

Synthesis of Conjugated Dienes *via* Palladium-Catalysed Aerobic Dehydrogenation of Unsaturated Acids and Amides

Meledath Sudhakaran Keerthana and Masilamani Jeganmohan*

Department of Chemistry, Indian Institute of Technology, Madras-600036, India

E-Mail: mjeganmohan@iitm.ac.in

Electronic Supporting Information (ESI)

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Experimental Section

General information: All reactions were carried out under the air atmosphere in flame-dried glassware. Syringes which were used to transfer anhydrous solvents or reagents were purged with nitrogen prior to use (three times). Dry solvents were used for the reaction. Column chromatographical purifications were performed using SiO₂ (120-200 mesh ASTM) from Avra Pvt. Ltd., India. Abbreviations for signal coupling are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Commercially available metal salts and acids were purchased from Sigma-Aldrich and Spectrochem. Pvt. Ltd., India and used without further purification. Starting Materials: Starting materials **1a-1p**, **3a-3f**, **5a-5e** and **7a-7f** were prepared by known literature procedures.¹⁻⁶

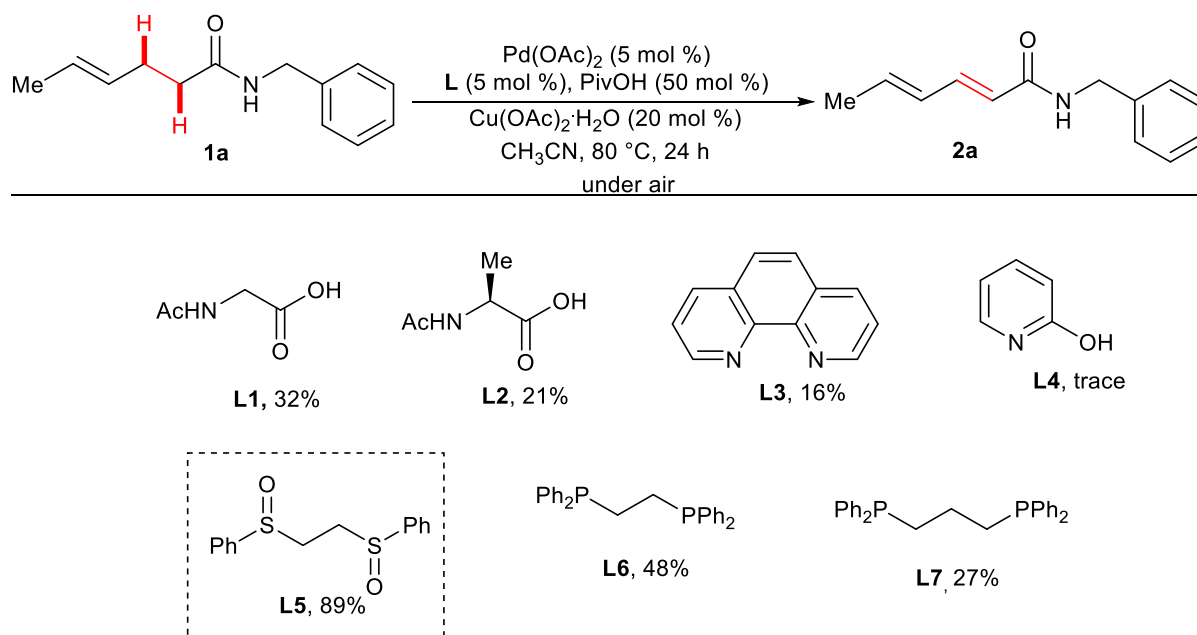
1. General Procedure for α β -Dehydrogenation of β -Substituted Amide **1.**

Pd(OAc)₂ (5.0 mol %), L5 (5 mol %), Cu(OAc)₂·H₂O (20 mol %), and Amide **1** (50.0 mg, 1 equiv.) were taken in a 15 mL Schlenk tube. Acetonitrile (1.0 mL) was added to the reaction mixture via syringe. Then, pivalic acid (50 mol %) was added to the solution, followed by the addition of Acetonitrile (2.0 mL). The tube was sealed using screw cap under air and the reaction mixture was allowed to stir at 100 °C for 24 h in an oil bath. After cooling to the ambient temperature, the reaction mixture was diluted with CH₂Cl₂, filtered through Celite, and the filtrate was concentrated. The crude residue was purified through a silica gel column using hexane and ethyl acetate as eluent to give pure product **2**.

Optimization Studies:

The reaction was initially studied using various ligands and most of them inhibited the reaction (Scheme 1). However, N-Ac-glycine **L1** gave the desired dehydrogenated product **2a** in 32% yield. When the ligand was switched from **L1** to N-Ac-alanine **L2** the reaction yield was reduced to 21%. Further, the reaction was probed using various phosphorous, sulphur and nitrogen-based ligands. When 1,10-phenanthroline **L3** was used, the dehydrogenated product **2a** was produced with a yield of 16%. A trace amount of product was observed when pyridin-2-ol **L4** was used. 1,2-bis(diphenylphosphanyl)ethane **L6** and 1,3-bis(diphenylphosphanyl)propane **L7** generated the product **2a** with a yield of 48 and 27% respectively. However, the best results were observed when 1,2-bis(phenylsulfinyl)ethane **L5** was used. In this case, the yield of the product **2a** was improved to 89%.

Scheme S1. Effect of ligands on α β -Dehydrogenation of β -Substituted Amide **1a**



The reaction was next screened with various additives such as AcOH, pivalic acid 1-adamantane carboxylic acid, mesitylenic acid, and benzoic acid (entries 1-5). AcOH gave the desired product **2a** in 58% yield (entry 1). In the case of pivalic acid, the expected product **2a** was isolated in 89% yield (entry 2). Other additives were found to be ineffective for the reaction. The reaction was further examined using various oxidants (20 mol %) such as Ag_2CO_3 , AgOAc , Ag_2O , benzoquinone, $\text{PhI}(\text{OAc})_2$, $\text{K}_2\text{S}_2\text{O}_8$, $(\text{NH}_4)_2\text{S}_2\text{O}_8$, $\text{Cu}(\text{OAc})_2$ and $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (entry 6-13). Among them, $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ was very effective, giving the dehydrogenated product **2a** in 89% yield (entry 2). AgOAc and $\text{PhI}(\text{OAc})_2$ gave the desired product in 20 and 35% respectively (entry 7 and 10). $\text{Cu}(\text{OAc})_2$ was partially effective, providing the desired product **2a** in 58% yield (entry 13). Other oxidants were found to be ineffective. The reaction was further examined with various solvents such as 1,2-dichloroethane, 1,2-dichlorobenzene, *N,N*-dimethylformamide (DMF), toluene, 1,2-dimethoxyethane (DME), methanol, $\text{CF}_3\text{CH}_2\text{OH}$, 1,4-dioxane, DMSO, CH_3CN and THF (entry 13-22). Among them, CH_3CN was very effective, giving the expected product **2a** in 89% yield (entry 2). DMF provided trace amount of product (entry 15). $\text{CF}_3\text{CH}_2\text{OH}$ gave the product **2a** in 24% yield (entry 20). 1,4-Dioxane and DMSO and were partially effective, yielding the dehydrogenated product **2a** in 58% and 43% yields, respectively (entry 21-22). Optimal yield of 89% for product **2a** was obtained when the reaction was carried out under air in the presence of 20 mol % of $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ along with 50 mol % of pivalic acid in acetonitrile solvent. Control experiment revealed that the reaction does not proceed in absence of ligand and pivalic

acid (entry 23-24). The yield of product **2a** was reduced to 20% in absence of $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ (entry 26). Trace amount of product was formed when O_2 was used as the sole oxidant (entry 27).

Scheme S2. α β -Dehydrogenation of β -Substituted Amide **1a**

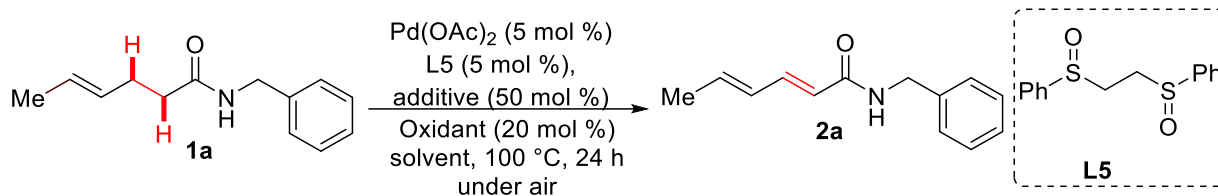


Table 1. Optimization Table

Entry	Catalyst	L (mol %)	Additive	Oxidant	Solvent	Yield
1	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	AcOH	$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$	CH_3CN	58%
2	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$	CH_3CN	89%
3	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	Adm-1-COOH	$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$	CH_3CN	NR
4	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	Mesitylene carboxylic acid	$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$	CH_3CN	NR
5	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	Benzoic acid	$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$	CH_3CN	NR
6	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	Ag_2CO_3	CH_3CN	NR
7	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	AgOAc	CH_3CN	20%
8	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	Ag_2O	CH_3CN	NR
9	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	Benzoquinone	CH_3CN	NR
10	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	$\text{PhI}(\text{OAc})_2$	CH_3CN	35%
11	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	$\text{K}_2\text{S}_2\text{O}_8$	CH_3CN	NR
12	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	$(\text{NH}_4)_2\text{S}_2\text{O}_8$	CH_3CN	NR
13	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	$\text{Cu}(\text{OAc})_2$	CH_3CN	58%
14	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$	1,2- dichloroethane	NR
15	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$	1,2- dichlorobenzene	NR
16	$\text{Pd}(\text{OAc})_2$	L5 (5 mol %)	PivOH	$\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$	DMF	trace

17	Pd(OAc) ₂	L5 (5 mol %)	PivOH	Cu(OAc) ₂ ·H ₂ O	toluene	NR
18	Pd(OAc) ₂	L5 (5 mol %)	PivOH	Cu(OAc) ₂ ·H ₂ O	DME	NR
19	Pd(OAc) ₂	L5 (5 mol %)	PivOH	Cu(OAc) ₂ ·H ₂ O	CH ₃ OH	NR
20	Pd(OAc) ₂	L5 (5 mol %)	PivOH	Cu(OAc) ₂ ·H ₂ O	CF ₃ CH ₂ OH	24%
21	Pd(OAc) ₂	L5 (5 mol %)	PivOH	Cu(OAc) ₂ ·H ₂ O	1,4-dioxane	58%
22	Pd(OAc) ₂	L5 (5 mol %)	PivOH	Cu(OAc) ₂ ·H ₂ O	DMSO	43%
23	Pd(OAc) ₂	L5 (5 mol %)	PivOH	Cu(OAc) ₂ ·H ₂ O	THF	NR
24	Pd(OAc) ₂	-	PivOH	Cu(OAc) ₂ ·H ₂ O	CH ₃ CN	NR
25	Pd(OAc) ₂	L5 (5 mol %)	-	Cu(OAc) ₂ ·H ₂ O	CH ₃ CN	NR
26	Pd(OAc) ₂	L5 (5 mol %)	PivOH	-	CH ₃ CN	20%
27	Pd(OAc) ₂	L5 (5 mol %)	PivOH	O ₂	CH ₃ CN	trace

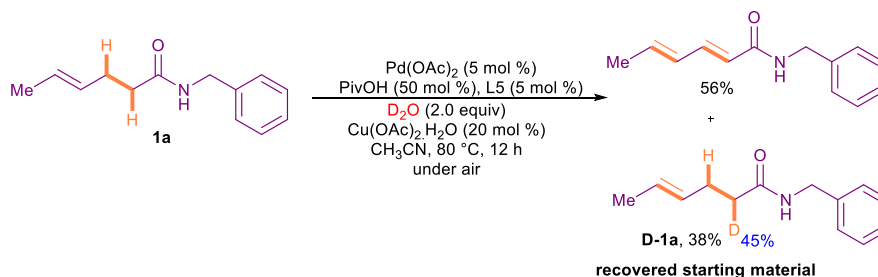
2. General Procedure for α β -Dehydrogenation of β -Substituted Acid **7**.

Pd(OAc)₂ (10.0 mol %), L5 (10 mol %), Cu(OAc)₂·H₂O (20 mol %), and Acid **7** (50.0 mg, 1 equiv.) were taken in a 15 mL Schlenk tube. Acetonitrile (1.0 mL) was added to the reaction mixture via syringe. Then, pivalic acid (50 mol %) was added to the solution, followed by the addition of Acetonitrile (2.0 mL). The tube was sealed using screw cap under air and the reaction mixture was allowed to stir at 100 °C for 24 h in oil bath. After cooling to the ambient temperature, the reaction mixture was diluted with CH₂Cl₂, filtered through Celite, and the filtrate was concentrated. The crude residue was purified through a silica gel column using hexane and ethyl acetate as eluent to give pure product **8**.

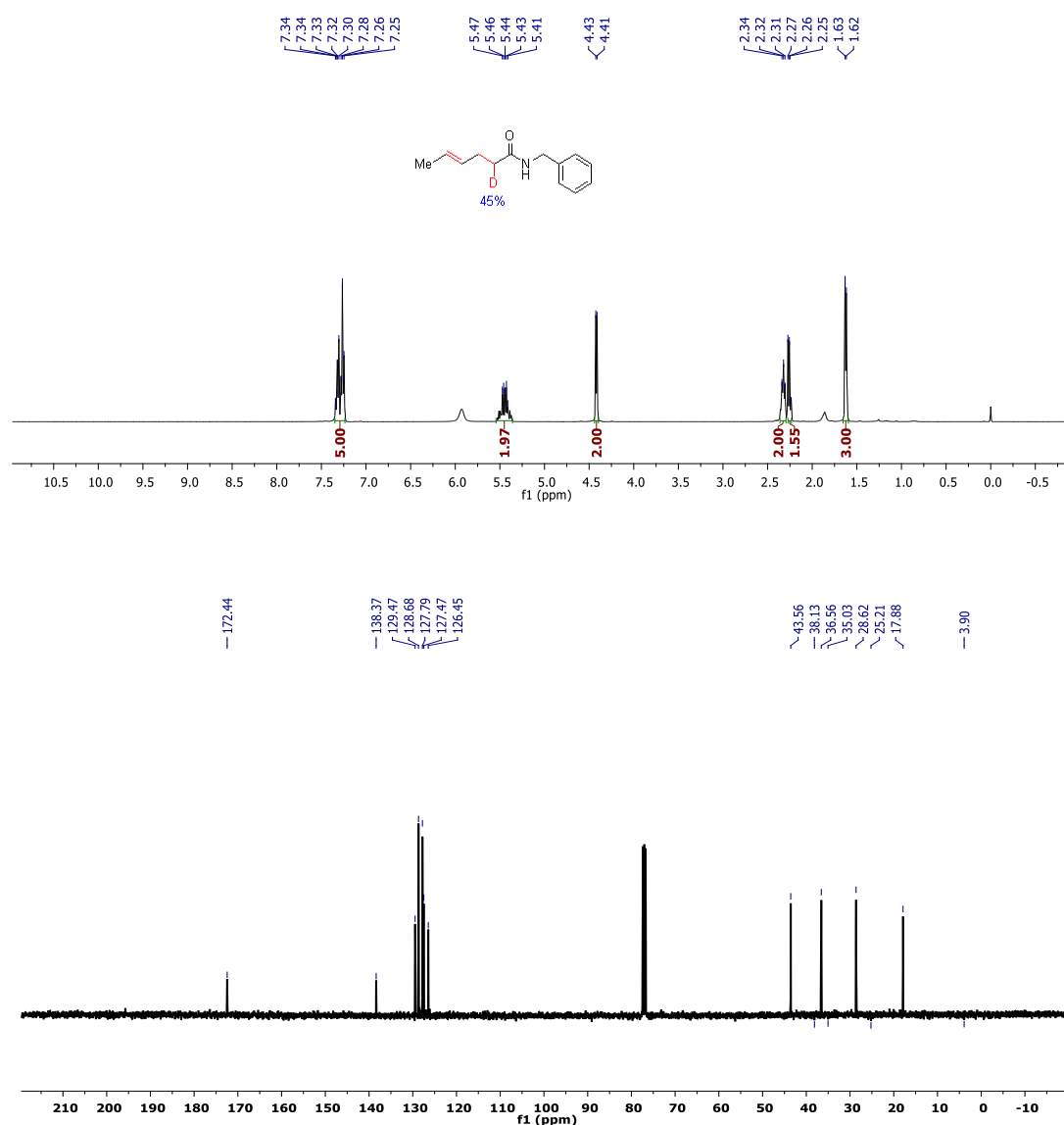
Mechanistic Investigation

Pd(OAc)₂ (5.0 mol %), Cu(OAc)₂·H₂O (20 mol %), and Amide **1a** (50.0 mg, 1 equiv.) were taken in a 15 mL Schlenk tube. Acetonitrile (1.0 mL) was added to the reaction mixture via syringe. Further, pivalic acid (50 mol %) was added to the solution. Then, D₂O (2 equiv.) was added followed by addition of acetonitrile (1 mL). The tube was sealed using screw cap under air and the reaction mixture was allowed to stir at 100 °C for 24 h in oil bath. After cooling to the ambient temperature, the reaction mixture was diluted with CH₂Cl₂, filtered through Celite and the filtrate was concentrated. The crude residue was purified through a silica gel column

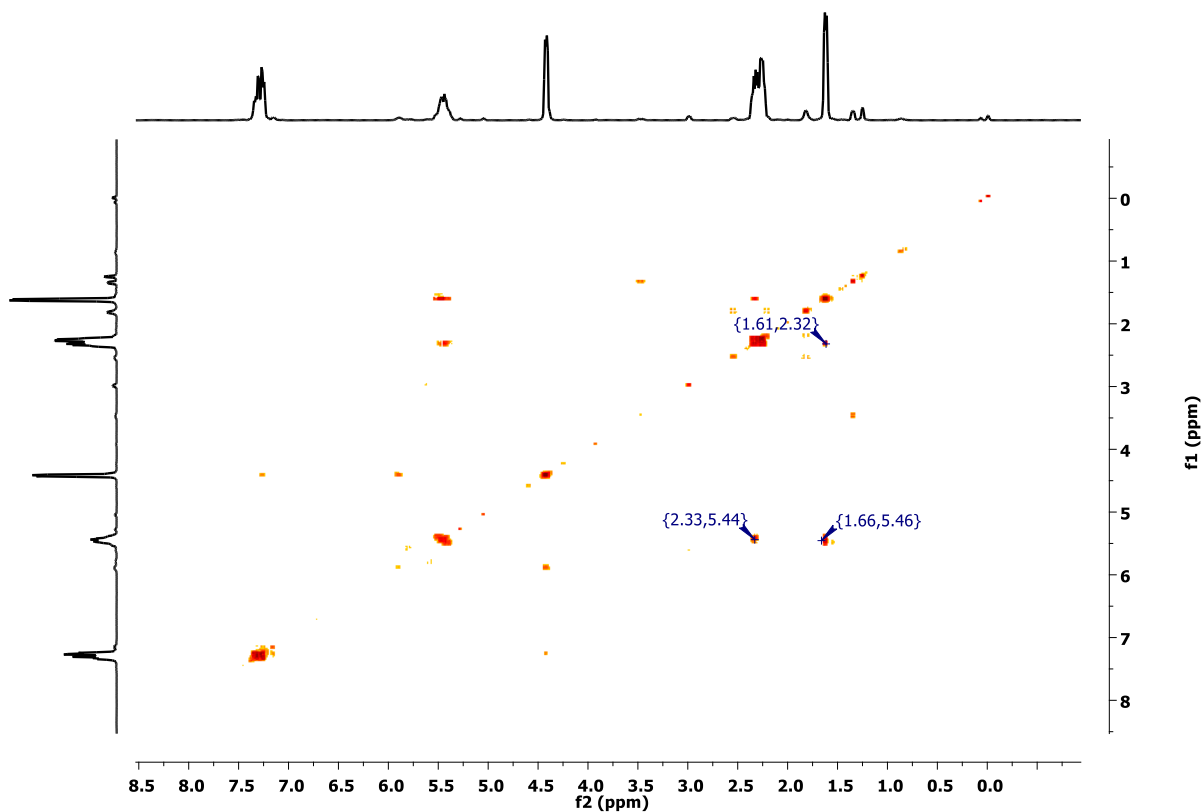
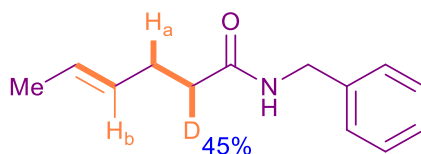
using hexane and ethyl acetate as eluent to give pure product **D-2a** in 56% yield. No deuterium incorporation was observed in the product. However, unreacted starting material **D-1a** was recovered in 38% yield with 45% deuterium incorporation at the α -carbon atom.



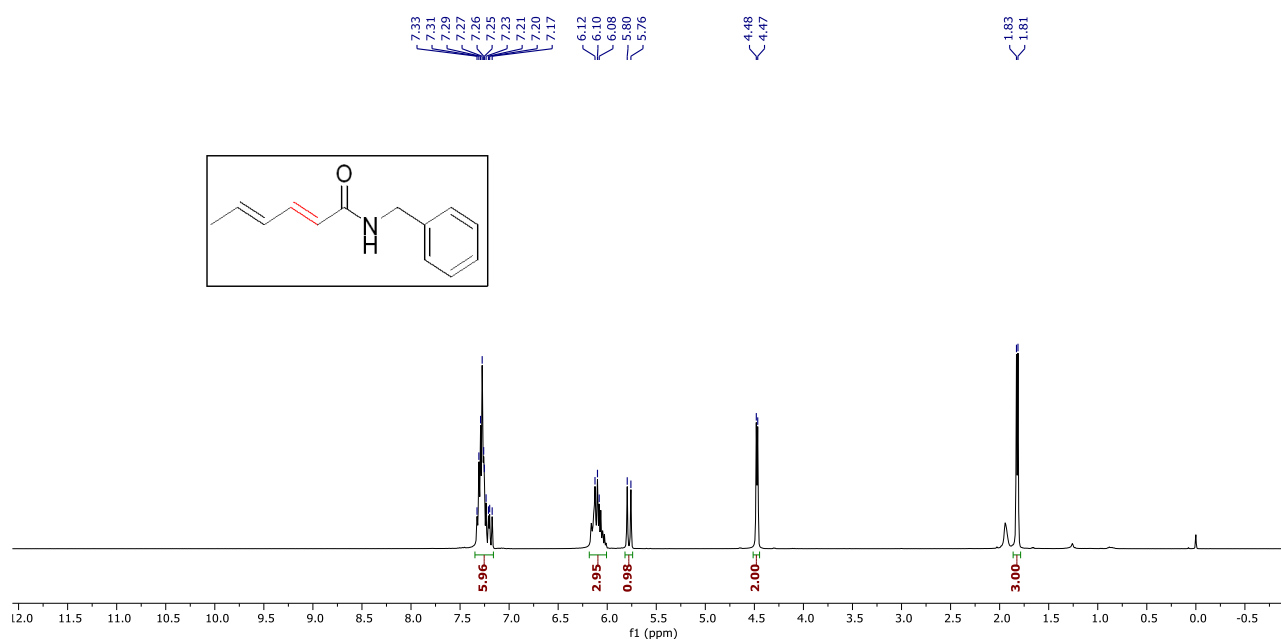
¹H and ¹³C NMR spectra of compound D-1a in CDCl₃ at 400MHz:



COSY Spectra of D-1a: From COSY NMR, it can be observed that proton H_a at ($\delta = 2.33$ ppm) is strongly correlated with alkene proton H_b at ($\delta = 5.4$ ppm). Similarly, the alkene proton H_b at ($\delta = 5.4$ ppm) shows a correlation with methyl proton at ($\delta = 1.6$ ppm).



¹H NMR spectra of compound D-2a in CDCl₃ at 400 MHz:

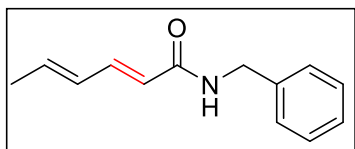


Reference:

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5. B. Jiang, M. Zhao, S.S. Li, Y. H. Xu, T.P. Loh, *Angew. Chem., Int. Ed.* 2018, **57**, 555–559.
6. J. Derosa, V.A.V. Puyl, V. T. Tran, M. Liu, K. M. Engle, *Chem. Sci.*, 2018, **9**, 5278-5283.

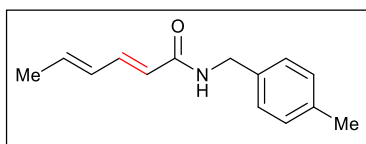
Spectral Data of All Compounds:

(2E,4E)-N-Benzylhexa-2,4-dienamide (2a):



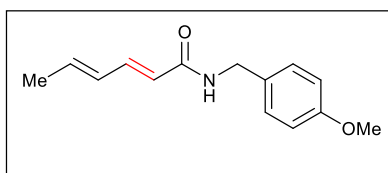
Prepared according to General Procedure 1; white solid; eluent (20% ethyl acetate :hexane); yield is 89%. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.35 – 7.23 (m, 5H), 6.45 – 5.96 (m, 3H), 5.78 (d, $J = 15.0$ Hz, 1H), 4.47 (d, $J = 5.8$ Hz, 2H), 1.82 (d, $J = 6.1$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 166.4, 141.6, 137.9, 129.7, 128.7, 127.8, 127.4, 121.3, 43.6, 18.6. HRMS (ESI/Q-TOF): $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{13}\text{H}_{15}\text{NONa}$ 224.1051; Found 224.1059.

(2E,4E)-N-(3,4-Dimethoxybenzyl)hexa-2,4-dienamide (2b):



Prepared according to General Procedure 1; half-white solid; eluent (25% ethyl acetate:hexane); yield is 85%. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.14 (d, $J = 12.5$ Hz, 5H), 6.37 – 5.87 (m, 2H), 5.75 (d, $J = 15.0$ Hz, 1H), 4.43 (d, $J = 5.7$ Hz, 2H), 2.32 (s, 3H), 1.82 (d, $J = 6.1$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 166.28, 141.46, 137.83, 137.14, 135.36, 129.71, 129.34, 129.25, 128.69, 127.88, 121.40, 43.43, 21.09, 18.57. HRMS (ESI/Q-TOF) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{14}\text{H}_{17}\text{NONa}$ 238.1208; Found 238.1219.

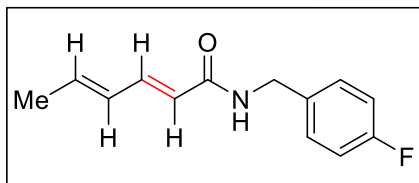
(2E,4E)-N-(4-Methoxybenzyl)hexa-2,4-dienamide (2c):



Prepared according to General Procedure 1; white solid; eluent (20% ethyl acetate:hexane); yield is 88%. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.17 (d, $J = 8.2$ Hz, 3H), 6.82 (d, $J = 8.2$ Hz, 2H), 6.07 (m, $J = 15.1, 8.8$ Hz, 2H), 5.78 (d, $J = 15.0$ Hz, 1H), 4.37 (d, $J = 5.7$ Hz, 2H), 3.76 (s, 3H), 1.81 (d, $J = 6.3$ Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 166.4, 158.9, 141.3,

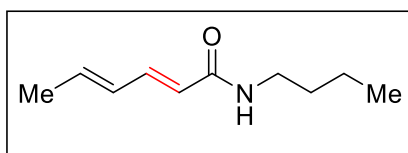
137.8, 130.5, 129.7, 129.1, 121.5, 114.0, 114.0, 55.2, 43.1, 18.5. **HRMS (ESI/Q-TOF) m/z:** $[M + Na]^+$ Calcd for $C_{14}H_{17}NO_2Na$ 254.1157; Found 254.1142.

