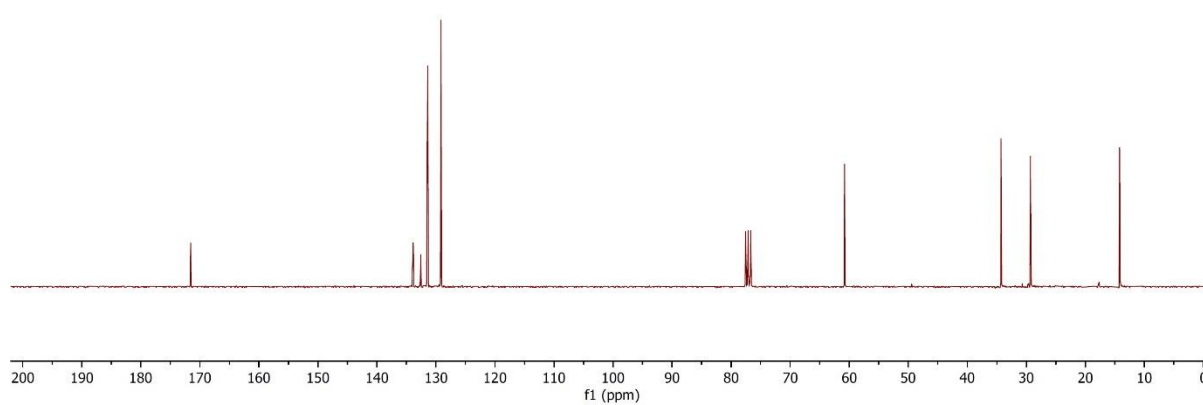
29.28

14.18



7a

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



DKM-DJB-266.1.fid
1H CDCl3 DKM-DJB-266 30/10/23

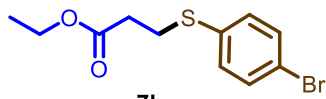
7.39
7.38
7.37
7.36
7.35
7.20
7.19
7.18
7.17

4.13
4.11
4.10
4.08

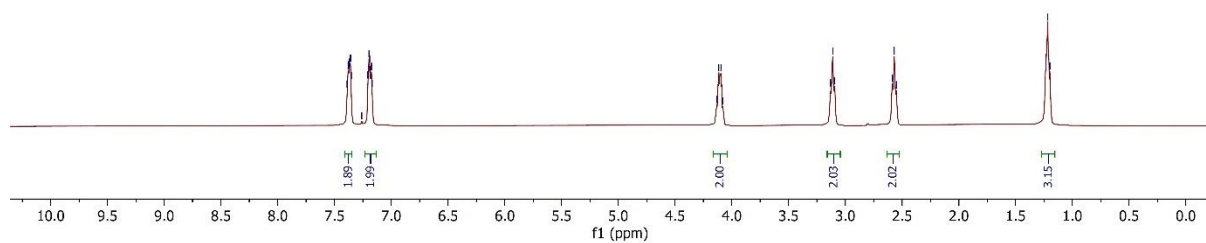
3.13
3.11
3.09

2.59
2.57
2.55

1.23
1.22
1.20



¹H NMR (400 MHz, CDCl₃)



DKM-DJB-266-13C.3.fid
13C CDCl3 DKM-DJB-266-13C 30/10/23

171.49

134.61
132.06
131.48

120.41

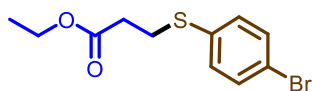
77.51
77.19
76.87

60.80

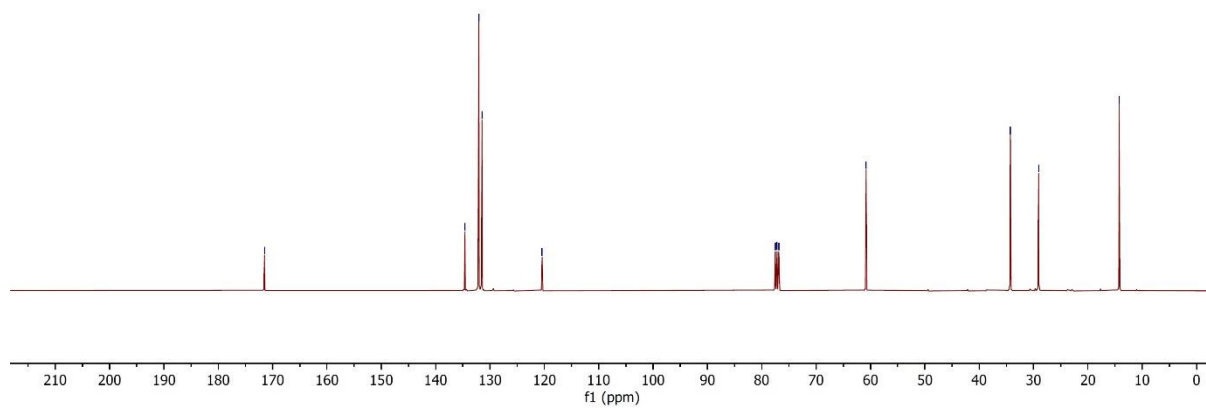
34.26

29.06

14.22



¹³C NMR (101 MHz, CDCl₃)



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

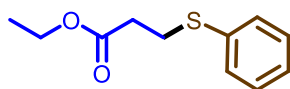
7.33
7.30
7.26
7.25
7.23
7.19
7.16
7.13
7.11

4.11
4.09
4.06
4.04

3.14
3.11
3.09

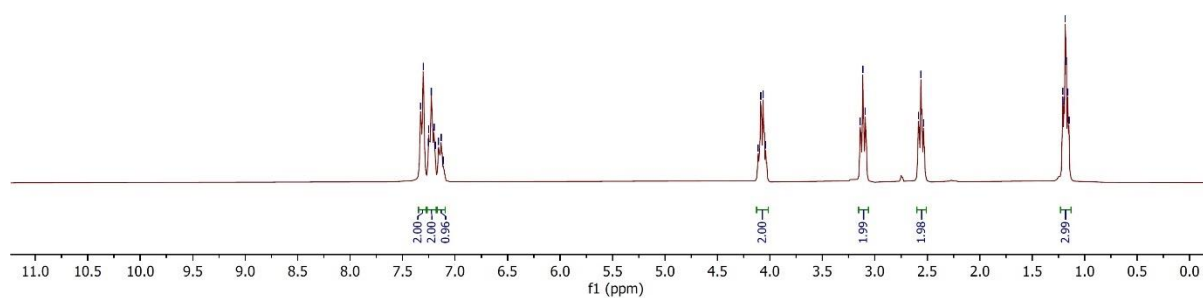
2.58
2.56
2.54

1.21
1.20
1.19
1.17
1.16
1.15



7c

¹H NMR (300 MHz, CDCl₃)



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

171.49

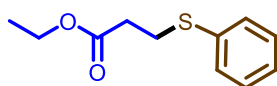
135.45
128.88
128.98
126.41

77.89
77.47
77.04

60.57

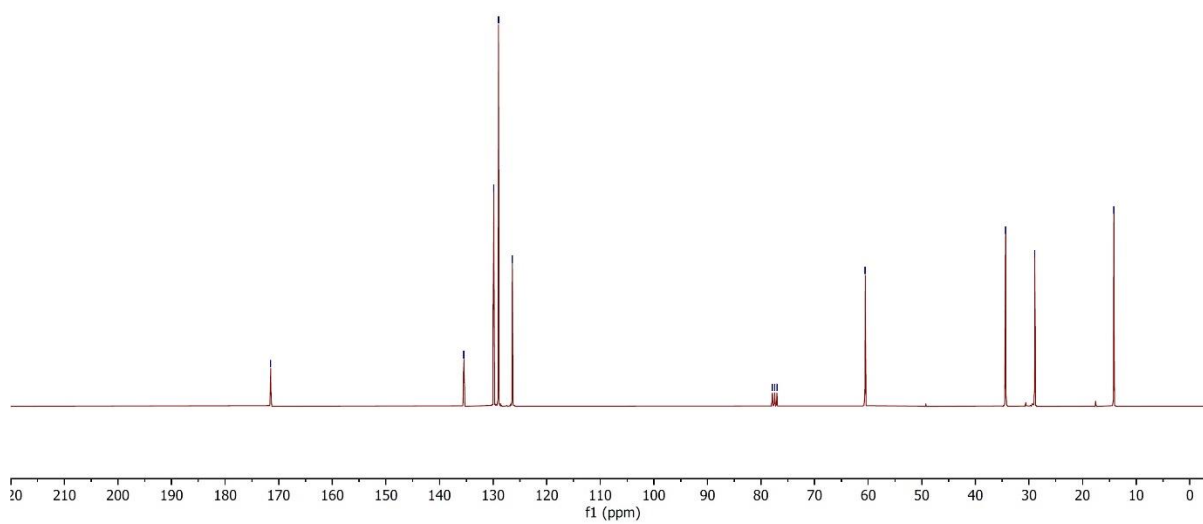
34.37
28.92

14.17



7c

¹³C NMR (75 MHz, CDCl₃)



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

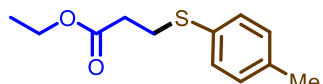
7.29
7.27
7.26
7.11
7.08

4.16
4.13
4.11
4.08

3.13
3.11
3.08

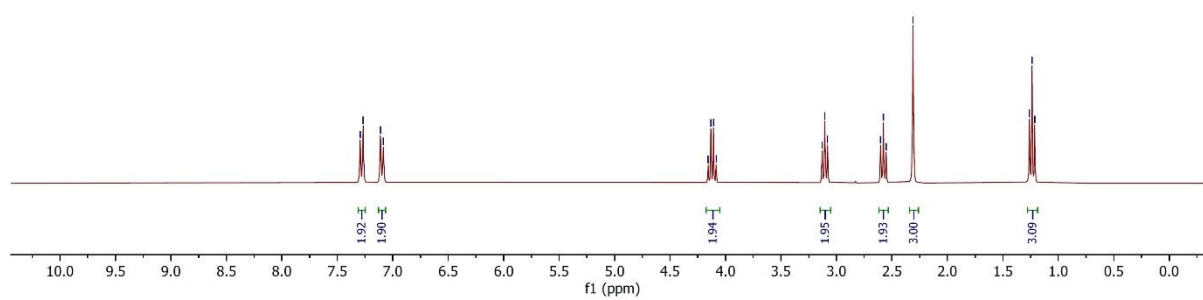
2.60
2.58
2.55
2.31

1.26
1.24
1.21



7d

¹H NMR (300 MHz, CDCl₃)



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

171.82

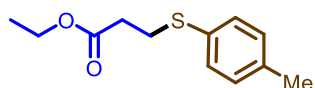
136.82
131.42
131.05
129.81

77.58
77.16
76.74

60.67

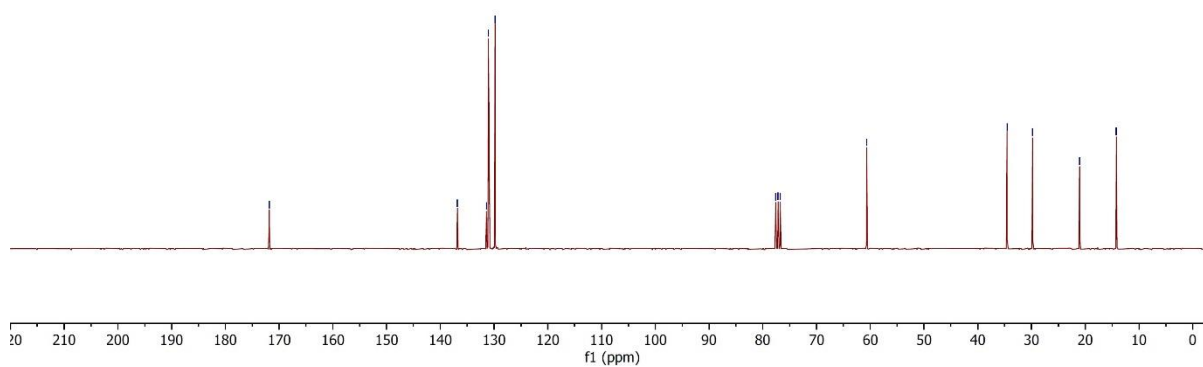
34.55
29.79

21.05
14.21



7d

¹³C NMR (75 MHz, CDCl₃)



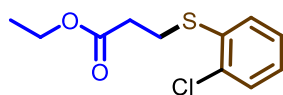
DKM-DJB-237

7.58
7.56
7.53
7.51
7.26
7.24
7.19
7.18
7.13
7.10

4.18
4.15
4.13
4.11

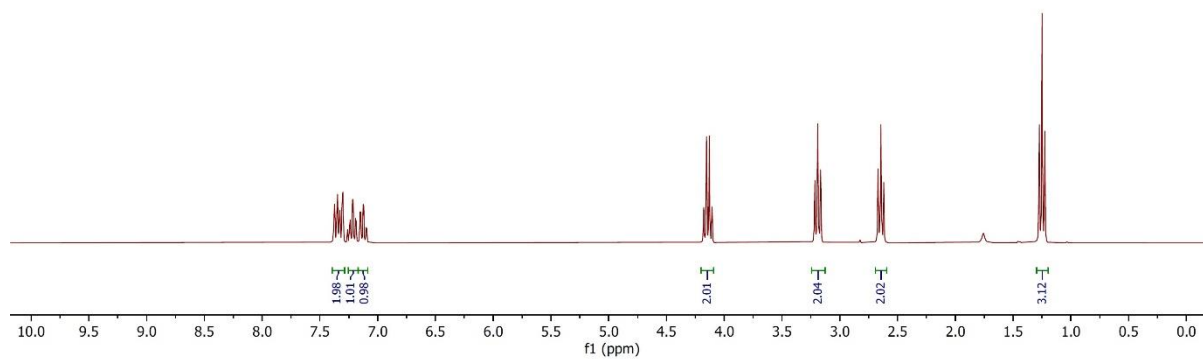
3.32
3.19
3.17
2.67
2.64
2.62

1.27
1.25
1.23



7e

¹H NMR (300 MHz, CDCl₃)



DKM-DJB-237-13C

171.53

134.76
134.22
129.86
129.30
127.21
127.10

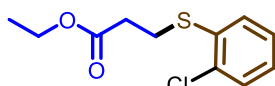
77.52
77.09
76.67

60.86

33.93

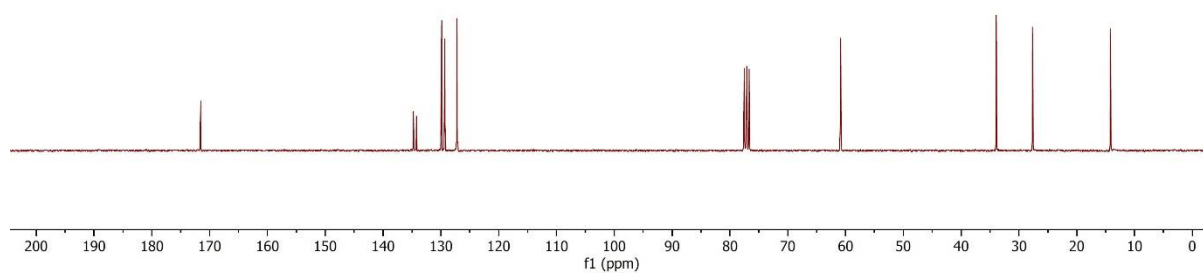
27.65

14.19



7e

¹³C NMR (75 MHz, CDCl₃)

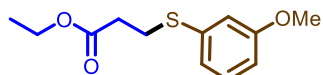


DKM-DJB-236

7.26
7.22
7.19
7.17
6.94
6.91
6.88
6.75
6.74
6.73
6.71

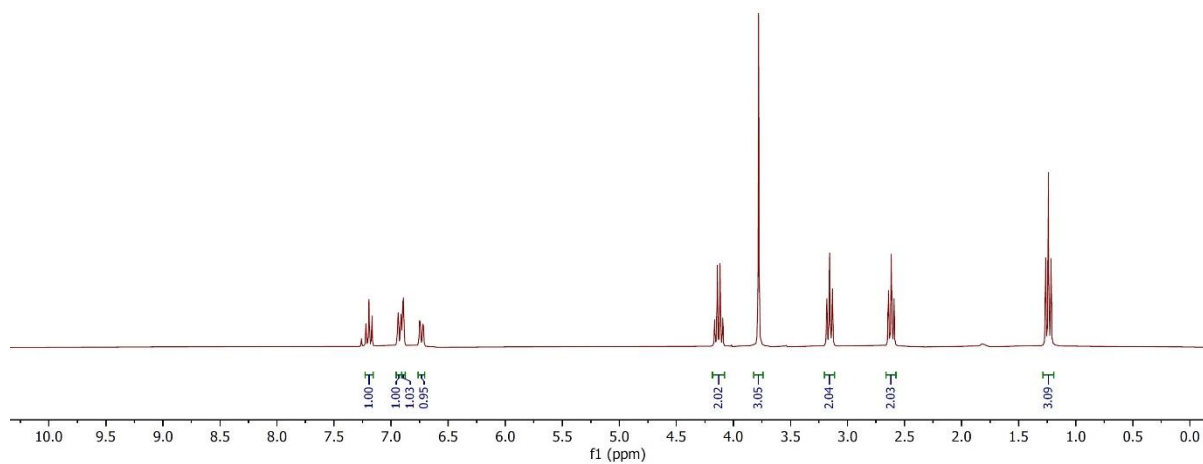
4.16
4.14
4.12
4.09
3.78
3.18
3.16
3.13
2.64
2.62
2.59

1.26
1.24
1.22



7f

^1H NMR (300 MHz, CDCl_3)



DKM-DJB-236-13C

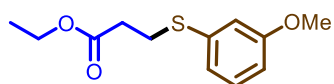
171.73
159.88
136.61
129.83
121.89
115.16
112.17

77.53
77.31
76.69

60.74
55.26

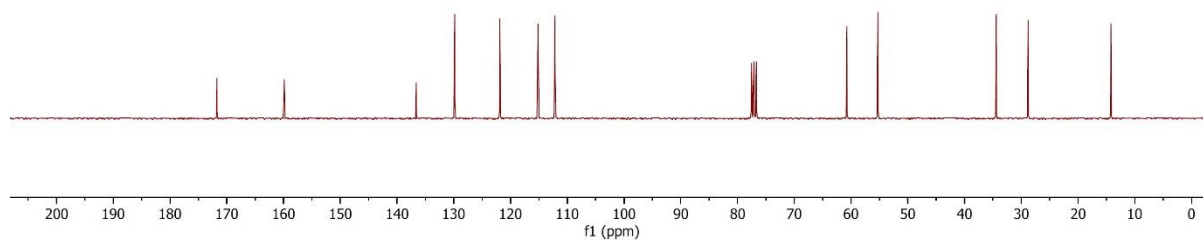
34.41
28.79

14.19



7f

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



DKM-DJB-238

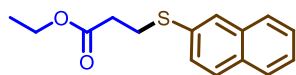
7.82
7.78
7.75
7.51
7.49
7.47
7.46
7.45
7.43
7.26

4.19
4.17
4.14
4.12

3.31
3.29
3.26

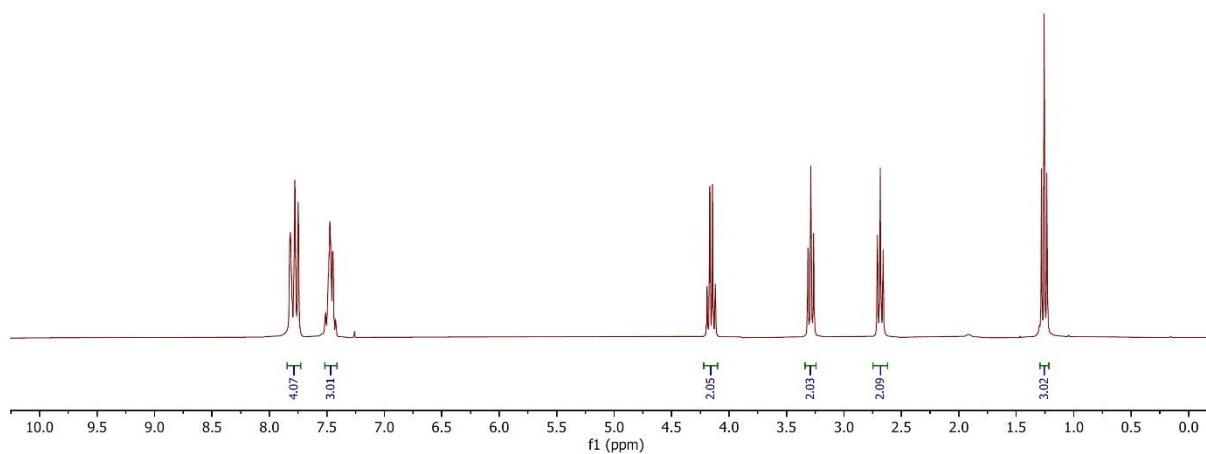
2.71
2.68
2.66

1.28
1.26
1.23



7g

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



DKM-DJB-238-13C

171.78

133.77
132.83
132.60
132.00
128.00
127.85
127.76
127.20
126.65
125.93

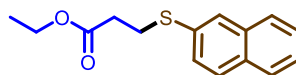
77.66
77.23
76.81

60.79

34.43

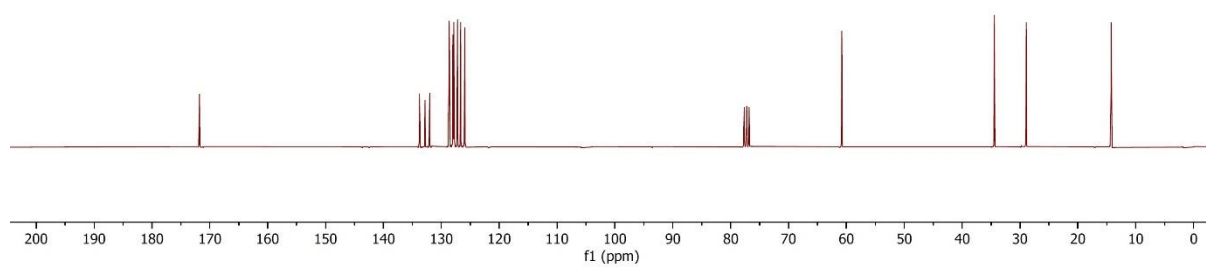
28.91

14.25



7g

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



DKM-DJB-239US.1.fid
1H CDCl3 DKM-DJB-239US 02/08/23

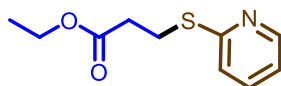
7.51
7.49
7.26
7.19
7.16
7.01
6.99

4.19
4.17
4.15
4.12

3.46
3.44
3.42

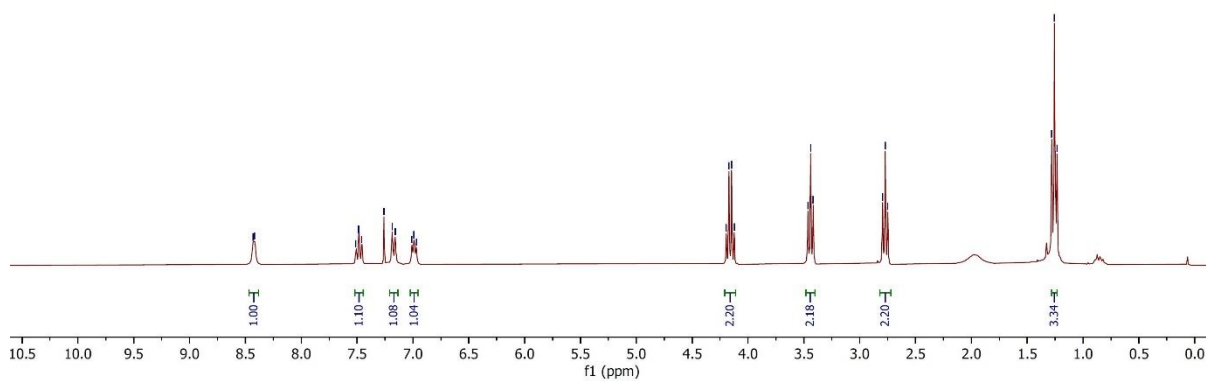
2.80
2.77
2.75

1.28
1.26
1.23



7h

¹H NMR (300 MHz, CDCl₃)



DKM-DJB-239US-13C.1.fid
1H CDCl3 DKM-DJB-239US-13C 18/9/23

172.11
158.19
149.13
136.31
122.57
119.58

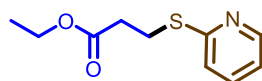
77.36
77.04
76.72

60.70

34.69

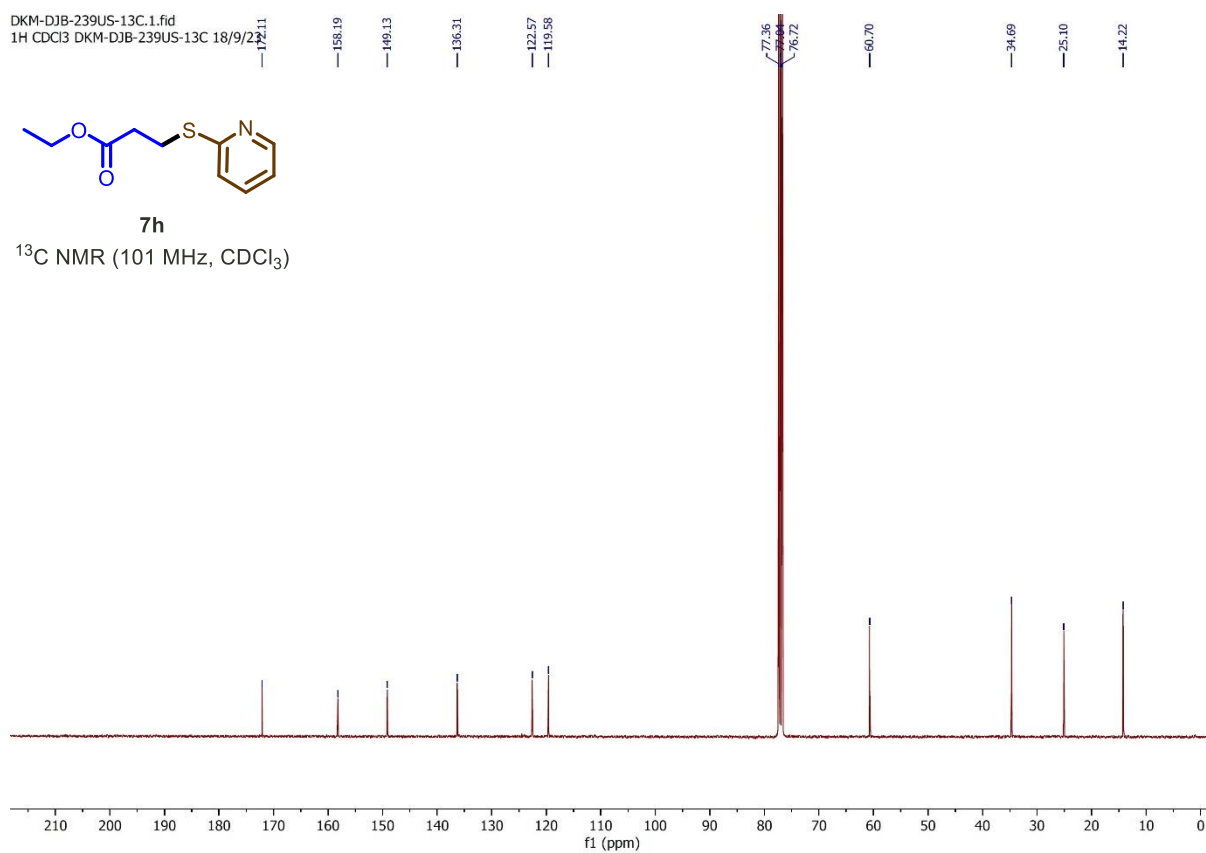
25.10

14.22



7h

¹³C NMR (101 MHz, CDCl₃)

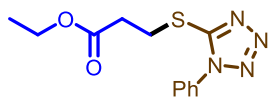


DKM-DJB-260LS.1.fid
1H CDCl3 DKM-DJB-260LS 30/10/23

7.95
7.94
7.53
7.52
7.26

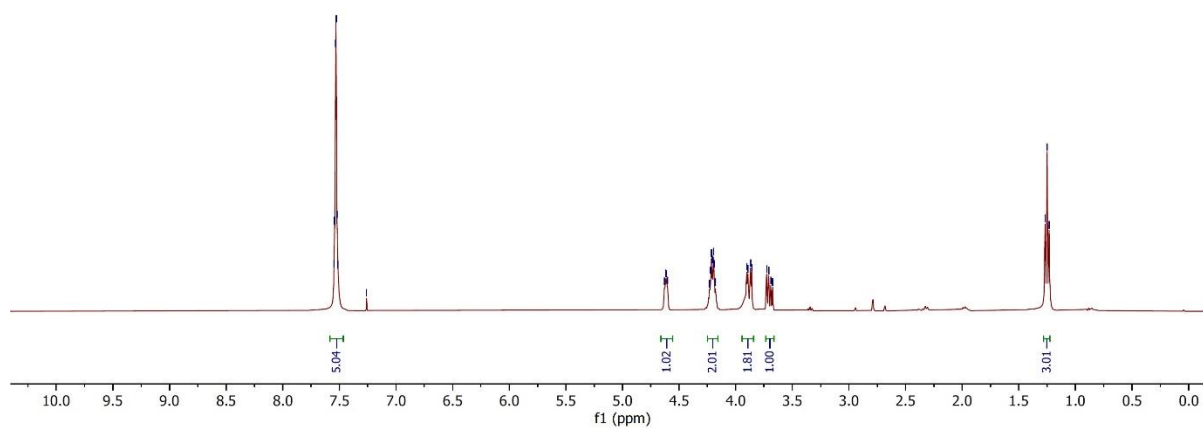
4.63
4.62
4.61
4.60
4.23
4.22
4.21
4.20
4.19
3.90
3.89
3.87
3.86
3.73
3.71
3.69
3.67

1.27
1.25
1.23



7i

¹H NMR (400 MHz, CDCl₃)



DKM-DJB-260LS-13C.2.fid

172.21

153.97

133.42

130.34

129.88

123.91

77.46

77.14

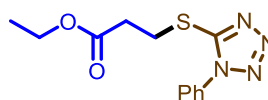
76.82

69.24

62.42

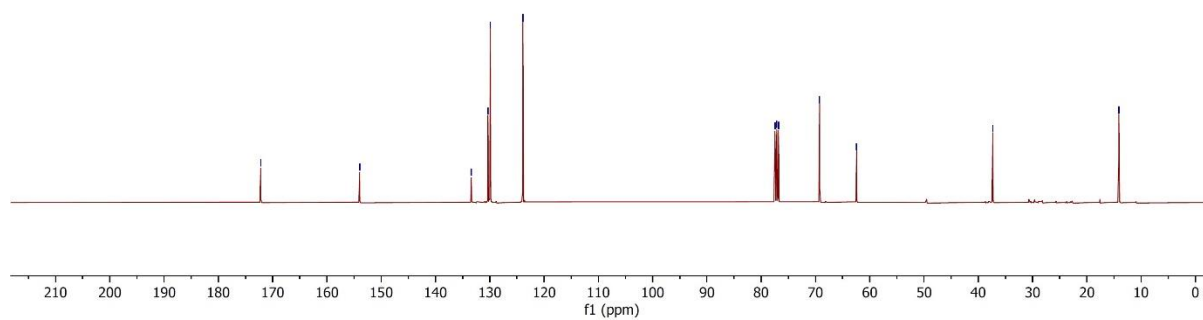
37.35

14.10



7i

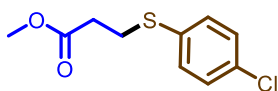
¹³C NMR (101 MHz, CDCl₃)



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

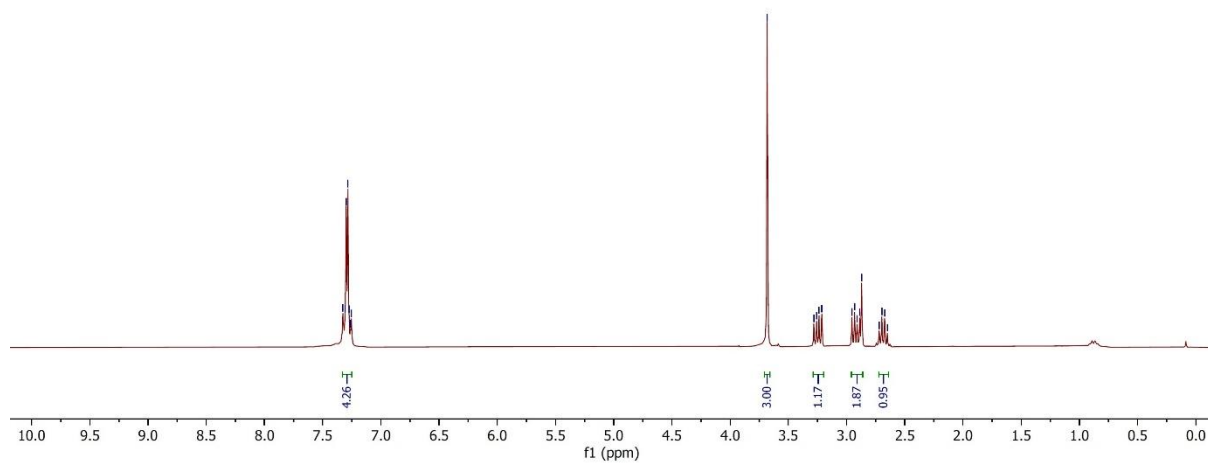
7.33
7.31
7.30
7.28
7.27
7.25

3.68
3.28
3.26
3.24
3.21
2.95
2.83
2.89
2.87
2.70
2.67
2.65



7j

¹H NMR (300 MHz, CDCl₃)



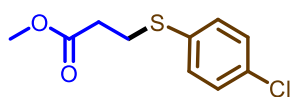
DKM-DJB-270.2.fid
13C_CDCl3_DKM-DJB-270_08.11.2021

134.21
132.55
131.46
129.12

77.48
77.06
76.63

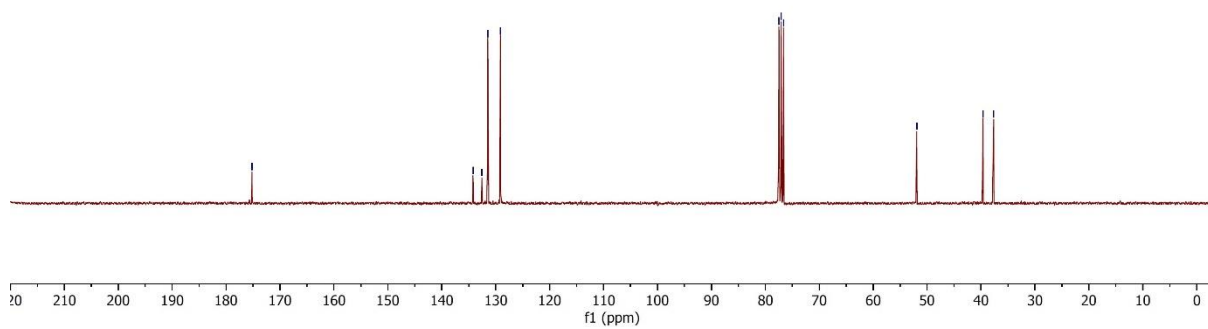
51.92

39.63
37.65

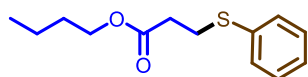


7j

¹³C NMR (75 MHz, CDCl₃)

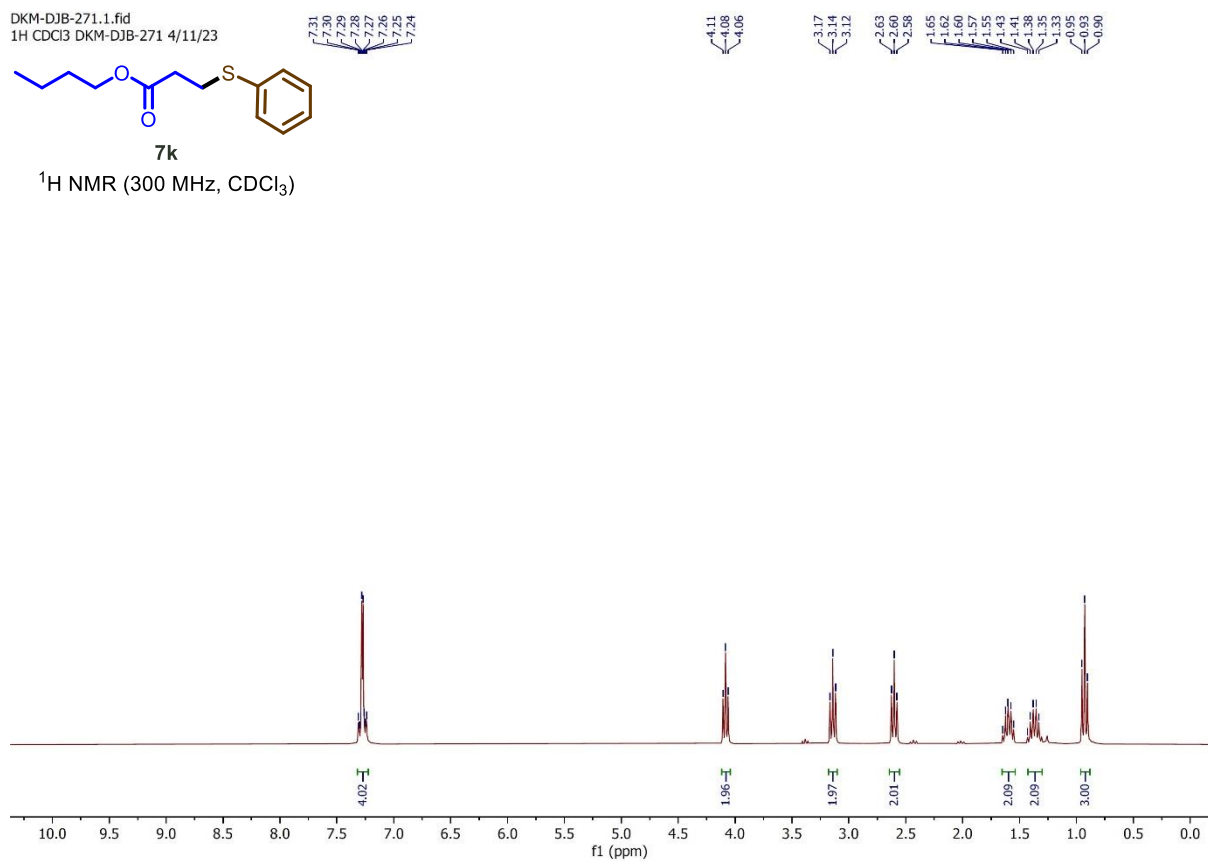


DKM-DJB-271-1.fid
1H CDCl3 DKM-DJB-271 4/11/23

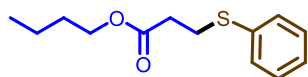


7k

¹H NMR (300 MHz, CDCl₃)

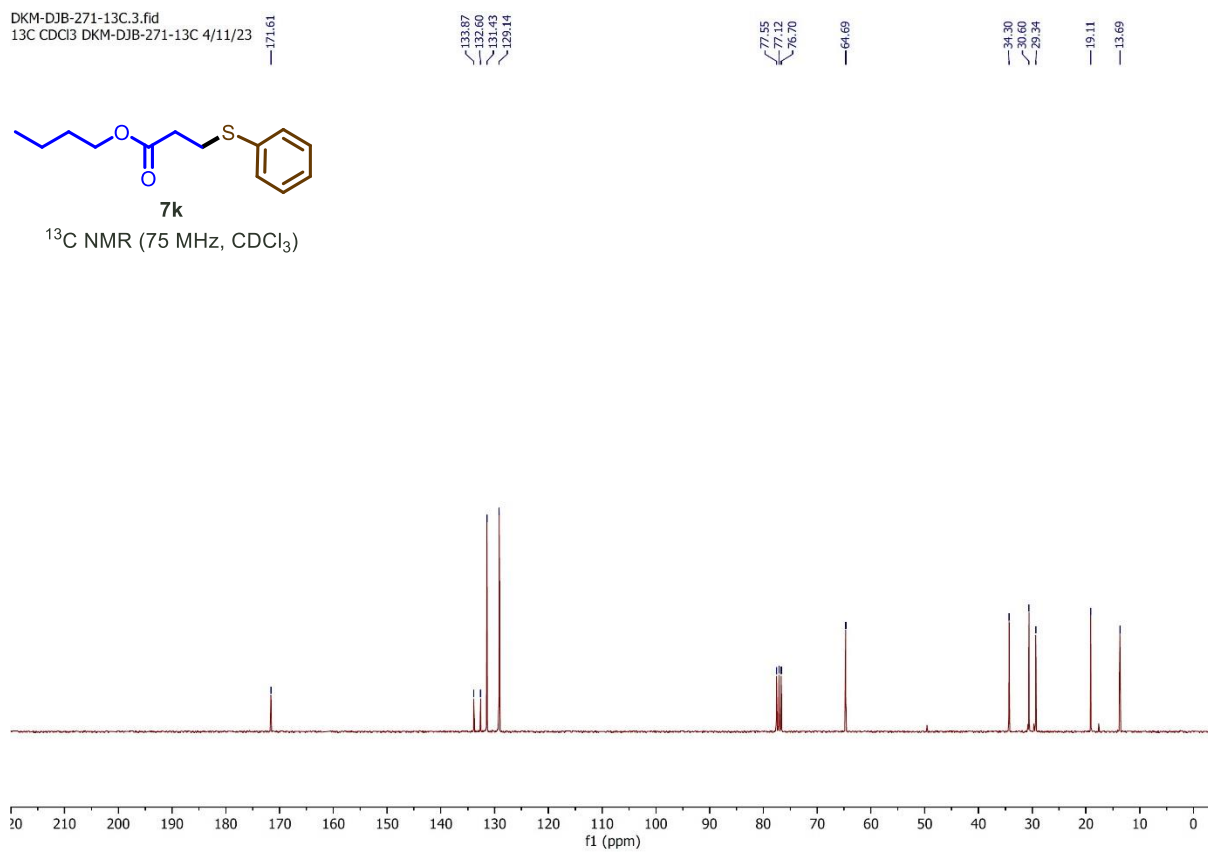


DKM-DJB-271-13C.3.fid
13C CDCl3 DKM-DJB-271-13C 4/11/23



7k

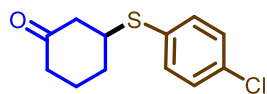
¹³C NMR (75 MHz, CDCl₃)



DKM-DJB-241.1.fid
1H CDCl3 DKM-DJB-241 01/09/23

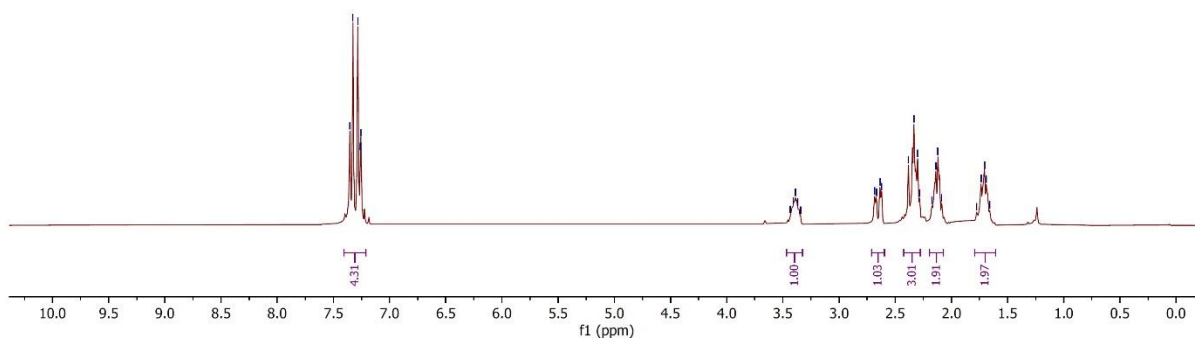
7.35
7.33
7.28
7.25

3.43
3.41
3.39
3.37
3.34
2.67
2.64
2.38
2.33
2.30
2.28
2.17
2.14
2.10
1.78
1.74
1.70
1.69
1.66



9a

¹H NMR (300 MHz, CDCl₃)



DKM-DJB-241-13C.1.fid

200.4

134.57
134.07
131.53
128.26

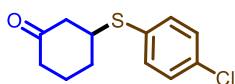
77.52
77.10
76.67

47.60
46.35

40.84

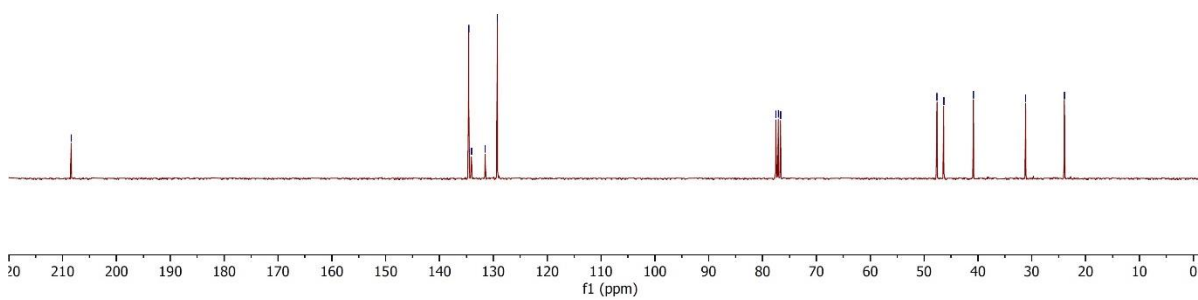
31.13

23.95



9a

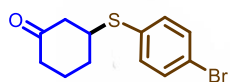
¹³C NMR (75 MHz, CDCl₃)



DKM-DJB-263.10.fid
1H-CDCl3-DKM-DJB-263-01-11-2023

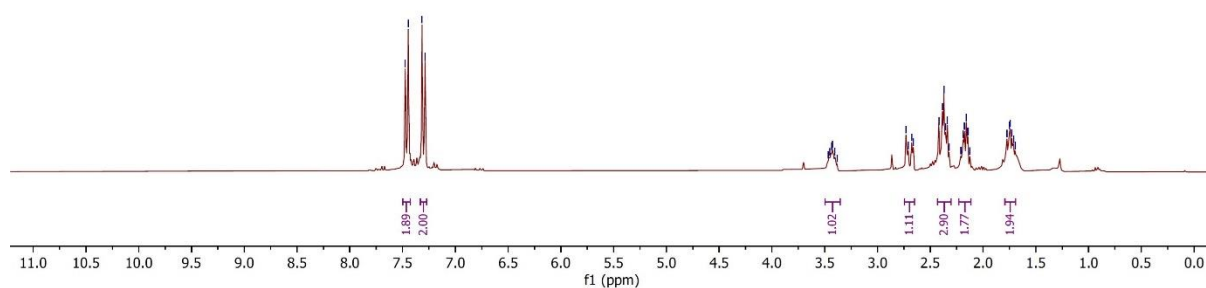
7.47
7.45
7.32
7.29

3.47
3.45
3.43
3.43
3.38
3.38
2.73
2.71
2.68
2.66
2.62
2.62
2.37
2.35
2.34
2.32
2.21
2.19
2.18
2.16
2.14
2.13
1.77
1.75
1.74
1.73
1.71
1.70



9b

¹H NMR (300 MHz, CDCl₃)



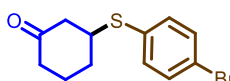
DKM-DJB-263-13C.1.fid

208.85

134.72
132.23
129.41
122.13

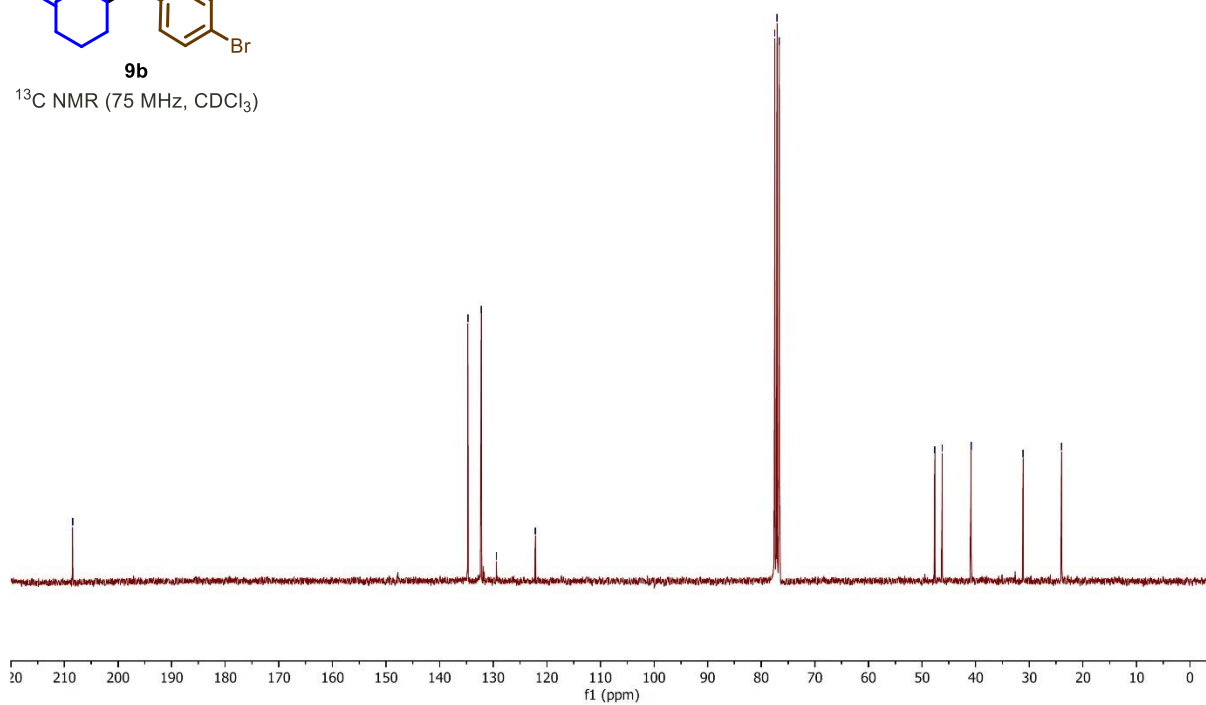
77.48
77.05
76.63

47.61
46.24
40.86
31.15
23.98



9b

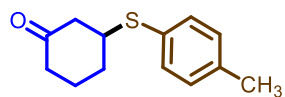
¹³C NMR (75 MHz, CDCl₃)



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

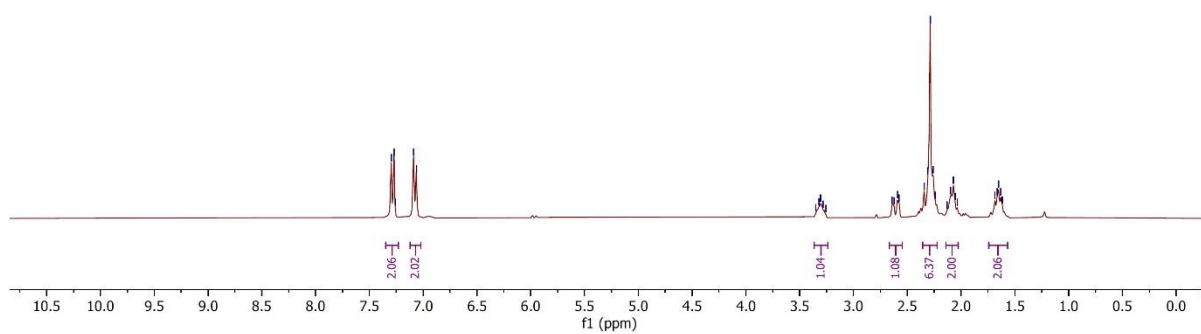
7.30
7.27
7.26
7.09
7.06

3.35
3.32
3.31
3.28
3.28
2.64
2.62
2.59
2.57
2.34
2.31
2.26
2.24
2.13
2.10
2.07
2.05
2.04
1.87
1.87
1.65
1.63



9c

¹H NMR (300 MHz, CDCl₃)



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

208.8

138.07
137.01
129.84
129.16

77.68
77.25
76.83

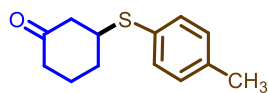
47.73
46.44

40.85

31.19

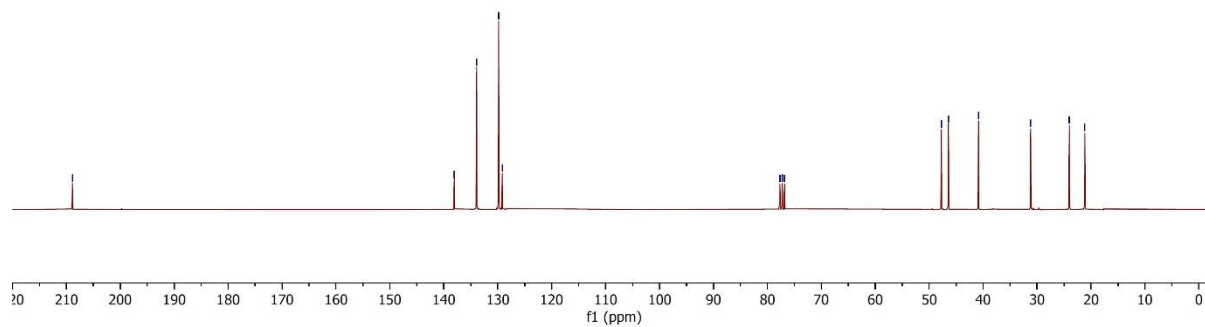
24.01

21.14



9c

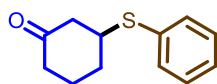
¹³C NMR (75 MHz, CDCl₃)



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

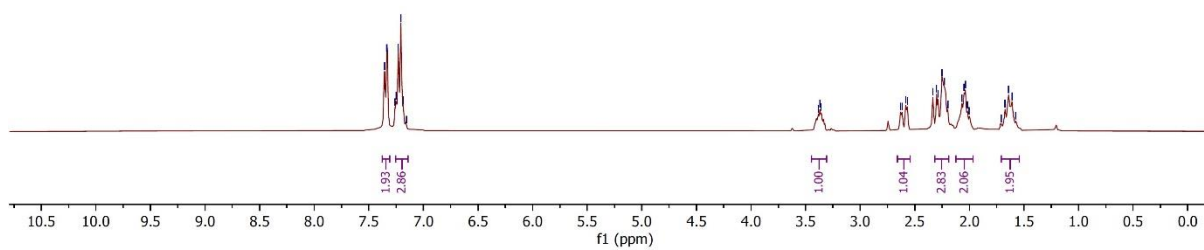
7.35
7.34
7.30
7.26
7.23
7.21
7.18
7.16

3.38
3.37
2.82
2.81
2.80
2.57
2.55
2.33
2.29
2.25
2.20
2.07
2.05
2.04
2.00
1.71
1.67
1.64
1.58



9d

¹H NMR (300 MHz, CDCl₃)



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

206

133.12
130.05
127.70

77.82
77.39
76.96

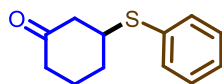
47.64

46.00

40.82

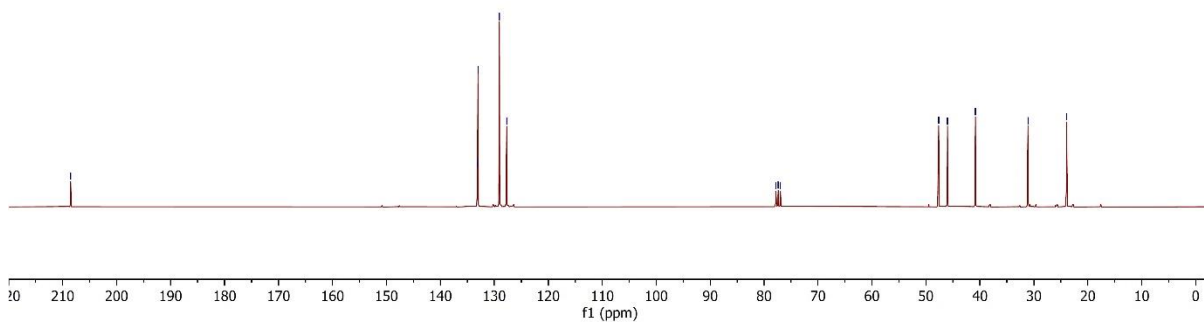
31.10

23.93



9d

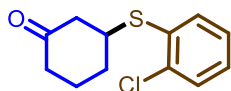
¹³C NMR (75 MHz, CDCl₃)



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

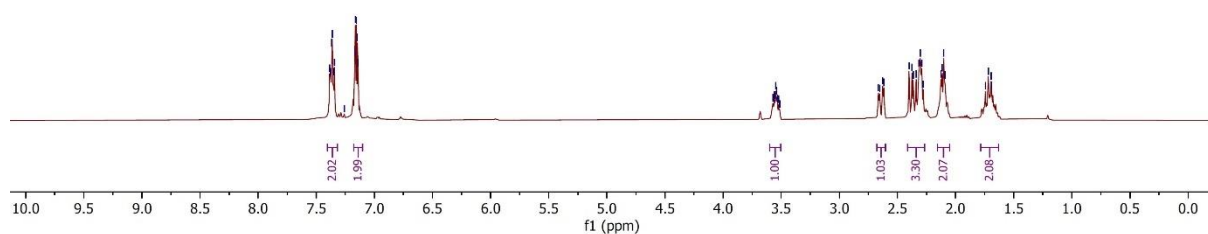
7.39
7.38
7.36
7.35
7.34
7.26
7.17
7.16
7.15
7.14

3.57
3.56
3.55
3.54
3.53
3.51
2.65
2.63
2.62
2.40
2.38
2.36
2.34
2.31
2.30
2.29
2.28
2.13
2.11
2.10
1.94
1.72
1.69



9e

¹H NMR (400 MHz, CDCl₃)



DKM-DJB-256.10.fid

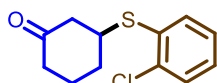
208.3

136.67
133.49
132.56
130.13
128.67
127.27

77.67
77.25
76.82

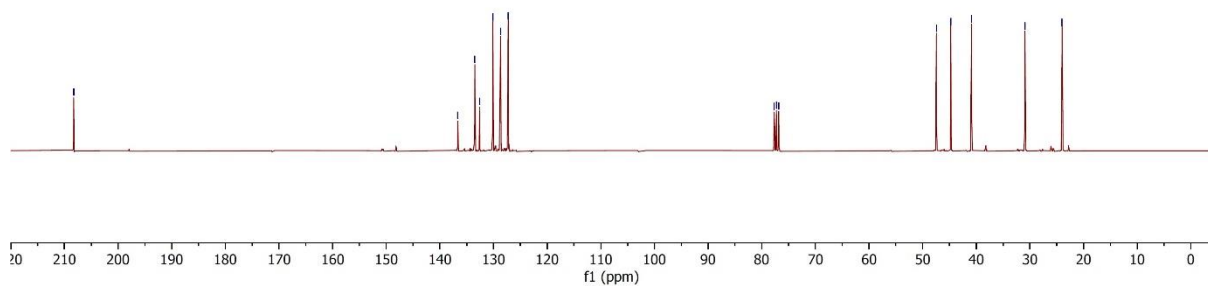
47.44
44.76
40.88

30.93
24.00



9e

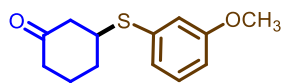
¹³C NMR (75 MHz, CDCl₃)



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

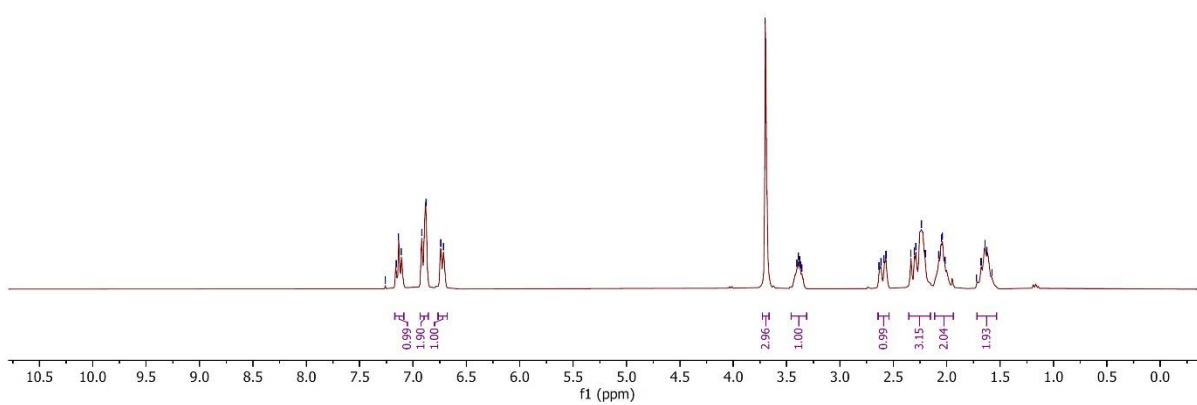
7.26
7.16
7.14
7.11
6.92
6.88
6.71

3.70
3.40
3.39
3.38
3.37
3.36
2.63
2.59
2.57
2.34
2.30
2.29
2.24
2.17
2.07
2.05
2.04
2.02
1.72
1.68
1.64
1.58



9f

¹H NMR (300 MHz, CDCl₃)



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

208.36

159.78

134.35
129.84
124.86

118.11
113.28

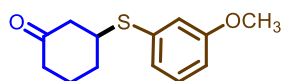
77.79
77.36
76.94

55.25

47.59
45.85
40.81

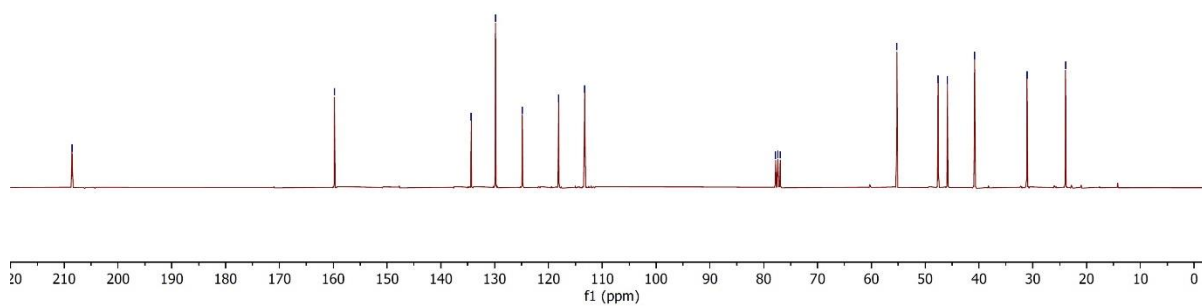
31.08

23.91



9f

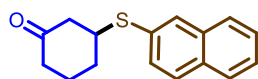
¹³C NMR (75 MHz, CDCl₃)



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

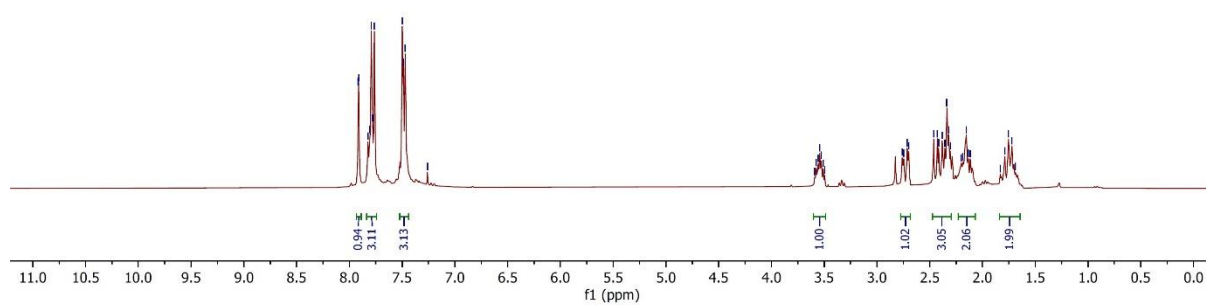
7.92
7.81
7.82
7.81
7.79
7.78
7.76
7.50
7.49
7.47
7.26

3.89
3.86
3.56
3.54
3.53
3.51
3.49
2.76
2.75
2.72
2.70
2.46
2.43
2.41
2.38
2.36
2.34
2.32
2.30
2.20
2.19
2.15
2.13
2.12
1.79
1.75
1.72
1.69



9g

¹H NMR (300 MHz, CDCl₃)



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

208.7

133.63
132.57
132.17
130.44
130.25
127.75
127.52
126.69
126.51

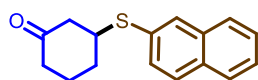
77.59
77.17
76.74

47.78
46.10

40.91

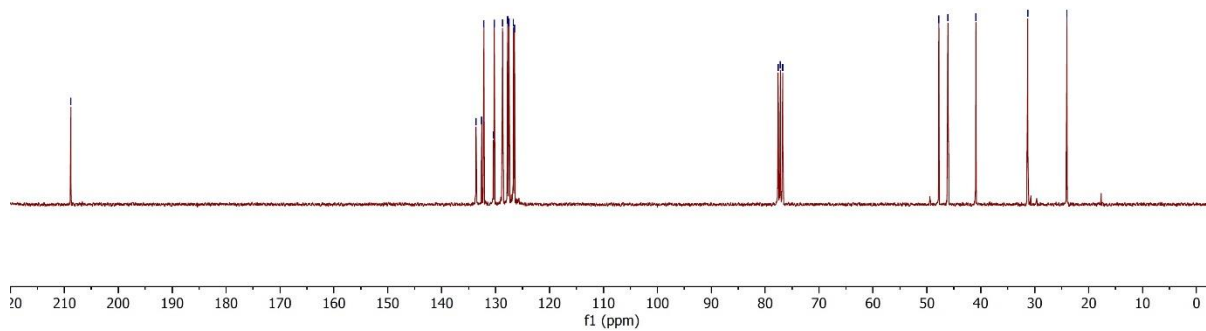
31.28

24.04



9g

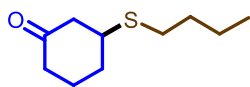
¹³C NMR (75 MHz, CDCl₃)



DKM-DJB-258.1.fid
1H CDCl3 DKM-DJB-258 30/10/23

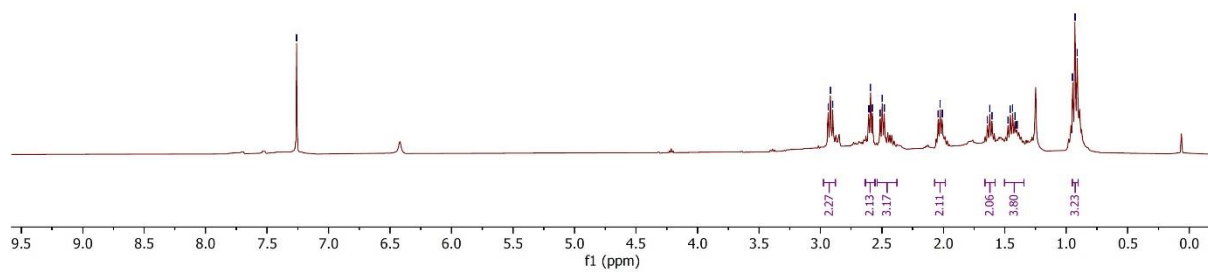
7.26

2.94
2.92
2.90
2.61
2.59
2.56
2.50
2.48
2.04
2.03
2.01
1.64
1.62
1.47
1.45
1.44
1.42
1.41
1.40
1.35
1.33
1.03
0.91



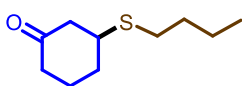
9h

¹H NMR (400 MHz, CDCl₃)



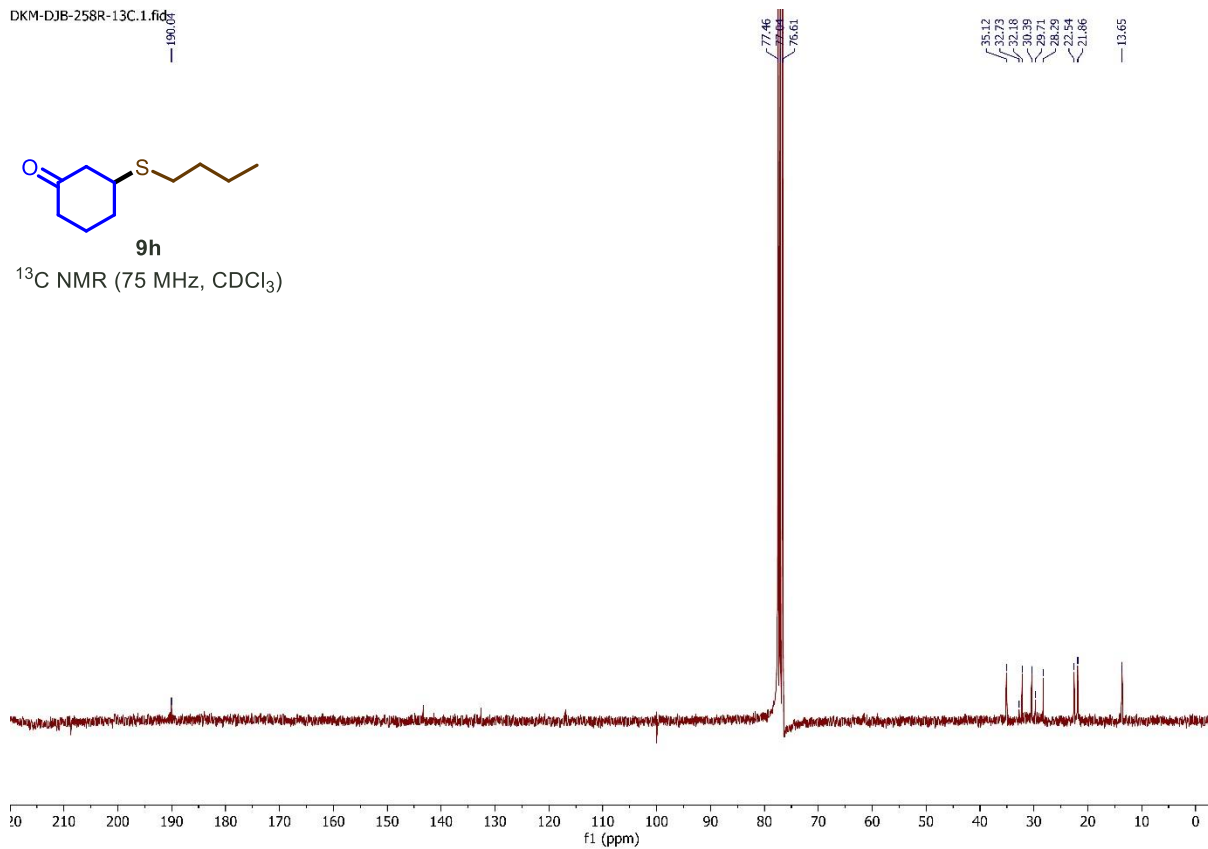
DKM-DJB-258R-13C.1.fid

194.06



9h

¹³C NMR (75 MHz, CDCl₃)



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.

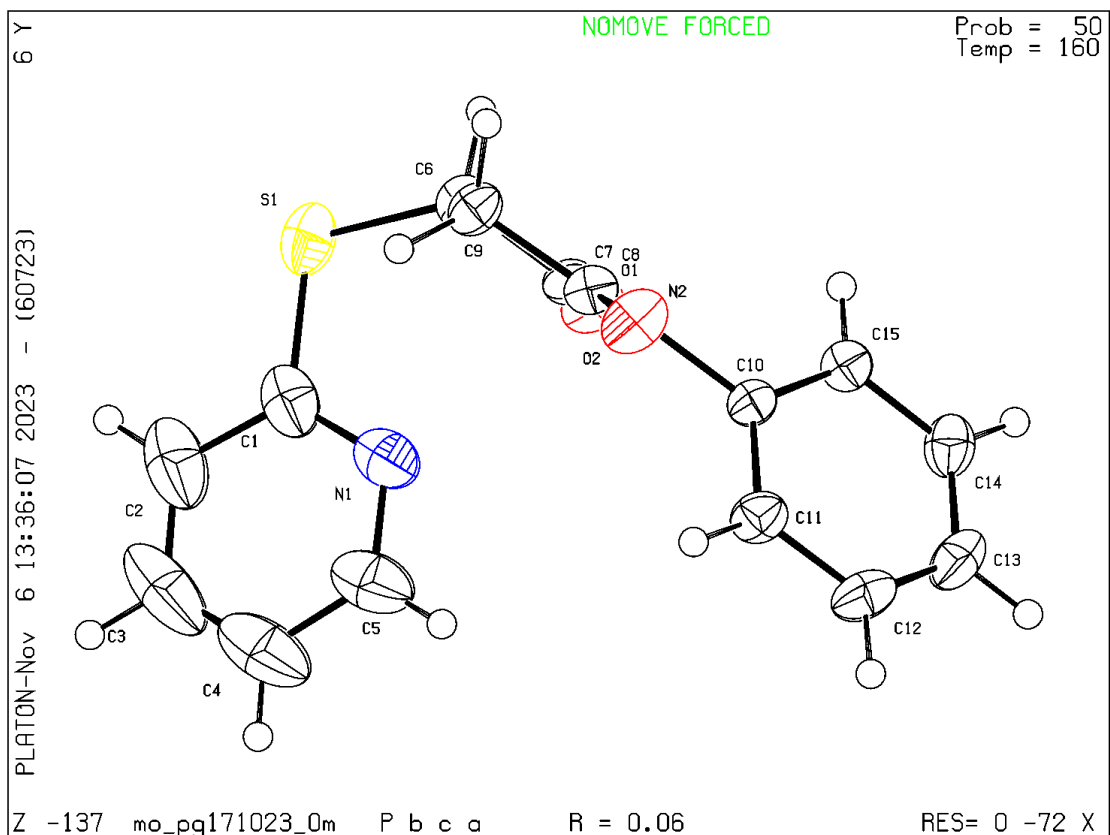


Table 1 Crystal data and structure refinement for mo_PG171023_0m.

| | |
|------------------------------------|---|
| Identification code | mo_PG171023_0m |
| Empirical formula | C ₁₅ H ₁₂ N ₂ O ₂ S |
| 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} /cm ³ | 1.376 |

| | |
|--|---|
| μ/mm^{-1} | 0.238 |
| F(000) | 1184.0 |
| Crystal size/ mm^3 | $0.6 \times 0.57 \times 0.2$ |
| Radiation | MoK α ($\lambda = 0.71073$) |
| 2 Θ range for data collection/ $^\circ$ | 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 [$R_{\text{int}} = 0.0738, R_{\text{sigma}} = 0.0407$] |
| Data/restraints/parameters | 2989/0/181 |
| Goodness-of-fit on F^2 | 1.108 |
| Final R indexes [$I \geq 2\sigma(I)$] | $R_1 = 0.0575, wR_2 = 0.1336$ |
| Final R indexes [all data] | $R_1 = 0.0643, wR_2 = 0.1379$ |
| Largest diff. peak/hole / $e \text{ \AA}^{-3}$ | 0.34/-0.31 |