(2E,4E)-N-(4-Fluorobenzyl)hexa-2,4-dienamide (2d):



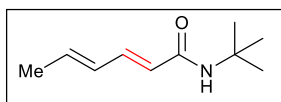
Prepared according to General Procedure 1; yellow-white solid; eluent (20% ethyl acetate:hexane); yield is 78%. **1H NMR (400 MHz, $CDCl_3$):** δ 7.38 – 7.10 (m, 3H), 6.97 (t, $J = 8.6$ Hz, 2H), 6.23 – 5.97 (m, 2H), 5.79 (d, $J = 15.0$ Hz, 1H), 4.42 (d, $J = 5.9$ Hz, 2H), 1.82 (d, $J = 6.0$ Hz, 3H). **$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$):** δ 166.4, 159.0, 141.3, 137.8, 130.5, 129.7, 129.1, 121.5, 114.0, 114.0, 55.2, 43.1, 18.5. **HRMS (ESI/Q-TOF) m/z:** $[M + Na]^+$ Calcd for $C_{13}H_{14}FNONa$ 242.0957; Found 242.0968.

(2E,4E)-N-Butylhexa-2,4-dienamide (2e):



Prepared according to General Procedure 1; yellow-white solid; eluent (25% ethyl acetate:hexane); yield is 75%. **1H NMR (400 MHz, $CDCl_3$):** δ 7.16 (dd, $J = 15.1, 10.7$ Hz, 1H), 6.30 – 5.77 (m, 3H), 3.31 (m, $J = 7.2, 5.7$ Hz, 2H), 1.81 (dd, $J = 6.8, 1.4$ Hz, 3H), 1.52 (m, $J = 6.9, 6.0, 1.6$ Hz, 2H), 1.46 – 1.26 (m, 2H), 0.91 (t, $J = 7.4$ Hz, 3H). **$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$):** δ 166.8, 140.5, 137.0, 129.8, 122.2, 39.3, 31.7, 20.1, 18.4, 13.7. **HRMS (ESI/Q-TOF) m/z:** $[M + Na]^+$ Calcd for $C_{10}H_{17}NONa$ 190.1208; Found 190.1211

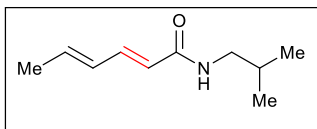
(2E,4E)-N-(Tert-butyl)hexa-2,4-dienamide (2f):



Prepared according to General Procedure 1; white solid; eluent (25% ethyl acetate in hexane); yield is 73%. **1H NMR (400 MHz, $CDCl_3$):** δ 7.35 – 6.84 (m, 1H), 6.56 – 5.96 (m, 3H), 5.82 (d, $J = 15.0$ Hz, 1H), 1.80 (d, $J = 6.4$ Hz, 3H), 1.38 (d, $J = 1.9$ Hz, 9H). **$^{13}C\{^1H\}$ NMR (101**

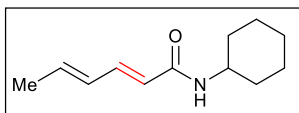
MHz, CDCl₃): 166.0, 140.1, 136.8, 129.7, 123.1, 51.1, 28.8, 28.8, 28.8, 18.4. **HRMS (ESI/Q-TOF) m/z**: [M + H]⁺ Calcd for C₁₀H₁₈NO 168.1388; Found 168.1396.

(2E,4E)-N-Isobutylhexa-2,4-dienamide (2g):



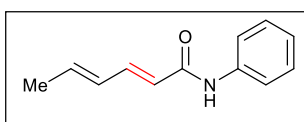
Prepared according to General Procedure 1; white solid; eluent (25% ethyl acetate:hexane); yield is 80%. **¹H NMR (400 MHz, CDCl₃)**: δ 7.37 – 6.66 (m, 2H), 6.30 – 5.60 (m, 3H), 3.12 (m, *J* = 5.7, 5.0 Hz, 3H), 1.81 (d, *J* = 6.7 Hz, 2H), 0.85 (m, 6H). **¹³C{¹H} NMR (101 MHz, CDCl₃)**: δ 166.9, 140.5, 137.0, 129.8, 122.2, 47.0, 28.5, 20.2, 20.1, 18.4. **HRMS (ESI/Q-TOF) m/z**: [M + Na]⁺ Calcd for C₁₀H₁₇NONa 190.1208; Found 190.1211

(2E,4E)-N-Cyclohexylhexa-2,4-dienamide (2h):



Prepared according to general procedure 1; white solid; eluent (25% ethyl acetate in hexane); yield is 64%. **¹H NMR (400 MHz, CDCl₃)**: 7.16 (dd, *J* = 15.0, 10.4 Hz, 1H), 6.34 – 5.47 (m, 3H), 3.84 (dt, *J* = 7.4, 3.5 Hz, 1H), 1.94 (dd, *J* = 12.8, 3.9 Hz, 2H), 1.82 (d, *J* = 6.4 Hz, 3H), 1.77 – 1.65 (m, 2H), 1.62 (m, *J* = 12.9, 3.8 Hz, 1H), 1.48 – 1.28 (m, 2H), 1.17 (m, *J* = 11.9, 3.7 Hz, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃)**: δ 165.5, 140.7, 137.2, 129.8, 122.2, 48.2, 33.2, 25.5, 24.9, 18.5. **HRMS (ESI/Q-TOF) m/z**: [M + Na]⁺ Calcd for C₁₂H₁₉NONa 216.1364; Found 216.1358.

(2E,4E)-N-Phenylhexa-2,4-dienamide (2i):

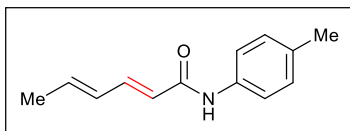


Prepared according to General Procedure 1; white solid; eluent (20% ethyl acetate:hexane); yield is 78%. **¹H NMR (400 MHz, CDCl₃)**: δ 8.39 – 7.46 (m, 3H), 7.30 (m, *J* = 10.9, 10.4, 4.7 Hz, 2H), 7.09 (t, *J* = 7.3 Hz, 1H), 6.26 – 5.64 (m, 3H), 1.96 – 1.53 (d, 3H). **¹³C{¹H} NMR (101**

MHz, CDCl₃): 166.5, 163.3, 141.7, 138.1, 134.3, 129.6, 129.4, 121.2, 115.5, 115.3, 42.9, 18.6.

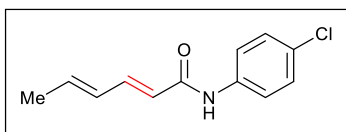
HRMS (ESI/Q-TOF) m/z: [M + Na]⁺ Calcd for C₁₂H₁₃NONa 210.0895; Found 210.0898.

(2E,4E)-N-(p-Tolyl)hexa-2,4-dienamide (2j):



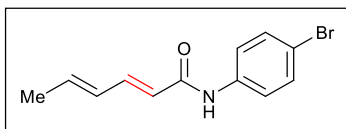
Prepared according to General Procedure 1; white solid; eluent (20% ethyl acetate:hexane); yield is 84%. **¹H NMR (400 MHz, CDCl₃):** δ 7.81 – 7.03 (m, 3H), 6.68 – 6.03 (m, 1H), 6.16 (m, 2H), 6.12 – 5.72 (m, 2H), 5.39 – 4.81 (m, 1H), 3.19 (d, *J* = 7.4 Hz, 3H), 1.86 (d, *J* = 5.1 Hz, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 164.9, 142.6, 138.7, 137.0, 135.3, 129.7, 129.0, 127.3, 124.2, 121.8, 120.0, 18.6, 14.0. **HRMS (ESI/Q-TOF) m/z:** [M + Na]⁺ Calcd for C₁₃H₁₅NONa 224.1051; Found 224.1062.

(2E,4E)-N-(4-Chlorophenyl)hexa-2,4-dienamide (2k):



Prepared according to General Procedure 1; white solid; eluent (20% ethyl acetate:hexane); yield is 74%. **¹H NMR (400 MHz, CDCl₃):** δ 7.97 – 7.52 (m, 3H), 7.30 – 7.09 (dd, *J* = 9.1, 5.6 Hz, 2H), 6.48 – 5.78 (m, 1H), 6.31 – 5.72 (m, 2H), 2.12 – 1.54 (m, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** 166.5, 141.7, 138.1, 134.3, 134.2, 129.6, 129.4, 129.4, 121.2, 115.5, 115.3, 42.8, 18.6.

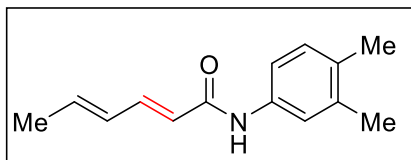
(2E,4E)-N-(4-Bromophenyl)hexa-2,4-dienamide (2l):



Prepared according to General Procedure 1; white solid; eluent (20% ethyl acetate : hexane); yield is 79%. **¹H NMR (400 MHz, CDCl₃):** δ 9.79 (s, 1H), 8.80 (d, *J* = 1.5 Hz, 1H), 8.74 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.10 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.62 – 7.37 (m, 4H), 6.24 – 5.94 (m, 2H), 1.82 (d, *J* = 6.4 Hz, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 166.1, 148.1, 142.4, 138.7,

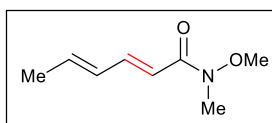
136.4, 131.3, 129.8, 127.5, 122.4, 121.8, 121.5, 116.8, 112.0. **HRMS (ESI/Q-TOF) m/z:** [M + H]⁺ Calcd for C₁₂H₁₃BrNO 266.0181; Found 266.0188.

(2E,4E)-N-(3,4-Dimethylphenyl)hexa-2,4-dienamide (2m):



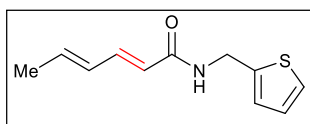
Prepared according to General Procedure 1; white solid; eluent (25% ethyl acetate:hexane); yield is 70%. ¹H NMR (400 MHz, CDCl₃): δ 7.51 (d, *J* = 45.1 Hz, 1H), 7.35 – 7.15 (m, 1H), 6.94 (d, *J* = 7.3 Hz, 2H), 6.25 – 5.79 (m, 3H), 2.27 (s, 3H), 2.17 (s, 3H), 1.82 (d, *J* = 6.4 Hz, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 164.9, 142.1, 138.2, 133.2, 131.1, 129.8, 127.1, 123.9, 121.9, 20.9, 18.6, 17.8. **HRMS (ESI/Q-TOF) m/z:** [M + Na]⁺ Calcd for C₁₄H₁₇NONa 238.1208; Found 238.1211.

(2E,4E)-N-Methoxy-N-methylhexa-2,4-dienamide (2n):



Prepared according to General Procedure 1; white solid; eluent (4% ethyl acetate in hexane); yield is 89%. ¹H NMR (400 MHz, CDCl₃): δ 7.44 – 7.09 (m, 1H), 6.82 (d, *J* = 8.2 Hz, 1H), 6.08 (m, 1H), 5.78 (m, *J* = 15.0 Hz, 1H), 4.37 (s, *J* = 5.8 Hz, 3H), 3.76 (s, 3H), 1.81 (d, *J* = 6.3 Hz, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 166.4, 141.3, 121.5, 114.0, 114.0, 55.2, 43.1, 18.5. **HRMS (ESI/Q-TOF) m/z:** [M + Na]⁺ Calcd for C₈H₁₃NO₂Na 178.0844; Found 178.0835.

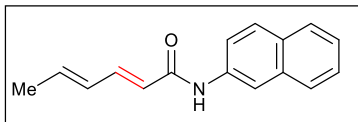
(2E,4E)-N-(Thiophen-2-ylmethyl)hexa-2,4-dienamide (2o):



Prepared according to General Procedure 1; white solid; eluent (18% ethyl acetate:hexane); yield is 68%. ¹H NMR (400 MHz, CDCl₃): δ 7.41 – 7.13 (m, 2H), 6.97 (t, *J* = 8.6 Hz, 2H), 6.23 – 6.01 (m, 2H), 5.79 (d, *J* = 15.0 Hz, 1H), 4.42 (d, *J* = 5.9 Hz, 2H), 1.82 (d, *J* = 6.0 Hz,

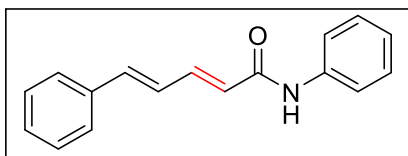
3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 166.5, 141.7, 138.1, 134.2, 129.6, 129.4, 121.2, 115.5, 115.3, 42.8, 18.6.

(2E,4E)-N-(Naphthalen-2-yl)hexa-2,4-dienamide (2p):



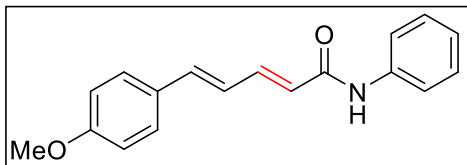
Prepared according to general procedure 2; white solid; eluent (20% ethyl acetate:hexane); yield is 66%. ^1H NMR (400 MHz, CDCl_3): δ 9.79 (s, 1H), 8.77 – 8.10 (d, J = 8.2 Hz, 2H), 7.74 – 7.11 (m, 1H), 7.38 (d, J = 4.2 Hz, 6H), 6.38 – 5.77 (m, 2H), 1.82 (d, J = 6.5 Hz, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 166.1, 148.1, 142.4, 138.7, 136.4, 129.8, 127.5, 123.1, 122.4, 121.8, 121.5, 116.7, 112.5, 111.99, 27.0. HRMS (ESI/Q-TOF) m/z : $[\text{M} + \text{H}]^+$ Calcd for $\text{C}_{16}\text{H}_{16}\text{NO}$ 238.1232; Found 238.1241.

(2E,4E)-N,5-Diphenylpenta-2,4-dienamide (4a):



Prepared according to general procedure 1; white solid; eluent (18% ethyl acetate:hexane); yield is 80%. ^1H NMR (400 MHz, CDCl_3): δ 9.85 (s, 1H), 8.08 (dd, J = 42.3, 5.8 Hz, 2H), 7.9 (dd, J = 15.7, 11.4 Hz, 1H), 7.88 (d, J = 8.2 Hz, 1H), 7.62 – 7.31 (m, 5H), 7.31 – 7.00 (m, 2H), 6.94 – 6.51 (m, 2H), 5.96 (d, J = 11.1 Hz, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 164.8, 148.1, 142.8, 140.7, 138.5, 136.6, 136.4, 134.7, 128.8, 128.0, 127.6, 127.5, 125.2, 121.6, 121.6, 120.8, 116.5. HRMS (ESI/Q-TOF) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{17}\text{H}_{15}\text{NONa}$ 272.1051; Found 272.1056.

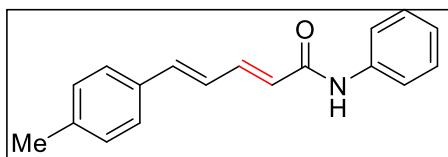
(2E,4E)-5-(4-Methoxyphenyl)-N-phenylpenta-2,4-dienamide (4b):



Prepared according to General Procedure 1; white solid; eluent (18% ethyl acetate:hexane); yield is 84%. ^1H NMR (400 MHz, CDCl_3): δ 9.84 (s, 1H), 8.79 (dd, J = 26.7, 5.8 Hz, 2H), 7.90 (d, J = 8.2 Hz, 1H), 7.59 – 7.33 (m, 5H), 6.96 – 6.74 (m, 4H), 6.27 (d, J = 14.8 Hz, 1H), 3.77 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 165.4, 147.4, 144.0, 143.9, 141.3, 138.4, 136.0,

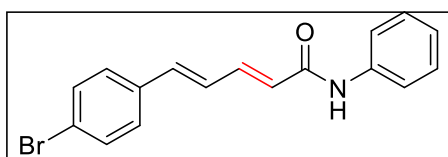
133.4, 130.9, 128.9, 127.9, 127.4, 121.3, 116.0, 110.1, 52.3. **HRMS (ESI/Q-TOF) m/z:** [M + H]⁺ Calcd for C₁₈H₁₈NO₂ 280.1338; Found 280.1332.

(2E,4E)-N-Phenyl-5-(p-tolyl)penta-2,4-dienamide (4c):



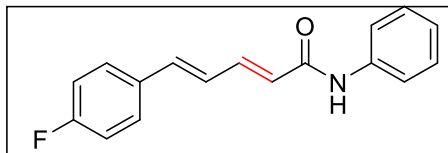
Prepared according to general procedure **1**; yellow solid; eluent (18% ethyl acetate:hexane); yield is 79%. **¹H NMR (400 MHz, CDCl₃):** δ 9.85 (s, 1H), 8.01 (dd, *J* = 39.9, 5.7 Hz, 2H), 7.95 (dd, *J* = 15.6, 11.4 Hz, 1H), 7.70 (d, *J* = 8.2 Hz, 1H), 7.54 – 7.33 (m, 4H), 7.08 (d, *J* = 7.8 Hz, 2H), 6.80 – 6.64 (m, 2H), 5.94 (d, *J* = 11.1 Hz, 1H), 2.28 (s, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 164.9, 148.1, 143.1, 140.8, 139.0, 138.9, 136.4, 133.9, 129.4, 128.6, 128.0, 127.5, 124.3, 121.6, 121.5, 120.2, 116.5, 21.4. **HRMS (ESI/Q-TOF) m/z:** [M + Na]⁺ Calcd for C₁₈H₁₇NONa 286.1208; Found 286.1213.

(2E,4E)-5-(4-Bromophenyl)-N-phenylpenta-2,4-dienamide (4d):



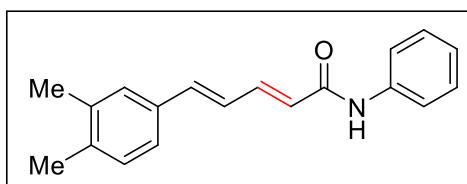
Prepared according to general procedure **1**; white solid; eluent (22% ethyl acetate:hexane); yield is 73%. **¹H NMR (400 MHz, CDCl₃):** 10.64 (s, 1H), 8.2 (m, 3H), 8.0 (dd, *J* = 8.3, 1.4 Hz, 1H), 7.88 (d, *J* = 8.5 Hz, 3H), 7.62 (d, *J* = 8.4 Hz, 2H), 7.52 (dt, *J* = 15.3, 7.5 Hz, 3H), 7.43 – 7.38 (m, 1H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 164.8, 152.1, 148.1, 142.8, 140.7, 138.5, 136.6, 136.4, 134.7, 128.7, 128.0, 127.6, 127.5, 125.2, 121.6, 120.8, 116.5.

(2E,4E)-5-(4-Fluorophenyl)-N-phenylpenta-2,4-dienamide (4e):



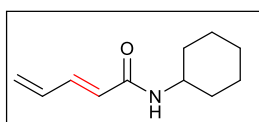
Prepared according to general procedure **1**; white solid; eluent (18% ethyl acetate:hexane); yield is 68%. **¹H NMR (400 MHz, CDCl₃):** δ 9.93 (s, 1H), 8.90 – 8.74 (m, 2H), 7.9 (d, *J* = 8.2 Hz, 1H), 7.75 (d, *J* = 15.5 Hz, 1H), 7.63 – 7.22 (m, 9H), 6.73 (d, *J* = 15.5 Hz, 1H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 164.2, 148.2, 142.1, 138.5, 137.3, 136.5, 134.8, 134.6, 130.9, 128.9, 128.7, 128.1, 127.5, 126.8, 126.5, 121.6, 116.9. **HRMS (ESI/Q-TOF) m/z:** [M + Na]⁺ Calcd C₁₇H₁₄FNONa 290.0957; Found 290.0964.

(2E,4E)-5-(3,4-Dimethylphenyl)-N-phenylpenta-2,4-dienamide (4f):



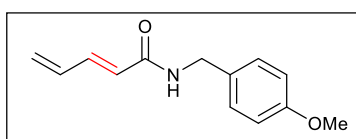
Prepared according to general procedure **1**; white solid; eluent (20% ethyl acetate:hexane); yield is 75%. **¹H NMR (400 MHz, CDCl₃):** δ 9.97 (s, 1H), 8.79 - 8.18 (m, 2H), 8.16 (d, *J* = 8.1 Hz, 1H), 7.53 - 7.15 (m, 8H), 6.29 (d, *J* = 5.4 Hz, 1H), 2.32 (s, 3H), 2.10 (s, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 167.8, 148.1, 139.8, 138.5, 138.1, 136.4, 134.7, 134.1, 131.8, 129.4, 128.4, 127.9, 127.4, 126.4, 121.6, 121.2, 116.8, 45.1, 21.2. **HRMS (ESI/Q-TOF) m/z:** [M + Na]⁺ Calcd C₁₉H₁₉NONa 300.1364; Found 300.1364.

(E)-N-Benzylpenta-2,4-dienamide (6a):



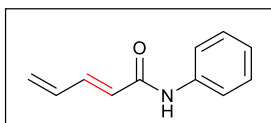
Prepared according to general procedure **1**; white solid; eluent (25% ethyl acetate:hexane); yield is 75%. **¹H NMR (400 MHz, CDCl₃):** δ 7.16 (dd, *J* = 15.0, 10.4 Hz, 1H), 6.28 - 5.64 (m, 4H), 3.85 (m, *J* = 7.4, 3.3 Hz, 1H), 1.99 - 1.86 (m, 2H), 1.71 (dt, *J* = 13.3, 3.9 Hz, 2H), 1.62 (dt, *J* = 12.9, 3.8 Hz, 1H), 1.46 - 1.27 (m, 2H), 1.17 (m, *J* = 11.8, 6.1 Hz, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 165.5, 140.7, 137.2, 129.8, 122.2, 48.2, 33.2, 25.5, 24.9, 18.5. **HRMS (ESI/Q-TOF) m/z:** [M + H]⁺ Calcd for C₁₁H₁₈NO 180.1388; Found 180.1395.

(E)-N-Isobutylpenta-2,4-dienamide (6b):



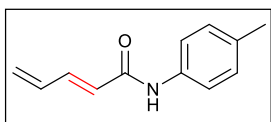
Prepared according to general procedure **1**; white solid; eluent (18% ethyl acetate:hexane); yield is 80%. **¹H NMR (400 MHz, CDCl₃):** δ 7.17 (d, *J* = 8.2 Hz, 3H), 6.82 (d, *J* = 8.2 Hz, 3H), 6.07 (m, *J* = 15.1, 8.8 Hz, 2H), 5.78 (d, *J* = 15.0 Hz, 1H), 4.37 (d, *J* = 5.7 Hz, 2H), 3.76 (s, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 166.4, 158.9, 141.3, 137.8, 130.5, 129.7, 129.1, 121.5, 114.0, 114.0, 55.2, 43.1.

(E)-N-(4-Bromophenyl)penta-2,4-dienamide (6c):



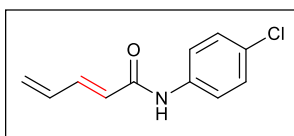
Prepared according to general procedure **1**; white solid; eluent (20% ethyl acetate:hexane); yield is 72%. ¹H NMR (400 MHz, CDCl₃): δ 7.65 – 7.47 (m, 3H), 7.38 (d, *J* = 8.9 Hz, 3H), 7.28 (m, 3H), 6.96 (s, 2H), 6.03 (d, *J* = 14.6 Hz, 1H). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 171.5, 146.9, 142.4, 136.2, 130.2, 129.2, 128.8, 127.7, 126.9, 124.9, 116.7. HRMS (ESI/Q-TOF) *m/z*: [M + H]⁺ Calcd for C₁₁H₁₂NO 174.0919; Found 174.0928.

(E)-N-(p-Tolyl)penta-2,4-dienamide (6d):



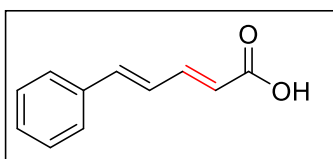
Prepared according to general procedure **1**; white solid; eluent (20% ethyl acetate:hexane); yield is 73%. ¹H NMR (400 MHz, CDCl₃): δ 9.84 (s, 1H), 8.79 (dd, *J* = 26.7, 5.8 Hz, 2H), 8.10 (d, *J* = 8.2 Hz, 1H), 7.49 – 7.36 (m, 3H), 6.82 (d, *J* = 15.8, 8.2 Hz, 1H), 6.27 (d, *J* = 14.8 Hz, 1H), 3.77 (s, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 170.6, 159.5, 143.0, 134.0, 130.4, 128.6, 127.7, 126.3, 125.0, 122.6, 120.9, 37.8.

(E)-N-(4-Chlorophenyl)penta-2,4-dienamide (6e):



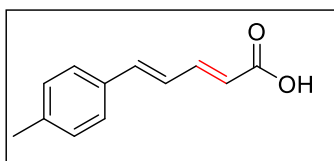
Prepared according to general procedure **1**; white solid; eluent (24% ethyl acetate:hexane); yield is 68%. ¹H NMR (400 MHz, CDCl₃): δ 7.76 – 7.68 (m, 1H), 7.46 (d, *J* = 8.3 Hz, 2H), 7.13 (d, *J* = 8.3 Hz, 2H), 6.72 (d, *J* = 11.3 Hz, 1H), 6.35 (t, *J* = 11.6 Hz, 1H), 5.98 (d, *J* = 15.2 Hz, 1H). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 171.8, 142.1, 137.6, 134.9, 131.8, 130.8, 127.7, 123.6, 123.1, 122.6, 122.0. HRMS (ESI/Q-TOF) *m/z*: [M + Na]⁺ Calcd C₁₁H₁₀ClNO 230.0349; Found 230.0354.

(2E,4E)-5-Phenylpenta-2,4-dienoic acid (8a):



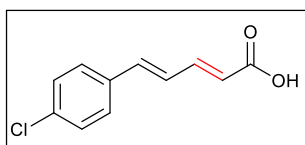
Prepared according to General Procedure 2; half white solid; eluent (28% ethyl acetate:hexane); yield is 86%. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.54 (dd, $J = 25.8, 10.0$ Hz, 3H), 7.38 (d, $J = 8.9$ Hz, 3H), 6.97 (t, $J = 14.8$ Hz, 2H), 6.03 (d, $J = 14.6$ Hz, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 171.5, 146.9, 142.9, 142.3, 141.7, 136.2, 136.0, 128.8, 127.7, 124.9, 116.7. HRMS (ESI/Q-TOF) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{11}\text{H}_{10}\text{O}_2\text{Na}$ 197.0578; Found 197.0576.

(2E,4E)-5-(*p*-Tolyl)penta-2,4-dienoic acid (8b):



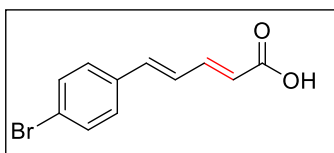
Prepared according to General Procedure 2; half white solid; eluent (30% ethyl acetate:hexane); yield is 84%. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.45 (dd, $J = 15.1, 10.3$ Hz, 1H), 7.31 (d, $J = 7.7$ Hz, 2H), 7.11 (d, $J = 7.8$ Hz, 2H), 6.83 (dd, $J = 20.7, 12.9$ Hz, 2H), 5.90 (d, $J = 15.4$ Hz, 1H), 2.30 (s, 3H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 170.6, 159.5, 143.0, 133.9, 132.1, 130.9, 128.6, 127.4, 127.1, 126.3, 120.9, 37.9. HRMS (ESI/Q-TOF) m/z : $[\text{M} + \text{Na}]^+$ Calcd for $\text{C}_{12}\text{H}_{12}\text{O}_2\text{Na}$ 211.0735; Found 211.0744.

(2E,4E)-5-(4-Chlorophenyl)penta-2,4-dienoic acid (8c):



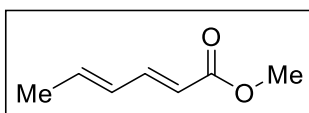
Prepared according to general procedure 2; Half white solid; eluent (28% ethyl acetate:hexane); yield is 77%. $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.82 – 7.51 (m, 1H), 7.46 (d, $J = 8.1$ Hz, 2H), 7.30 – 7.01 (m, 3H), 6.72 (d, $J = 11.3$ Hz, 1H), 6.35 (t, $J = 11.6$ Hz, 1H), 5.98 (d, $J = 15.2$ Hz, 1H). $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3): δ 171.5, 148.6, 146.9, 142.4, 136.2, 129.2, 128.8, 127.7, 126.9, 124.9, 116.7.

(2E,4E)-5-(4-Bromophenyl)penta-2,4-dienoic acid (8d):



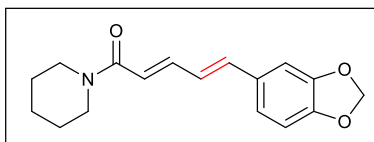
Prepared according to general procedure **2**; Half white solid; eluent (25% ethyl acetate:hexane); yield is 79%. **¹H NMR (400 MHz, CDCl₃):** δ 7.76 – 7.68 (m, 1H), 7.46 (d, *J* = 8.3 Hz, 2H), 7.13 (d, *J* = 8.3 Hz, 2H), 6.72 (d, *J* = 11.3 Hz, 1H), 6.35 (t, *J* = 11.6 Hz, 1H), 5.98 (d, *J* = 15.2 Hz, 1H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 171.8, 142.1, 137.6, 134.9, 131.8, 130.8, 127.7, 123.6, 123.1, 122.6, 122.0. **HRMS (ESI/Q-TOF) m/z:** [M + Na]⁺ Calcd for C₁₁H₉BrO₂Na 274.9684; Found 274.9690.

Methyl (2*E*,4*E*)-hexa-2,4-dienoate (8e):



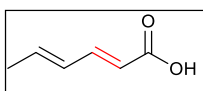
Prepared according to general procedure **2**; Yellow solid; eluent (15% ethyl acetate:hexane); yield is 38%. **¹H NMR (400 MHz, CDCl₃):** δ 7.51 – 7.03 (m, 1H), 6.44 – 6.04 (m, 2H), 5.78 (d, *J* = 15.4 Hz, 1H), 3.73 (d, *J* = 1.4 Hz, 3H), 1.85 (d, *J* = 5.7 Hz, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 167.7, 145.2, 139.4, 129.7, 118.5, 51.4, 18.6, 18.6.

(2*E*,4*E*)-5-(Benzo[d][1,3]dioxol-5-yl)-1-(piperidin-1-yl)penta-2,4-dien-1-one (10)



Prepared according to general procedure **1**; white solid; eluent (20% ethyl acetate:hexane); yield is 69%. **¹H NMR (400 MHz, CDCl₃):** δ 7.16 (dd, *J* = 15.1, 10.7 Hz, 2H), 6.30 – 5.81 (m, 4H), 3.31 (m, *J* = 7.2, 5.7 Hz, 3H), 1.81 (dd, *J* = 6.8, 1.4 Hz, 3H), 1.62 – 1.43 (m, 2H), 1.46 – 1.28 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 166.8, 140.5, 137.0, 129.8, 122.2, 39.3, 31.7, 20.1, 18.4, 13.7. **HRMS (ESI/Q-TOF) m/z:** [M + Na]⁺ Calcd C₁₇H₁₉NO₃Na 308.1263; Found 308.1269.

(2*E*,4*E*)-hexa-2,4-dienoic acid (12)

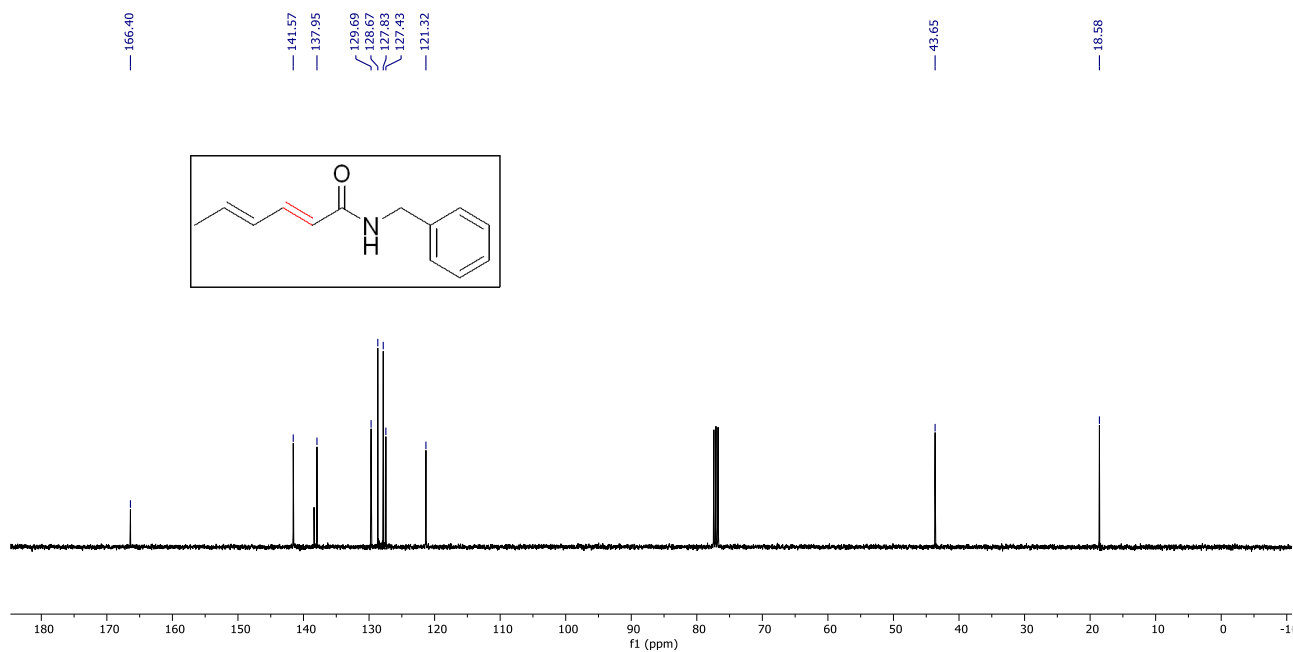


Prepared according to general procedure **2**; Half white solid; eluent (25% ethyl acetate:hexane); yield is 82%. **¹H NMR (400 MHz, CDCl₃):** δ 7.43 – 7.26 (m, 1H), 6.33 – 6.09 (m, 2H), 5.78 (d, *J* = 15.4 Hz, 1H), 1.88 (d, *J* = 5.0 Hz, 3H). **¹³C{¹H} NMR (101 MHz, CDCl₃):** δ 172.9,

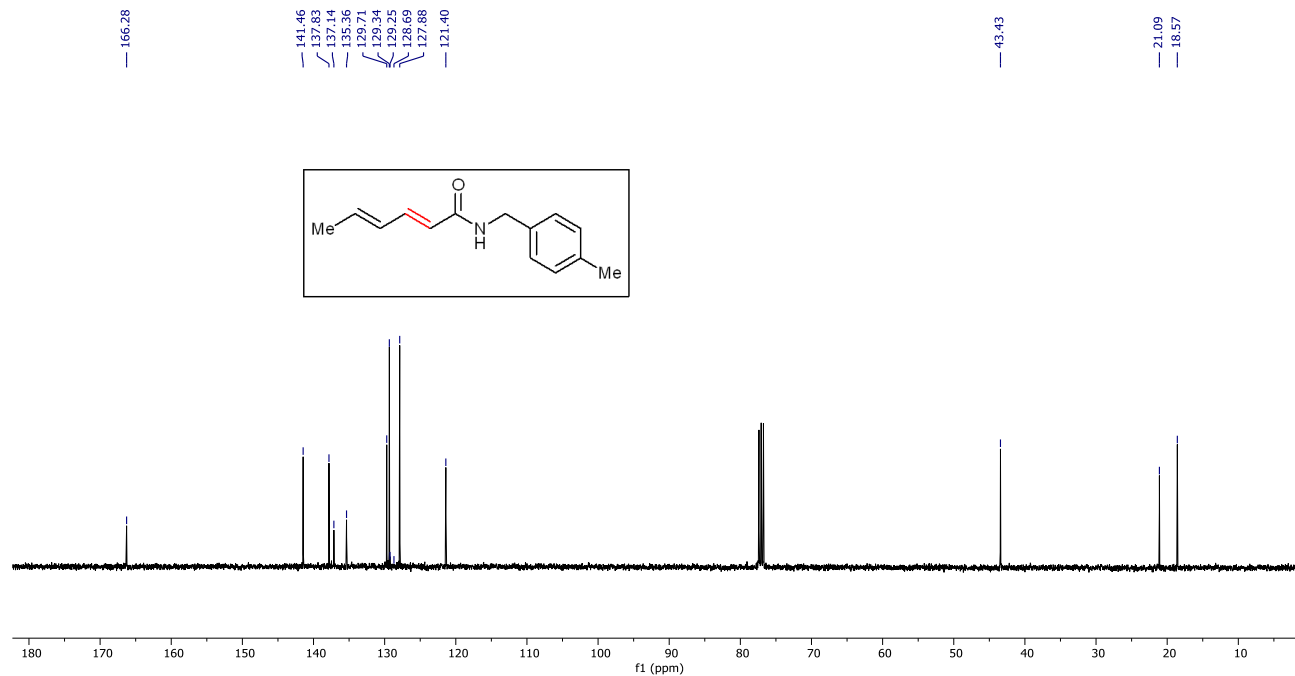
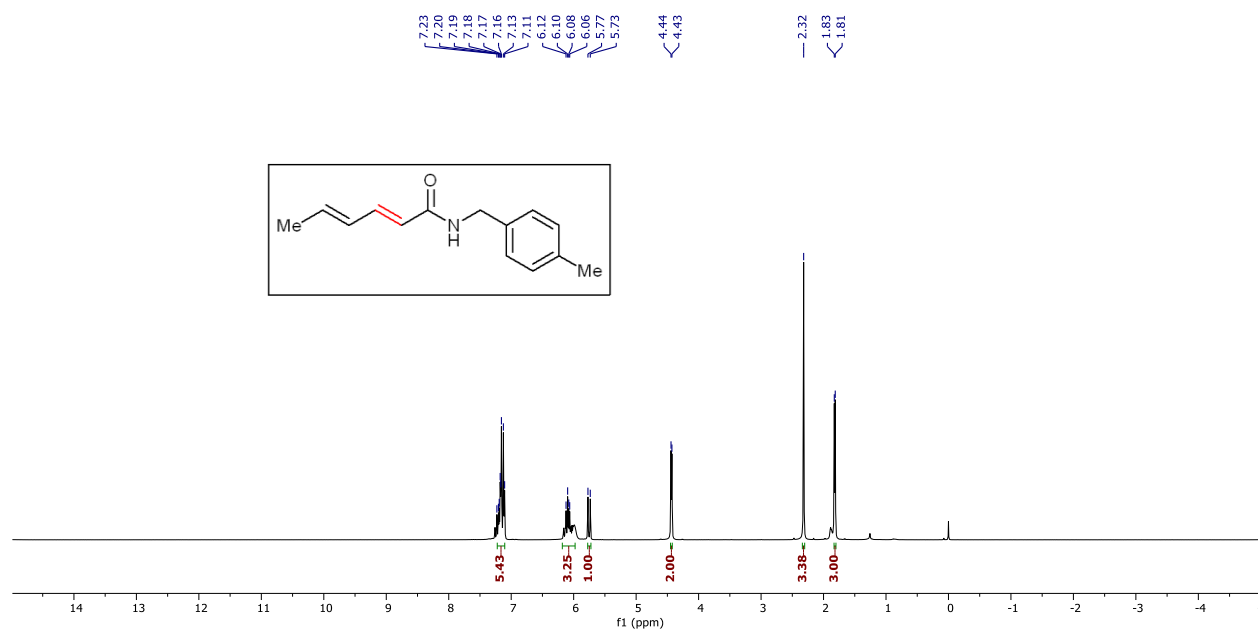
147.4, 140.8, 129.7, 118.2, 18.7. **HRMS (ESI/Q-TOF) m/z:** [M + Na]⁺ Calcd for C₆H₈O₂Na
135.0422; Found 135.0429.

Copies of NMR Spectra

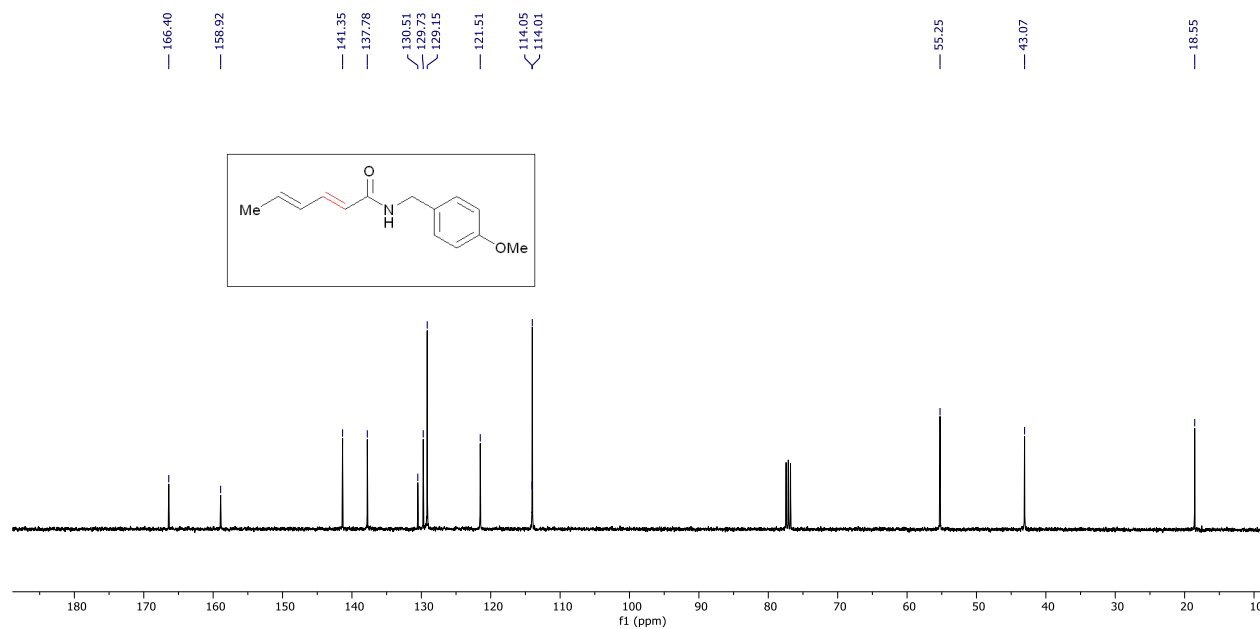
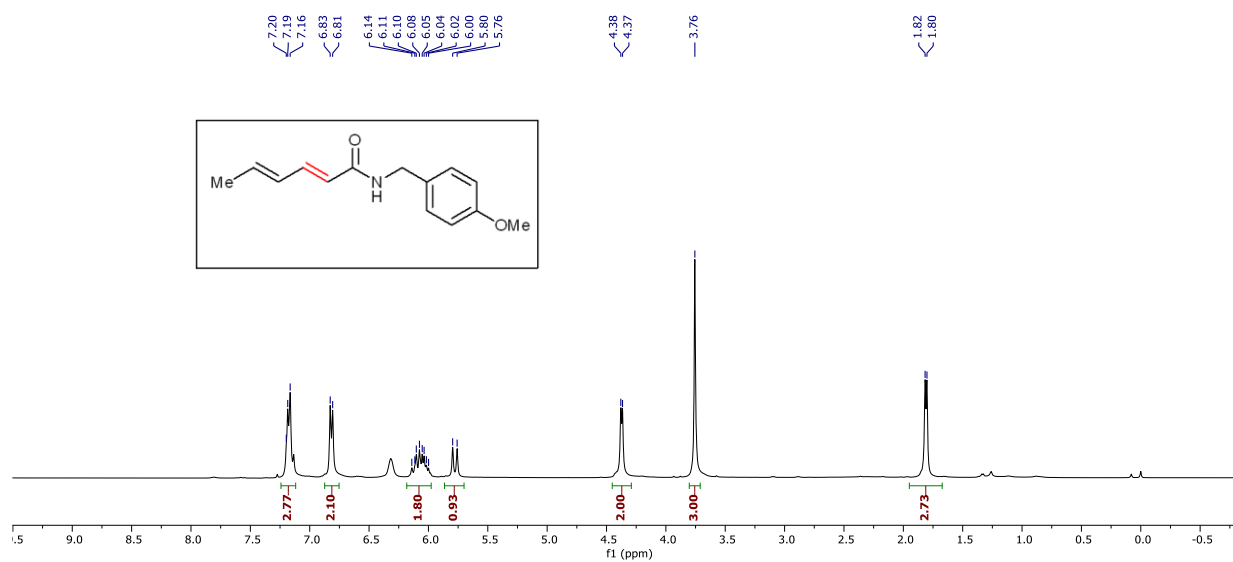
^1H and ^{13}C spectra of compound 2a:



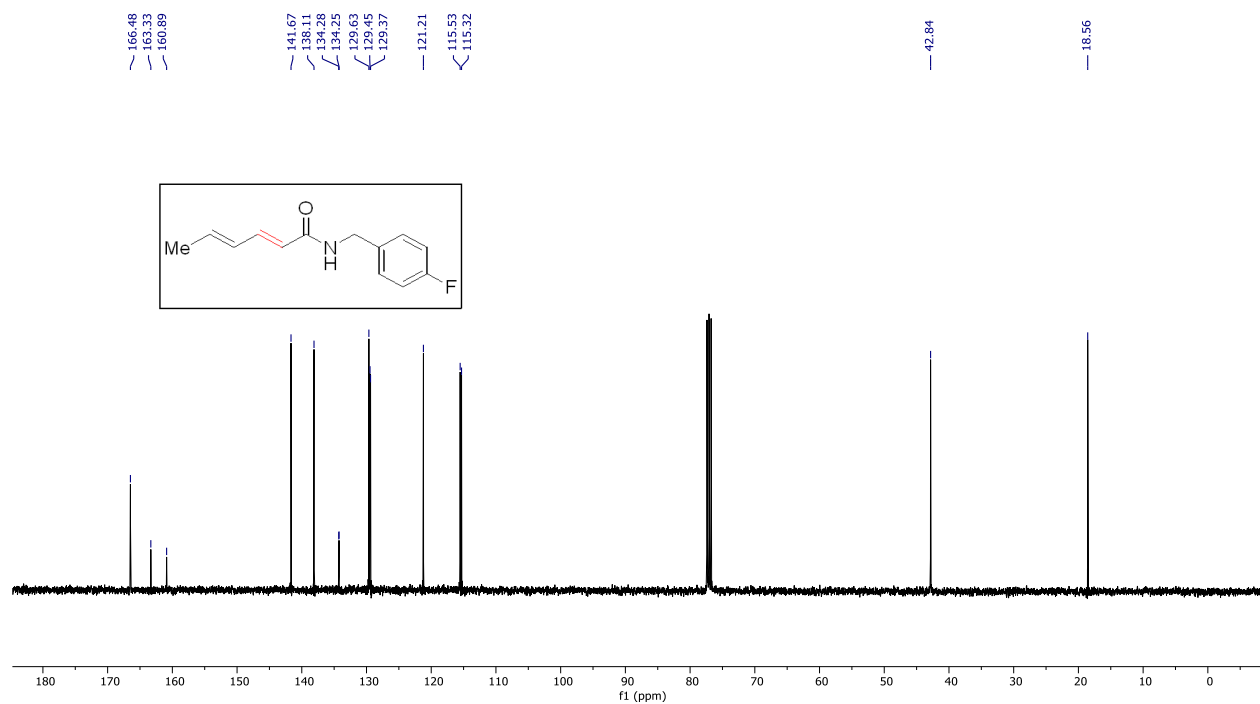
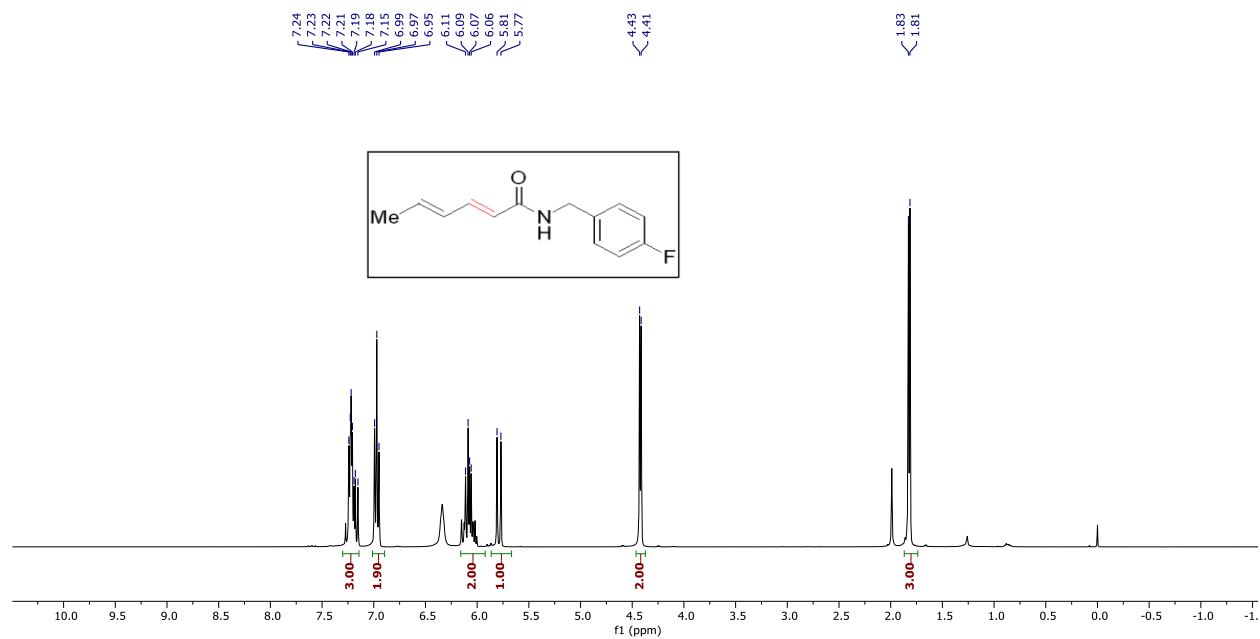
¹H and ¹³C spectra of compound 2b:



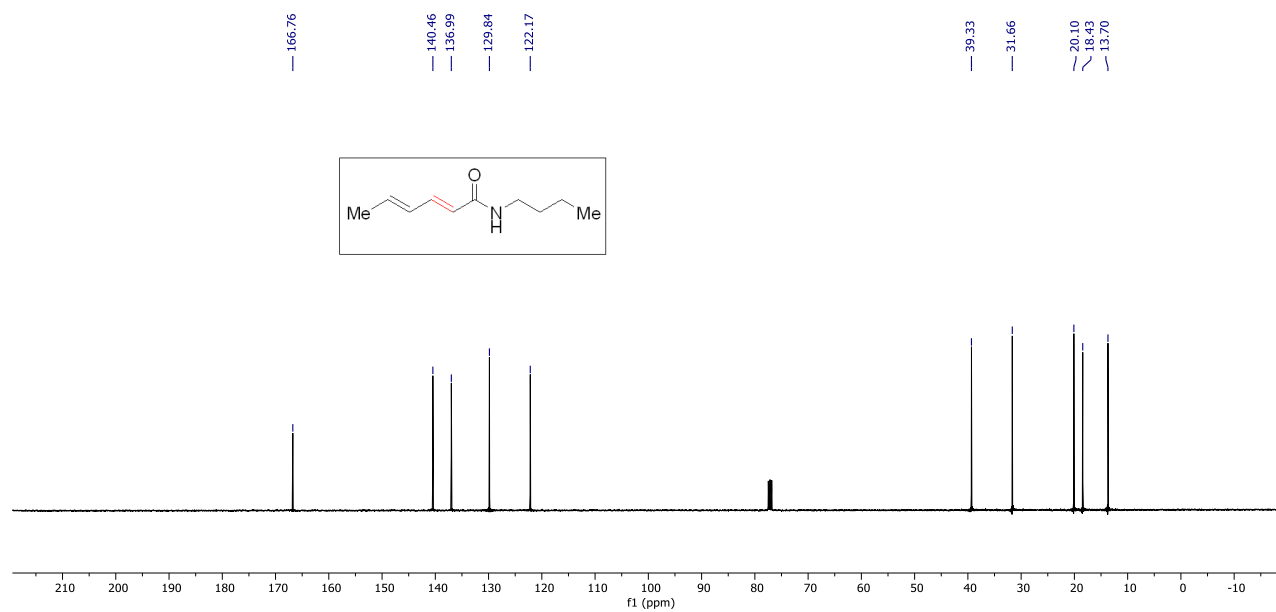
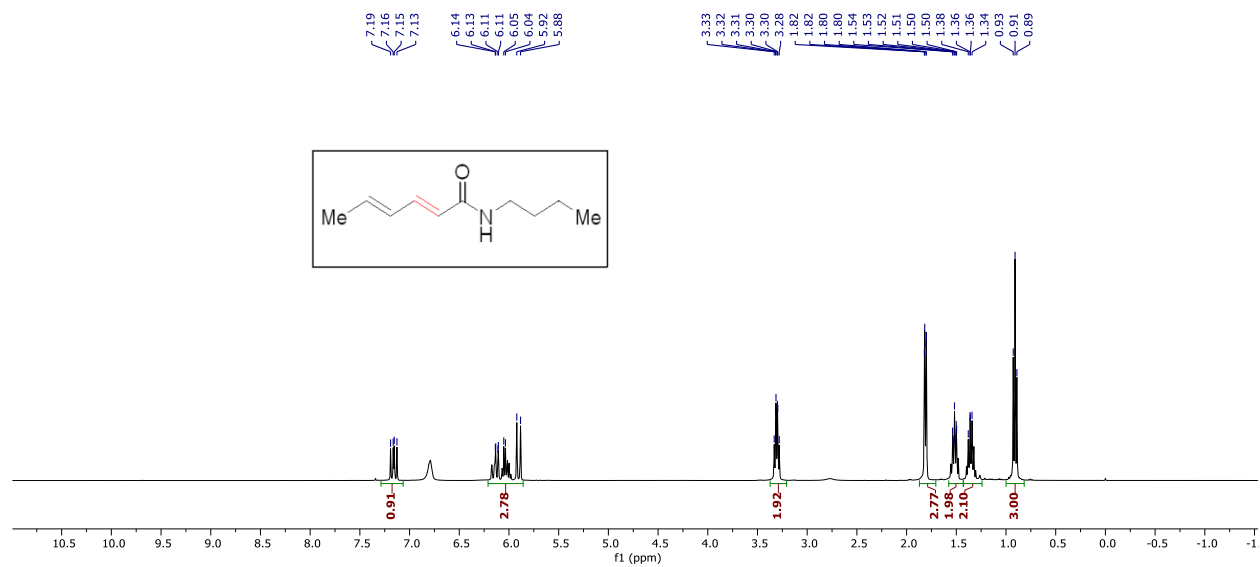
^1H and ^{13}C spectra of compound 2c:



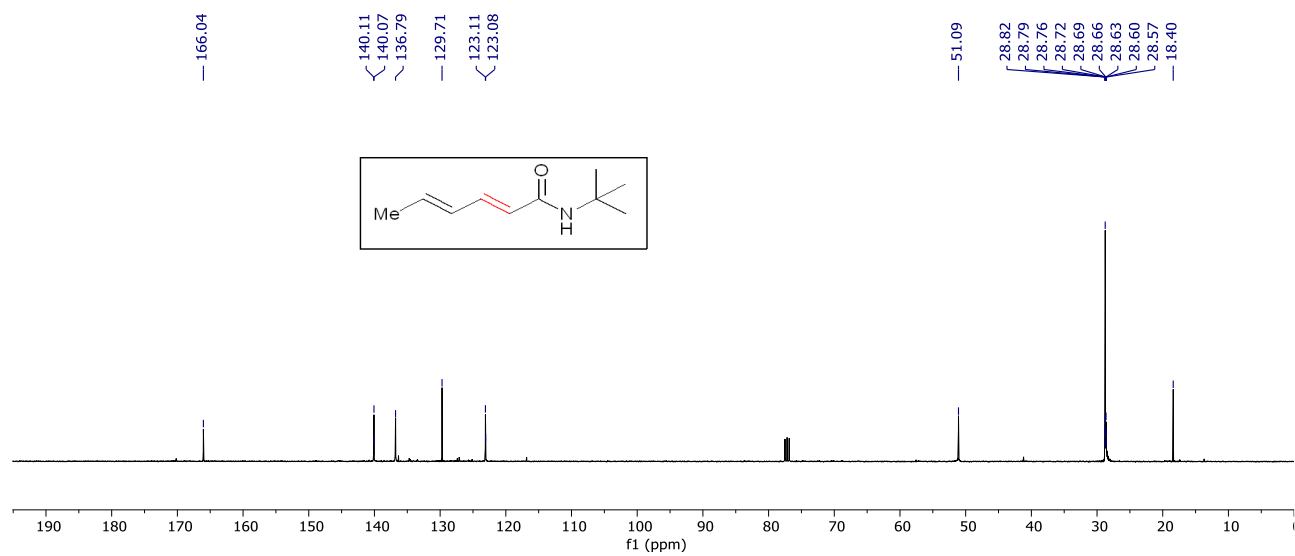
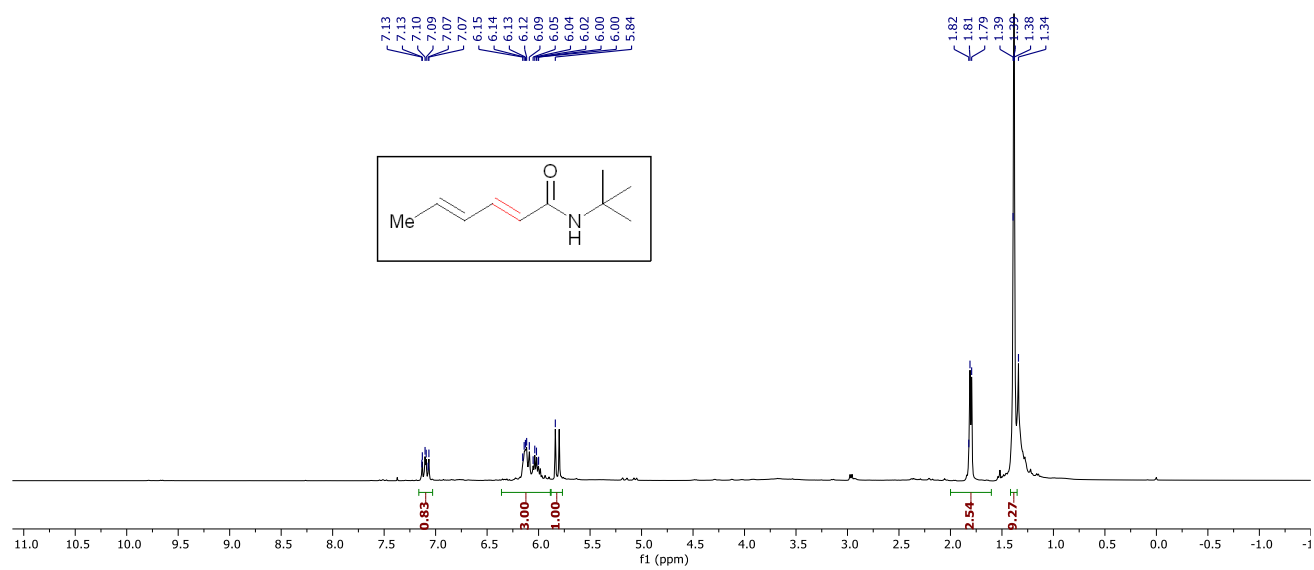
^1H and ^{13}C spectra of compound 2d:



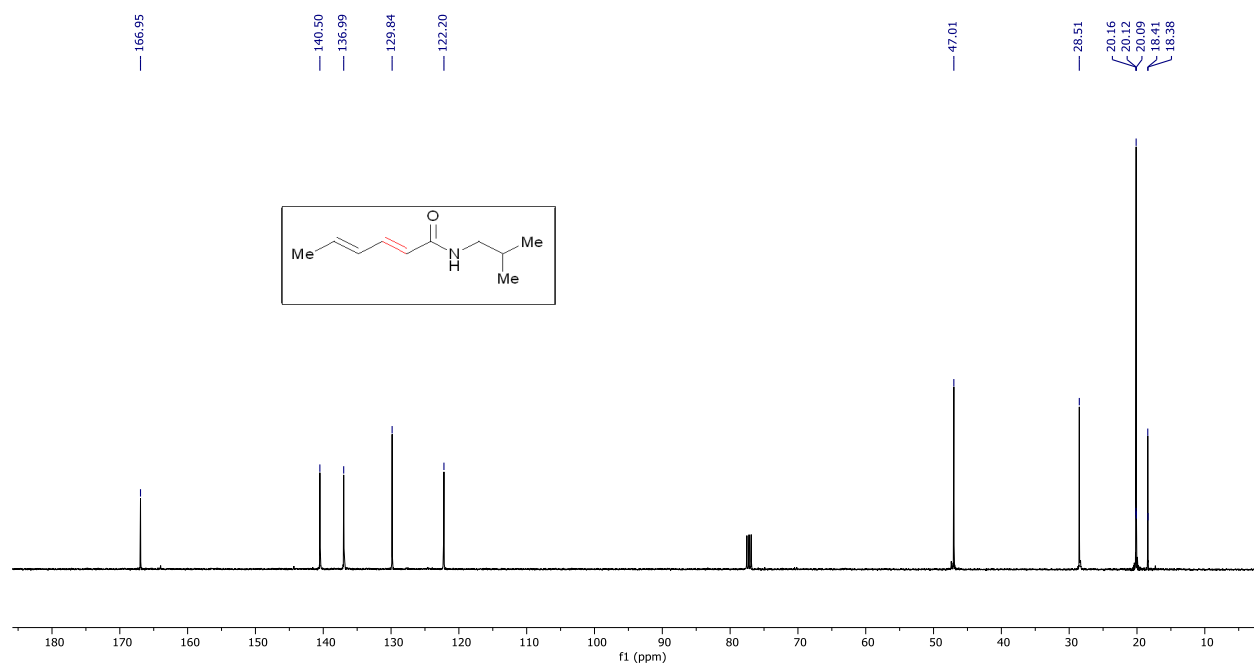
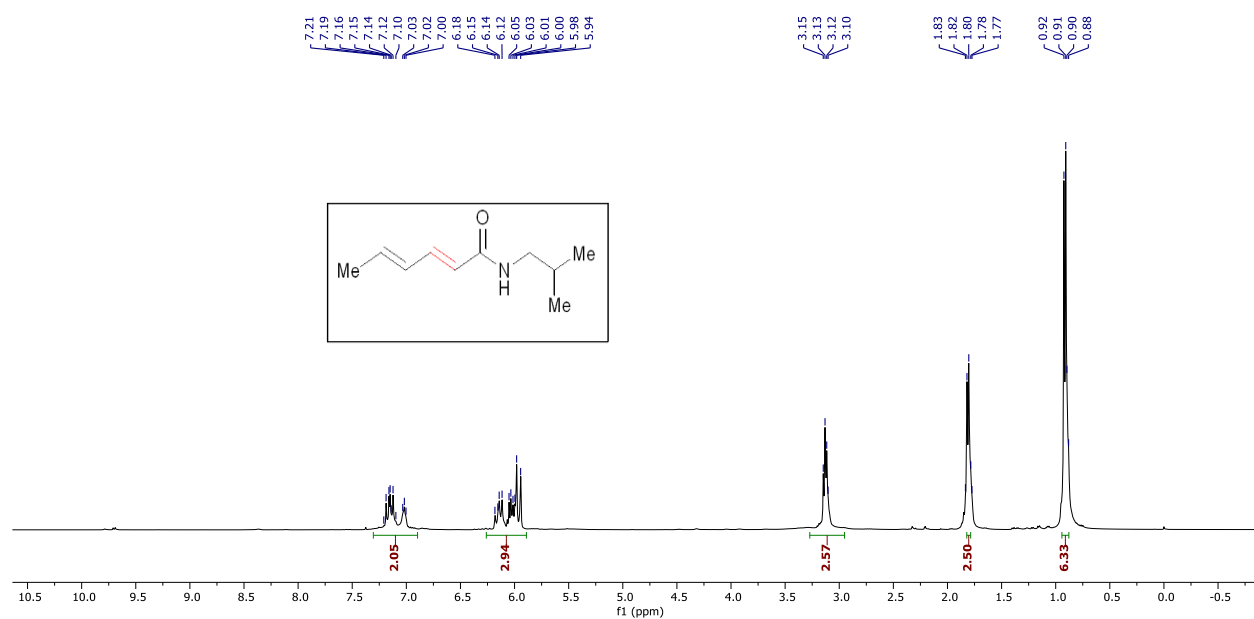
¹H and ¹³C spectra of compound 2e:



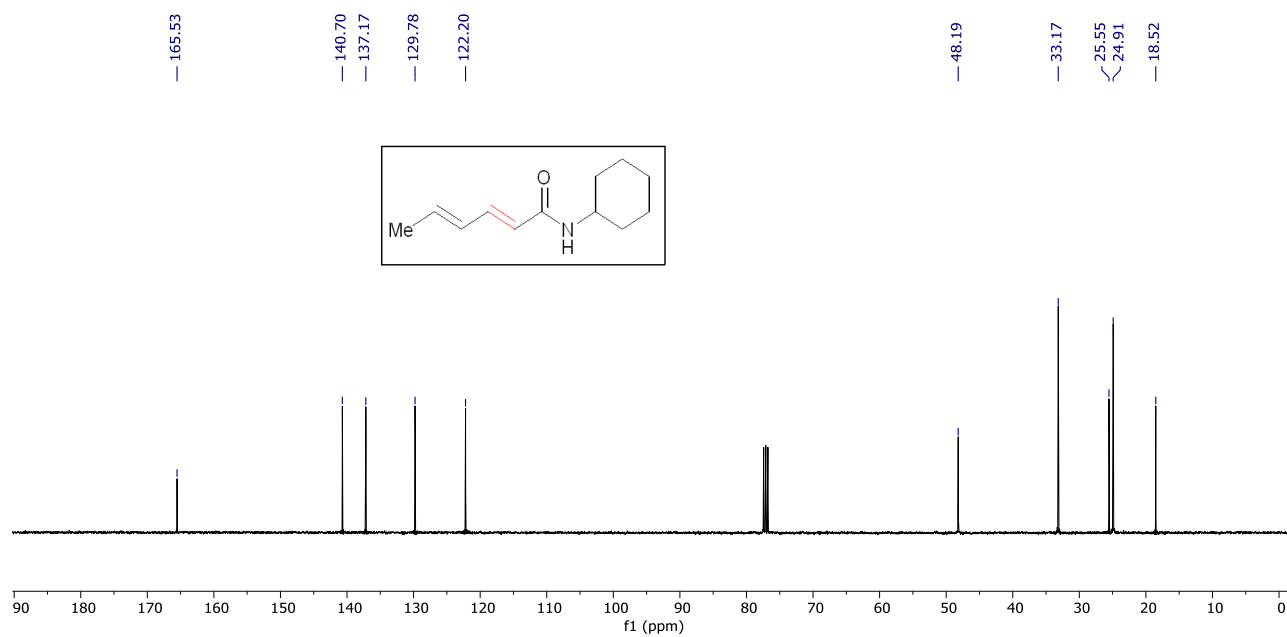
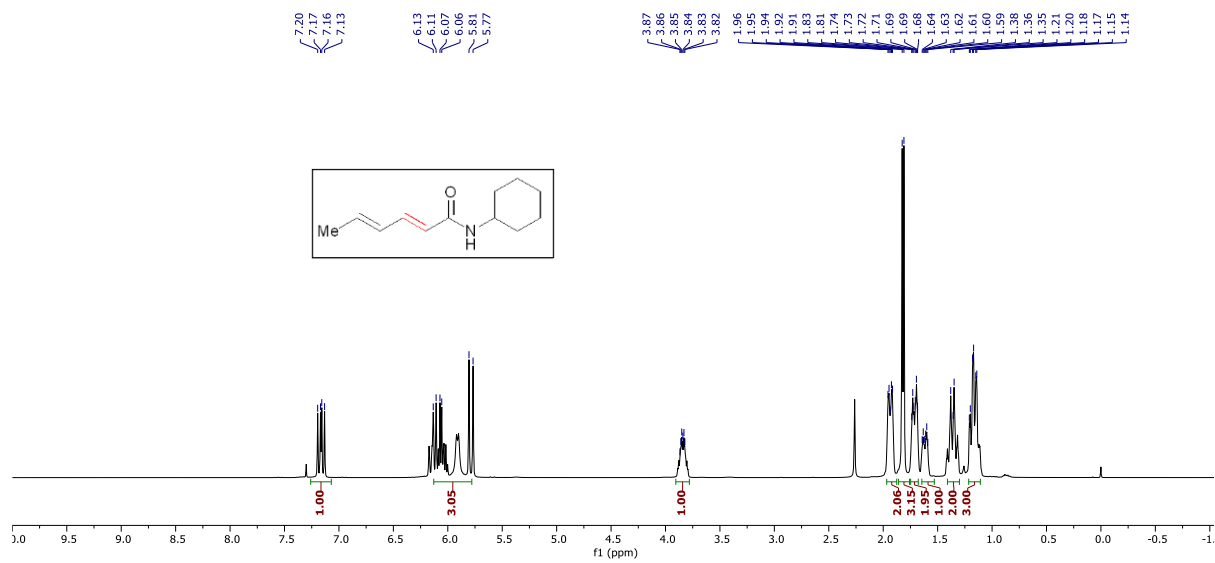
^1H and ^{13}C spectra of compound 2f:



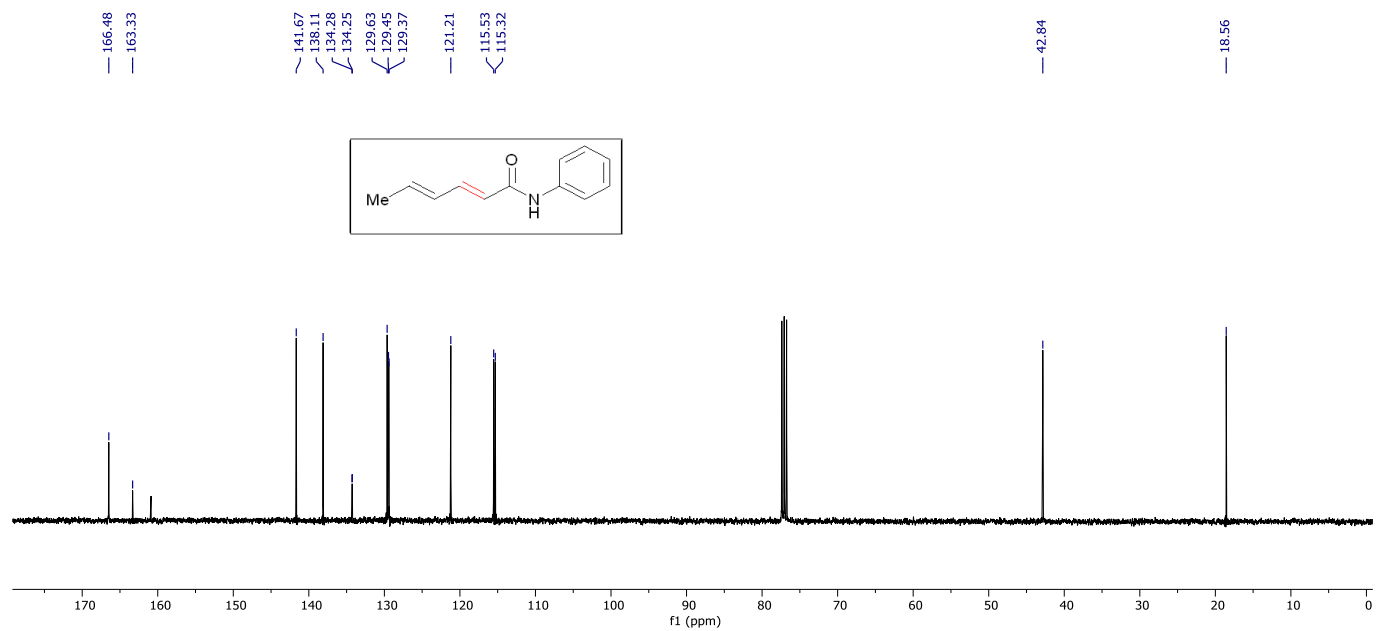
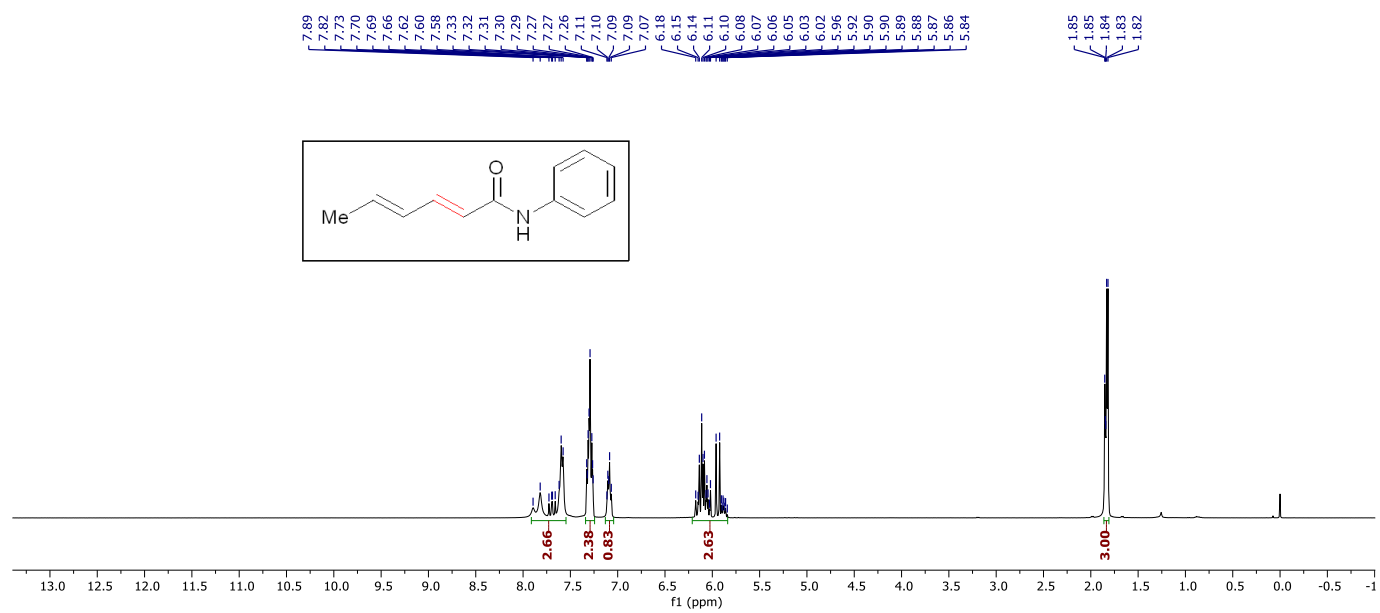
¹H and ¹³C spectra of compound 2g:



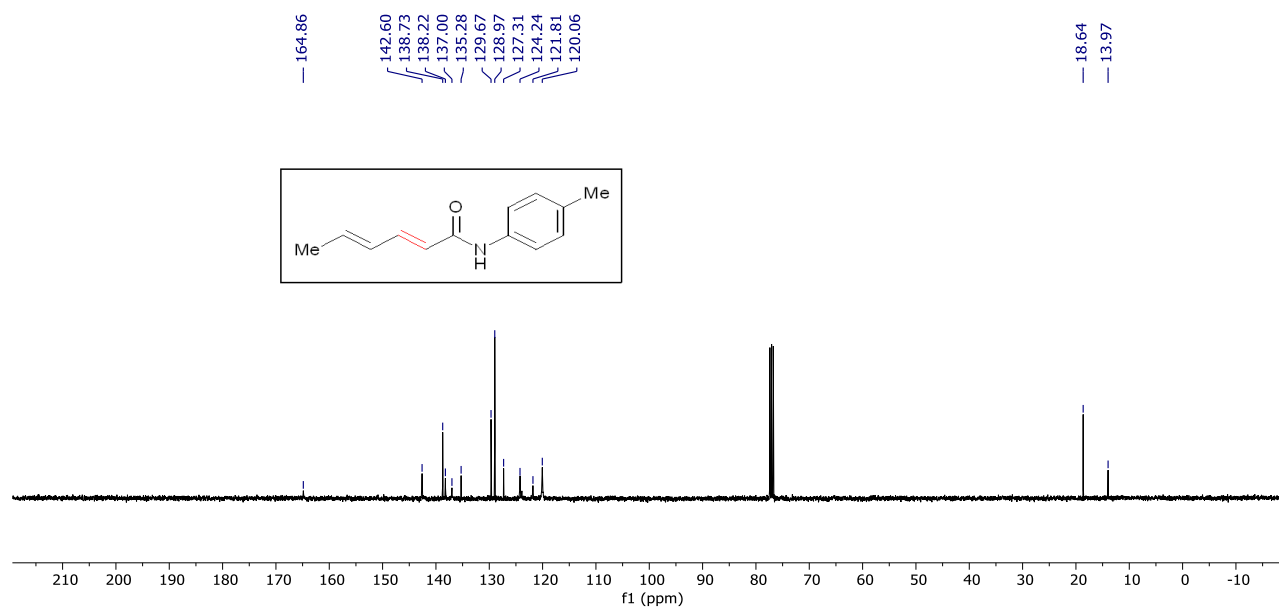
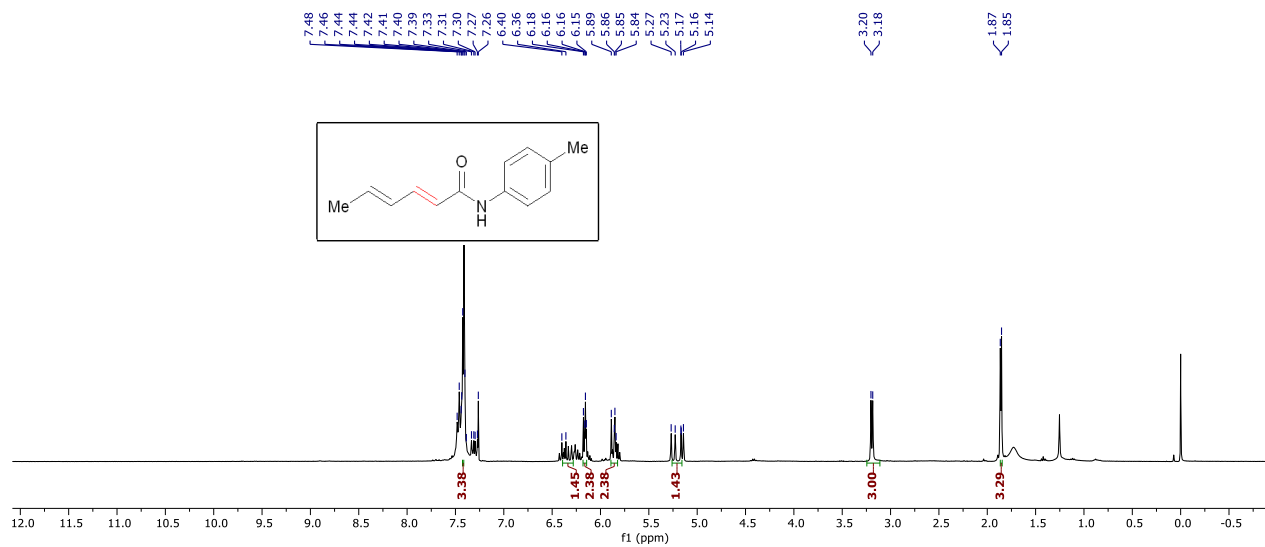
¹H and ¹³C spectra of compound 2h:



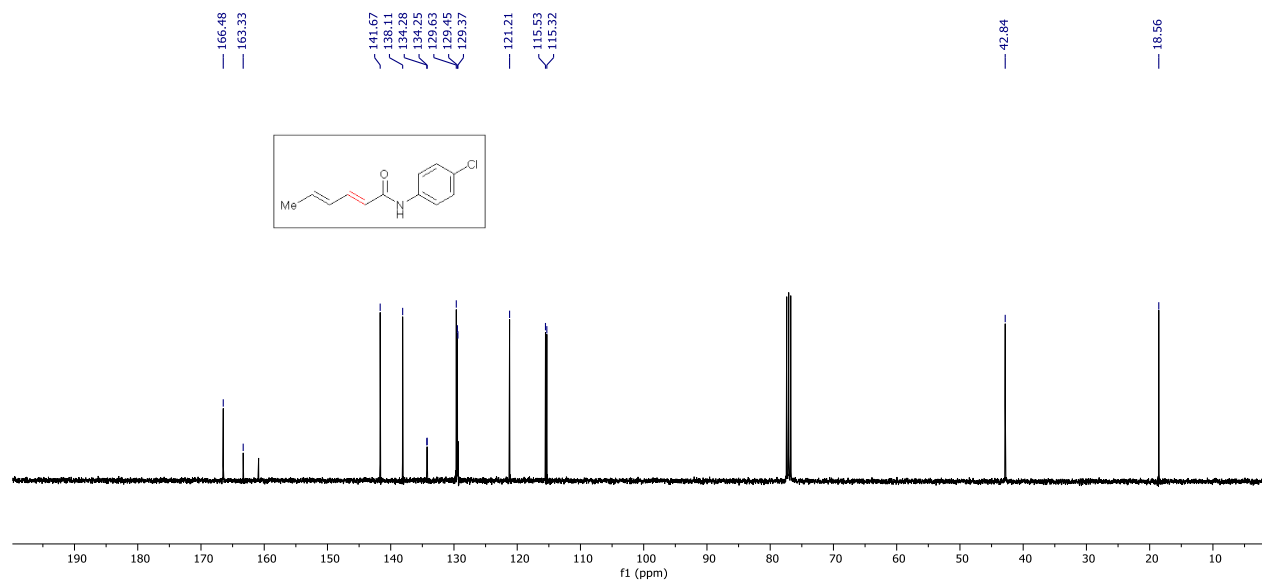
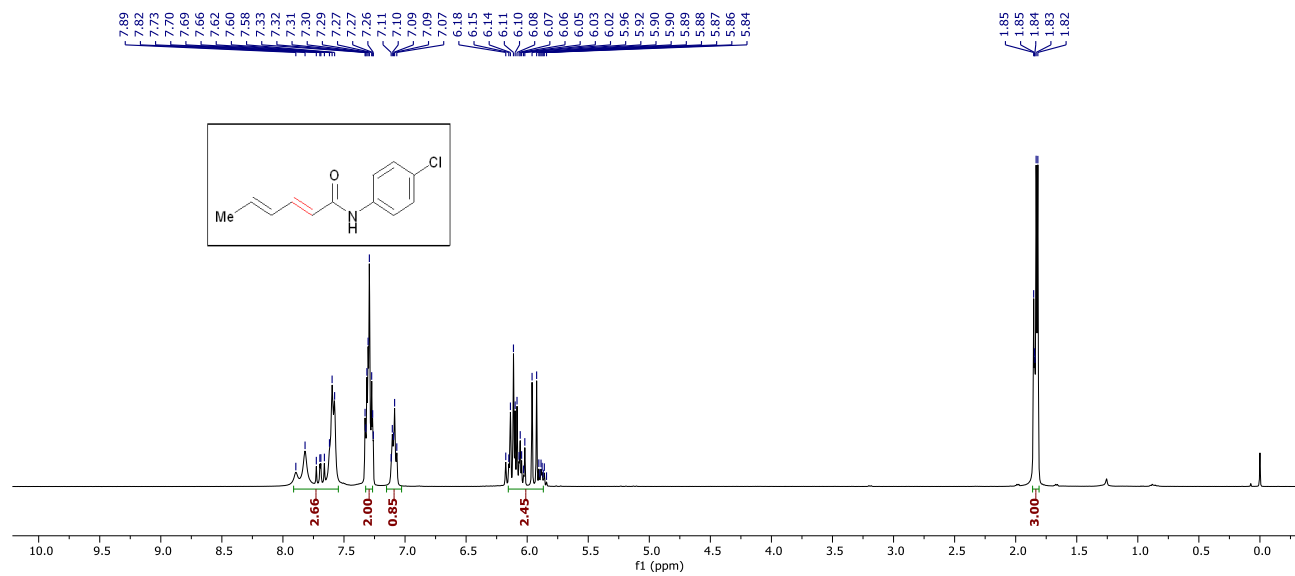
^1H and ^{13}C spectra of compound 2i:



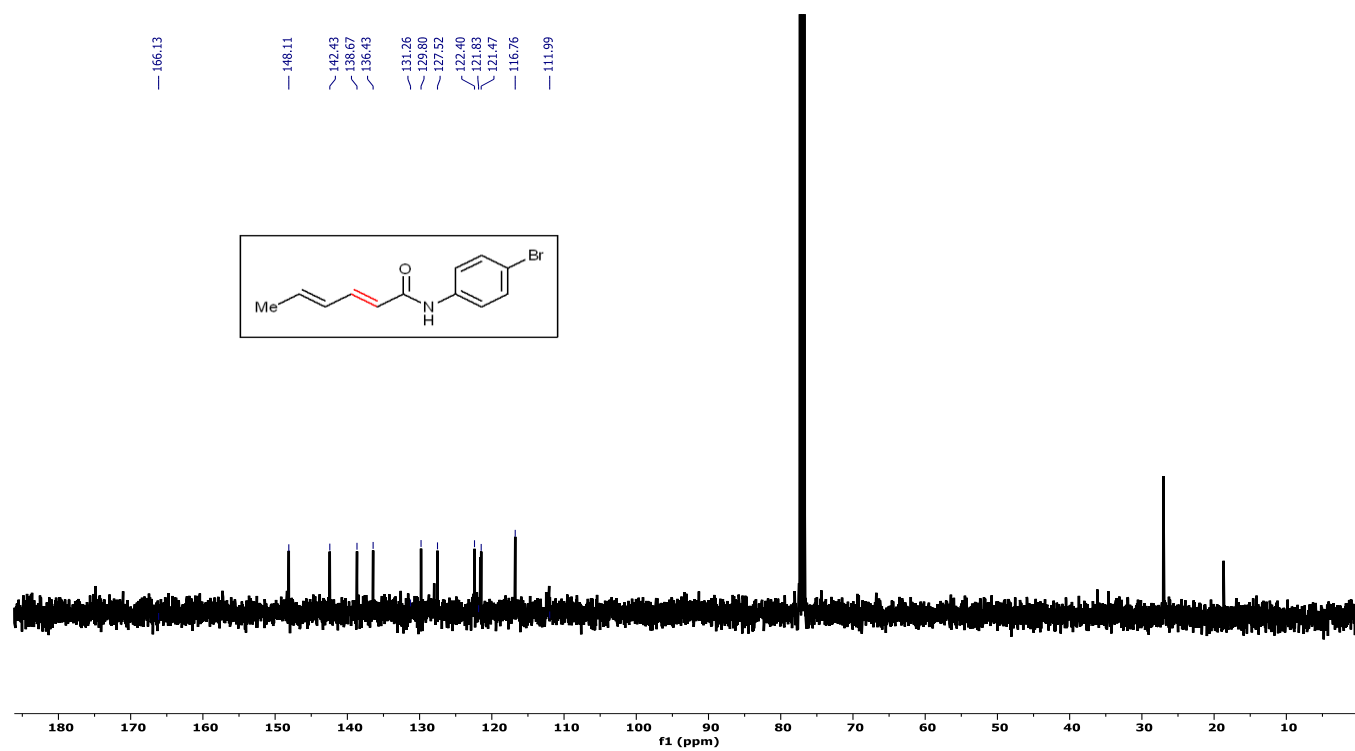
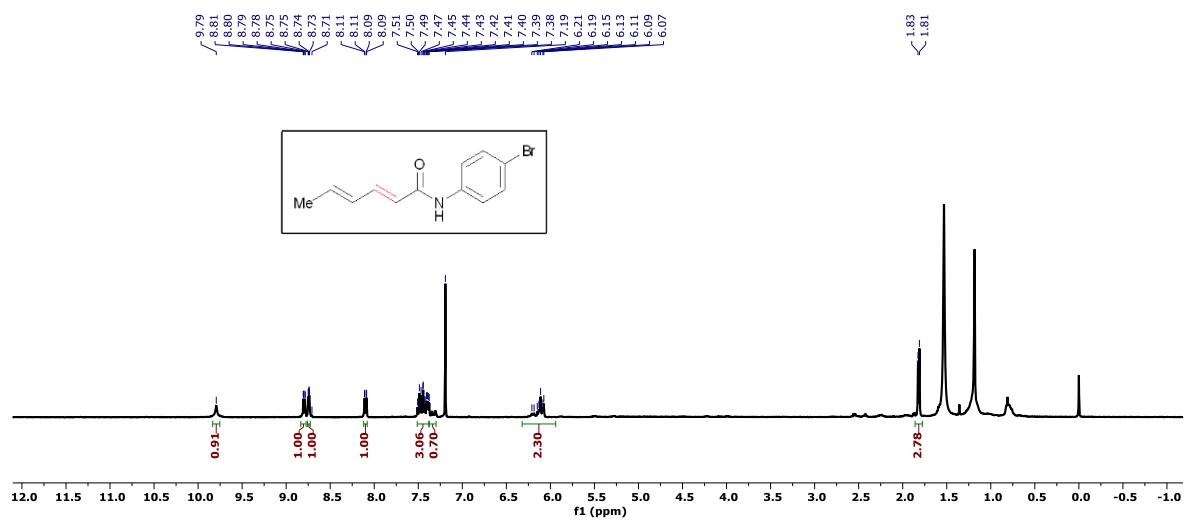
^1H and ^{13}C spectra of compound 2j:



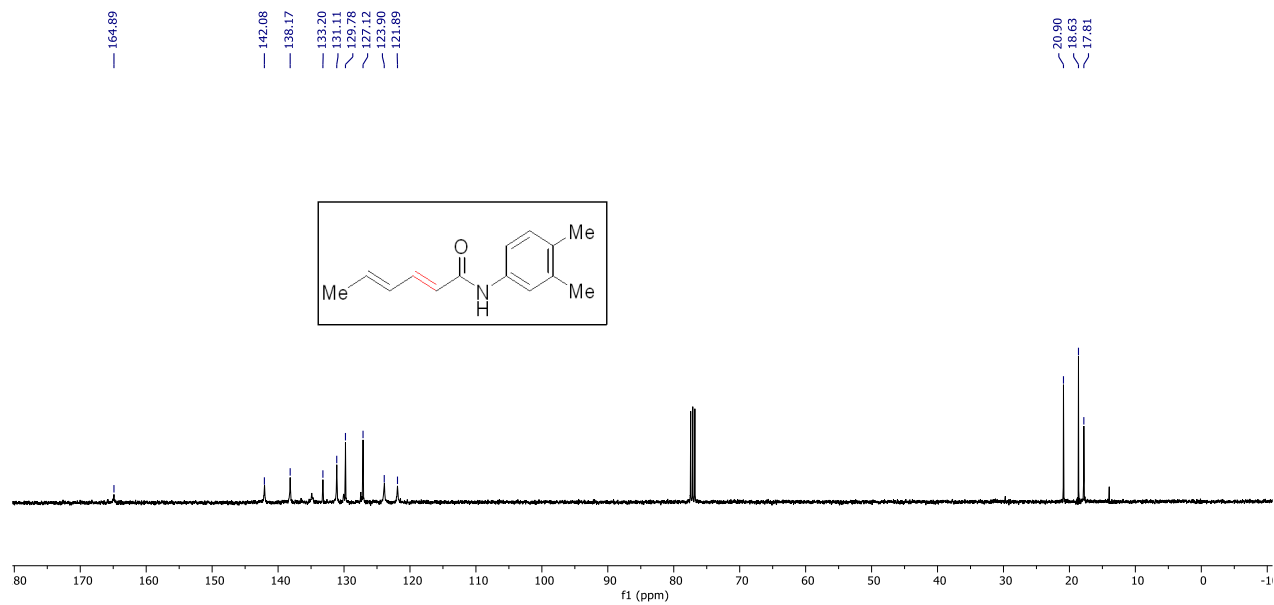
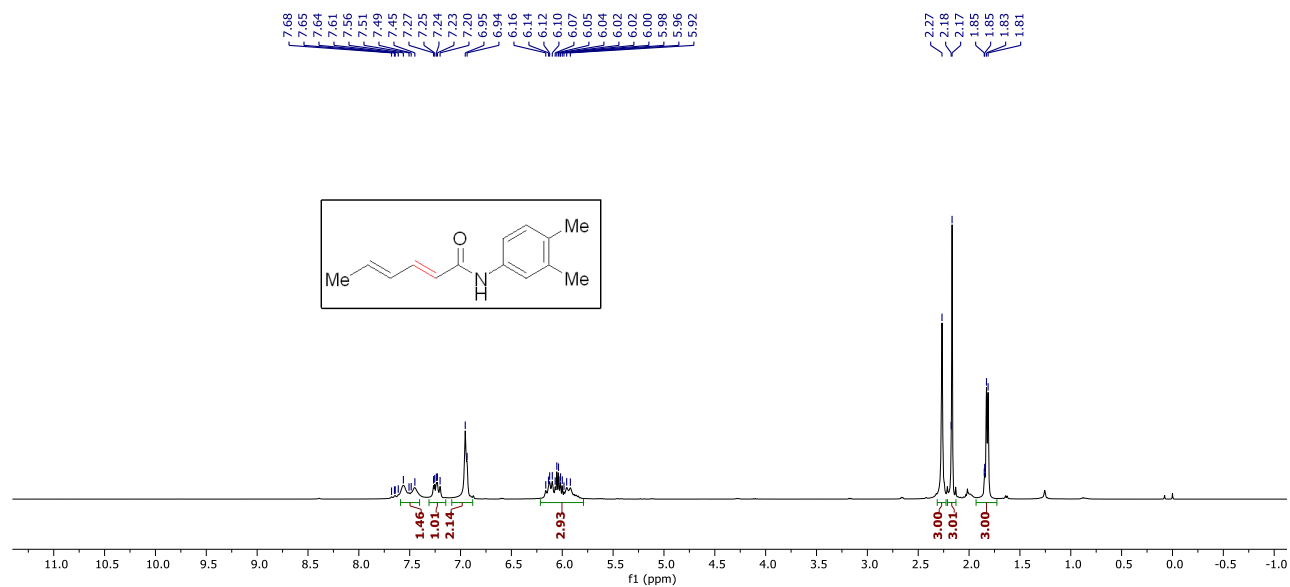
¹H and ¹³C spectra of compound 2k:



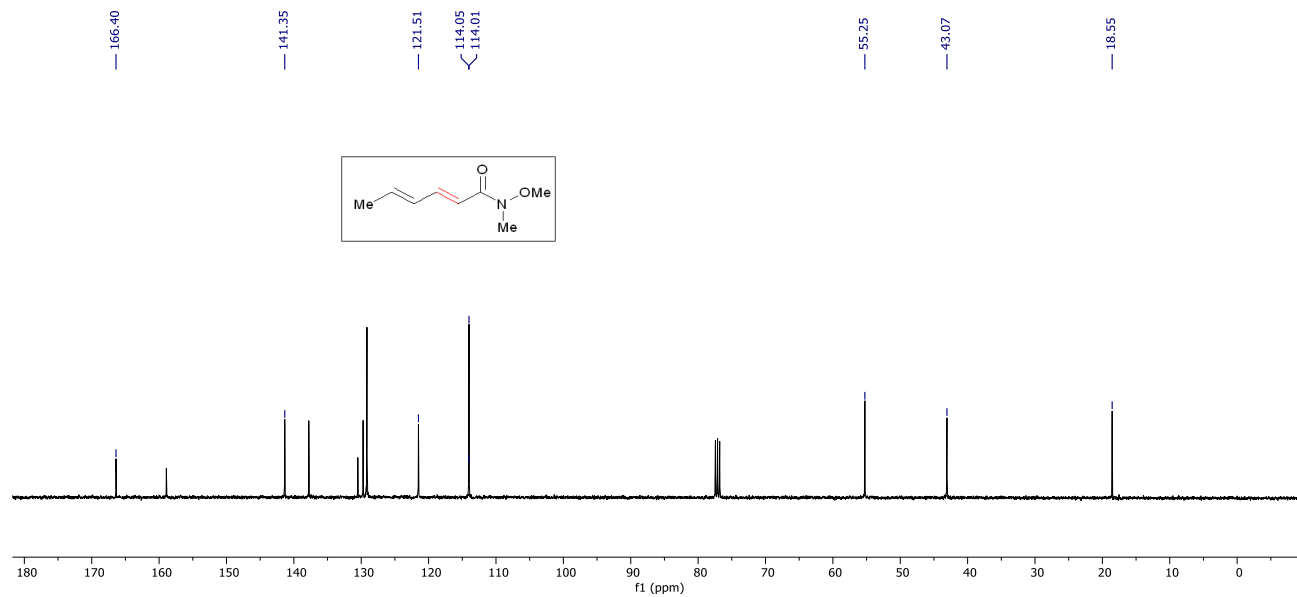
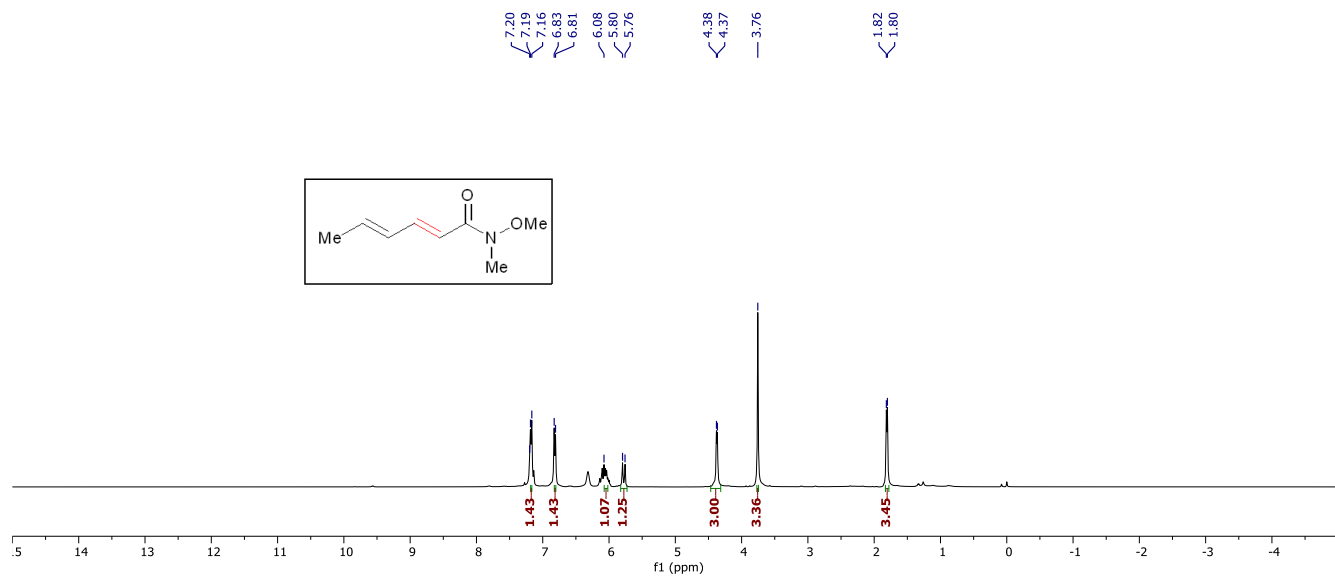
^1H and ^{13}C spectra of compound 2l:



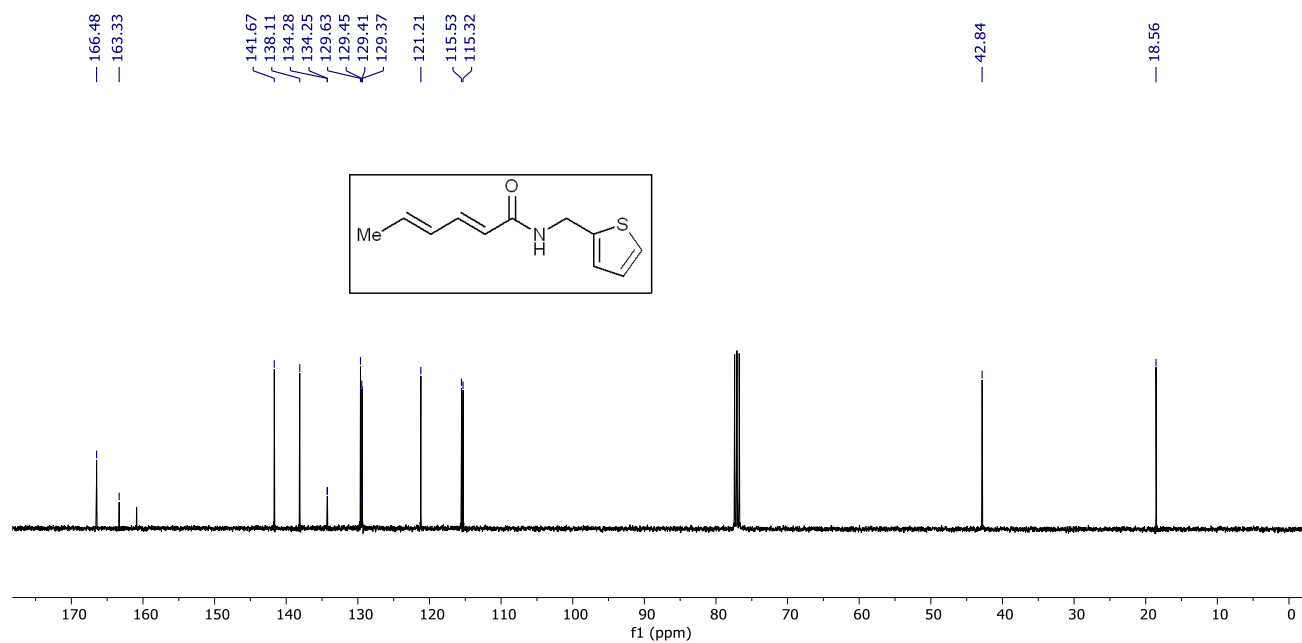
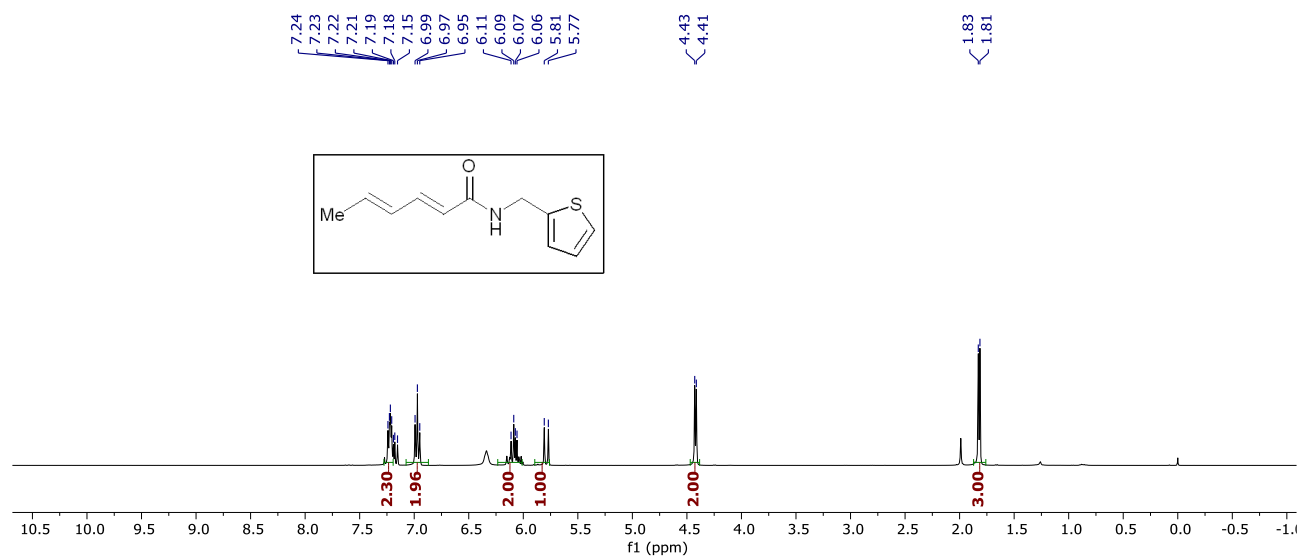
¹H and ¹³C spectra of compound 2m:



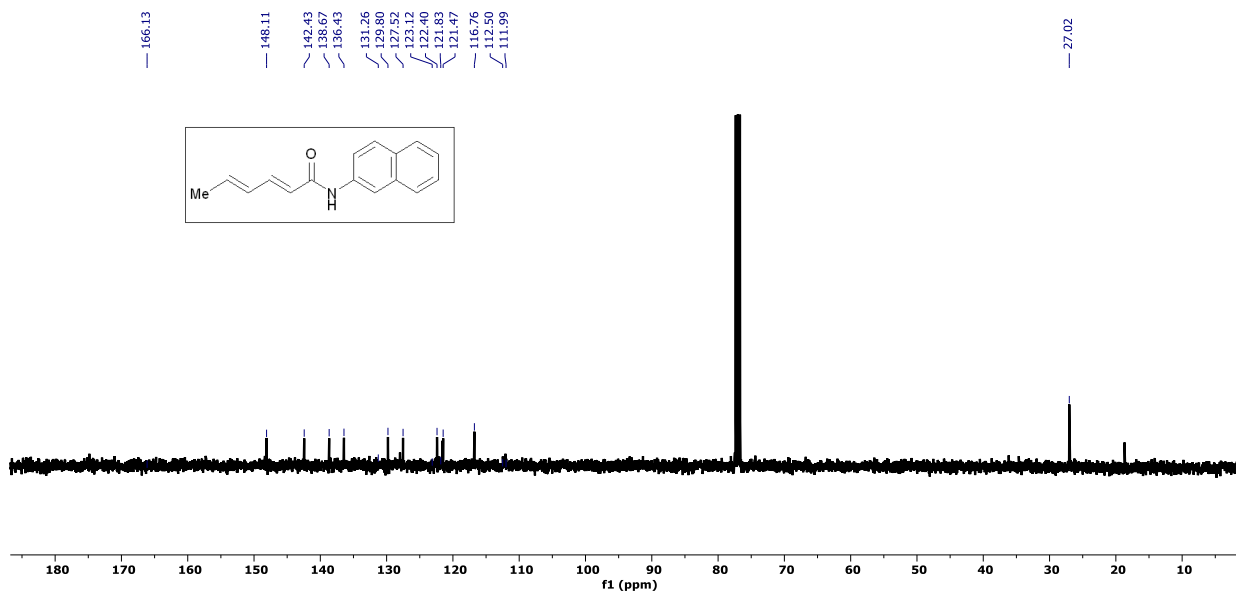
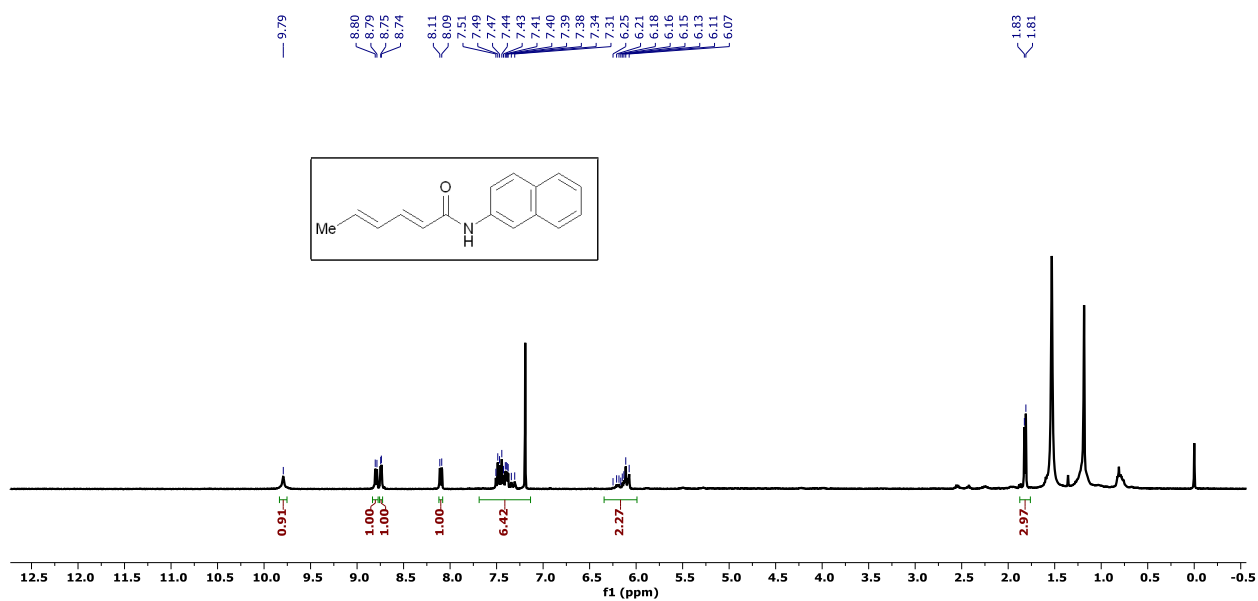
^1H and ^{13}C spectra of compound 2n:



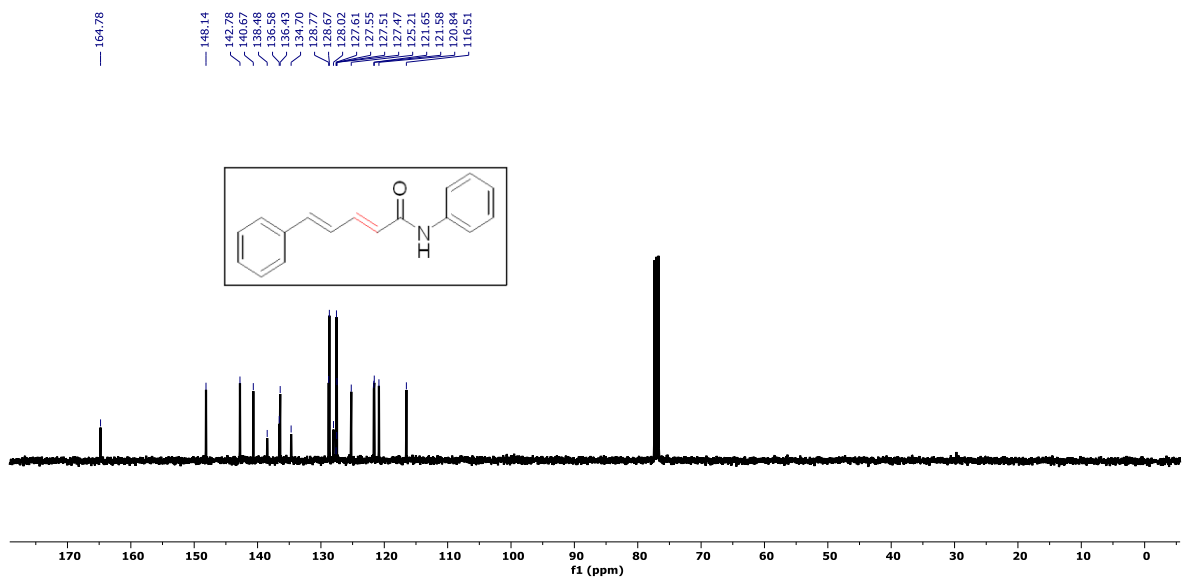
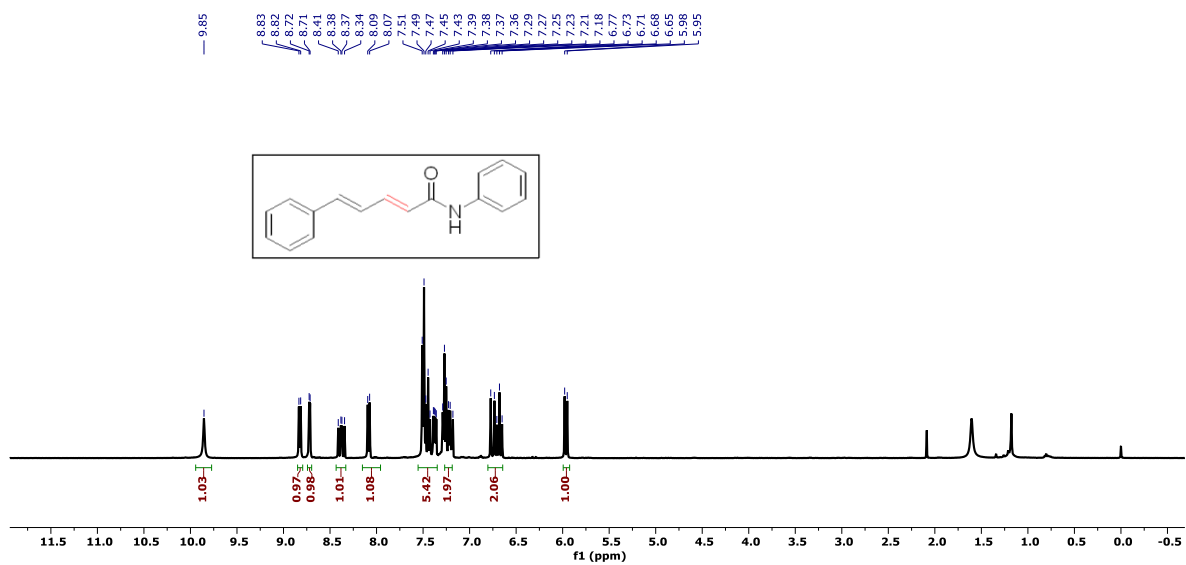
^1H and ^{13}C spectra of compound 2o:



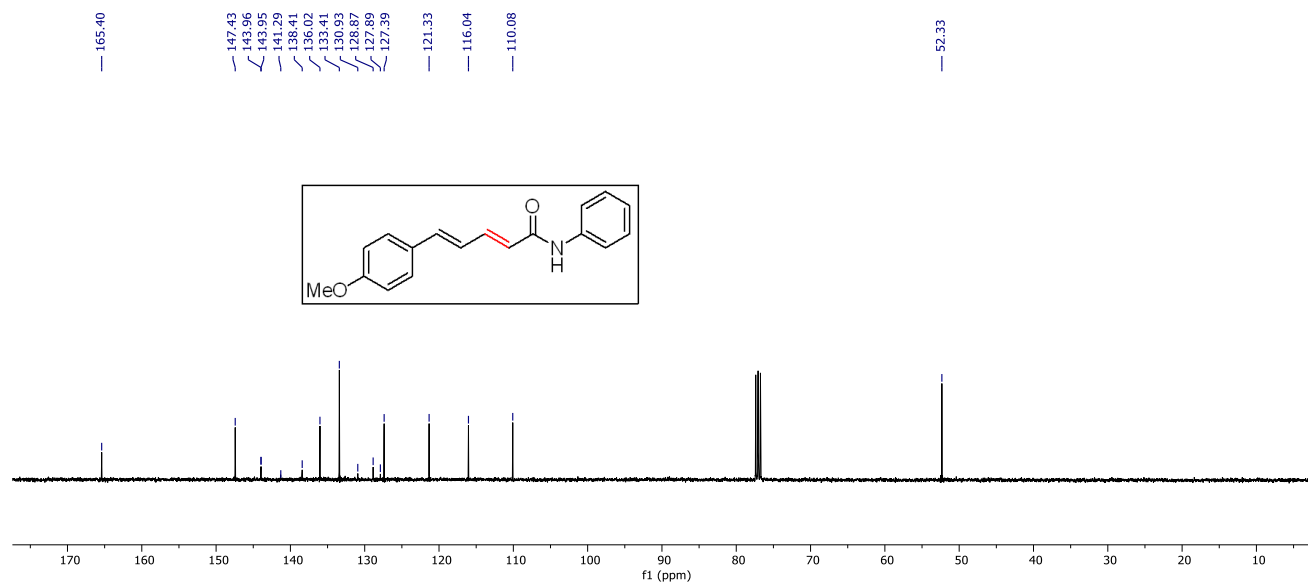
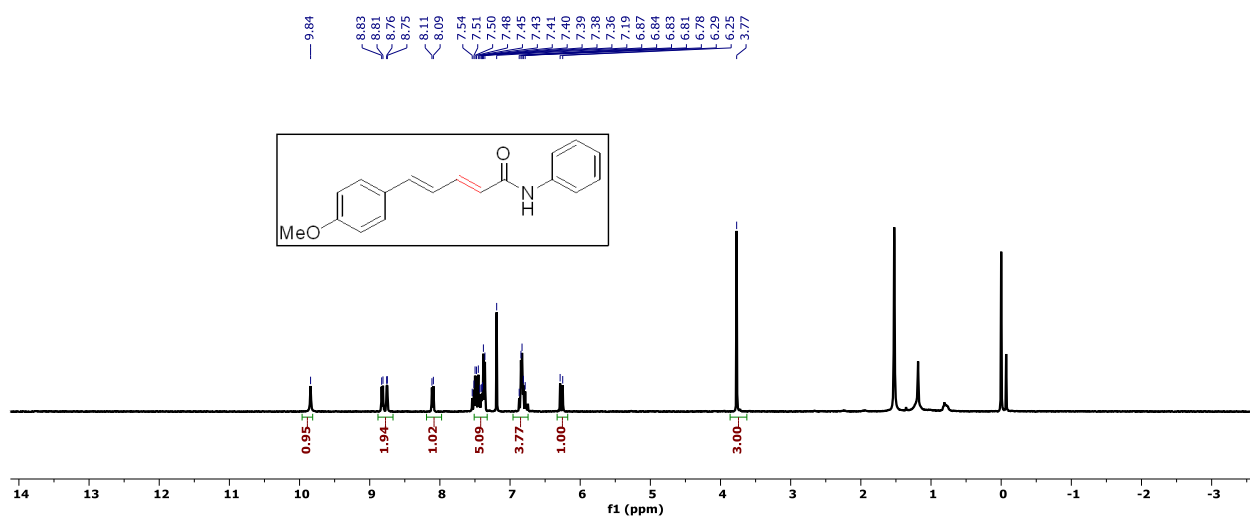
^1H and ^{13}C spectra of compound 2p:



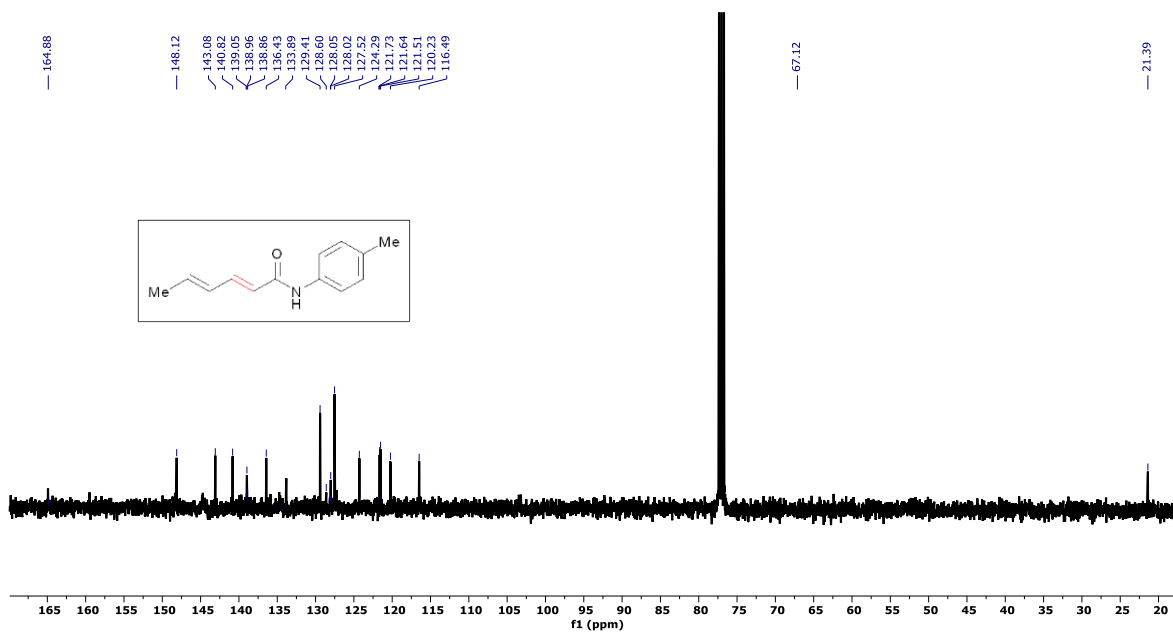
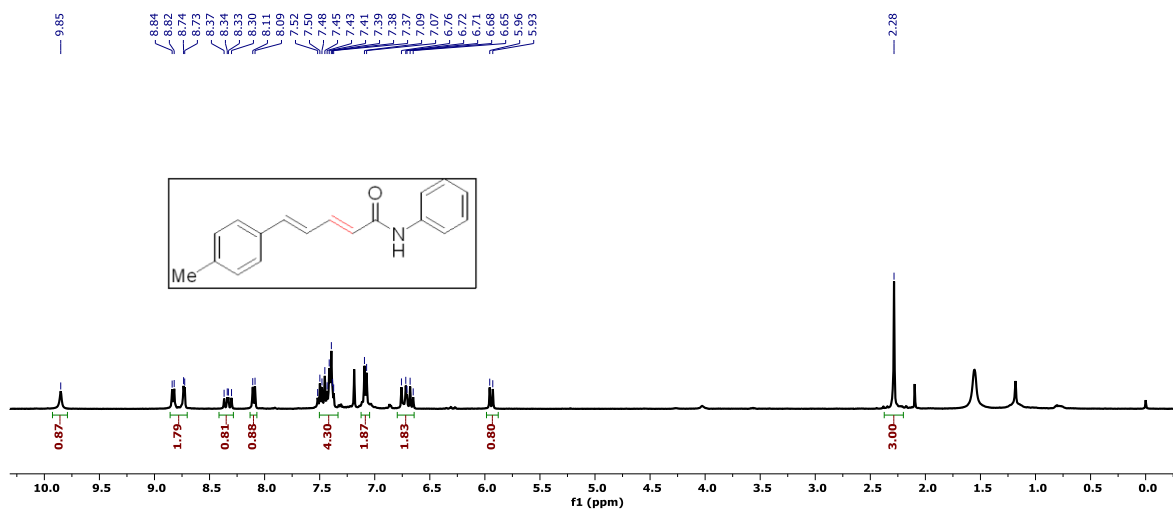
^1H and ^{13}C spectra of compound 4a:



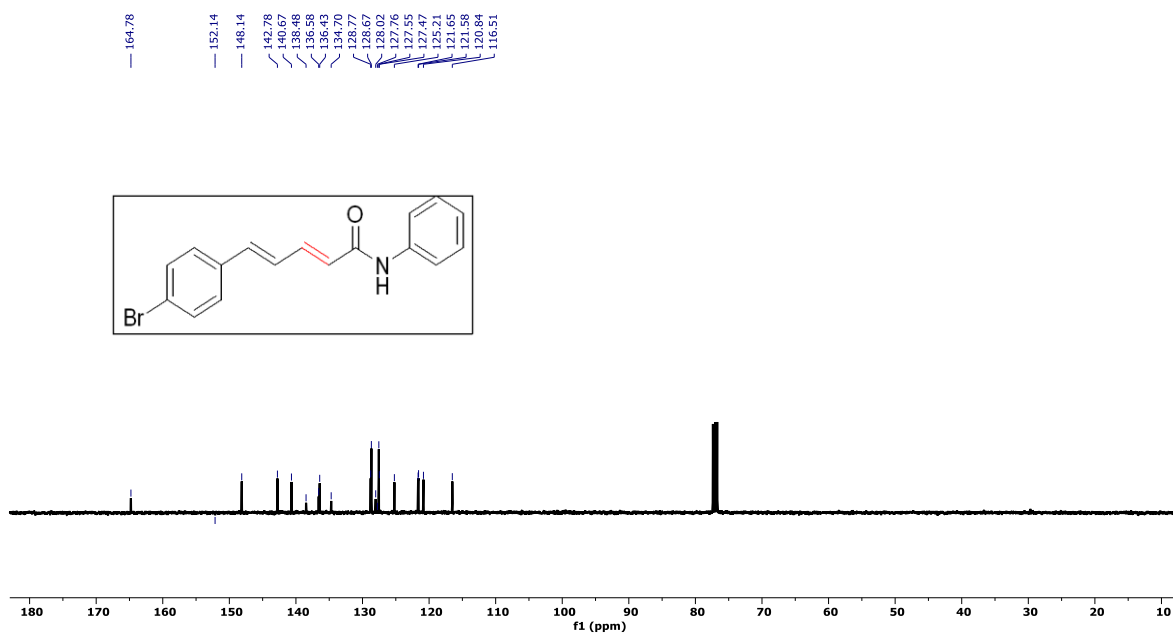
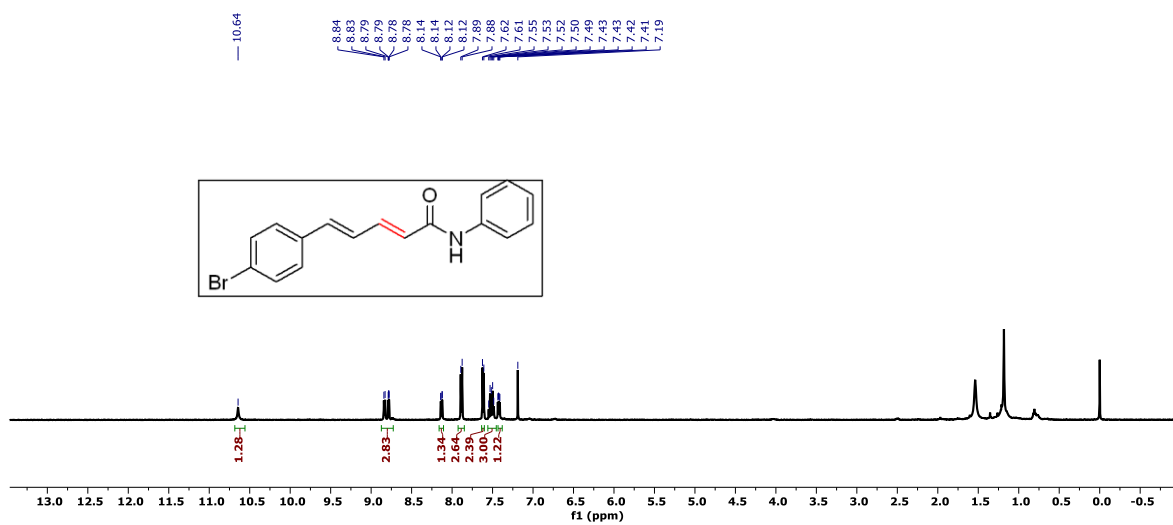
^1H and ^{13}C spectra of compound 4b:



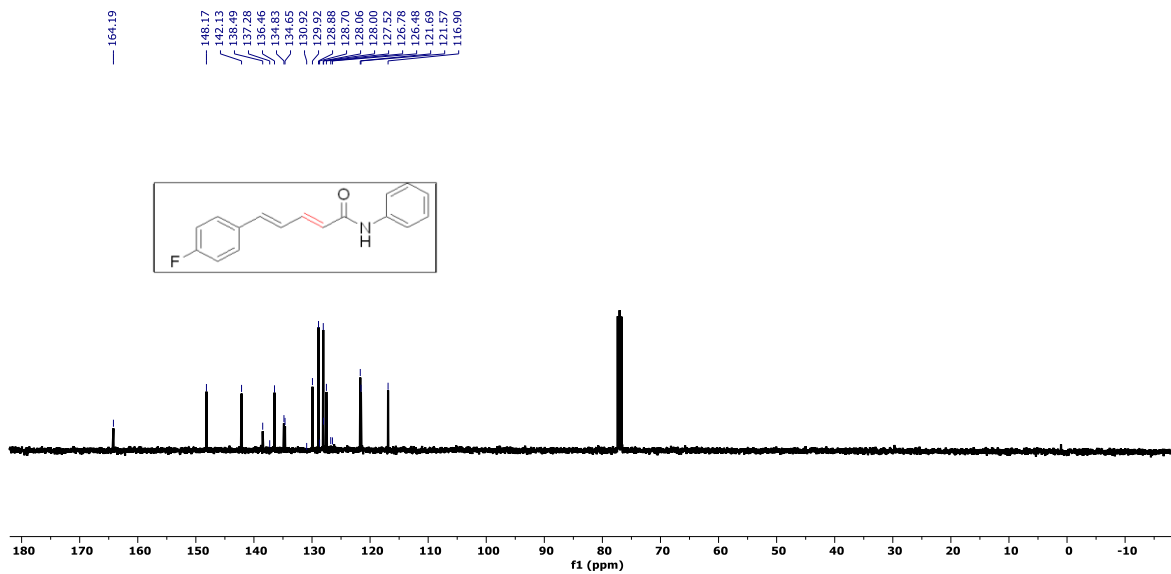
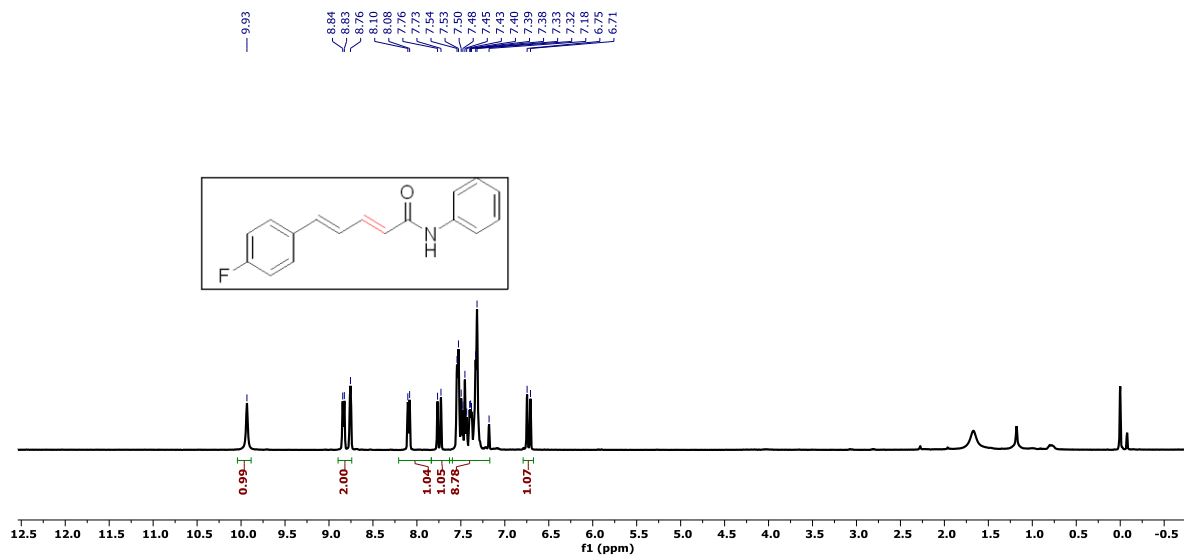
^1H and ^{13}C spectra of compound 4c:



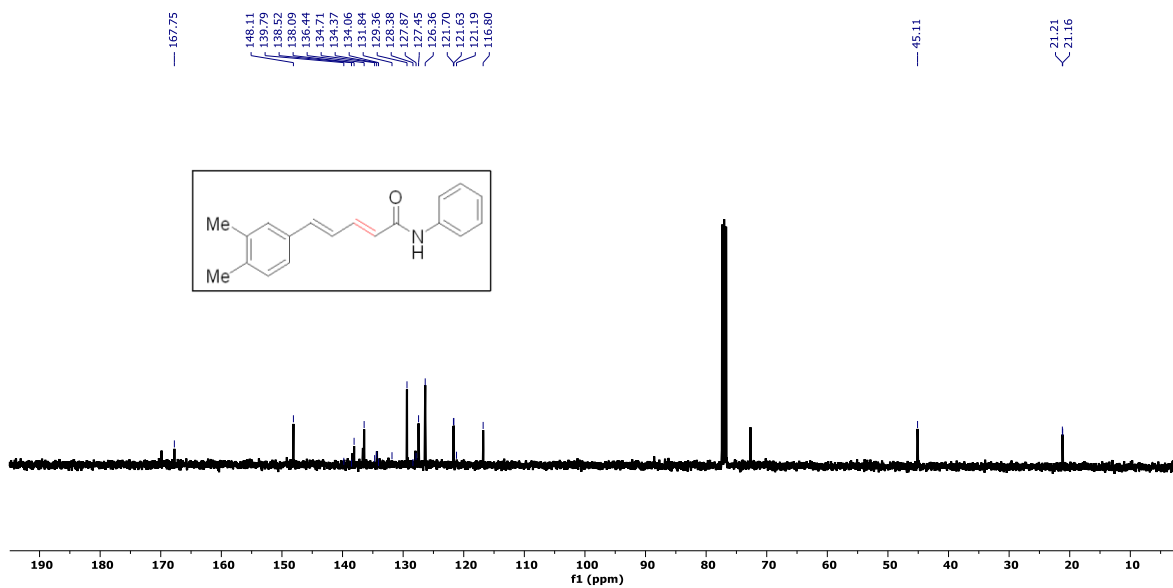
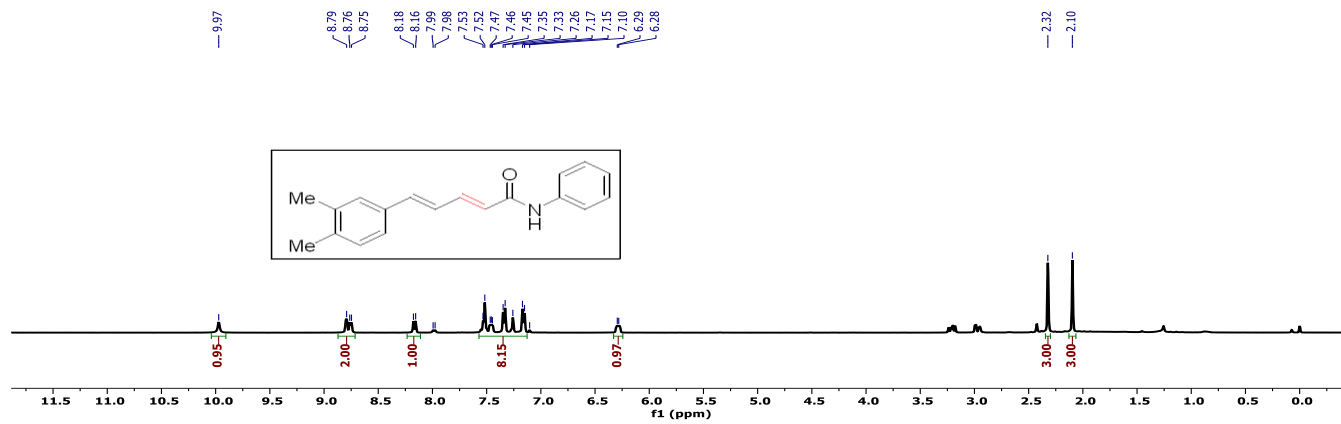
^1H and ^{13}C spectra of compound 4d:



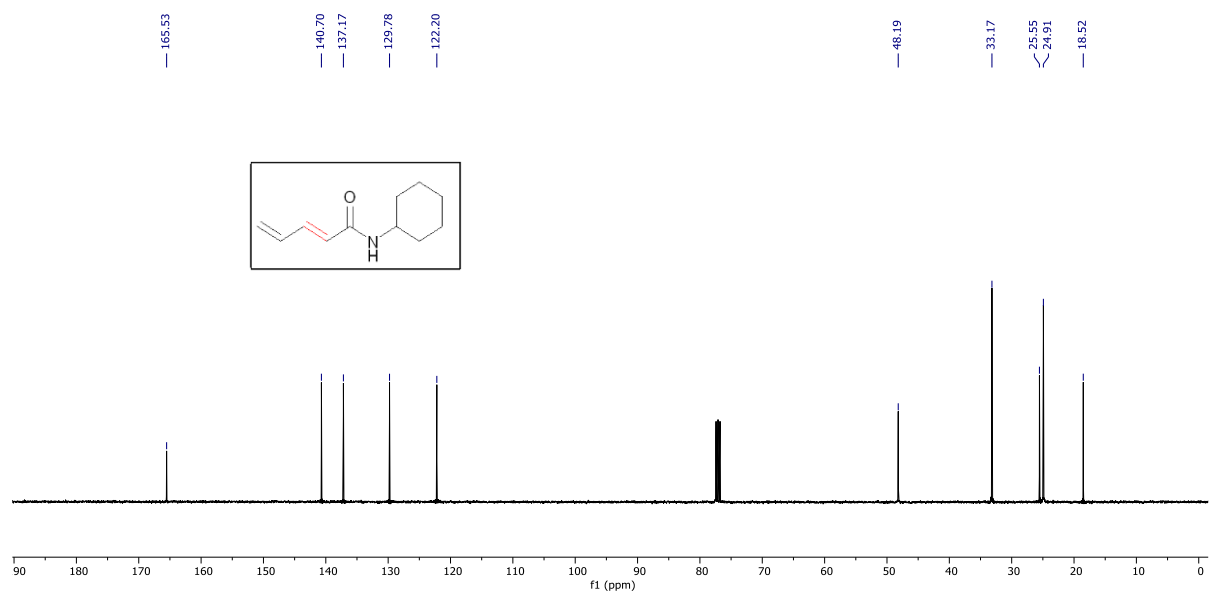
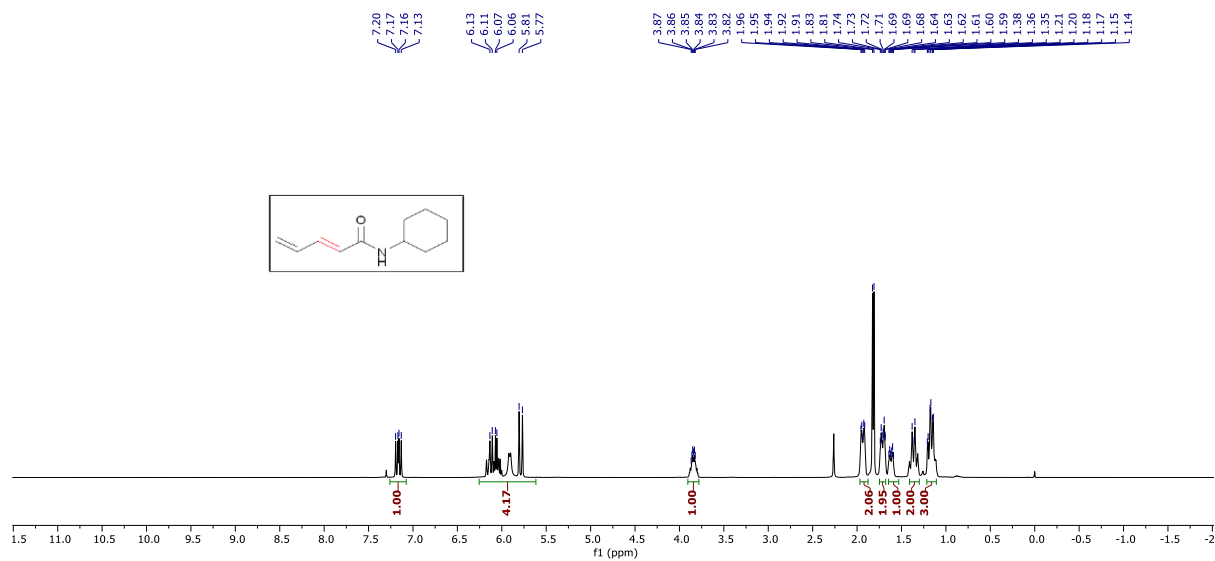
^1H and ^{13}C spectra of compound 4e:



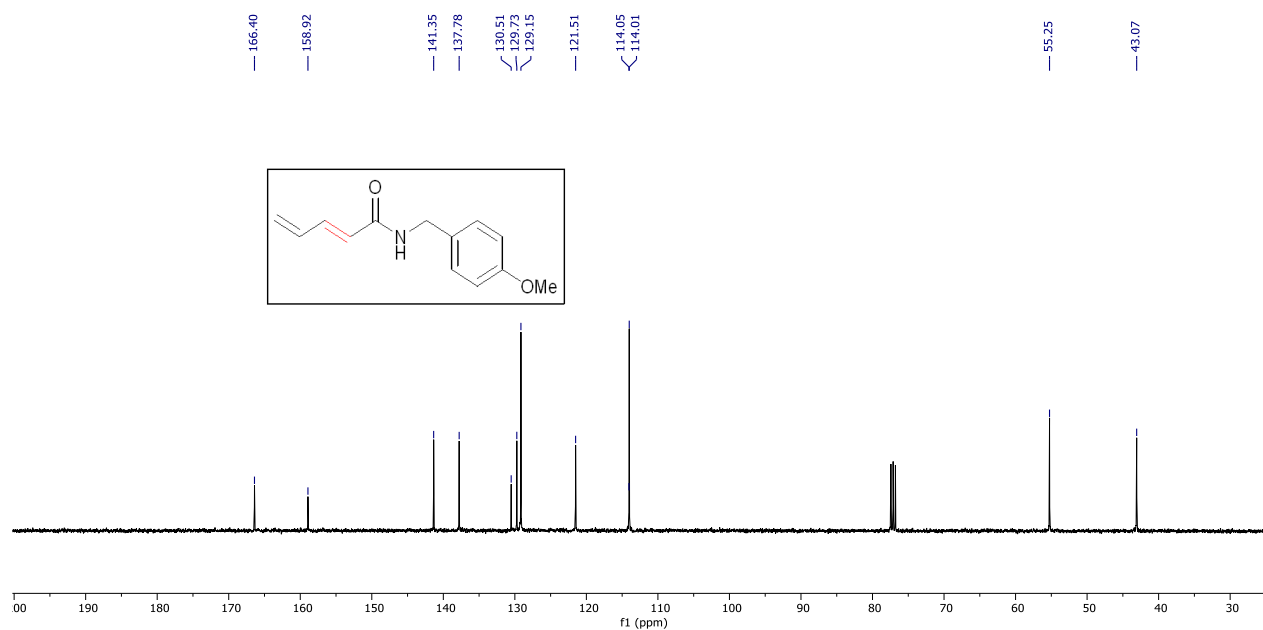
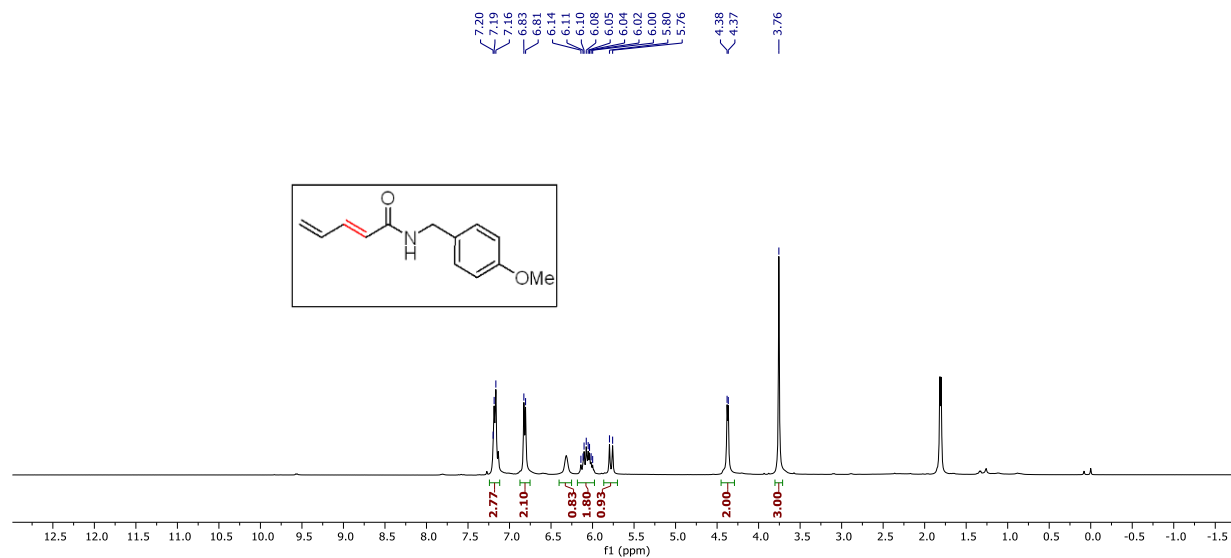
^1H and ^{13}C spectra of compound 4f:



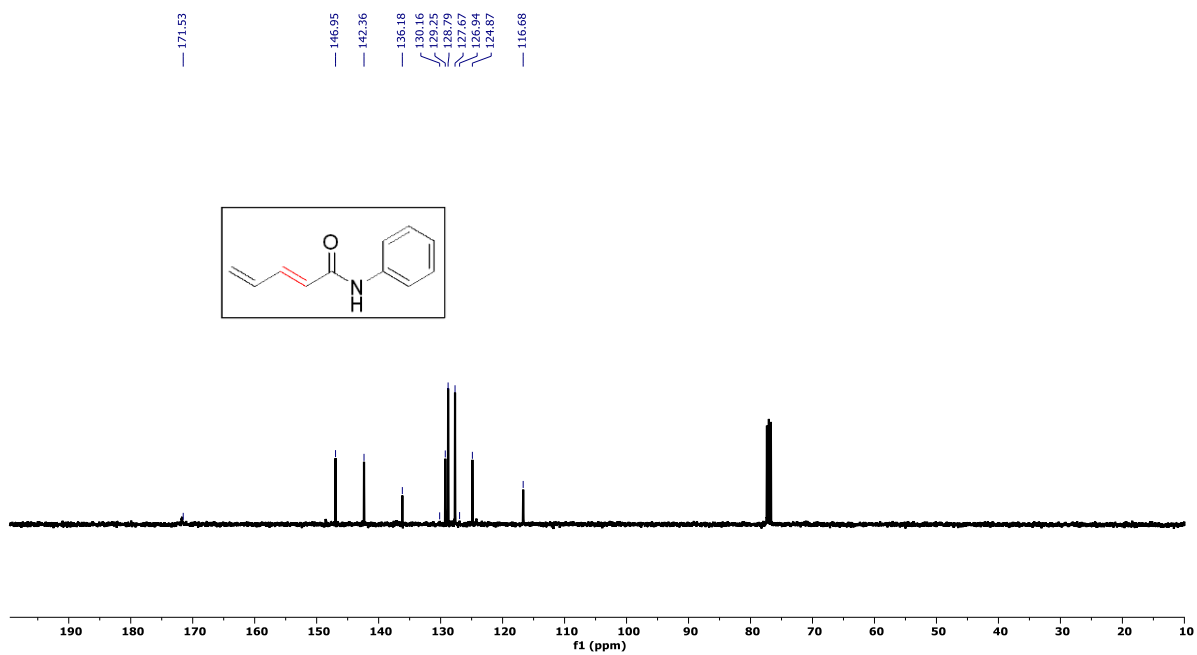
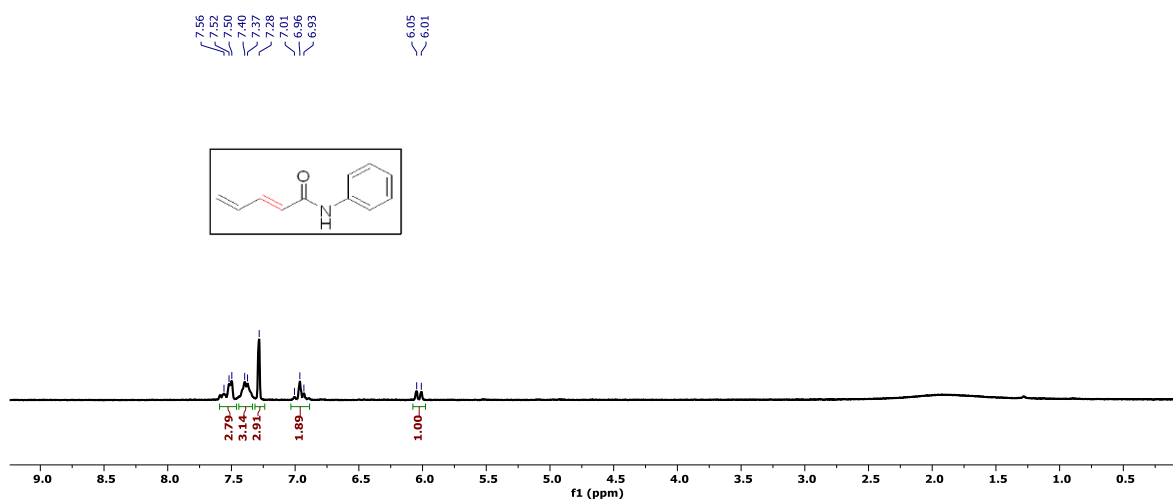
^1H and ^{13}C spectra of compound 6a:



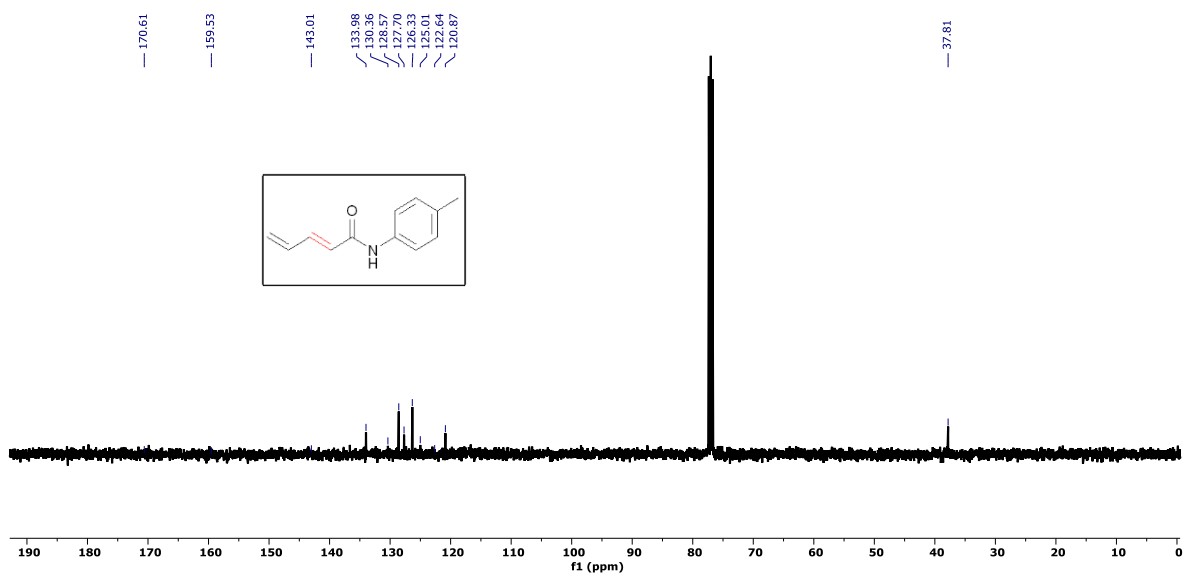
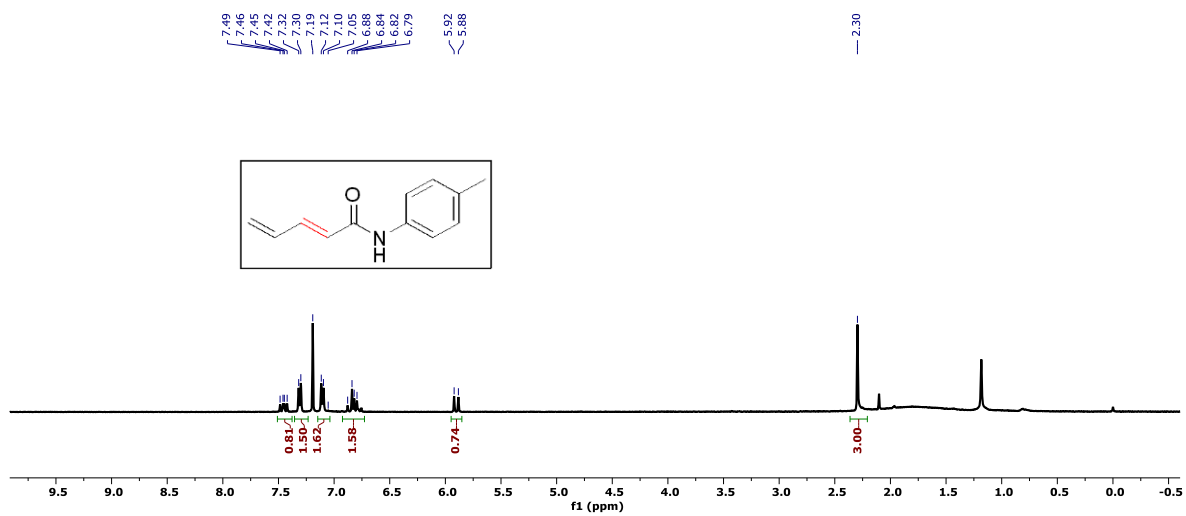
^1H and ^{13}C spectra of compound 6b:



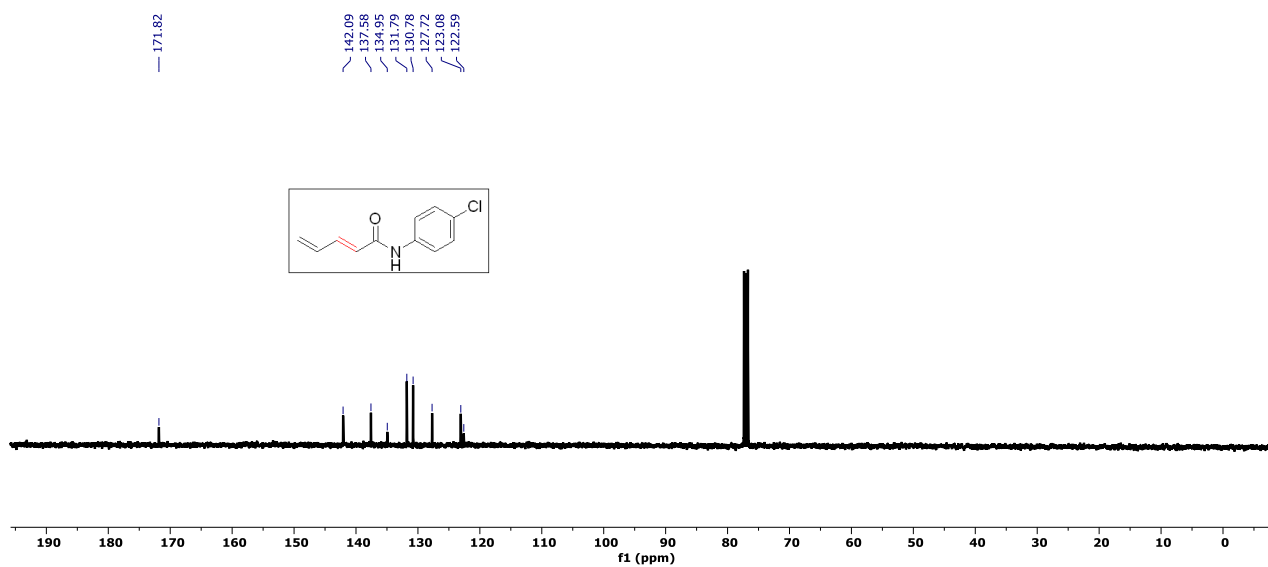
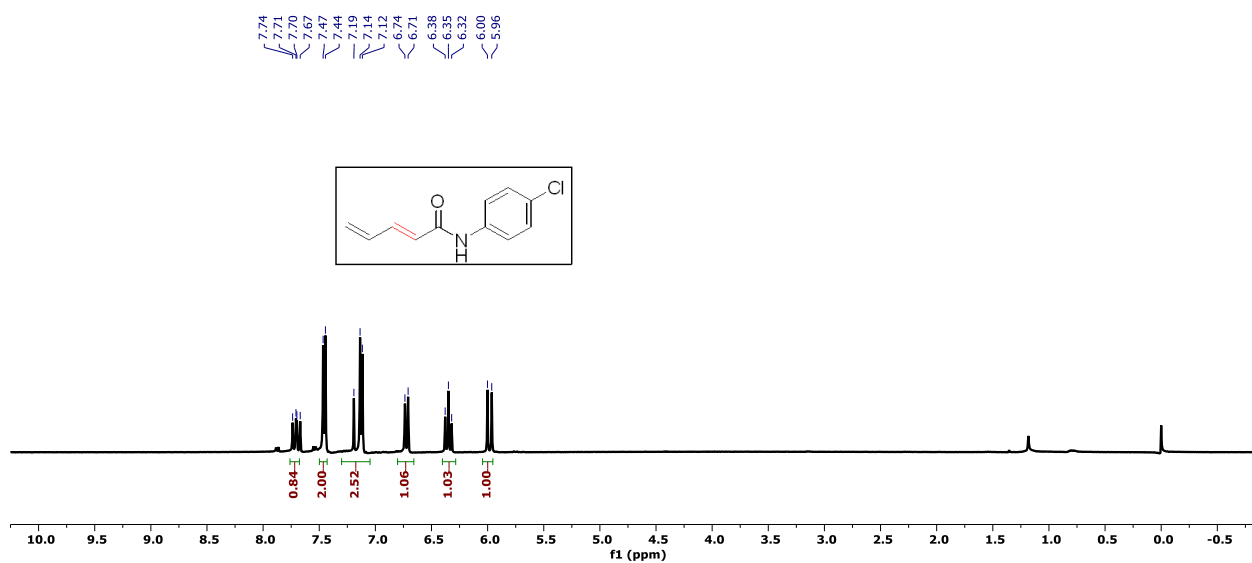
^1H and ^{13}C spectra of compound 6c:



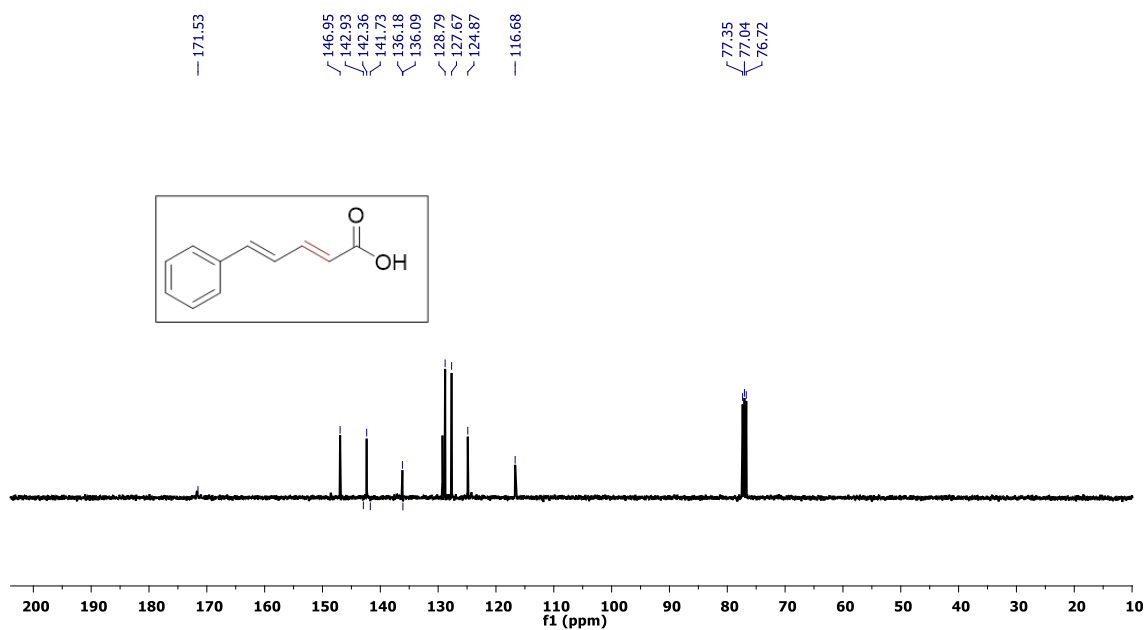
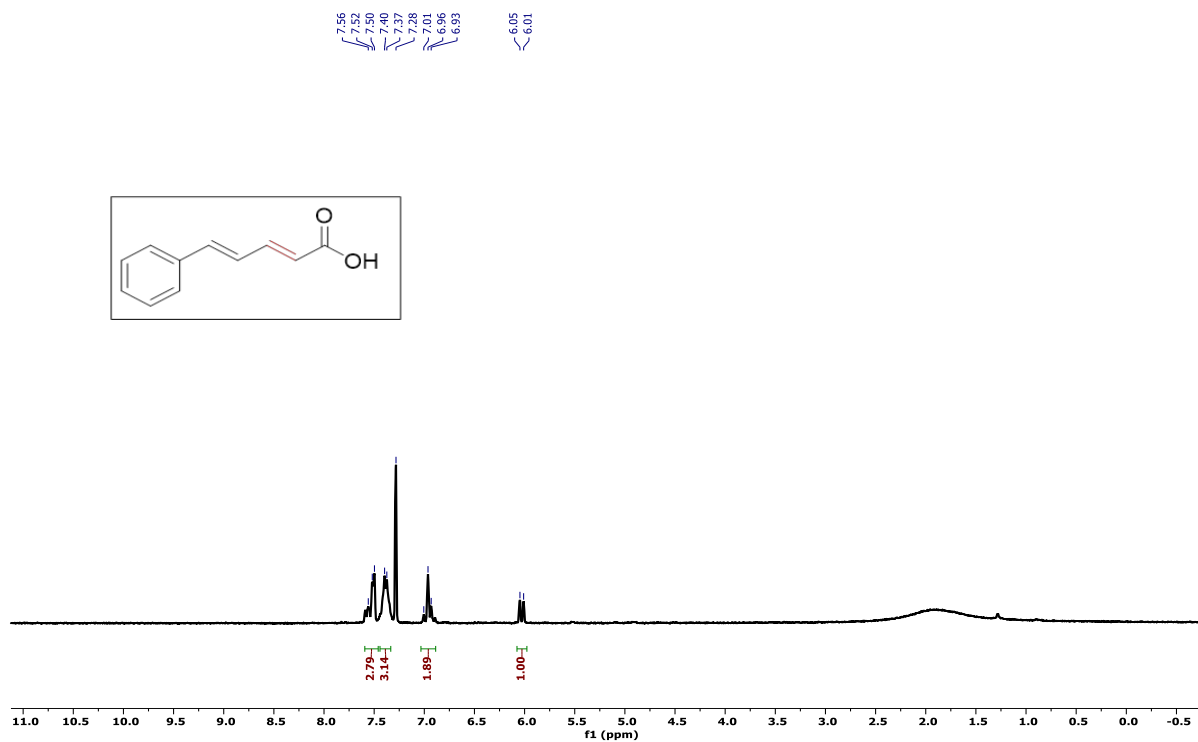
^1H and ^{13}C spectra of compound 6d:



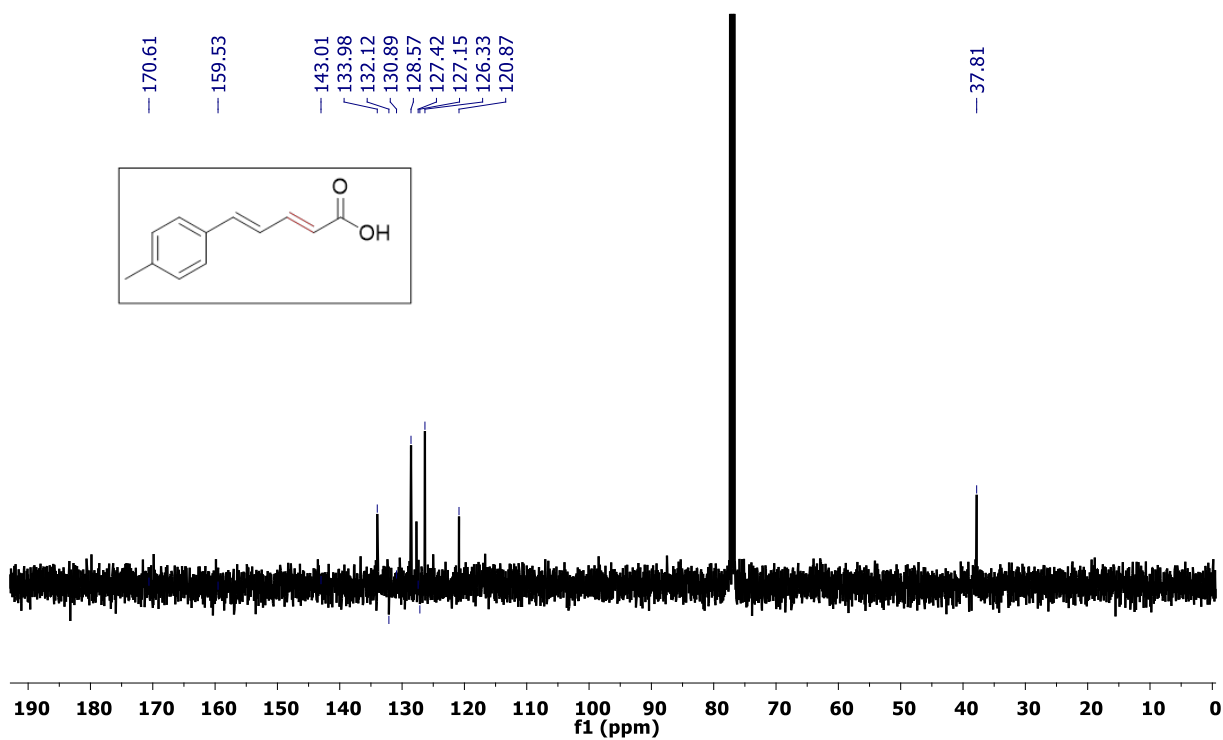
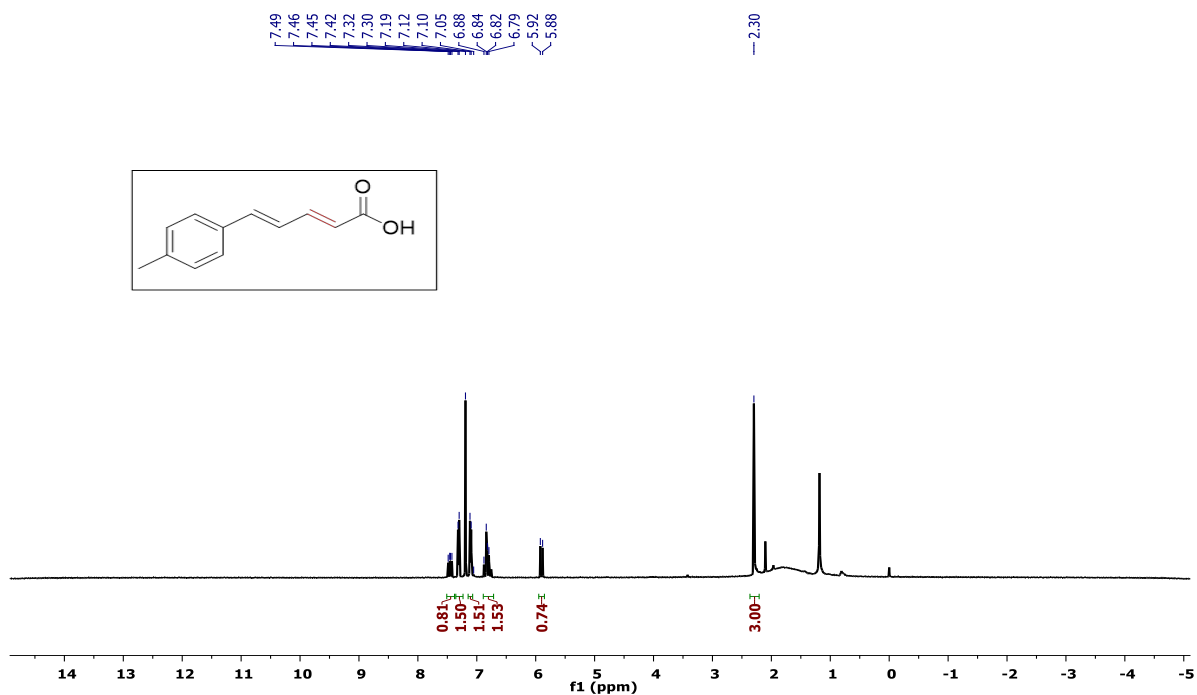
^1H and ^{13}C spectra of compound 6c:



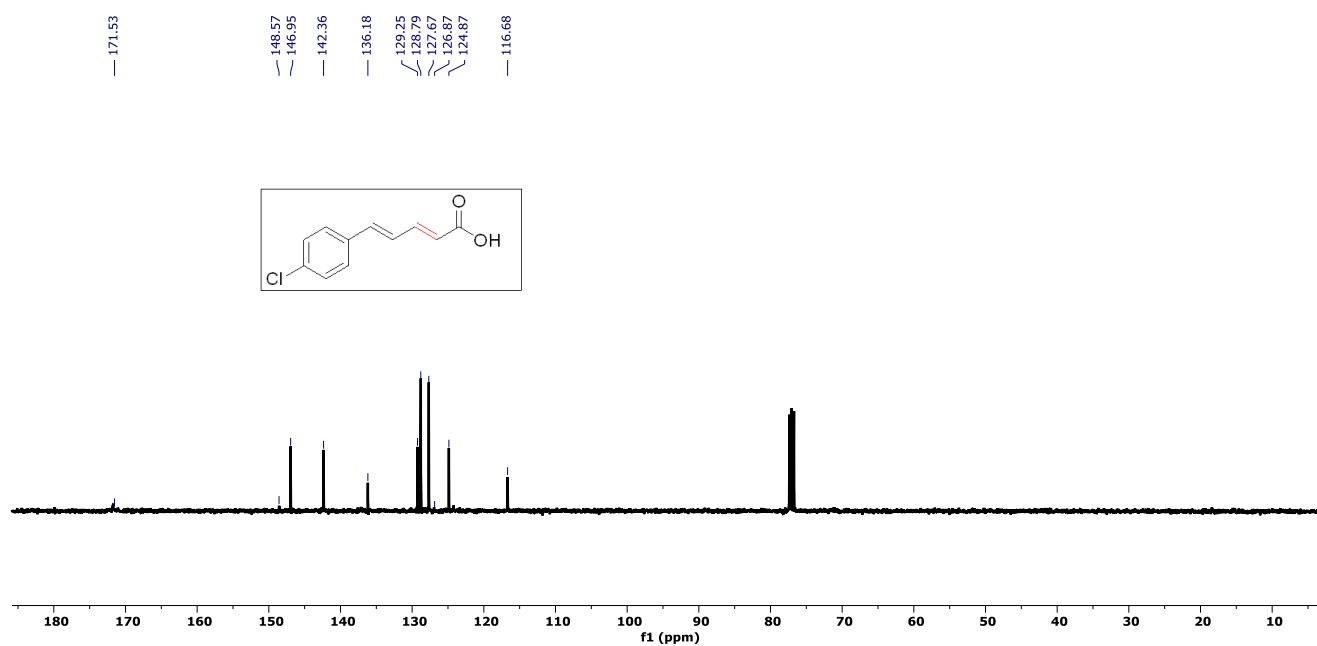
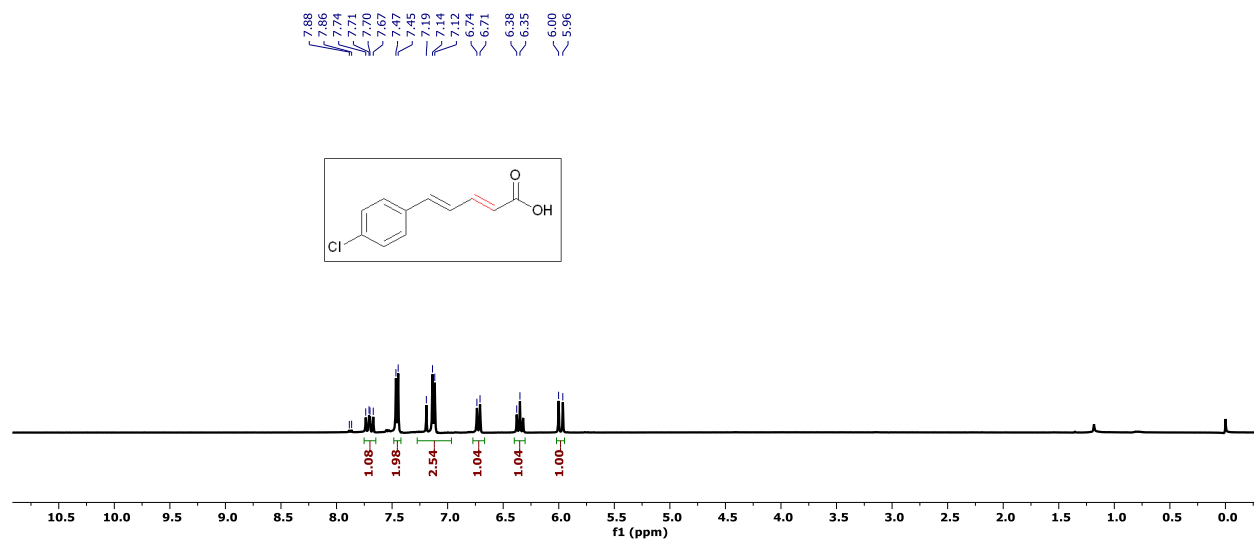
^1H and ^{13}C spectra of compound 8a:



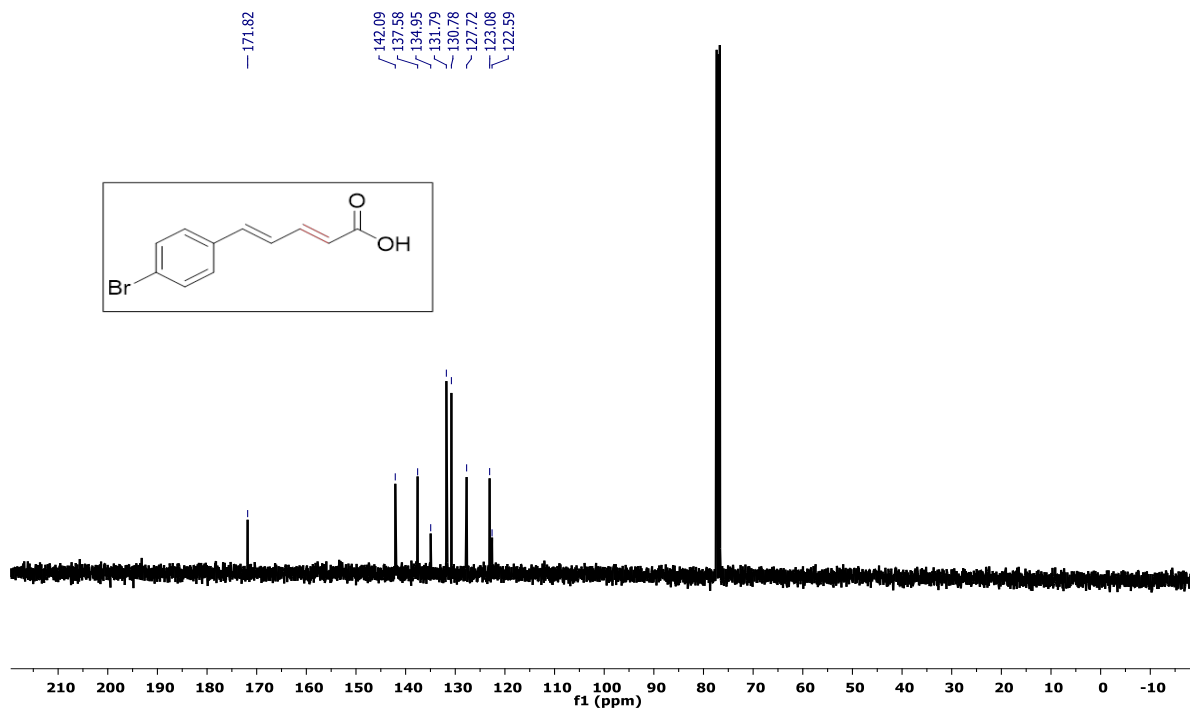
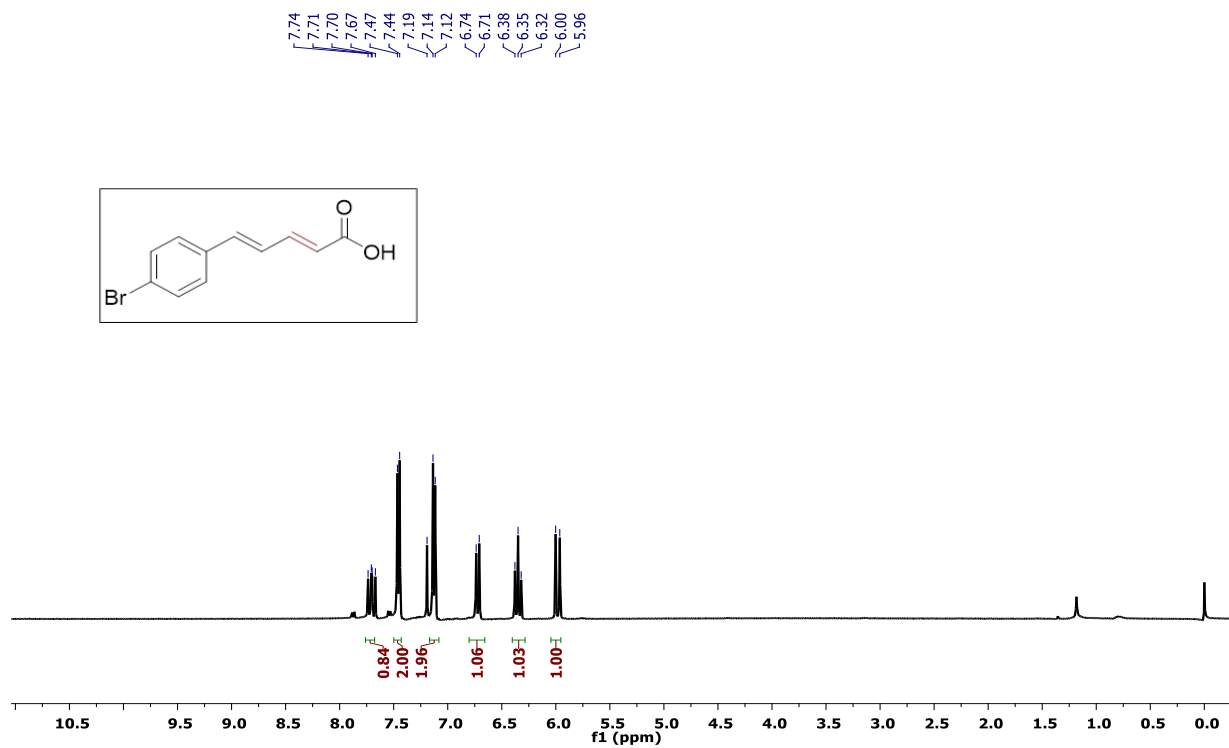
^1H and ^{13}C spectra of compound 8b:



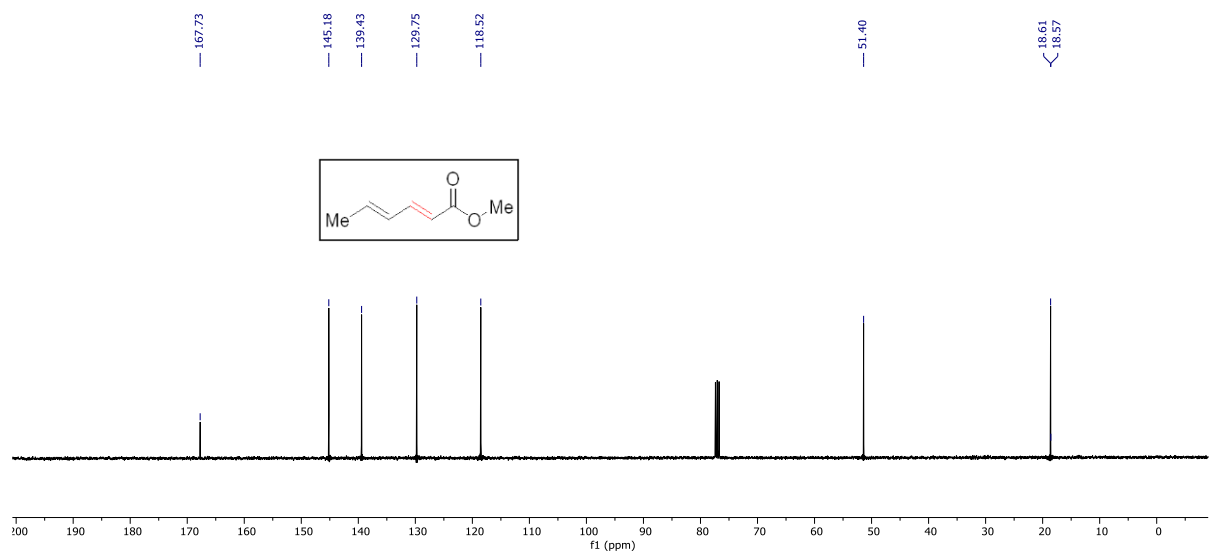
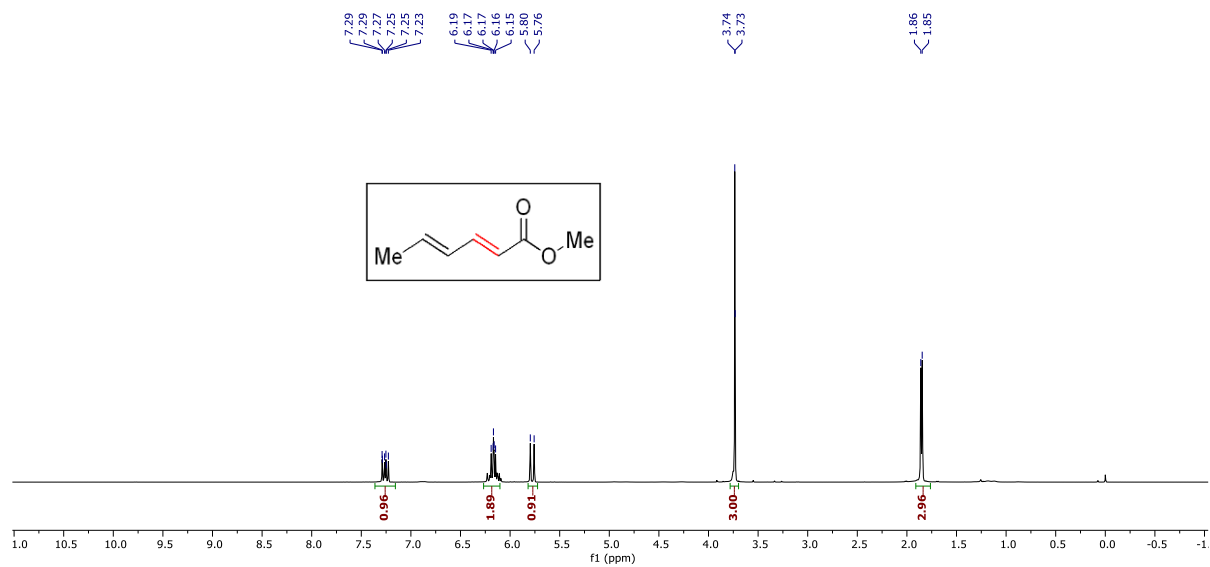
^1H and ^{13}C spectra of compound 8c:



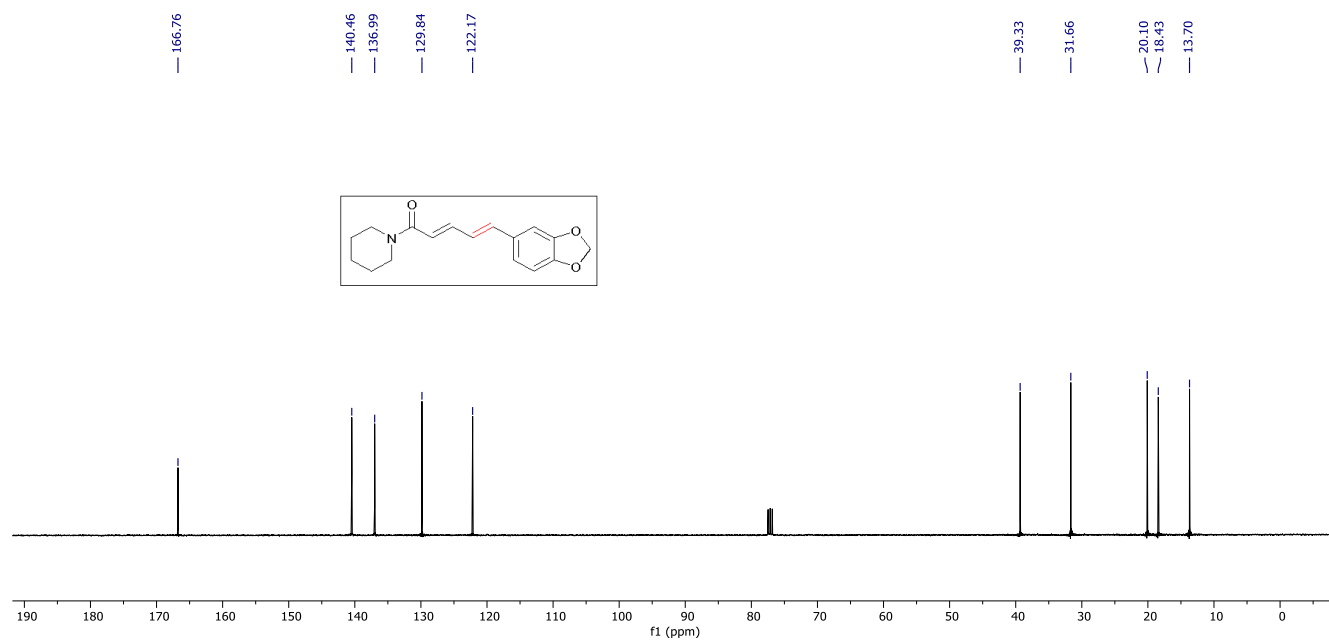
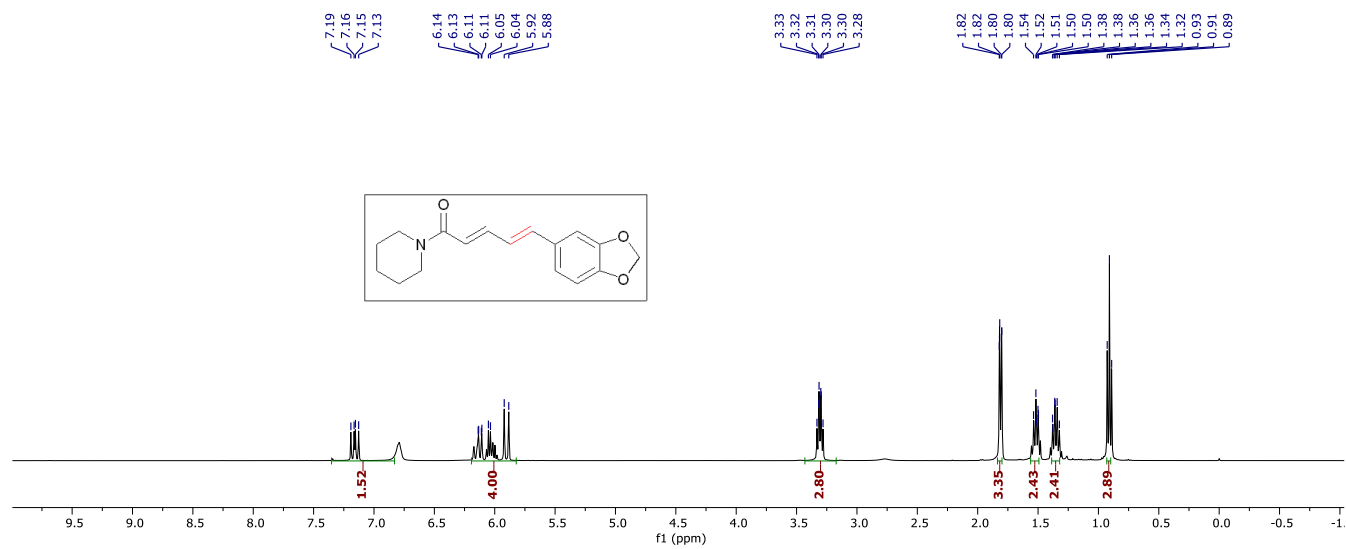
^1H and ^{13}C spectra of compound 8d:



^1H and ^{13}C spectra of compound 8e:



^1H and ^{13}C spectra of compound 10:



^1H and ^{13}C spectra of compound 12:

