Decatungstate-Photocatalyzed Transfer Hydrogenation of Alkenes Using Alcohol as the Hydrogen Source

Teng Zhang[1], Zheng-Feng Zhang[2], Xuan Lan[3], Si-Yi Li[3], Ming-Der Su[2], Yonggui Robin Chi[1], Lixin Wu[4] and Cheuk-Wai So[1]

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[1] School of Chemistry, Chemical Engineering and Biotechnology, Nanyang Technological University, 637371, Singapore.

[2] Department of Applied Chemistry, National Chiayi University, Chiayi 60004, Taiwan.

[3] College of Chemistry and Materials Science, Zhejiang Normal University, Jinhua, 321004, PR China.

[4]State Key Laboratory of Supramolecular Structure and Materials and College of Chemistry, Jilin University, Changchun 130012, P. R. China.

Correspondence to: midesu@mail.ncyu.edu.tw; robinchi@ntu.edu.sg; CWSo@ntu.edu.sg

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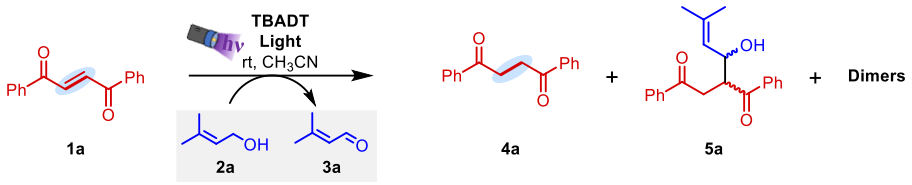
1. General Information

Reagents and solvents were bought from Sigma Aldrich, TCI, KANTO and BLD pharm (Shanghai, China), which used as received without any further purification. Catalyst TBADT was synthesized according to the literature procedure.[1] The olefins and alkynes were bought from Sigma Aldrich, TCI, and BLD pharm (Shanghai, China). All the products were purified by column chromatography, using 230 - 400 mesh silica gel. TLC plates (Merck silica gel 60 F254) were used for thin-layer chromatography (TLC) analysis of reaction mixtures, visualized by UV light (254 nm and 365 nm). Photochemical reactions were placed in a 10 mL Schlenk tube equipped with a magnetic stir bar, irradiated with one Kessil LED light (40 W, PR160-370 nm) from 3 cm away. High-resolution mass spectra (HRMS) were recorded on a JEOC AccuTOF LC-plus 4G mass spectrometer using ESI (electrospray ionization). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance III 600 spectrometer equipped with a BBFO probehead (^{1}H: 600 MHz, ^{13}C: 150 MHz), Bruker DPX 400 MHz spectrometer (^{1}H: 400 MHz, ^{13}C: 100 MHz), JEOL JNM-ECZL500 (^{1}H: 500 MHz, ^{13}C: 125 MHz) and JEOL JNM-ECA400 (^{1}H: 400 MHz, ^{13}C: 100 MHz). Chemical shifts are reported relative to $SiMe_4$ for ^{1}H ($CDCl_3$: δ = 7.26 ppm, CD_3CN: 1.94 ppm, Acetone-d_6: 2.05 ppm, DMSO-d_6: 2.50 ppm, $SiMe_4$: δ = 0.00 ppm) and ^{13}C ($CDCl_3$: δ = 77.16 ppm, CD_3CN: 118.26 ppm, Acetone-d_6: 29.84 ppm, DMSO-d_6: 39.52 ppm, $SiMe_4$: δ = 0.00 ppm). NMR multiplicities are abbreviated, where s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, h = hextet and m = multiplet.

2. Optimization of Reaction Conditions

2.1 Irradiation Wavelength Optimization



In a glovebox, 1,2-dibenzoylethylene (0.1 mmol), TBADT (5 mol%), prenol (0.5 mmol, 5 equiv.), and anhydrous acetonitrile (2 mL) were placed in a 10 mL Schlenk tube equipped with a magnetic stir bar. The reaction mixture was then irradiated with an LED light (Kessil 40 W) for 6 hours. The yield of the target product was determined based on ^{1}H NMR analysis, using 1,3,5-trimethoxybenzene as the internal standard.

We used the yields of **4a** in control experiments to optimize the irradiation wavelength of the LED light. The experimental results showed no reaction in the absence of light (Entry 1). With 370 and 400 nm LED lamps, lower yields were observed (Entry 2 and 4). Using 427 nm LED lamp, moderate yield was obtained (Entry 5). Based on the above results, 390 nm (Entry 3) was chosen as the optimal irradiation wavelength.

Table S1. LED light irradiation wavelength optimization.

Entry	Light Source (40 W)	Catalyst (mol%)	Reaction Time (h)	*Yield of **4a**
1	No	5	6	n.d.
2	370 nm LED	5	6	84%
3	390 nm LED	5	6	89%
4	400 nm LED	5	6	78%
5	427 nm LED	5	6	57%

*Yield of the target product was determined based on ^{1}H NMR analysis, using 1,3,5-trimethoxybenzene as the internal standard. n.d.: not detected.

2.2 Irradiation Time Optimization

The irradiation time was optimized by a kinetic experiment. In Figure S1, the kinetic plot showed a plateau at six hours. In addition, no product of **4a** was observed without catalyst.

Table S2. LED light irradiation time optimization.

Entry	Light Source (40 W)	Catalyst (mol%)	Reaction Time (min)	*Yield of **4a**
1	390 nm LED	5	30	24%
2	390 nm LED	5	60	46%
3	390 nm LED	5	100	56%
4	390 nm LED	5	156	68%
5	390 nm LED	5	221	83%
6	390 nm LED	5	365	89%
7	390 nm LED	5	483	86%
8	390 nm LED	No	360	n.d.

*Yield of the target product was determined based on ^{1}H NMR analysis, using 1,3,5-trimethoxybenzene as the internal standard. n.d.: not detected.

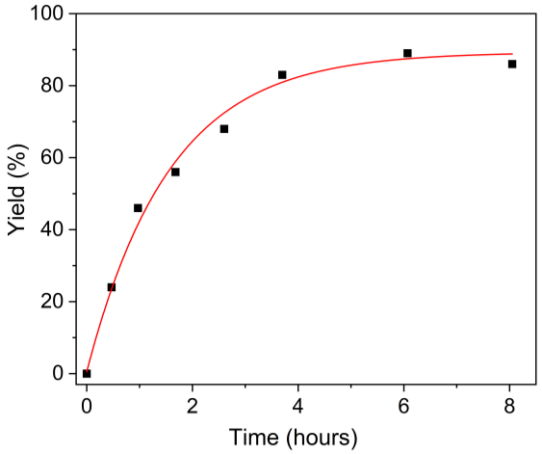
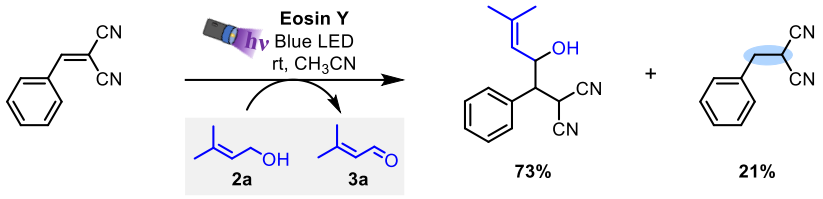


Figure S1. Time profile for the yield of **4a**.

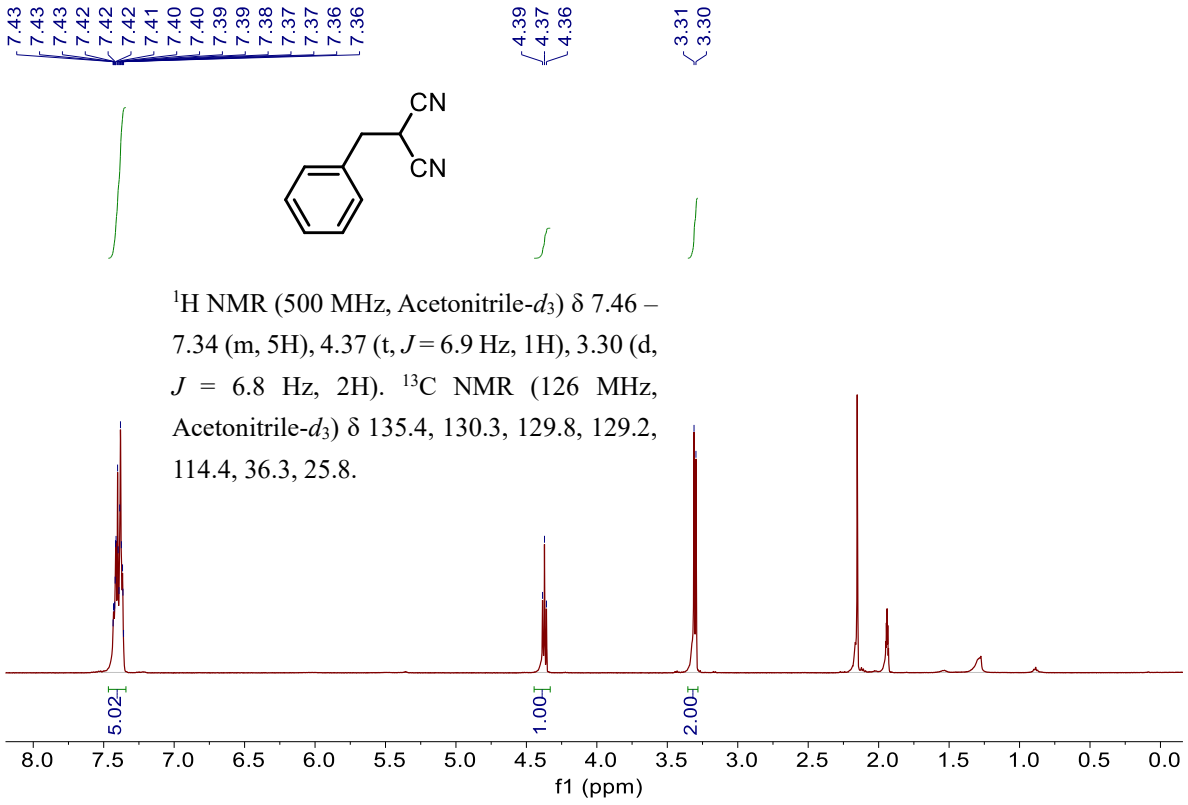
Notably, limited solubility of the substrate was observed with acetonitrile/water as solvent. Furthermore, employing two or five equivalents of alcohol provided comparable yields of **4a**, however, the reaction with two equivalents of alcohol required a longer reaction time. Additionally, no **4a** was detected in the absence of light or photocatalyst, indicating both components are required. Finally, the kinetic profile detailed in Scheme S1 confirmed that the photocatalytic reaction can be completed within 6 hours under the optimized conditions. Given the optimized reaction conditions, hydrogenated product **4a** was afforded an excellent yield (89%) in the 5 mol% TBADT-catalyzed reaction of 1,2-dibenzoylethylene (**1a**) with five equivalents of prenol in acetonitrile under 390 nm LED lamp irradiation.

3. Eosin Y Catalyzed Transfer Hydrogenation of 2-Benzylidenemalononitrile



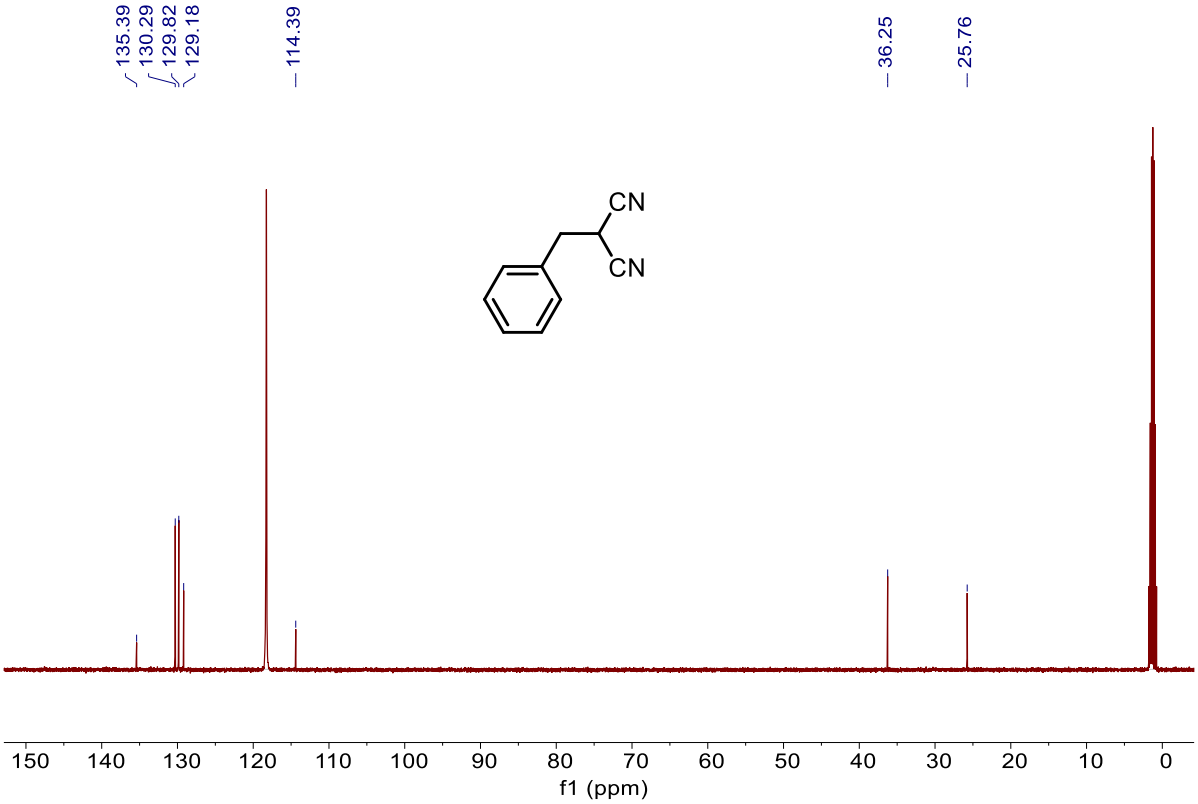
Scheme S1. EosinY photocatalyzed hydrogenation of 2-benzylidenemalononitrile.

In a glovebox, 2-benzylidenemalononitrile (0.2 mmol), EosinY (10 mol%), prenol (1.0 mmol, 5 equiv.), and anhydrous acetonitrile (5 mL) were placed in a 10 mL Schlenk tube equipped with a magnetic stir bar. The reaction mixture was then irradiated with a 456 nm LED light (Kessil 40 W) at 60 °C for 12 hours. Then, the solvent was removed under reduced pressure using a rotary evaporator, and the resulting residue was purified by silica gel column chromatography (hexane/ethyl acetate 5:1) to obtain the product as a colorless solid (6.4 mg, 21%).



^{1}H NMR (500 MHz, Acetonitrile-d_3) δ 7.46 – 7.34 (m, 5H), 4.37 (t, J = 6.9 Hz, 1H), 3.30 (d, J = 6.8 Hz, 2H). ^{13}C NMR (126 MHz, Acetonitrile-d_3) δ 135.4, 130.3, 129.8, 129.2, 114.4, 36.3, 25.8.

^{1}H NMR (500 MHz, CD_3CN)

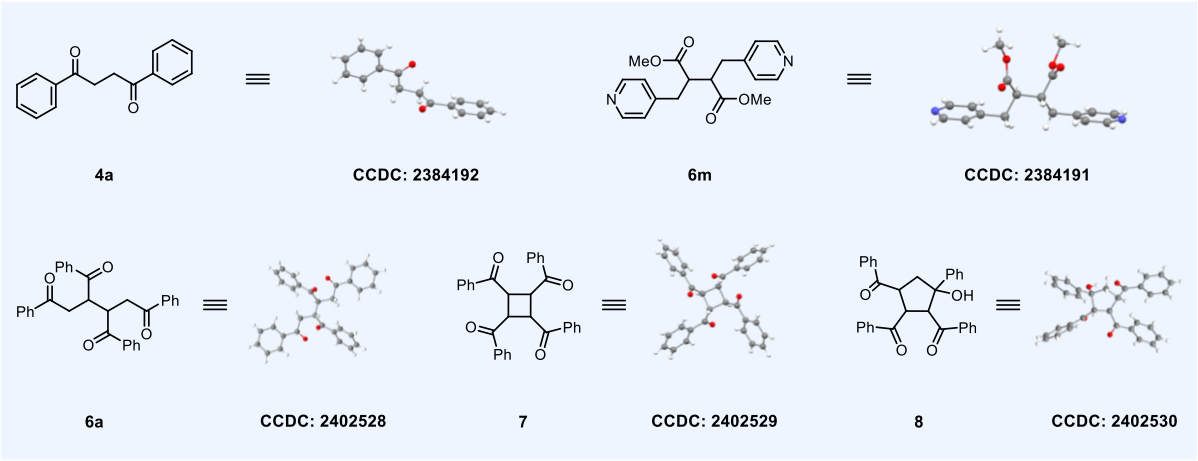


^{13}C NMR (126 MHz, CD_3CN)

4. X-ray Data Collection and Structural Refinement

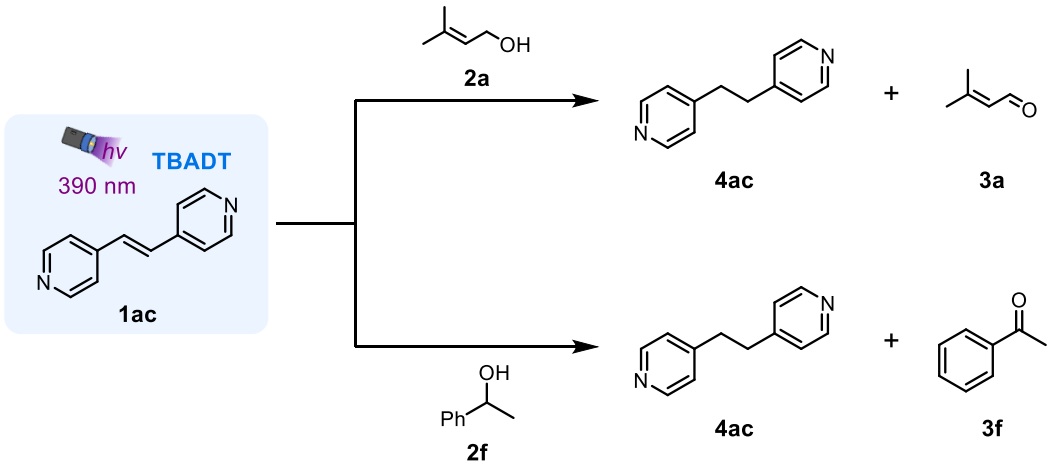
The X-ray diffraction intensity data were measured at 100 K with a Bruker APEX II diffractometer equipped with a CCD detector, employing Mo K α radiation (λ = 0.71073 Å), with the SMART suite of programs. All data were processed and corrected for Lorentz and polarization effects with SAINT and for absorption effects with SADABS.[2] Structural solution and refinement were carried out with the SHELXTL suite of programs.[3] CCDC-2384191, 2384192, and 2402528-2402530 contain the supplementary crystallographic data for this paper. The data can be obtained free of charge from the Cambridge Crystallography Data Center via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: + 44 1223 336033.

Table S3. Crystal structures of the synthesized alkanes.



5. Mechanism Study

5.1 Transfer hydrogenation of 1,2-di(4-pyridyl)ethylene by using alcohol as the hydrogen source



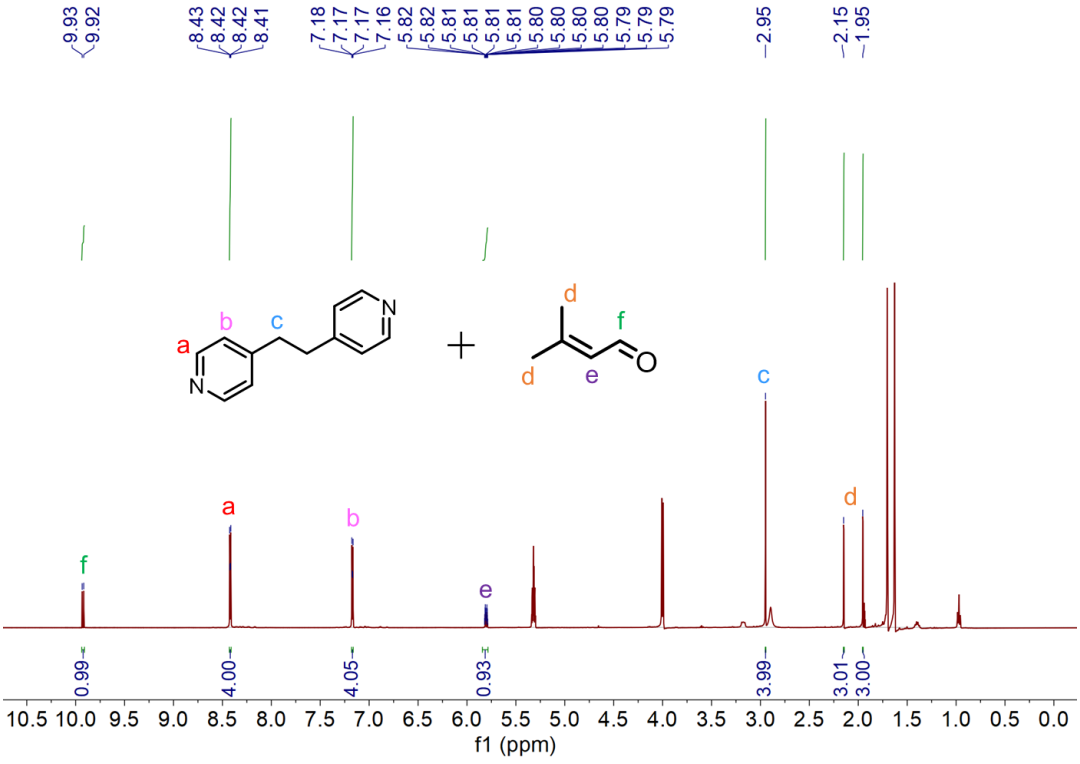


Figure S2. The crude ^{1}H NMR spectrum of TBADT (5 mol%) catalyzed transfer hydrogenation of 1,2-di(4-pyridyl)ethylene using prenol as the hydrogen source in CD_3CN.

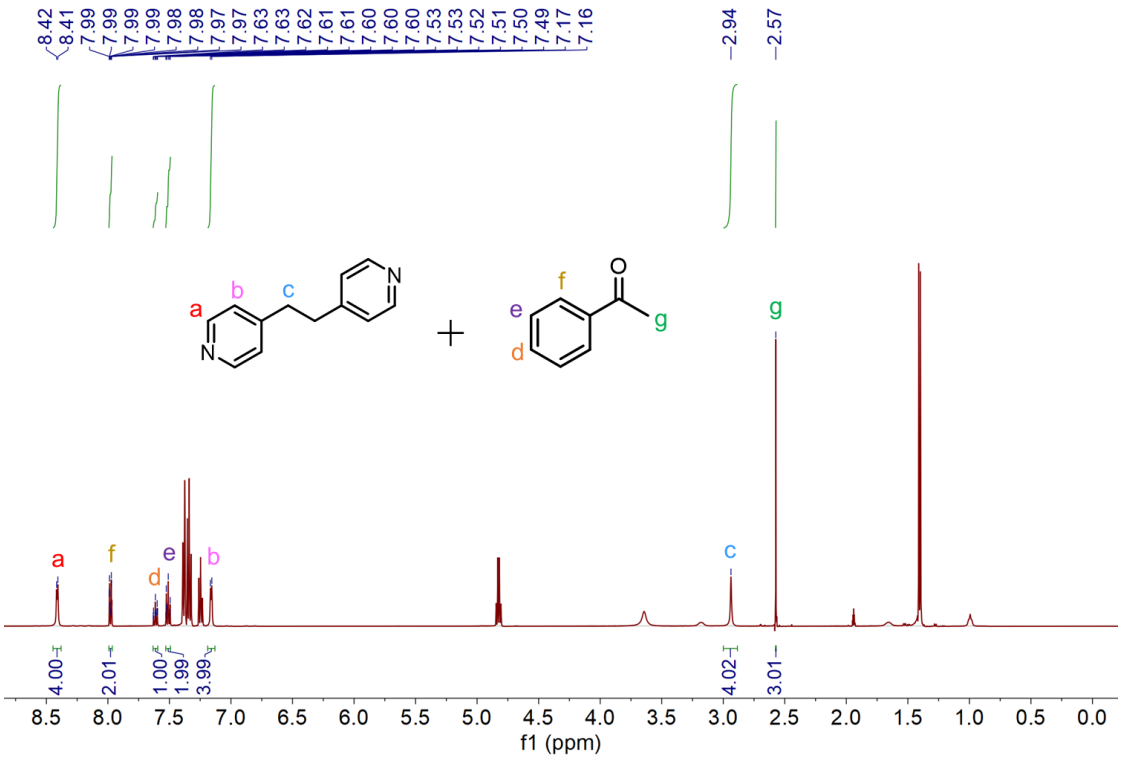
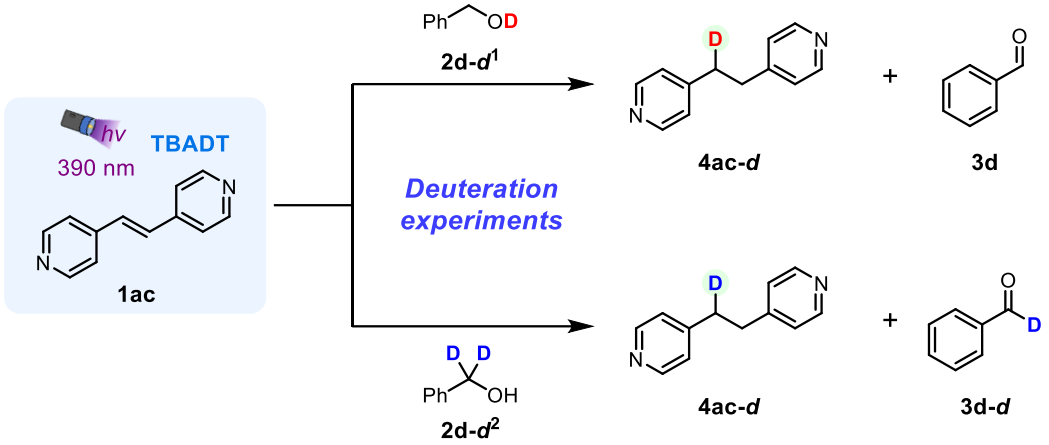


Figure S3. The crude ^{1}H NMR spectrum of TBADT (5 mol%) catalyzed transfer hydrogenation of 1,2-di(4-pyridyl)ethylene using 1-phenylethanol as the hydrogen source in CD_3CN.

5.2 Deuterium Labeling Experiments



Scheme S2. Deuteration experiments.

The HRMS (ESI) m/z calculated for **4ac-*d*** [M + H^+]: 186.1142, found: 186.1154.

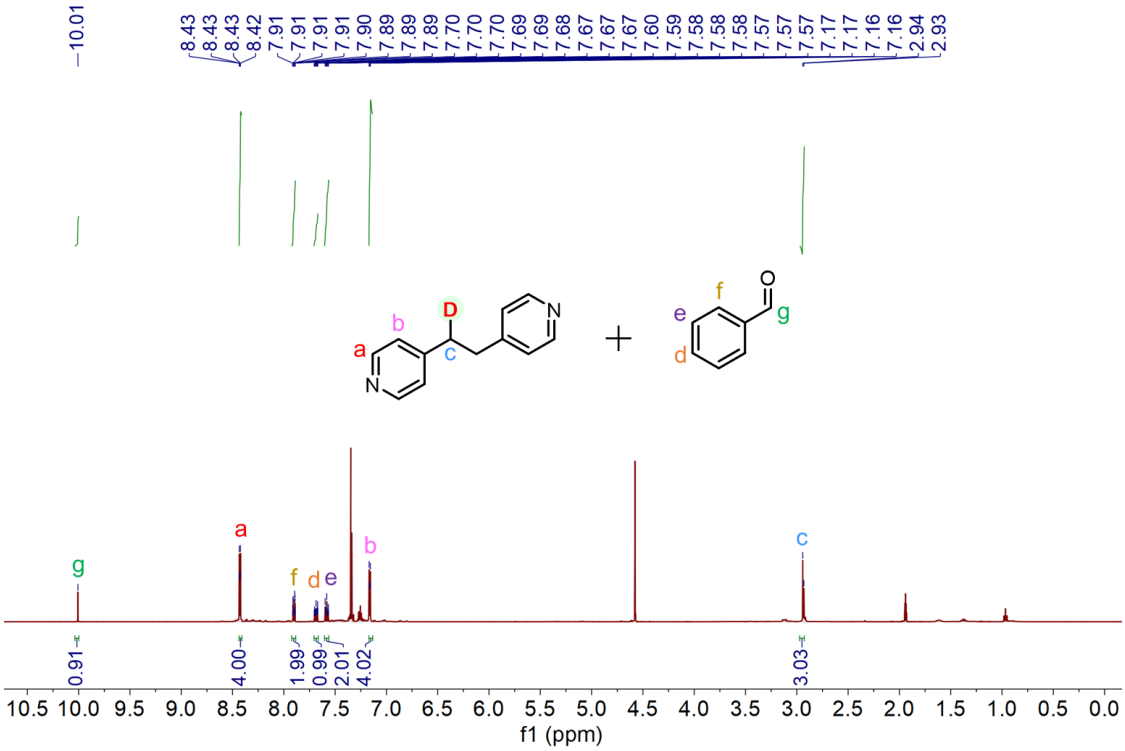


Figure S4. The crude ^{1}H NMR spectrum of TBADT (5 mol%) catalyzed transfer hydrogenation of 1,2-di(4-pyridyl)ethylene using benzyl alcohol-d_1 as the hydrogen source in CD_3CN.

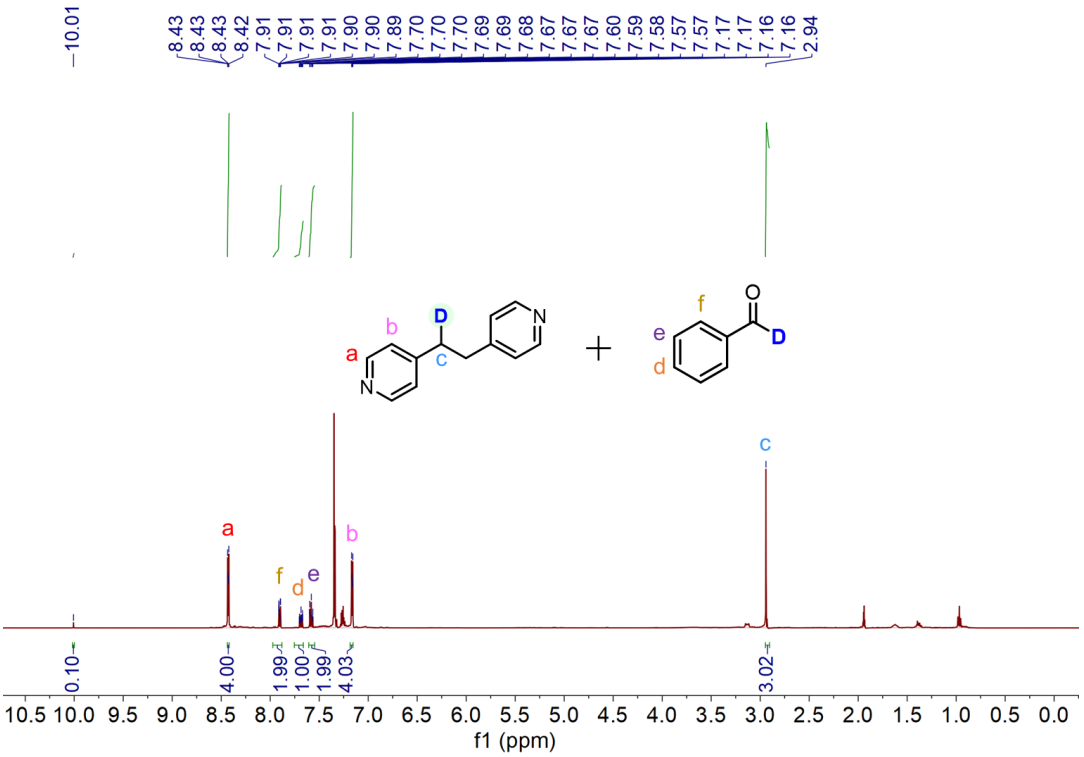


Figure S5. The crude ^{1}H NMR spectrum of TBADT (5 mol%) catalyzed transfer hydrogenation of 1,2-di(4-pyridyl)ethylene using phenylmethan-d_2-ol as the hydrogen source in CD_3CN.

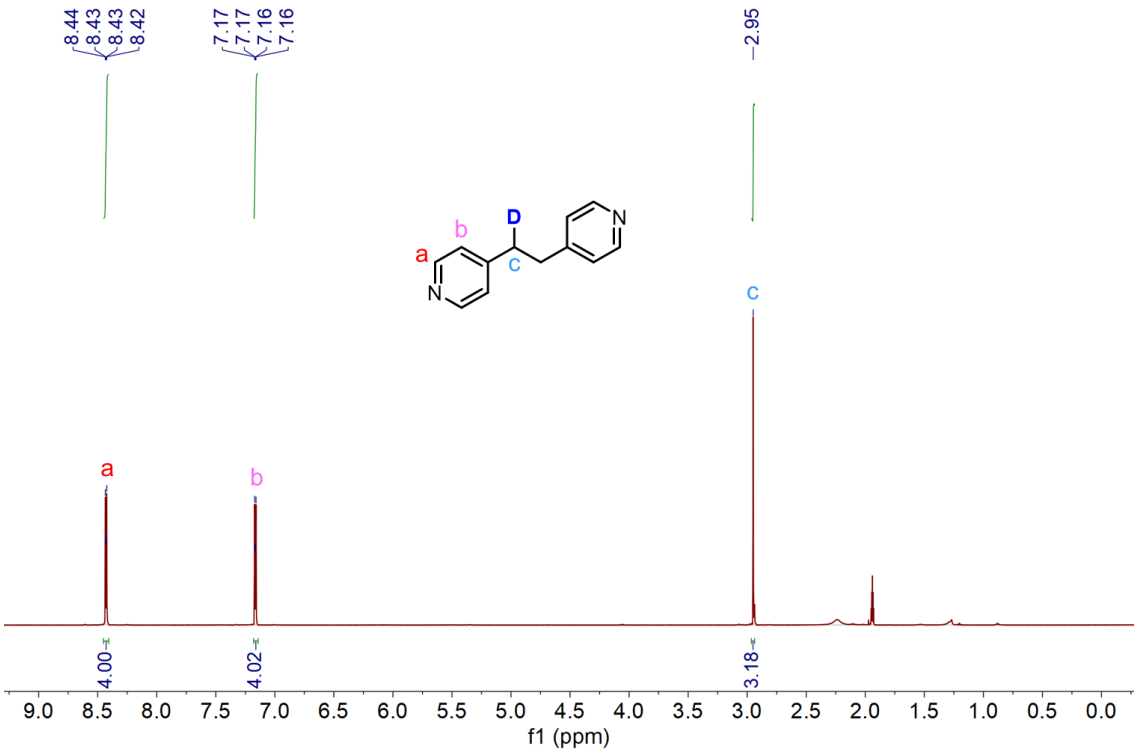
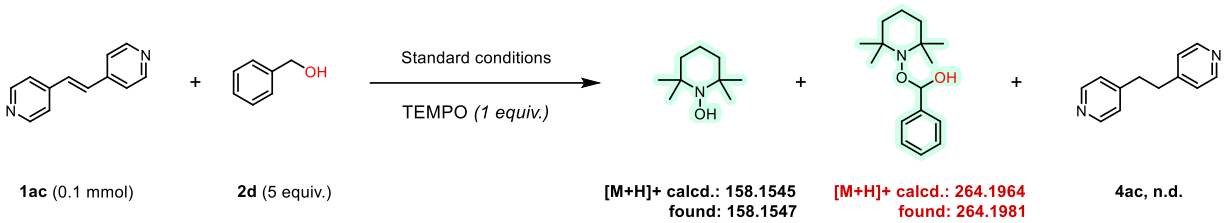


Figure S6. ^{1}H NMR spectrum of the isolated **4ac-*d*** in CD_3CN.

5.3 Radical Trapping Experiments

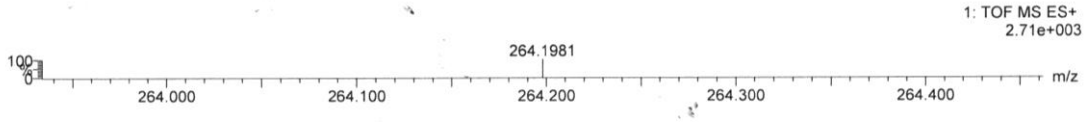


1ac (0.1 mmol) **2d** (5 equiv.) **[M+H]+ calcd.: 158.1545 found: 158.1547** **[M+H]+ calcd.: 264.1964 found: 264.1981** **4ac, n.d.**

Elemental Composition Report

Single Mass Analysis
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 2

Monoisotopic Mass, Even Electron Ions
2 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 15-16 H: 24-26 N: 1-2 O: 0-2



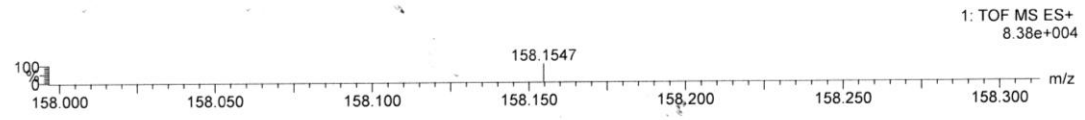
Minimum:				-1.5				
Maximum:		5.0	20.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
264.1981	264.1964	1.7	6.4	4.5	21.7	n/a	n/a	C16 H26 N O2

Figure S7. High-resolution mass spectrometry (HRMS) analysis of TEMPO trapped phenylmethanol radical.

Elemental Composition Report

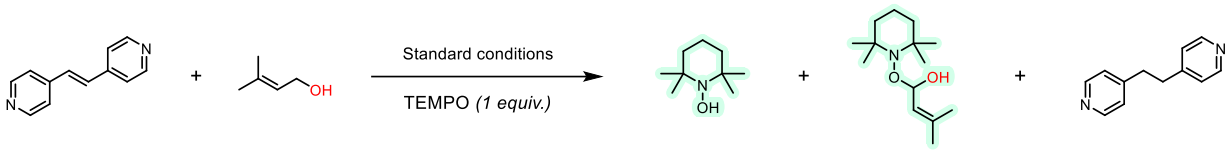
Single Mass Analysis
Tolerance = 20.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
141 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-18 H: 0-40 N: 0-3 O: 0-5



Minimum:				-1.5				
Maximum:		5.0	20.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
158.1547	158.1545	0.2	1.3	0.5	33.3	n/a	n/a	C9 H20 N O

Figure S8. High-resolution mass spectrometry (HRMS) analysis of TEMPO trapped hydrogen atom.



1ac (0.1 mmol) **2a** (5 equiv.) **[M+H]+ calcd.: 158.1545 found: 158.1548** **[M+H]+ calcd.: 242.2120 found: 242.2120** **4ac, n.d.**

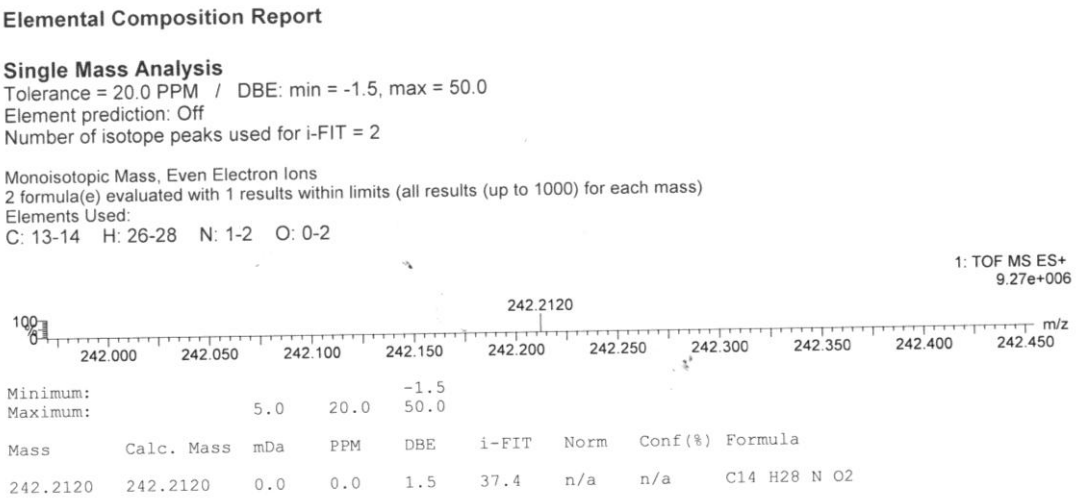


Figure S9. High-resolution mass spectrometry (HRMS) analysis of TEMPO trapped 3-methyl-2-butenol (prenol) radical.

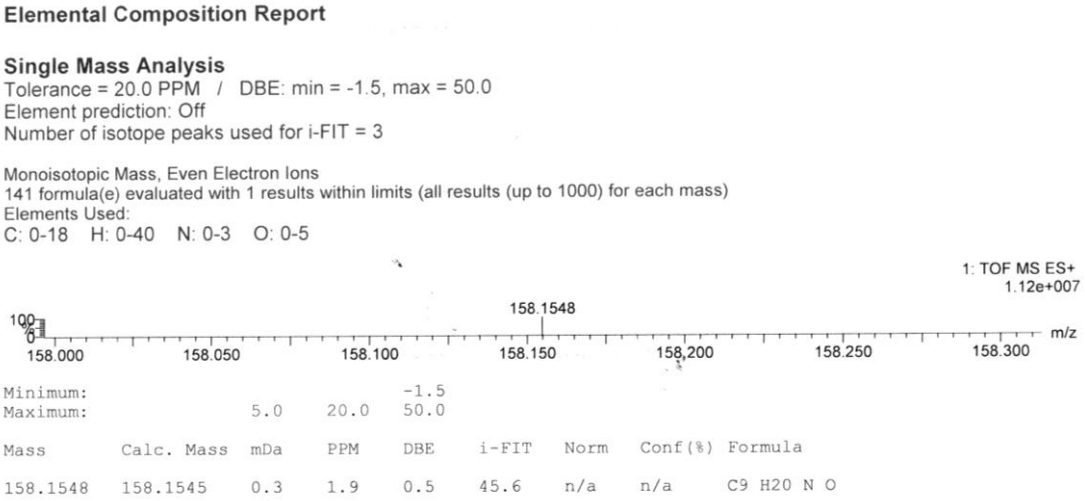
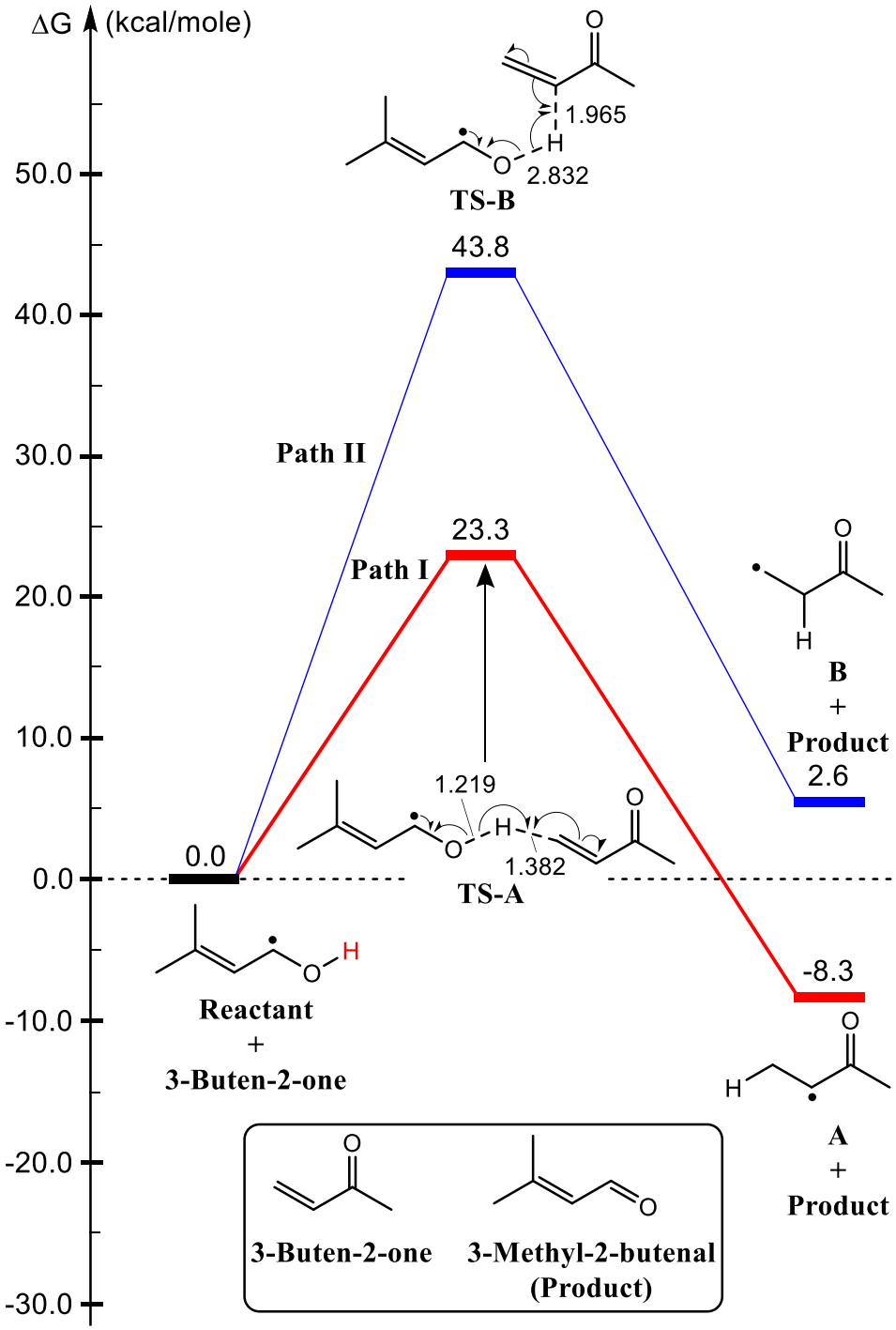


Figure S10. High-resolution mass spectrometry (HRMS) analysis of TEMPO trapped hydrogen atom.

6. Density functional theory (DFT) calculations

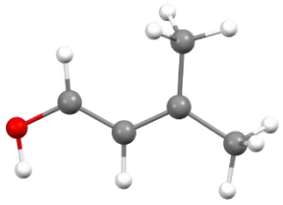
All density functional theory (DFT) calculations were performed using Gaussian 16 B.01 software,[4] and the computed activation and reaction energy profiles are expressed as Gibbs free energy, with units in kcal/mol. Geometry optimization of all stationary and transition points in the reaction pathway was carried out using the M06-2X[5]/def2-SVP[6] level of theory. Vibrational frequency calculations were performed to establish the nature of stationary points. The local minima are confirmed by zero imaginary frequencies, and transition states are characterized by an imaginary frequency.

Theoretical Calculations of Pathways I and II



*Gibbs free energy represented in kcal/mol.

Table S1
M06-2X/def2-SVP

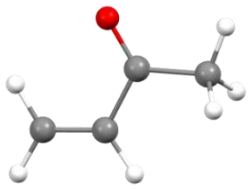


Reactant

G = -270.682404 Hartree

Atomic Number	Coordinates (Angstroms) X	Y	Z
C	0.61572400	-0.02206500	0.40107900
C	1.99020200	-0.14455700	0.20550400
C	-0.19718200	0.88658100	-0.47399500
C	-0.08923500	-0.78469000	1.47861600
H	-0.13009300	0.59363600	-1.53618000
H	0.15465800	1.93099200	-0.41179000
H	-1.25809300	0.87383400	-0.19040000
H	0.60315200	-1.41500400	2.05302500
H	-0.87564900	-1.43703000	1.06035800
H	-0.59660100	-0.10509700	2.18549300
C	2.72393200	0.51701500	-0.76050900
H	2.54070900	-0.82175300	0.87149400
H	2.28219300	1.21210700	-1.47534000
O	4.05590400	0.38488000	-0.93363600
H	4.40211700	-0.23656900	-0.28179100

Table S2
M06-2X/def2-SVP

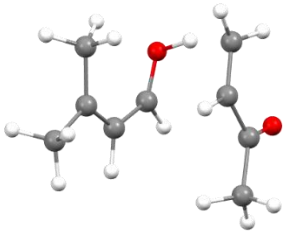


3-Buten-2-one

G = -230.888618 Hartree

Atomic Number	Coordinates (Angstroms) X	Y	Z
C	0.86820800	0.60732200	-0.89832300
C	2.07684900	0.12588200	-0.60815800
H	0.39784200	0.32620500	-1.84433300
H	0.33166900	1.27415000	-0.22160200
C	2.77883800	-0.78903100	-1.56868800
H	2.58614300	0.37942000	0.32562500
O	2.27543000	-1.10897400	-2.61769700
C	4.14267800	-1.28002600	-1.14453800
H	4.06442900	-1.82217900	-0.19012100
H	4.81242600	-0.42302500	-0.97708700
H	4.55774700	-1.93619400	-1.91694800

Table S3
M06-2X/def2-SVP



TS-A
G = -501.533828 Hartree

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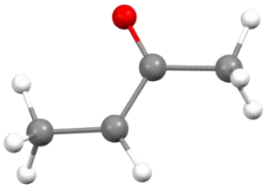
C	-1.13007400	0.85628100	1.68265200
C	-2.30252500	-0.06581300	1.52463700
C	-1.46885600	2.26075100	2.08380000
C	0.69293800	-0.77387200	1.06472600
O	0.00091200	-1.80281600	0.79537900
H	-0.58403700	2.90815100	2.12655100
H	-1.96011600	2.26967300	3.07056800
H	-2.19195300	2.69733900	1.37467900
H	-2.01793400	-1.12077500	1.58290400
H	-2.77966400	0.10567900	0.54299400
H	-3.06510100	0.15807900	2.28539600
C	0.17154800	0.50450500	1.45845000
H	1.79005200	-0.86425600	0.99955800
H	0.93531200	1.27194000	1.61573400
H	-0.50947400	-1.67627600	-0.30477300
C	-0.80254300	-1.33714100	-1.61274000
C	-0.21099700	-0.07837200	-1.59885900
H	-0.21651400	-2.14876900	-2.05801500
H	-1.89141300	-1.41838300	-1.66649300
C	1.24388100	0.06530600	-1.72223400
H	-0.80385400	0.82142900	-1.41260800
O	1.96540600	-0.90858500	-1.87627100
C	1.81010200	1.47126800	-1.69344800
H	1.27928100	2.10801500	-0.97198100
H	1.69959600	1.92744500	-2.68955800
H	2.87841200	1.42628500	-1.45125200

Table S4
M06-2X/def2-SVP

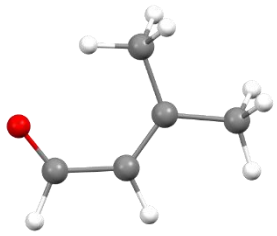


Compound A

G = -231.457942 Hartree

Atomic Number	Coordinates (Angstroms) X	Y	Z
H	2.48396900	0.30665400	0.35093100
C	0.72473800	0.68352800	-0.97810100
C	2.04160300	0.10256200	-0.62756800
H	0.43918400	0.37355400	-1.99053400
H	0.75143700	1.78467900	-0.92927100
C	2.76949000	-0.73996500	-1.55866800
O	2.31637500	-0.99728900	-2.66556200
C	4.10938500	-1.28611200	-1.10579100
H	3.99053900	-1.89411400	-0.19698700
H	4.79900500	-0.46372400	-0.86521700
H	4.53533500	-1.90067500	-1.90634500
H	-0.05308000	0.36125200	-0.26626800

Table S5
M06-2X/def2-SVP

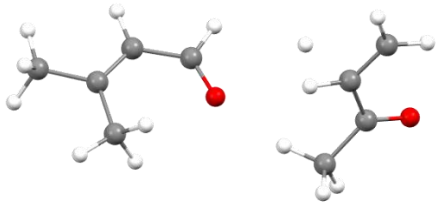


Product

G = -270.126367 Hartree

Atomic Number	Coordinates (Angstroms) X	Y	Z
C	1.00574300	0.72656000	-1.36611800
C	2.18082200	0.18613200	-0.98141800
C	0.35886700	0.50581900	-2.70184000
C	0.24455900	1.62308900	-0.43153700
C	3.05487900	-0.71103500	-1.76680300
O	2.86886400	-1.11468500	-2.88902800
H	0.75777800	1.75533000	0.52884600
H	-0.75881900	1.20935500	-0.24381500
H	0.09509700	2.61176400	-0.89342000
H	0.94383400	-0.15598100	-3.34414500
H	0.21820900	1.47700000	-3.20279100
H	-0.64790000	0.08364700	-2.55378300
H	2.55566900	0.41907200	0.01928200
H	3.97709800	-1.02232700	-1.21762100

Table S6
M06-2X/def2-SVP

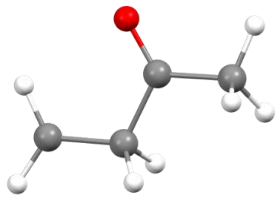


TS-B

G = -501.501178 Hartree

Atomic Number	Coordinates (Angstroms) X	Y	Z
C	2.87901700	3.65276400	0.49822400
C	2.94612200	3.12969300	-0.90945300
C	3.99169300	4.56515700	0.91901300
C	0.76082200	2.45486800	1.03614800
O	0.58183100	1.90880200	-0.03273600
H	3.88195600	4.91003600	1.95432200
H	4.03083800	5.44067300	0.25157700
H	4.95924800	4.04917200	0.81359300
H	2.04471800	3.41055300	-1.47230000
H	2.95919900	2.03061300	-0.91599600
H	3.83551600	3.51031400	-1.42783600
C	1.88684800	3.33762500	1.35998300
H	0.02867000	2.29446200	1.86369300
H	1.89989700	3.75288400	2.37047100
H	-1.66929200	0.85812900	1.32837500
C	-3.76547700	1.12841100	-0.39534900
C	-2.48851900	0.70698400	-0.45182500
H	-4.55620000	0.40266100	-0.19026200
H	-4.03361700	2.17717800	-0.52934200
C	-2.18302700	-0.76625900	-0.35055800
H	-1.66194900	1.37472100	-0.70997100
O	-3.04365800	-1.56995400	-0.08819500
C	-0.75311200	-1.15811000	-0.62612000
H	-0.06543600	-0.48628000	-0.09353100
H	-0.55405300	-1.02692300	-1.70158800
H	-0.59313600	-2.20604900	-0.34993300

Table S7
M06-2X/def2-SVP

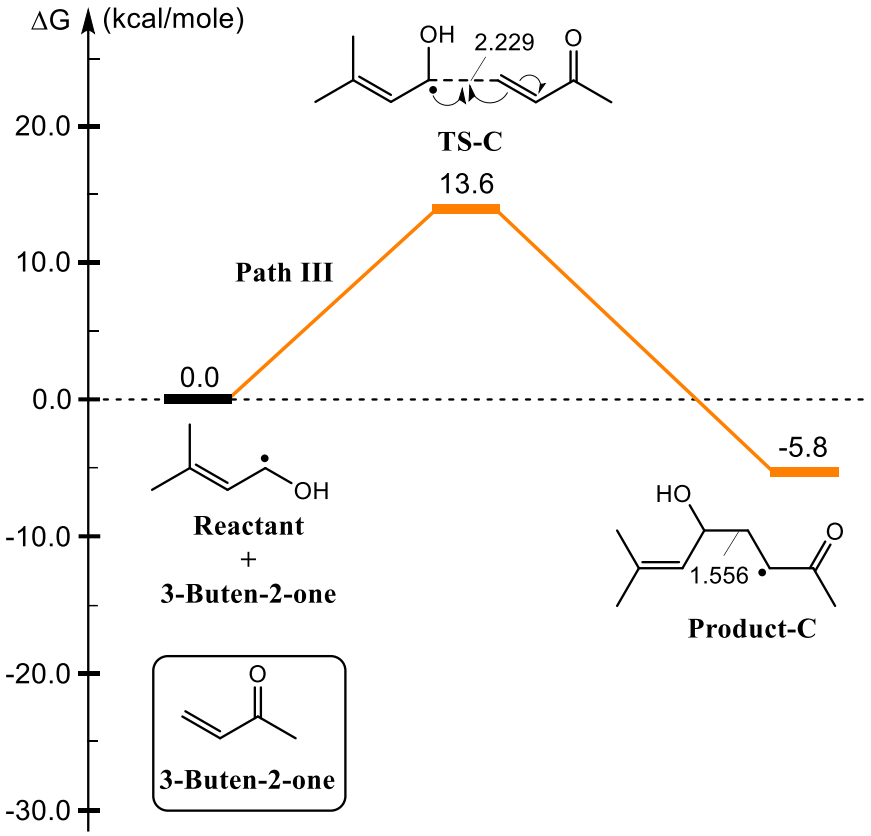


Compound B

G = -231.440521 Hartree

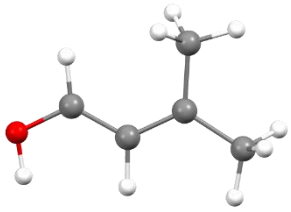
Atomic Number	Coordinates (Angstroms) X	Y	Z
C	0.86261700	0.80147600	-1.34103700
C	1.95961900	0.02934400	-0.70954800
H	0.68739200	0.70027100	-2.41097800
H	0.19546500	1.41191100	-0.73484500
C	2.85426700	-0.69315900	-1.70890100
H	2.60175400	0.65909000	-0.06476000
O	2.59566500	-0.73487000	-2.88350400
C	4.09131400	-1.35029600	-1.13702200
H	3.85459400	-1.91192400	-0.22247500
H	4.81829100	-0.56994200	-0.86422200
H	4.53785400	-2.01083900	-1.88835600
H	1.57412700	-0.74836200	-0.01771100

Theoretical Calculations of Pathway III



*Gibbs free energy represented in kcal/mol.

Table S8
M06-2X/def2-SVP

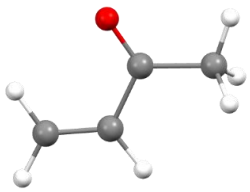


Reactant

G = -270.682404 Hartree

Atomic Number	Coordinates (Angstroms) X	Y	Z
C	0.61572400	-0.02206500	0.40107900
C	1.99020200	-0.14455700	0.20550400
C	-0.19718200	0.88658100	-0.47399500
C	-0.08923500	-0.78469000	1.47861600
H	-0.13009300	0.59363600	-1.53618000
H	0.15465800	1.93099200	-0.41179000
H	-1.25809300	0.87383400	-0.19040000
H	0.60315200	-1.41500400	2.05302500
H	-0.87564900	-1.43703000	1.06035800
H	-0.59660100	-0.10509700	2.18549300
C	2.72393200	0.51701500	-0.76050900
H	2.54070900	-0.82175300	0.87149400
H	2.28219300	1.21210700	-1.47534000
O	4.05590400	0.38488000	-0.93363600
H	4.40211700	-0.23656900	-0.28179100

Table S9
M06-2X/def2-SVP

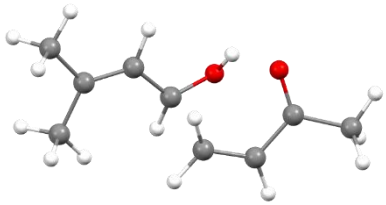


3-Buten-2-one

G = -230.888618 Hartree

Atomic Number	Coordinates (Angstroms) X	Y	Z
C	0.86820800	0.60732200	-0.89832300
C	2.07684900	0.12588200	-0.60815800
H	0.39784200	0.32620500	-1.84433300
H	0.33166900	1.27415000	-0.22160200
C	2.77883800	-0.78903100	-1.56868800
H	2.58614300	0.37942000	0.32562500
O	2.27543000	-1.10897400	-2.61769700
C	4.14267800	-1.28002600	-1.14453800
H	4.06442900	-1.82217900	-0.19012100
H	4.81242600	-0.42302500	-0.97708700
H	4.55774700	-1.93619400	-1.91694800

Table S10
M06-2X/def2-SVP

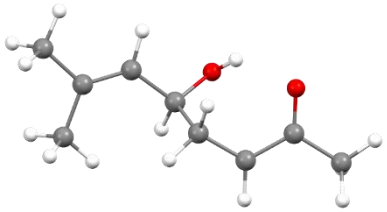


TS-C

G = -501.549297 Hartree

Atomic Number	Coordinates (Angstroms) X	Y	Z
C	2.16048800	-0.22774800	-0.05982700
C	1.10797000	0.57796700	0.23069100
C	2.62073100	-1.34757100	0.82855600
C	2.94818300	-0.04640900	-1.32413100
H	2.06702500	-1.41159400	1.77261900
H	3.69096200	-1.23507300	1.06516900
H	2.51575500	-2.31355100	0.30740800
H	2.55513000	0.77461800	-1.93717700
H	2.93507500	-0.96865900	-1.92837000
H	4.00630500	0.16427000	-1.09731700
C	0.24629200	0.49272000	1.38053200
H	0.58860300	-0.02326200	2.28105500
O	-0.60020600	1.49249500	1.66654600
H	-1.09945700	1.73100600	0.86311100
H	0.82758100	1.34695000	-0.49883800
C	-1.13199900	-1.11015800	0.67183600
C	-2.34559800	-0.79802100	1.23045600
C	-3.12524400	0.30152000	0.67150900
C	-4.54803200	0.47095200	1.15264200
O	-2.64069500	1.08446600	-0.14289500
H	-0.93765000	-0.76700300	-0.34442300
H	-2.70957400	-1.29472200	2.13274500
H	-4.54014700	0.73784200	2.22047600
H	-5.11004100	-0.46839300	1.05241900
H	-5.03568200	1.26870400	0.58183600
H	-0.55229100	-1.96665500	1.02286900

Table S11
M06-2X/def2-SVP

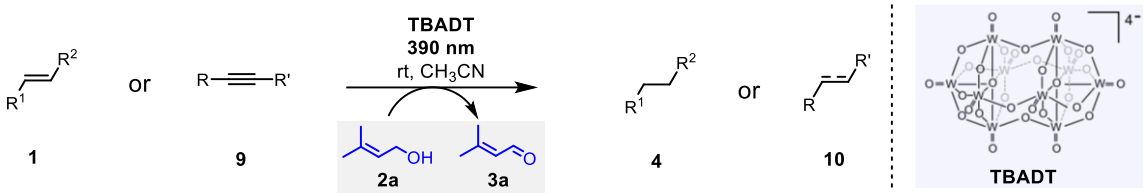


Product C

G = -501.580228 Hartree

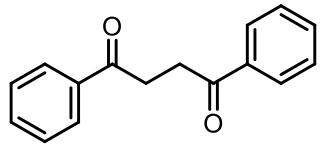
Atomic Number	Coordinates (Angstroms) X	Y	Z
C	0.61471000	-0.09806700	0.69575300
C	1.74687000	-0.18711400	-0.01291400
C	-0.70120100	0.35319900	0.11945600
C	0.57376400	-0.45939900	2.15572800
H	-1.43564300	-0.46773800	0.15766300
H	-0.62565100	0.69064400	-0.92157000
H	-1.11970700	1.17746400	0.71825900
H	1.55409700	-0.79303300	2.51926800
H	-0.15786500	-1.26278700	2.33891400
H	0.25368300	0.40461200	2.76000100
C	1.93164500	0.12288600	-1.47852700
H	1.26625700	0.94999600	-1.77428200
O	3.23643100	0.55718900	-1.75080600
H	3.79199800	-0.21781700	-1.92369200
H	2.65891300	-0.53705000	0.48412700
C	1.55945500	-1.12287800	-2.33403800
C	1.88589200	-0.94593600	-3.76959400
C	3.23177000	-1.19893300	-4.24350500
C	3.52069200	-1.01712400	-5.71598400
O	4.11622300	-1.53310500	-3.45916200
H	2.13800200	-1.97317600	-1.93825900
H	1.13695400	-0.57117200	-4.47117100
H	3.55956500	0.05849200	-5.94630200
H	2.73462800	-1.46489300	-6.33919400
H	4.49277200	-1.46419300	-5.95118600
H	0.49076800	-1.33378700	-2.18670000

7. General procedure for Transfer Hydrogenation of Alkenes and Alkynes

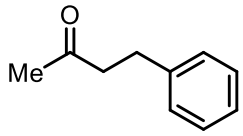


In a glovebox, alkenes or alkynes (0.1 mmol), TBADT (5 mol%), prenol (0.5 mmol, 5 equiv.), and anhydrous acetonitrile (2 mL) were placed in a 10 mL Schlenk tube equipped with a magnetic stir bar. The reaction mixture was subsequently irradiated with an LED light (Kessil 40 W) at room temperature for 6 – 24 hours, with 24 hours being sufficient for all substrates. Then, the solvent was removed under reduced pressure using a rotary evaporator, and the resulting residue was purified by silica gel column chromatography. The appropriate eluent system (hexane, hexane/ethyl acetate, DCM/methanol or ethyl acetate/methanol) was selected based on the polarity of the target product, yielding the corresponding products.

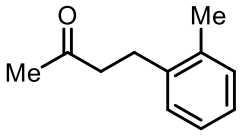
8. Characterization of Hydrogenated Products



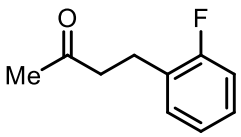
1,4-Diphenylbutane-1,4-dione (4a) Prepared from 1,2-dibenzoylethylene (**1a**, 23.6 mg, 0.1 mmol) according to the general procedure, compound **4a** was purified by silica gel column chromatography (hexane/ethyl acetate 10:1) and obtained as a colorless solid (20.6 mg, yield 86%). ^{1}H NMR (500 MHz, Acetone-d_6 / DMSO-d_6): δ 8.09 – 8.04 (m, 4H), 7.68 – 7.63 (m, 2H), 7.58 – 7.53 (m, 4H), 3.46 (s, 4H). ^{13}C NMR (126 MHz, Acetone-d_6 / DMSO-d_6): δ 198.8, 137.4, 133.5, 129.1, 128.4, 32.8. HRMS (ESI, m/z) calculated for $C_{16}H_{15}O_2$ $[M+H]^+$: 239.1067, found: 239.1074.



4-Phenylbutan-2-one (4b) Prepared from 4-phenyl-3-buten-2-one (**1b**, 14.7 mg, 0.1 mmol) according to the general procedure, compound **4b** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (11.3 mg, yield 76%). ^{1}H NMR (500 MHz, Acetonitrile-d_3): δ 7.33 – 7.26 (m, 2H), 7.25 – 7.16 (m, 3H), 2.87 – 2.80 (m, 2H), 2.79 – 2.73 (m, 2H), 2.08 (s, 3H). ^{13}C NMR (126 MHz, Acetonitrile-d_3): δ 208.6, 142.6, 129.3, 129.2, 126.9, 45.4, 30.3, 30.0. HRMS (ESI, m/z) calculated for $C_{10}H_{13}O$ $[M+H]^+$: 149.0961, found: 149.0967.

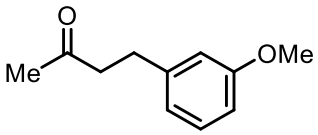


4-(*O*-tolyl)butan-2-one (4c) Prepared from 4-(*o*-tolyl)-3-buten-2-one (**1c**, 16.2 mg, 0.1 mmol) according to the general procedure, compound **4c** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (9.3 mg, yield 57%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.19 – 7.08 (m, 4H), 2.89 (t, *J* = 7.6 Hz, 2H), 2.72 (t, *J* = 7.6 Hz, 2H), 2.32 (s, 3H), 2.17 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 208.1, 139.1, 135.9, 130.3, 128.6, 126.3, 126.2, 43.9, 30.1, 27.1, 19.3. HRMS (ESI, m/z) calculated for $C_{11}H_{15}O$ $[M+H]^+$: 163.1117, found: 163.1101.

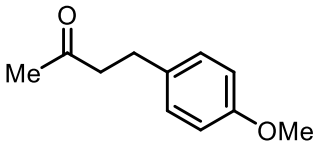


4-(2-Fluorophenyl)butan-2-one (4d) Prepared from 4-(2-fluorophenyl)but-3-en-2-one (**1d**, 16.4 mg, 0.1 mmol) according to the general procedure, compound **4d** was purified by

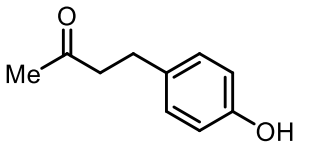
silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (12.1 mg, yield 73%). ^{1}H NMR (600 MHz, Chloroform-*d*): δ 7.23 – 7.13 (m, 2H), 7.07 – 6.96 (m, 2H), 2.91 (t, *J* = 7.6 Hz, 2H), 2.76 (t, *J* = 7.6 Hz, 2H), 2.14 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*): δ 207.7, 161.2 (d, *J* = 244.9 Hz), 130.8 (d, *J* = 4.9 Hz), 128.0 (d, *J* = 8.1 Hz), 127.9 (d, *J* = 15.6 Hz), 124.2 (d, *J* = 3.5 Hz), 115.4 (d, *J* = 21.9 Hz), 43.7, 30.0, 23.5. ^{19}F NMR (565 MHz, Chloroform-*d*): δ -118.60. HRMS (ESI, m/z) calculated for $C_{10}H_{12}FO$ $[M+H]^+$: 167.0867, found: 167.0862.



4-(3-Methoxyphenyl)butan-2-one (4e) Prepared from 4-(3-methoxyphenyl)but-3-en-2-one (**1e**, 17.6 mg, 0.1 mmol) according to the general procedure, compound **4e** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (10.9 mg, yield 61%). ^{1}H NMR (600 MHz, Chloroform-*d*): δ 7.22 – 7.16 (m, 1H), 6.82 – 6.66 (m, 3H), 3.79 (s, 3H), 2.87 (t, *J* = 7.6 Hz, 2H), 2.76 (t, *J* = 7.7 Hz, 2H), 2.14 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*): δ 208.0, 159.8, 142.7, 129.6, 120.7, 114.2, 111.5, 55.2, 45.2, 30.2, 29.9. HRMS (ESI, m/z) calculated for $C_{11}H_{15}O_2$ $[M+H]^+$: 179.1067, found: 179.1063.

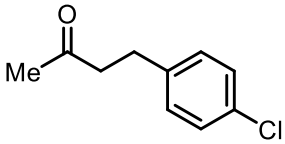


4-(4-Methoxyphenyl)butan-2-one (4f) Prepared from 4-(4-methoxyphenyl)but-3-en-2-one (**1f**, 17.6 mg, 0.1 mmol) according to the general procedure, compound **4f** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (11.2 mg, yield 63%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.16 – 7.04 (m, 2H), 6.88 – 6.75 (m, 2H), 3.78 (s, 3H), 2.84 (t, *J* = 7.5 Hz, 2H), 2.72 (t, *J* = 7.4 Hz, 2H), 2.12 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 208.2, 158.1, 133.1, 129.3, 114.0, 55.4, 45.6, 30.2, 29.0. HRMS (ESI, m/z) calculated for $C_{11}H_{15}O_2$ $[M+H]^+$: 179.1067, found: 179.1061.

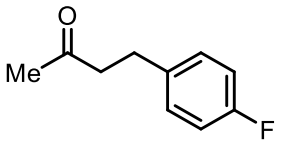


4-(4-Hydroxyphenyl)butan-2-one (4g) Prepared from 4-(4-hydroxyphenyl)but-3-en-2-one (**1g**, 16.2 mg, 0.1 mmol) according to the general procedure, compound **4g** was purified by silica gel column chromatography (hexane/ethyl acetate 5:1) and obtained as a colorless solid (8.9 mg, yield 54%). ^{1}H NMR (600 MHz, Chloroform-*d*): δ 7.06 – 6.97 (m, 2H), 6.79 – 6.71 (m, 2H), 6.22 (s, 1H), 2.82 (t, *J* = 7.5 Hz, 2H), 2.74 (t, *J* = 7.5 Hz, 2H), 2.14 (s, 3H). ^{13}C NMR

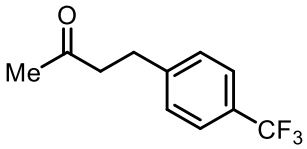
(151 MHz, Chloroform-*d*): δ 209.9, 154.3, 132.6, 129.5, 115.5, 45.6, 30.3, 29.0. HRMS (ESI, m/z) calculated for $C_{10}H_{13}O_2$ $[M+H]^+$: 165.0910, found: 165.0903.



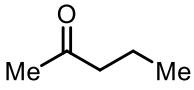
4-(4-Chlorophenyl)butan-2-one (4h) Prepared from 4-(4-chlorophenyl)but-3-en-2-one (**1h**, 18.1 mg, 0.1 mmol) according to the general procedure, compound **4h** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (14.3 mg, yield 79%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.29 – 7.18 (m, 2H), 7.15 – 7.06 (m, 2H), 2.86 (t, J = 7.5 Hz, 2H), 2.73 (t, J = 7.4 Hz, 2H), 2.13 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 207.5, 139.6, 132.0, 129.8, 128.7, 45.0, 30.2, 29.1. HRMS (ESI, m/z) calculated for $C_{10}H_{12}ClO$ $[M+H]^+$: 183.0571, found: 183.0569.



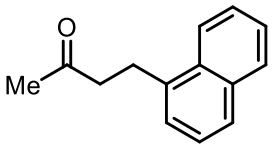
4-(4-Fluorophenyl)butan-2-one (4i) Prepared from 4-(4-fluorophenyl)but-3-en-2-one (**1i**, 16.4 mg, 0.1 mmol) according to the general procedure, compound **4i** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (13.5 mg, yield 81%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.19 – 7.05 (m, 2H), 7.02 – 6.85 (m, 2H), 2.86 (t, J = 7.4 Hz, 2H), 2.73 (t, J = 7.5 Hz, 2H), 2.13 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 207.7, 161.5 (d, J = 243.8 Hz), 136.8 (d, J = 3.3 Hz), 129.8 (d, J = 7.8 Hz), 115.3 (d, J = 21.2 Hz), 45.3, 30.2, 29.0. ^{19}F NMR (377 MHz, Chloroform-*d*): δ -117.3. HRMS (ESI, m/z) calculated for $C_{10}H_{12}FO$ $[M+H]^+$: 167.0867, found: 167.0863.



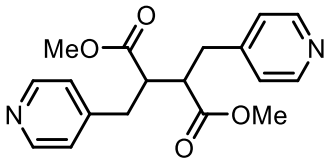
4-(4-(Trifluoromethyl)phenyl)butan-2-one (4j) Prepared from 4-(4-(trifluoromethyl)phenyl)but-3-en-2-one (**1j**, 21.5 mg, 0.1 mmol) according to the general procedure, compound **4j** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (18.3 mg, yield 85%). ^{1}H NMR (600 MHz, Chloroform-*d*): δ 7.59 – 7.44 (m, 2H), 7.35 – 7.20 (m, 2H), 2.95 (t, J = 7.5 Hz, 2H), 2.78 (t, J = 7.5 Hz, 2H), 2.15 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*): δ 207.3, 145.3,128.8, 128.6 (q, J_{C-F} = 32.4 Hz), 125.5 (q, J = 3.8 Hz), 124.4 (q, J_{C-F} = 271.8 Hz), 44.7, 30.2, 29.5. ^{19}F NMR (565 MHz, Chloroform-*d*): δ -62.39. HRMS (ESI, m/z) calculated for $C_{11}H_{12}F_3O$ $[M+H]^+$: 217.0835, found: 217.0848.



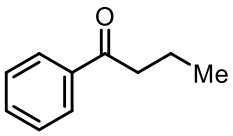
Pentan-2-one (4k) Prepared from 3-penten-2-one (**1k**, 8.4 mg, 0.1 mmol) according to the general procedure, compound **4k** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless oil (5.7 mg, yield 66%). ^{1}H NMR (500 MHz, Chloroform-*d*): δ 2.39 (t, *J* = 7.3 Hz, 2H), 2.12 (s, 3H), 1.59 (h, *J* = 7.4 Hz, 2H), 0.91 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*): δ 209.3, 45.8, 29.9, 17.4, 13.8. HRMS (ESI, m/z) calculated for $C_5H_{11}O$ $[M+H]^+$: 87.0804, found: 87.0810.



4-(Naphthalen-1-yl)butan-2-one (4l) Prepared from 4-(naphthalen-1-yl)but-3-en-2-one (**1l**, 19.7 mg, 0.1 mmol) according to the general procedure, compound **4l** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (14.1 mg, yield 71%). ^{1}H NMR (600 MHz, Chloroform-*d*): δ 8.02 – 7.95 (m, 1H), 7.89 – 7.84 (m, 1H), 7.73 (d, *J* = 8.2 Hz, 1H), 7.55– 7.46 (m, 2H), 7.39 (dd, *J* = 8.1, 7.0 Hz, 1H), 7.33 (dd, *J* = 6.9, 1.2 Hz, 1H), 3.36 (dd, *J* = 8.5, 7.1 Hz, 2H), 2.90 (dd, *J* = 8.6, 7.1 Hz, 2H), 2.17 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*): δ 208.1, 137.2, 134.1, 131.7, 129.1, 127.1, 126.2, 126.1, 125.7, 125.7, 123.6, 44.6, 30.2, 26.9. HRMS (ESI, m/z) calculated for $C_{14}H_{15}O$ $[M+H]^+$: 199.1117, found: 199.1106.

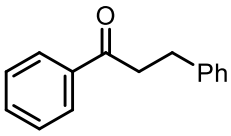


Dimethyl 2,3-bis(pyridin-4-ylmethyl)succinate (6m) Prepared from methyl 3-(pyridin-4-yl)acrylate (**1m**, 16.3 mg, 0.1 mmol) according to the general procedure, compound **6m** was purified by silica gel column chromatography (ethyl acetate/methanol 10:1) and obtained as a colorless solid (14.9 mg, yield 91%). ^{1}H NMR (500 MHz, Acetonitrile-d_3): δ 8.42 – 8.39 (m, 4H), 7.10 – 7.07 (m, 4H), 3.51 (s, 6H), 3.02 – 2.80 (m, 6H). ^{13}C NMR (126 MHz, Acetonitrile-d_3): δ 173.7, 150.5, 148.9, 125.3, 52.4, 48.3, 35.2. HRMS (ESI, m/z) calculated for $C_{18}H_{21}N_2O_4$ $[M+H]^+$: 329.1496, found: 329.1494.

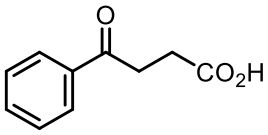


1-Phenylbutan-1-one (4n) Prepared from 1-phenylbut-2-en-1-one (**1n**, 14.6 mg, 0.1 mmol) according to the general procedure, compound **4n** was purified by silica gel column

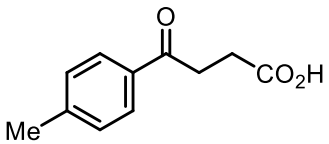
chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless oil (6.1 mg, yield 41%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 8.01 – 7.89 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.40 (m, 2H), 2.95 (t, *J* = 7.3 Hz, 2H), 1.77 (h, *J* = 7.4 Hz, 2H), 1.00 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 200.6, 137.2, 133.0, 128.7, 128.1, 40.6, 17.9, 14.0. HRMS (ESI, m/z) calculated for $C_{10}H_{13}O$ $[M+H]^+$: 149.0961, found: 149.0956.



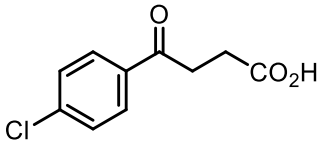
1,3-Diphenylpropan-1-one (4o) Prepared from chalcone (**1o**, 20.8 mg, 0.1 mmol) according to the general procedure, compound **4o** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (6.7 mg, yield 32%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 8.00 – 7.92 (m, 2H), 7.59 – 7.52 (m, 1H), 7.49 – 7.42 (m, 2H), 7.33 – 7.17 (m, 5H), 3.31 (t, *J* = 7.3 Hz, 2H), 3.07 (t, *J* = 7.4 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 199.4, 141.4, 137.0, 133.2, 128.8, 128.7, 128.6, 128.2, 126.3, 40.6, 30.3. HRMS (ESI, m/z) calculated for $C_{15}H_{15}O$ $[M+H]^+$: 211.1117, found: 211.1118.



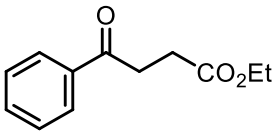
4-Oxo-4-phenylbutanoic acid (4p) Prepared from 4-oxo-4-phenylbut-2-enoic acid (**1p**, 17.6 mg, 0.1 mmol) according to the general procedure, compound **4p** was purified by silica gel column chromatography (DCM/methanol 10:1) and obtained as a colorless solid (12.6 mg, yield 71%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 10.48 (s, 1H), 8.05 – 7.91 (m, 2H), 7.62 – 7.52 (m, 1H), 7.51 – 7.42 (m, 2H), 3.31 (t, *J* = 6.5 Hz, 2H), 2.81 (t, *J* = 6.5 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 198.0, 179.0, 136.5, 133.5, 128.8, 128.2, 33.3, 28.2. HRMS (ESI, m/z) calculated for $C_{10}H_{11}O_3$ $[M+H]^+$: 179.0703, found: 179.0700.



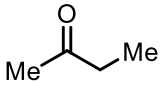
4-Oxo-4-(*p*-tolyl)butanoic acid (4q) Prepared from 4-oxo-4-(*p*-tolyl)but-2-enoic acid (**1q**, 19.0 mg, 0.1 mmol) according to the general procedure, compound **4q** was purified by silica gel column chromatography (DCM/methanol 10:1) and obtained as a colorless solid (12.8 mg, yield 67%). ^{1}H NMR (500 MHz, Acetonitrile-d_3): δ 9.13 (s, 1H), 7.92 – 7.85 (m, 2H), 7.35 – 7.28 (m, 2H), 3.24 (t, *J* = 6.4 Hz, 2H), 2.65 (t, *J* = 6.3 Hz, 2H), 2.40 (s, 3H). ^{13}C NMR (126 MHz, Acetonitrile-d_3): δ 199.0, 174.7, 145.1, 135.2, 130.2, 128.9, 34.0, 28.3, 21.6. HRMS (ESI, m/z) calculated for $C_{11}H_{13}O_3$ $[M+H]^+$: 193.0865, found: 193.0871.



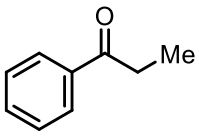
4-(4-Chlorophenyl)-4-oxobutanoic acid (4r) Prepared from 4-(4-chlorophenyl)-4-oxobut-2-enoic acid (**1r**, 21.1 mg, 0.1 mmol) according to the general procedure, compound **4r** was purified by silica gel column chromatography (DCM/methanol 10:1) and obtained as a colorless solid (15.7 mg, yield 74%). ^{1}H NMR (500 MHz, Acetonitrile-d_3): δ 9.02 (brs, 1H), 8.00 – 7.93 (m, 2H), 7.55 – 7.49 (m, 2H), 3.25 (t, J = 6.3 Hz, 2H), 2.66 (t, J = 6.4 Hz, 2H). ^{13}C NMR (126 MHz, Acetonitrile-d_3): δ 198.5, 174.3, 139.8, 136.3, 130.6, 129.8, 34.1, 28.2. HRMS (ESI, m/z) calculated for $C_{10}H_{10}ClO_3$ $[M+H]^+$: 213.0313, found: 213.0312.



Ethyl 4-oxo-4-phenylbutanoate (4s) Prepared from ethyl 4-oxo-4-phenylbut-2-enoate (**1s**, 20.4 mg, 0.1 mmol) according to the general procedure, compound **4s** was purified by silica gel column chromatography (hexane/ethyl acetate 10:1) and obtained as a colorless solid (17.9 mg, yield 87%). ^{1}H NMR (500 MHz, Acetonitrile-d_3): δ 8.02 – 7.95 (m, 2H), 7.65 – 7.58 (m, 1H), 7.54 – 7.46 (m, 2H), 4.09 (q, J = 7.1 Hz, 2H), 3.29 (t, J = 6.4 Hz, 2H), 2.66 (t, J = 6.3 Hz, 2H), 1.21 (t, J = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, Acetonitrile-d_3): δ 199.5, 173.6, 137.7, 134.1, 129.6, 128.8, 61.1, 34.1, 28.9, 14.5. HRMS (ESI, m/z) calculated for $C_{12}H_{15}O_3$ $[M+H]^+$: 207.1016, found: 207.1016.

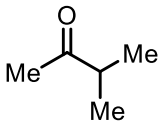


Butan-2-one (4t) Prepared from 3-buten-2-one (**1t**, 7.0 mg, 0.1 mmol) according to the general procedure, compound **4t** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (6.2 mg, yield 86%). ^{1}H NMR (400 MHz, Chloroform-d): δ 2.45 (q, J = 7.3 Hz, 2H), 2.14 (s, 3H), 1.05 (t, J = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-d): δ 209.7, 37.0, 29.6, 8.0.

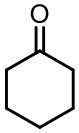


Propiophenone (4u) Prepared from 1-phenylprop-2-en-1-one (**1u**, 13.2 mg, 0.1 mmol) according to the general procedure, compound **4u** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless oil (11.3 mg, yield 84%). ^{1}H NMR (500 MHz, Chloroform-d): δ 8.00 – 7.94 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.43 (m, 2H), 3.01

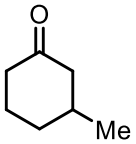
(q, J = 7.2 Hz, 2H), 1.23 (t, J = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*): δ 201.0, 137.1, 133.0, 128.7, 128.1, 31.9, 8.4. HRMS (ESI, m/z) calculated for $C_9H_{11}O$ $[M+H]^+$: 135.0804, found: 135.0803.



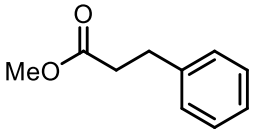
3-Methylbutan-2-one (4v) Prepared from 3-methyl-3-buten-2-one (**1v**, 8.4 mg, 0.1 mmol) according to the general procedure, compound **4v** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless oil (5.5 mg, yield 64%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 2.59 (hept, J = 6.9 Hz, 1H), 2.14 (s, 3H), 1.10 (d, J = 7.0 Hz, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 213.0, 41.8, 27.6, 18.3. HRMS (ESI, m/z) calculated for $C_5H_{11}O$ $[M+H]^+$: 87.0804, found: 87.0808.



Cyclohexanone (4w) Prepared from 2-cyclohexen-1-one (**1w**, 9.6 mg, 0.1 mmol) according to the general procedure, compound **4w** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless oil (7.4 mg, yield 76%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 2.37 – 2.28 (m, 4H), 1.91 – 1.79 (m, 4H), 1.77 – 1.67 (m, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 212.2, 42.1, 27.1, 25.1. HRMS (ESI, m/z) calculated for $C_6H_{11}O$ $[M+H]^+$: 99.0804, found: 99.0809.

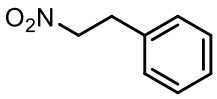


3-Methylcyclohexan-1-one (4x) Prepared from 3-methyl-2-cyclohexenone (**1x**, 11.0 mg, 0.1 mmol) according to the general procedure, compound **4x** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless oil (4.8 mg, yield 43%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 2.43 – 2.29 (m, 2H), 2.28 – 2.17 (m, 1H), 2.11 – 1.77 (m, 4H), 1.74 – 1.57 (m, 1H), 1.41 – 1.22 (m, 1H), 1.01 (d, J = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 212.1, 50.1, 41.3, 34.3, 33.5, 25.4, 22.2. HRMS (ESI, m/z) calculated for $C_7H_{13}O$ $[M+H]^+$: 113.0961, found: 113.0963.

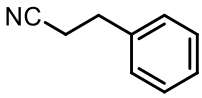


Methyl 3-phenylpropanoate (4y) Prepared from methyl cinnamate (**1y**, 16.2 mg, 0.1 mmol) according to the general procedure, compound **4y** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless oil (6.7 mg, yield 41%). ^{1}H

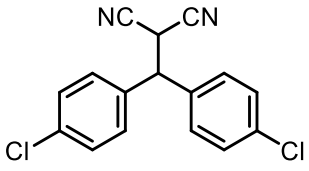
NMR (500 MHz, Acetonitrile-d_3): δ 7.29 – 7.24 (m, 2H), 7.22 – 7.15 (m, 3H), 3.58 (s, 3H), 2.88 (t, J = 7.7 Hz, 2H), 2.59 (t, J = 7.6 Hz, 2H). ^{13}C NMR (126 MHz, Acetonitrile-d_3): δ 174.0, 141.9, 129.4, 129.3, 127.1, 52.0, 36.1, 31.5. HRMS (ESI, m/z) calculated for $C_{10}H_{13}O_2$ $[M+H]^+$: 165.0910, found: 165.0909.



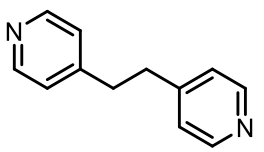
(2-Nitroethyl)benzene (4z) Prepared from trans-*β*-nitrostyrene (**1z**, 14.7 mg, 0.1 mmol) according to the general procedure, compound **4z** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless oil (6.9 mg, yield 46%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.40 – 7.11 (m, 5H), 4.60 (t, J = 7.4 Hz, 2H), 3.31 (t, J = 7.4 Hz, 2H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 135.8, 129.1, 128.7, 127.5, 76.4, 33.5. HRMS (ESI, m/z) calculated for $C_8H_{10}NO_2$ $[M+H]^+$: 152.0706, found: 152.0700.



3-Phenylpropanenitrile (4aa) Prepared from cinnamonitrile (**1aa**, 12.9 mg, 0.1 mmol) according to the general procedure, compound **4aa** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless oil (5.0 mg, yield 38%). ^{1}H NMR (500 MHz, Chloroform-*d*): δ 7.37 – 7.32 (m, 2H), 7.30 – 7.25 (m, 1H), 7.25 – 7.21 (m, 2H), 2.97 (t, 2H), 2.63 (t, J = 7.4 Hz, 2H). ^{13}C NMR (126 MHz, Chloroform-*d*): δ 138.2, 129.0, 128.4, 127.4, 119.3, 31.7, 19.5. HRMS (ESI, m/z) calculated for $C_9H_{10}N$ $[M+H]^+$: 132.0808, found: 132.0808.

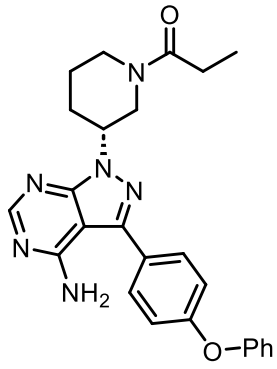


2-(Bis(4-chlorophenyl)methyl)malononitrile (4ab) Prepared from 2-(bis(4-chlorophenyl)methylene)malononitrile (**1ab**, 30.0 mg, 0.1 mmol) according to the general procedure, compound **4ab** was purified by silica gel column chromatography (hexane/ethyl acetate 10:1) and obtained as a colorless solid (12.9 mg, yield 43%). ^{1}H NMR (500 MHz, Acetone-d_6): δ 7.58 – 7.55 (m, 4H), 7.49 – 7.46 (m, 4H), 5.54 (d, J = 9.6 Hz, 1H), 5.10 (d, J = 9.5 Hz, 1H). ^{13}C NMR (126 MHz, Acetone-d_6): δ 137.9, 134.7, 130.7, 130.1, 113.7, 50.5, 28.9. HRMS (ESI, m/z) calculated for $C_{16}H_{10}Cl_2N_2$ $[M+H]^+$: 301.0294, found: 301.0292.

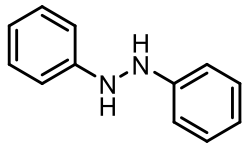


1,2-Di(pyridin-4-yl)ethane (4ac) Prepared from 1,2-di(pyridin-4-yl)ethene (**1ac**, 18.2 mg, 0.1 mmol) according to the general procedure, compound **4ac** was purified

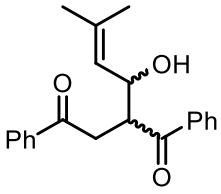
by silica gel column chromatography (ethyl acetate/methanol 6:1) and obtained as a colorless solid (17.3 mg, yield 94%). ^{1}H NMR (500 MHz, Acetone-d_6 / DMSO-d_6): δ 8.51 – 8.40 (m, 4H), 7.27 – 7.18 (m, 4H), 2.97 (s, 4H). ^{13}C NMR (126 MHz, Acetone-d_6 / DMSO-d_6): δ 150.4, 150.2, 124.5, 35.6. HRMS (ESI, m/z) calculated for $C_{12}H_{12}N_2$ $[M+H]^+$: 185.1079, found: 185.1084.



(R)-1-(3-(4-amino-3-(4-phenoxyphenyl)-1H-pyrazolo[3,4-d]pyrimidin-1-yl)piperidin-1-yl)propan-1-one (4ad) Prepared from (R)-1-(3-(4-amino-3-(4-phenoxyphenyl)-1H-pyrazolo[3,4-d]pyrimidin-1-yl)piperidin-1-yl)prop-2-en-1-one (**1ad**, 44.1 mg, 0.1 mmol) according to the general procedure, compound **4ad** was purified by silica gel column chromatography (DCM/methanol 10:1) and obtained as a colorless solid (24.8 mg, yield 56%). ^{1}H NMR (600 MHz, Chloroform-*d*): δ 8.31 (d, *J* = 24.3 Hz, 1H), 7.68 – 7.58 (m, 2H), 7.44 – 7.31 (m, 2H), 7.20 – 7.01 (m, 5H), 4.89 – 4.75 (m, 1.5H), 4.60 (d, *J* = 13.1 Hz, 0.5H), 4.08 – 4.00 (m, 0.5H), 3.88 (d, *J* = 13.6 Hz, 0.5H), 3.64 (dd, *J* = 13.2, 10.7 Hz, 0.5H), 3.32 – 3.18 (m, 0.5H), 3.11 (td, *J* = 13.1, 2.8 Hz, 0.5H), 2.72 (td, *J* = 12.8, 3.0 Hz, 0.5H), 2.45 – 2.14 (m, 4H), 2.00 – 1.87 (m, 1H), 1.73 – 1.61 (m, 1H), 1.14 (dt, *J* = 12.3, 7.4 Hz, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*): δ 172.5, 158.6, 158.5, 158.1, 156.4, 156.3, 155.8, 155.6, 154.2, 154.1, 144.0, 143.9, 130.0, 127.9, 127.6, 124.1, 119.6, 119.1, 98.6, 53.4, 52.5, 49.8, 45.8, 45.5, 41.7, 30.3, 30.0, 26.6, 25.2, 24.1, 9.5. HRMS (ESI, m/z) calculated for $C_{25}H_{26}N_6O_2$ $[M+H]^+$: 443.2195, found: 443.2194. Spectroscopic data correspond to those reported in the literature.[7]

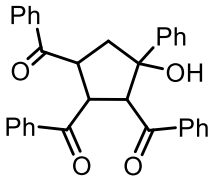


1,2-Diphenylhydrazine (4ae) Prepared from azobenzene (**1ae**, 18.2 mg, 0.1 mmol) according to the general procedure, compound **4ae** was purified by silica gel column chromatography (hexane/ethyl acetate 15:1) and obtained as a colorless solid (15.8 mg, yield 86%). ^{1}H NMR (500 MHz, Acetonitrile-d_3): δ 7.19 – 7.14 (m, 4H), 6.82 – 6.78 (m, 4H), 6.77 – 6.71 (m, 2H), 6.35 (s, 2H). ^{13}C NMR (126 MHz, Acetonitrile-d_3): δ 150.6, 130.0, 119.8, 113.0. HRMS (ESI, m/z) calculated for $C_{12}H_{12}N_2$ $[M+H]^+$:185.1079, found:185.1084.



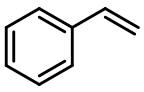
2-(1-Hydroxy-3-methylbut-2-en-1-yl)-1,4-diphenylbutane-1,4-dione (5a)

Prepared from 1,2-dibenzoylethylene (**1a**, 23.6 mg, 0.1 mmol) according to the general procedure, using anthraquinone as a photocatalyst, compound **5a** was purified by silica gel column chromatography (hexane/ethyl acetate 5:1) and obtained as a colorless solid (21.6 mg, yield 67%). ^{1}H NMR (500 MHz, Acetonitrile-d_3): δ 7.54 – 7.47 (m, 2H), 7.41 – 7.21 (m, 5H), 7.06 – 6.93 (m, 3H), 6.04 (t, J = 7.3 Hz, 1H) (5.49 (t, J = 8.0 Hz, 1H)), 5.37 – 5.27 (m, 1H), 4.59 (d, J = 7.3 Hz, 2H) (4.55 (d, J = 7.3 Hz, 2H)), 3.24 (d, J = 7.3 Hz, 2H) (3.20 (d, J = 8.0 Hz, 2H)), 1.77 – 1.72 (m, 3H), 1.72 – 1.67 (m, 3H). ^{13}C NMR (126 MHz, Acetonitrile-d_3): δ 172.3, 171.9, 157.8, 154.1, 151.6, 140.1, 139.9, 135.7, 134.7, 130.6, 130.5, 129.9, 129.8, 129.6, 129.4, 126.7, 123.6, 123.0, 119.6, 119.6, 118.9, 116.8, 111.0, 108.8, 62.3, 33.7, 32.2, 25.8, 18.1. HRMS (ESI, m/z) calculated for $C_{21}H_{23}O_3$ $[M+H]^+$: 323.1647, found: 323.1653.



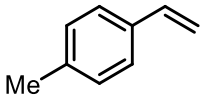
(4-Hydroxy-4-phenylcyclopentane-1,2,3-triyl)tris(phenylmethanone) (8)

Prepared from 1,2-dibenzoylethylene (**1a**, 23.6 mg, 0.1 mmol) according to the general procedure, using 2,4,6-triphenylpyrylium as a photocatalyst, compound **8** was purified by silica gel column chromatography (hexane/ethyl acetate 5:1) and obtained as a colorless solid. ^{1}H NMR (500 MHz, Acetonitrile-d_3 / DMSO-d_6): δ 8.00 – 7.95 (m, 2H), 7.90 – 7.85 (m, 2H), 7.57 – 7.47 (m, 2H), 7.45 – 7.37 (m, 6H), 7.35 – 7.25 (m, 3H), 7.17 – 7.12 (m, 2H), 7.11 – 7.04 (m, 3H), 5.59 (dd, J = 10.3, 8.0 Hz, 1H), 5.21 (s, 1H), 4.69 (d, J = 9.5 Hz, 1H), 4.36 (ddd, J = 11.1, 8.0, 5.5 Hz, 1H), 3.12 – 3.05 (m, 1H), 2.28 (dd, J = 14.0, 5.5 Hz, 1H). ^{13}C NMR (126 MHz, Acetonitrile-d_3/ DMSO-d_6): δ 202.6, 200.5, 199.9, 145.7, 138.2, 137.5, 136.7, 134.0, 133.9, 133.1, 129.3, 129.3, 129.3, 129.2, 128.6, 128.5, 128.5, 127.4, 126.0, 84.0, 62.8, 49.8, 49.8, 49.1. HRMS (ESI, m/z) calculated for $C_{32}H_{27}O_4$ $[M+H]^+$: 475.1904, found: 475.1912.

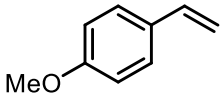


Styrene (10a) Prepared from ethynylbenzene (**9a**, 10.2 mg, 0.1 mmol) according to the general procedure, compound **10a** was purified by silica gel column chromatography (hexane) and obtained as a colorless oil (6.7 mg, yield 64%). ^{1}H NMR (500 MHz, Chloroform-d): δ 7.44 – 7.39 (m, 2H), 7.36 – 7.30 (m, 2H), 7.28 – 7.22 (m, 1H), 6.72 (dd, J = 17.6, 10.9 Hz, 1H), 5.75 (dd, J = 17.6, 1.0

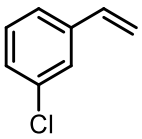
Hz, 1H), 5.24 (dd, *J* = 10.9, 1.0 Hz, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*): δ 137.7, 137.0, 128.7, 127.9, 126.4, 113.9. HRMS (ESI, m/z) calculated for C_8H_9 $[M+H]^+$: 105.0699, found: 105.0698.



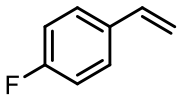
1-Methyl-4-vinylbenzene (10b) Prepared from 1-ethynyl-4-methylbenzene (**9b**, 11.6 mg, 0.1 mmol) according to the general procedure, compound **10b** was purified by silica gel column chromatography (hexane) and obtained as a colorless oil (7.2 mg, yield 61%). ^{1}H NMR (500 MHz, Chloroform-*d*): δ 7.35 – 7.30 (m, 2H), 7.17 – 7.12 (m, 2H), 6.70 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.71 (dd, *J* = 17.6, 1.0 Hz, 1H), 5.20 (dd, *J* = 10.9, 1.0 Hz, 1H), 2.35 (s, 3H). ^{13}C NMR (126 MHz, Chloroform-*d*): δ 137.8, 136.9, 135.0, 129.4, 126.3, 112.9, 21.3. HRMS (ESI, m/z) calculated for C_9H_{11} $[M+H]^+$: 119.0855, found: 119.0867.



1-Methoxy-4-vinylbenzene (10c) Prepared from 1-ethynyl-4-methoxybenzene (**9c**, 13.2 mg, 0.1 mmol) according to the general procedure, compound **10c** was purified by silica gel column chromatography (hexane/ethyl acetate 30:1) and obtained as a colorless oil (9.6 mg, yield 72%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.44 – 7.31 (m, 2H), 6.95 – 6.81 (m, 2H), 6.68 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.63 (d, *J* = 17.6 Hz, 1H), 5.15 (d, *J* = 10.9 Hz, 1H), 3.82 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 159.5, 136.3, 130.5, 127.5, 114.0, 111.7, 55.4. HRMS (ESI, m/z) calculated for $C_9H_{11}O$ $[M+H]^+$: 135.0804, found: 135.0804.

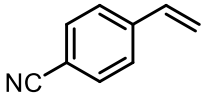


1-Chloro-3-vinylbenzene (10d) Prepared from 1-chloro-3-ethynylbenzene (**9d**, 13.7 mg, 0.1 mmol) according to the general procedure, compound **10d** was purified by silica gel column chromatography (hexane/ethyl acetate 30:1) and obtained as a colorless oil (12.6 mg, yield 91%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.39 (s, 1H), 7.31 – 7.16 (m, 3H), 6.65 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.76 (d, *J* = 17.6 Hz, 1H), 5.30 (d, *J* = 10.9 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 139.5, 135.7, 134.6, 129.9, 127.9, 126.3, 124.6, 115.5. HRMS (ESI, m/z) calculated for C_8H_8Cl $[M+H]^+$: 139.0309, found: 139.0310.

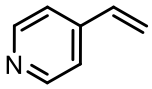


1-Fluoro-4-vinylbenzene (10e) Prepared from 1-ethynyl-4-fluorobenzene (**9e**, 12.0 mg, 0.1 mmol) according to the general procedure, compound **10e** was purified by silica gel column

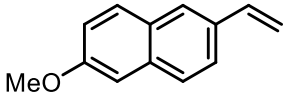
chromatography (hexane/ethyl acetate 30:1) and obtained as a colorless oil (10.4 mg, yield 86%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.44 – 7.32 (m, 2H), 7.10 – 6.94 (m, 2H), 6.69 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.67 (d, *J* = 17.6 Hz, 1H), 5.23 (d, *J* = 10.9 Hz, 1H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 162.6 (d, *J* = 246.7 Hz), 135.8, 133.9 (d, *J* = 3.3 Hz), 127.9 (d, *J* = 8.0 Hz), 115.5 (d, *J* = 21.6 Hz), 113.7. ^{19}F NMR (377 MHz, Chloroform-*d*) δ -114.34. HRMS (ESI, m/z) calculated for C_8H_8F $[M+H]^+$: 123.0605, found: 123.0601.



4-Vinylbenzonitrile (10f) Prepared from 4-ethynylbenzonitrile (**9f**, 12.7 mg, 0.1 mmol) according to the general procedure, compound **10f** was purified by silica gel column chromatography (hexane/ethyl acetate 30:1) and obtained as a colorless oil (9.6 mg, yield 74%). ^{1}H NMR (600 MHz, Chloroform-*d*): δ 7.65 – 7.57 (m, 2H), 7.51 – 7.45 (m, 2H), 6.72 (dd, *J* = 17.6, 10.9 Hz, 1H), 5.87 (d, *J* = 17.6 Hz, 1H), 5.45 (d, *J* = 10.9 Hz, 1H). ^{13}C NMR (151 MHz, Chloroform-*d*): δ 142.0, 135.5, 132.5, 126.9, 119.0, 117.9, 111.3. HRMS (ESI, m/z) calculated for C_9H_8N $[M+H]^+$: 130.0651, found: 130.0650.

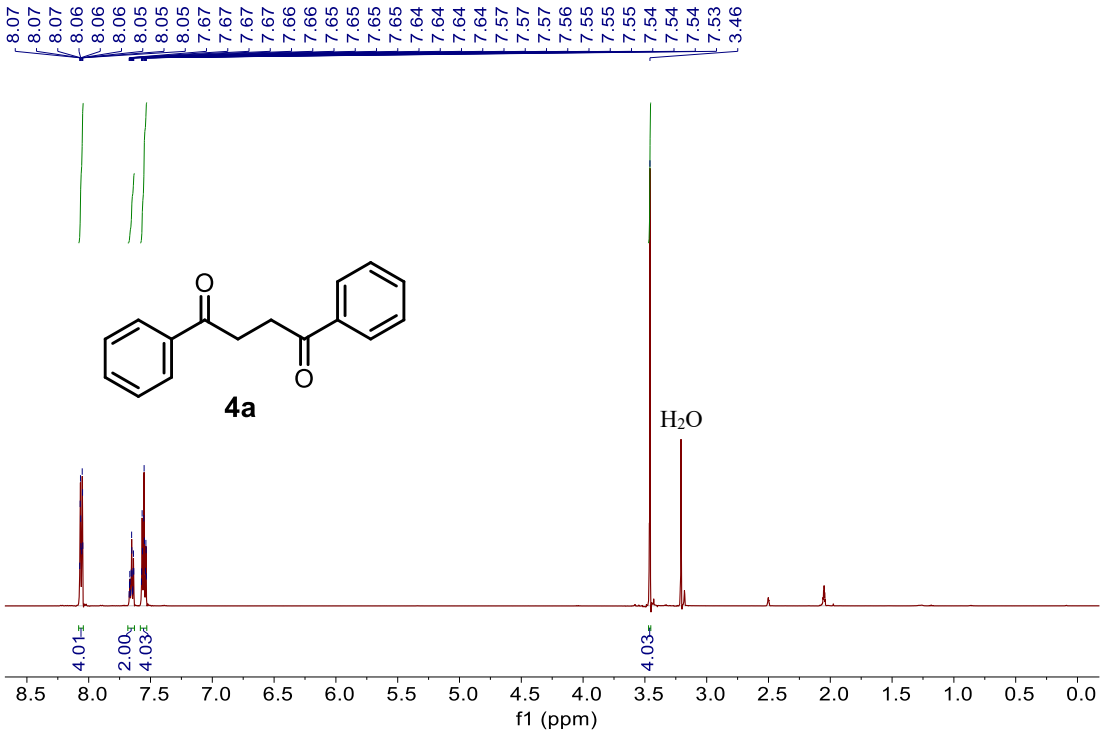


3-Vinylpyridine (10g) Prepared from 4-ethynylpyridine (**9g**, 10.3 mg, 0.1 mmol) according to the general procedure, compound **10g** was purified by silica gel column chromatography (hexane/ethyl acetate 10:1) and obtained as a light yellow oil (8.2 mg, yield 78%). ^{1}H NMR (500 MHz, Chloroform-*d*): δ 8.62 – 8.51 (m, 2H), 7.29 – 7.23 (m, 2H), 6.66 (ddd, *J* = 17.0, 10.6, 7.8 Hz, 1H), 6.01 – 5.92 (m, 1H), 5.51 – 5.45 (m, 1H). ^{13}C NMR (126 MHz, Chloroform-*d*): δ 150.3, 144.8, 134.9, 120.9, 118.8. HRMS (ESI, m/z) calculated for C_7H_8N $[M+H]^+$: 106.0651, found: 106.0652.

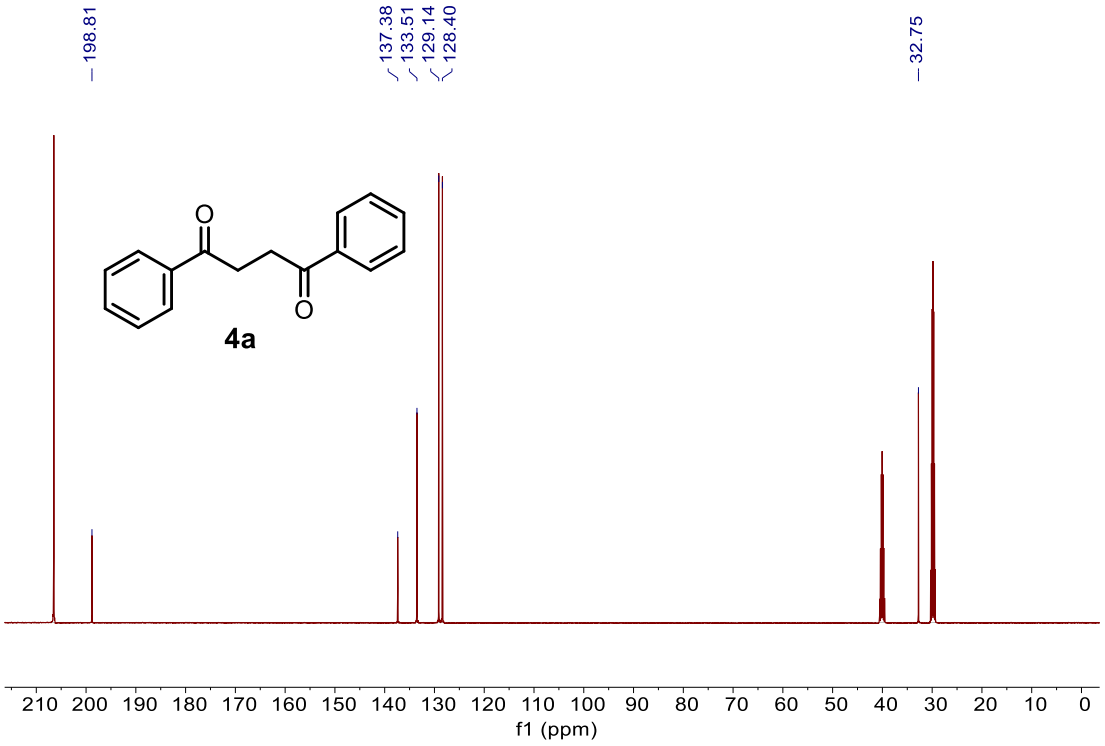


2-Methoxy-6-vinylnaphthalene (10h) Prepared from 2-ethynyl-6-methoxynaphthalene (**9h**, 18.2 mg, 0.1 mmol) according to the general procedure, compound **10h** was purified by silica gel column chromatography (hexane/ethyl acetate 30:1) and obtained as a colorless solid (12.3 mg, yield 67%). ^{1}H NMR (400 MHz, Chloroform-*d*): δ 7.77 – 7.57 (m, 4H), 7.14 (m, 2H), 6.87 (dd, *J* = 17.5, 10.9 Hz, 1H), 5.84 (d, *J* = 17.6 Hz, 1H), 5.30 (d, *J* = 10.8 Hz, 1H), 3.93 (s, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*): δ 157.9, 137.1, 134.4, 133.1, 129.7, 129.1, 127.1, 126.3, 123.9, 119.1, 113.2, 106.0, 55.4. HRMS (ESI, m/z) calculated for $C_{13}H_{13}O$ $[M+H]^+$: 185.0961, found: 185.0977.

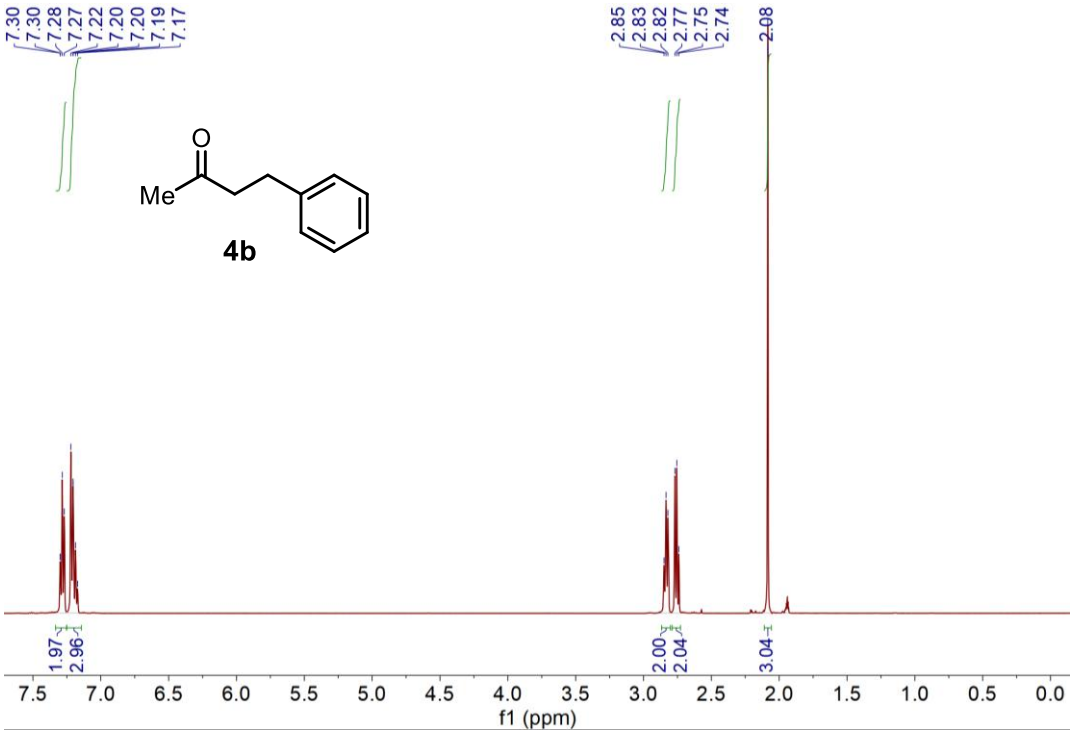
9. NMR Spectra



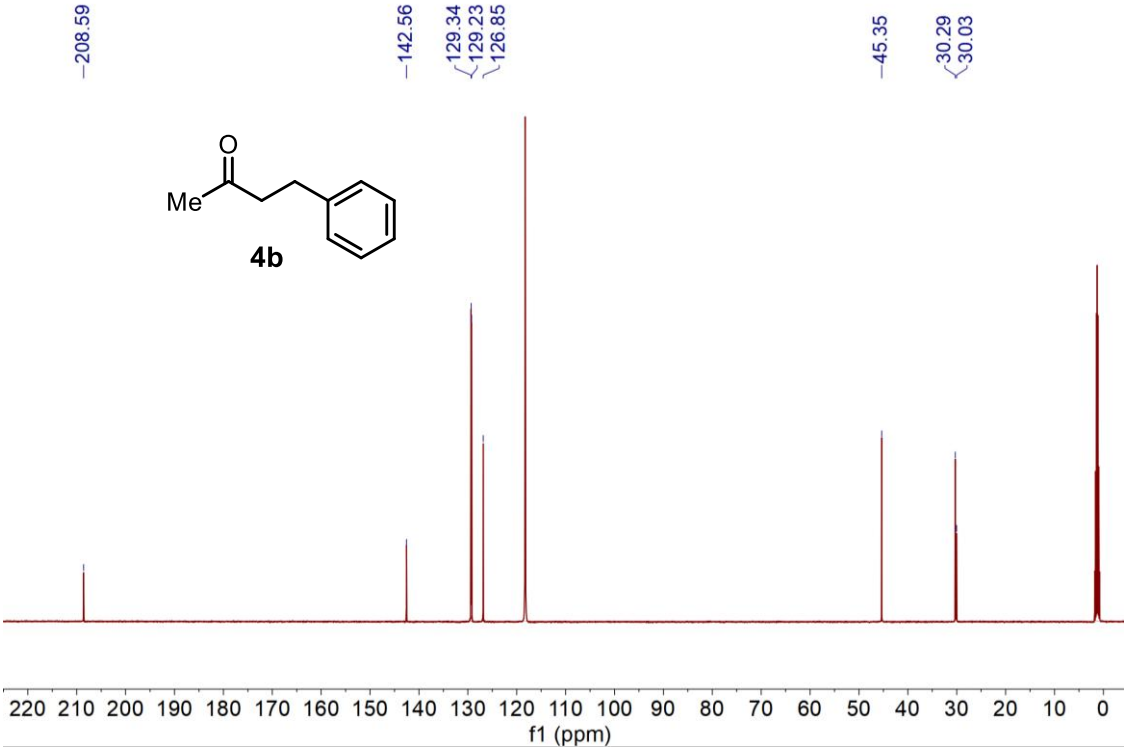
^{1}H NMR (500 MHz, Acetone-d_6 / DMSO-d_6)



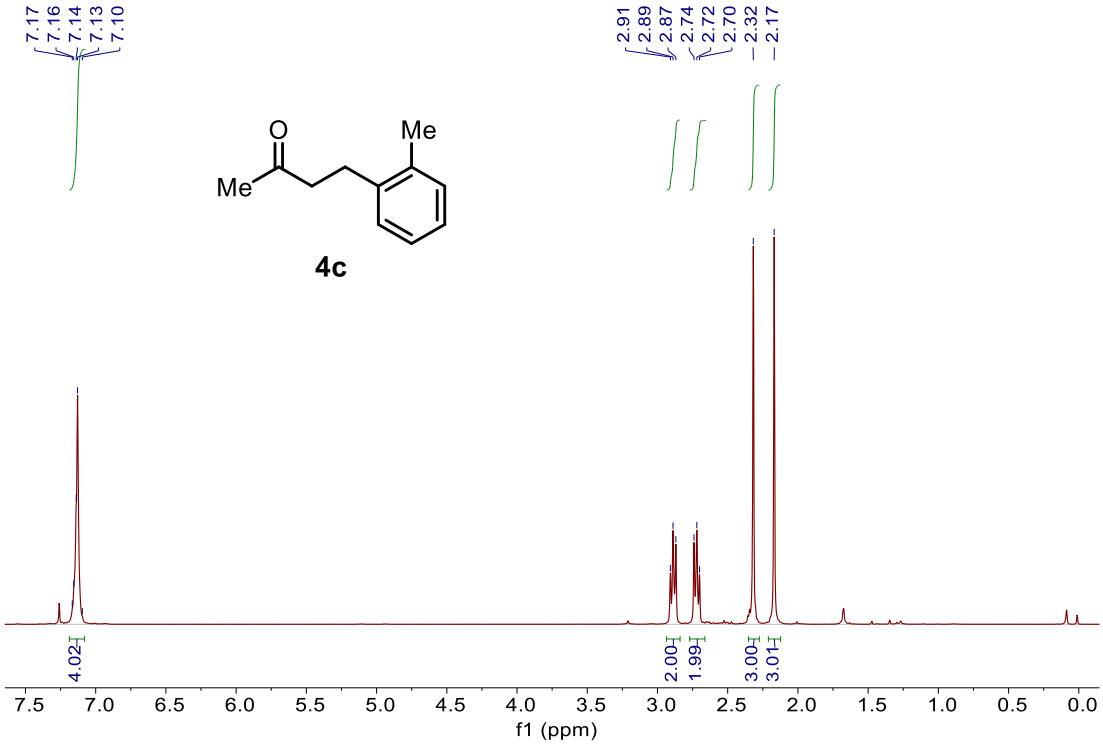
^{13}C NMR (126 MHz, Acetone-d_6 / DMSO-d_6)



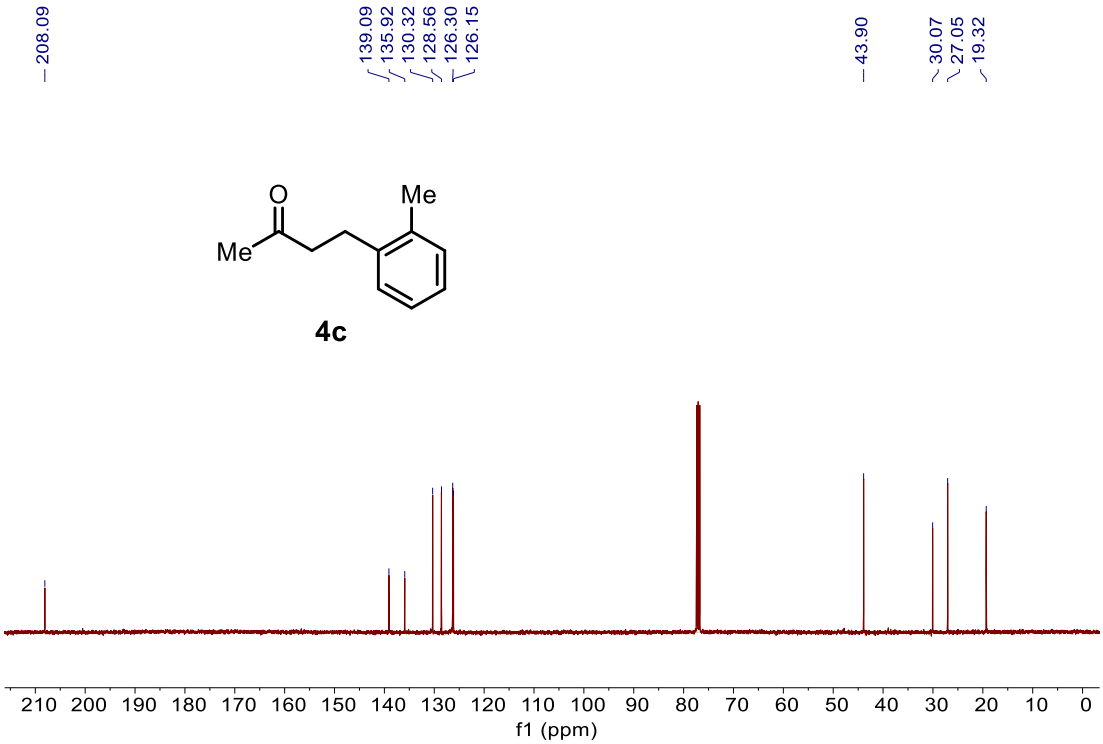
^{1}H NMR (500 MHz, CD_3CN)



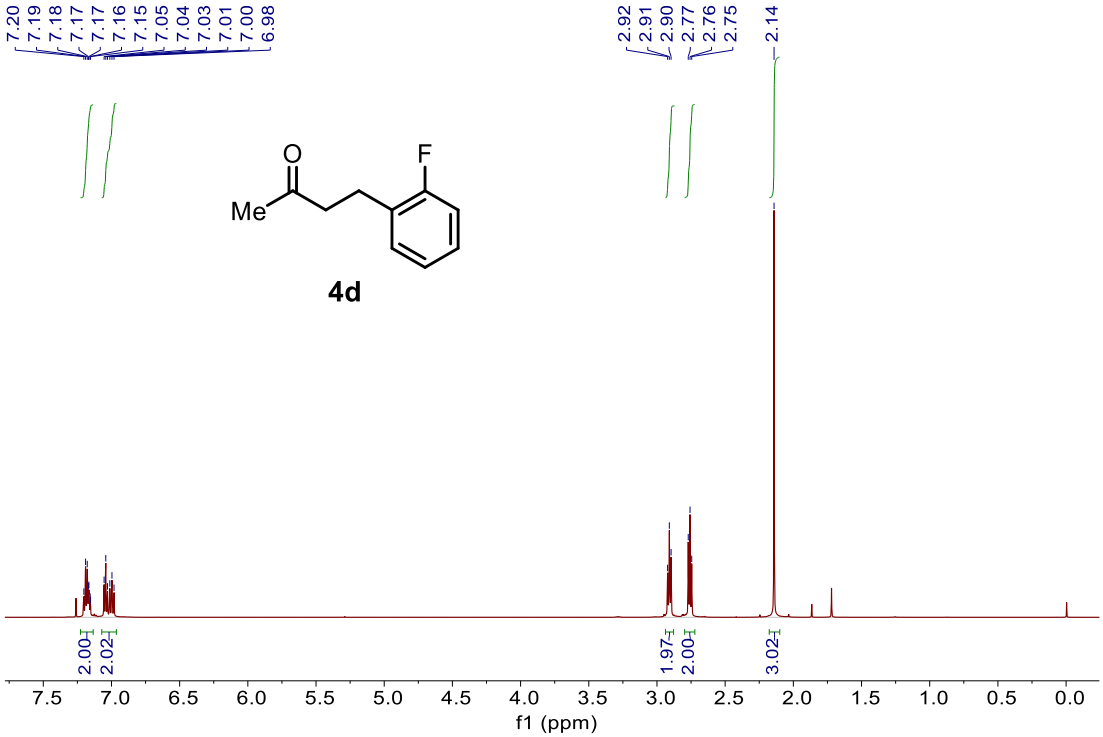
^{13}C NMR (126 MHz, CD_3CN)



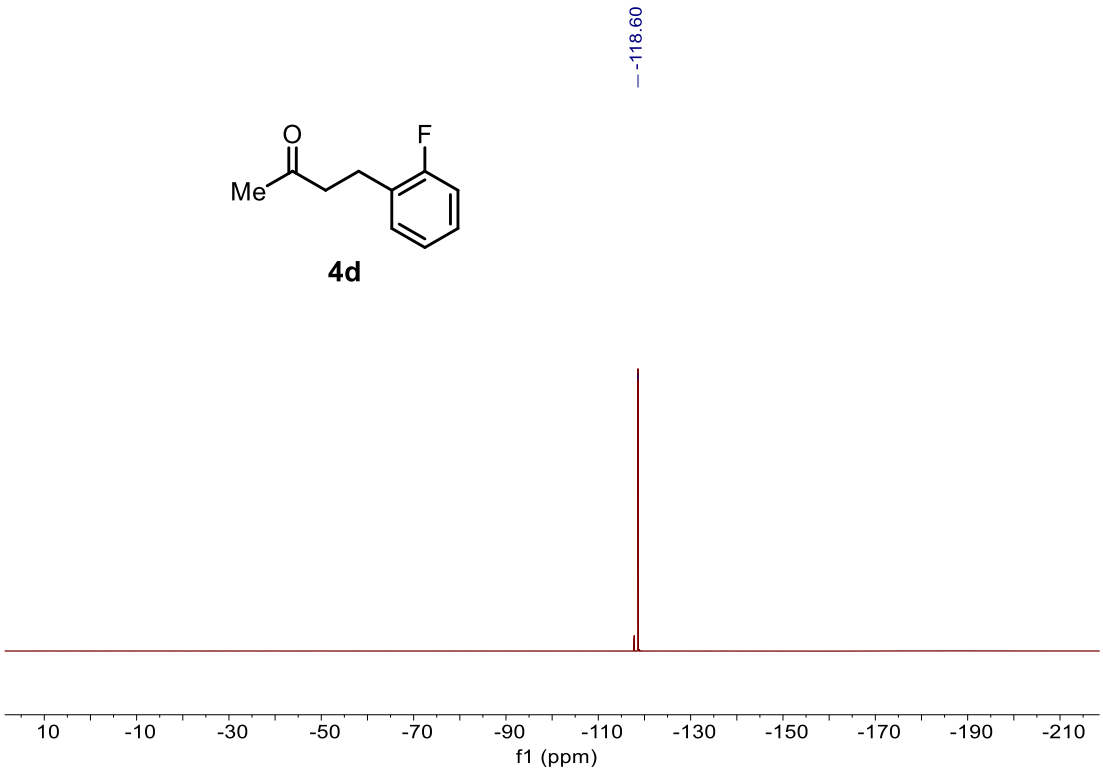
^{1}H NMR (400 MHz, Chloroform-*d*)



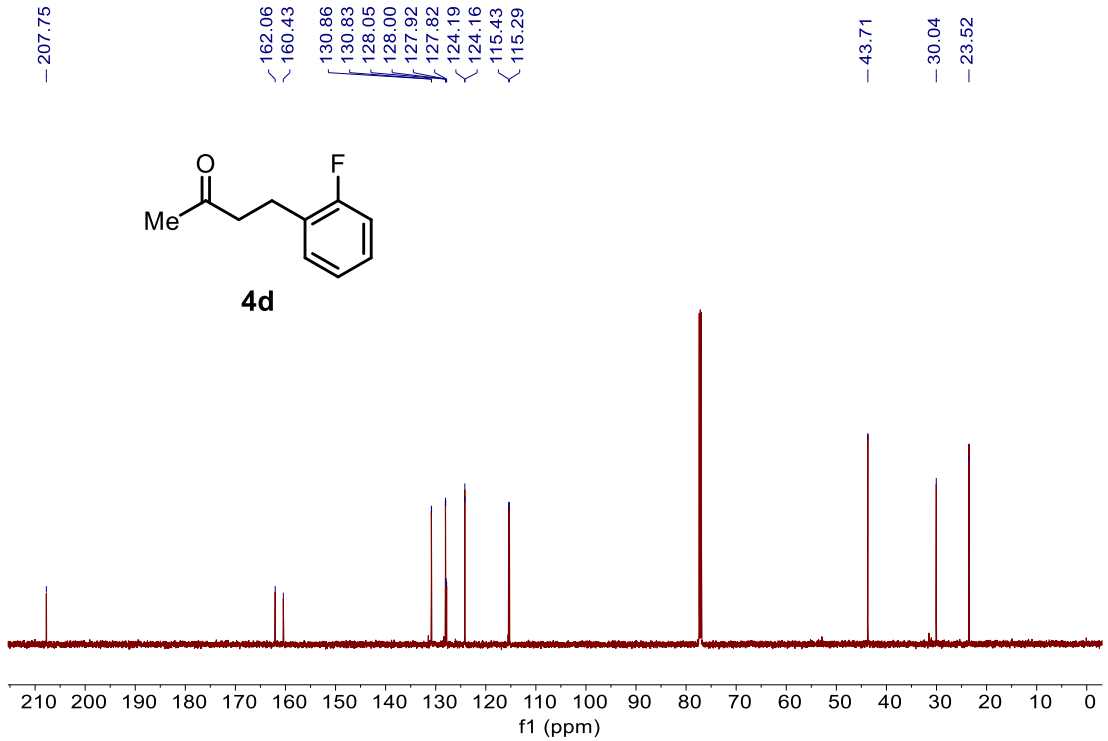
^{13}C NMR (101 MHz, Chloroform-*d*)



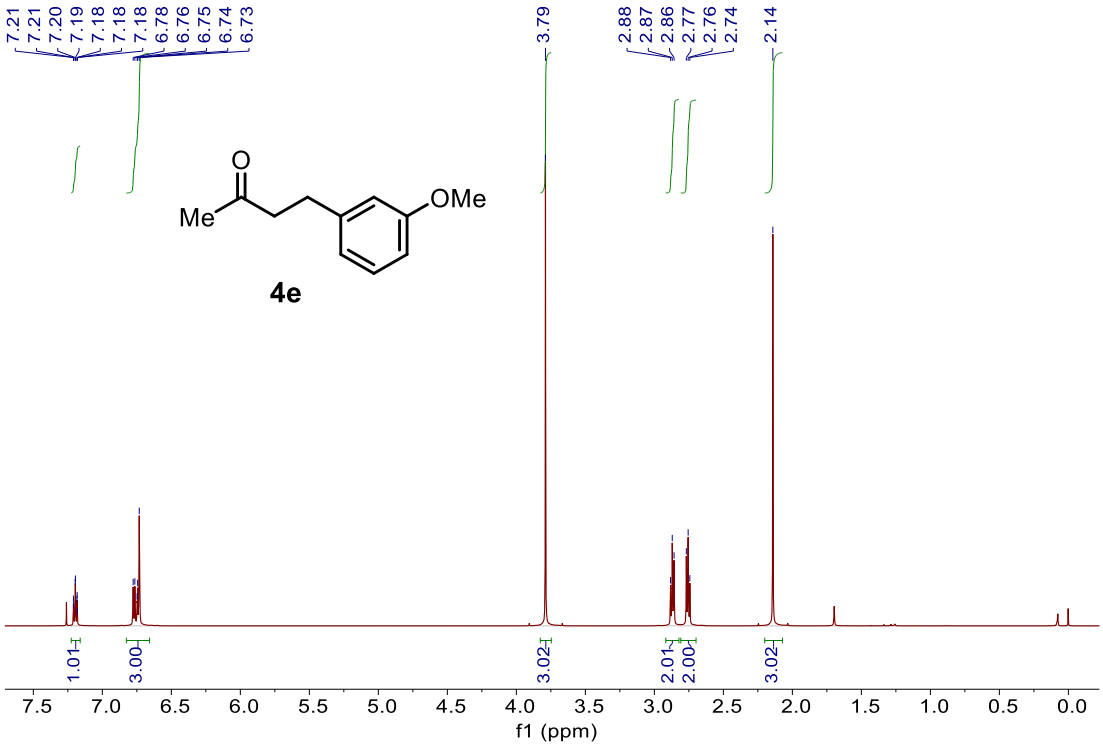
^{1}H NMR (600 MHz, Chloroform-*d*)



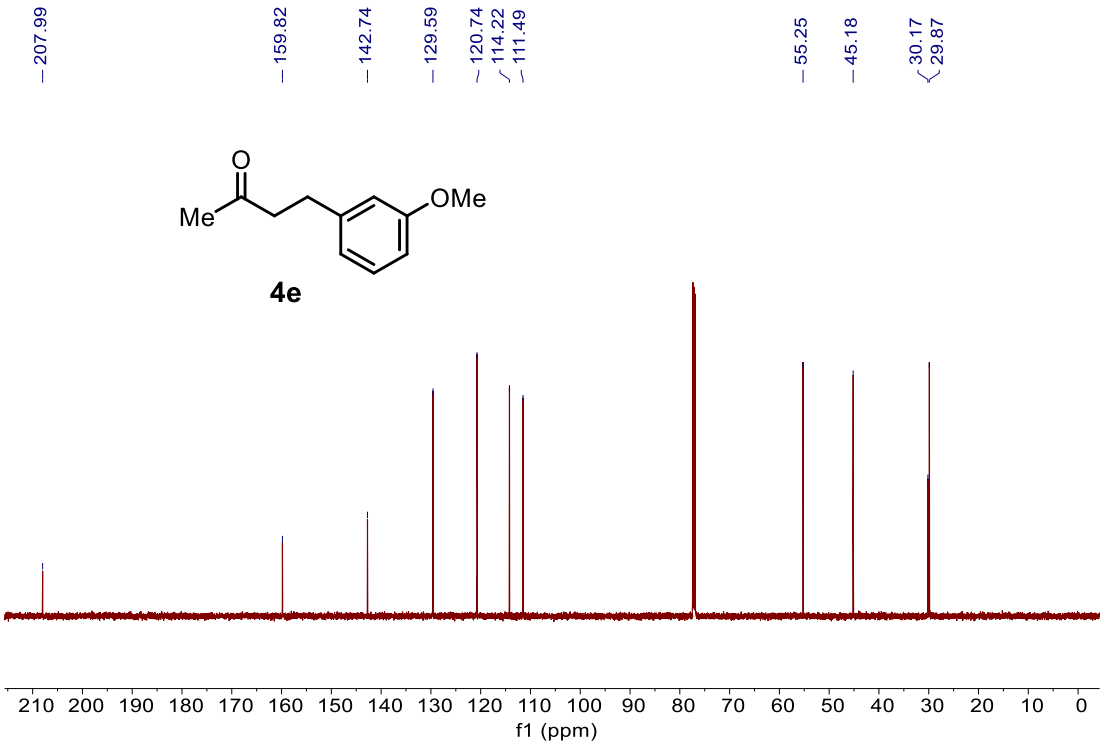
^{19}F NMR (565 MHz, Chloroform-*d*)



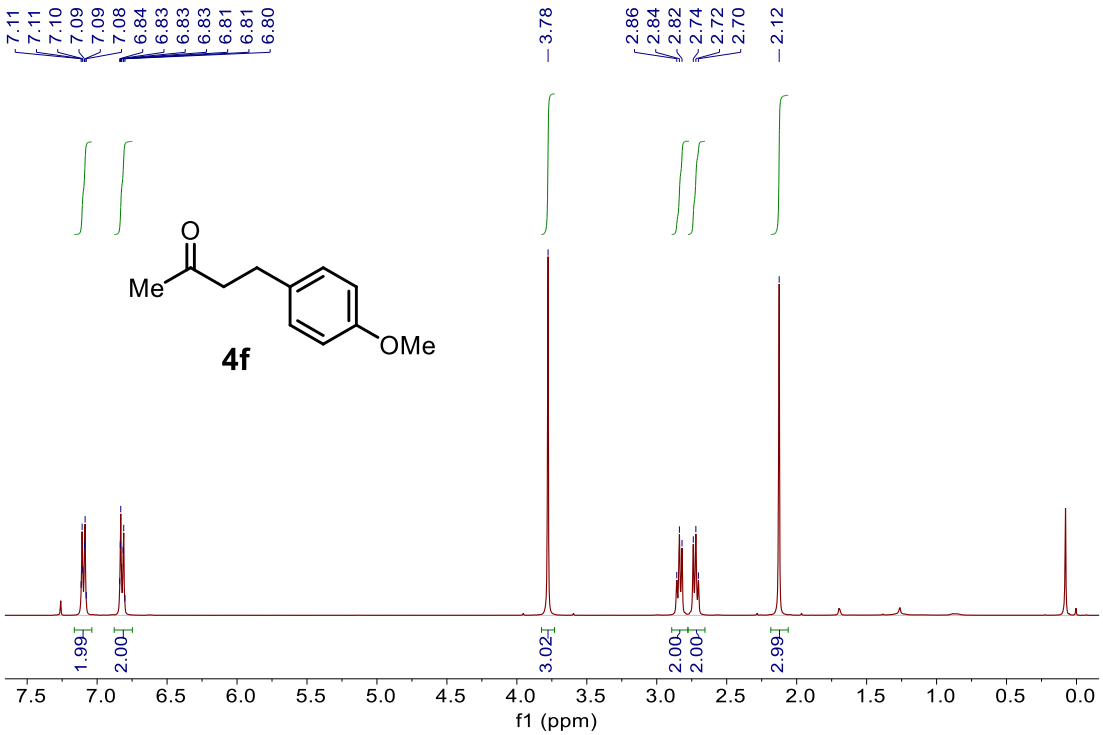
^{13}C NMR (151 MHz, Chloroform-*d*)



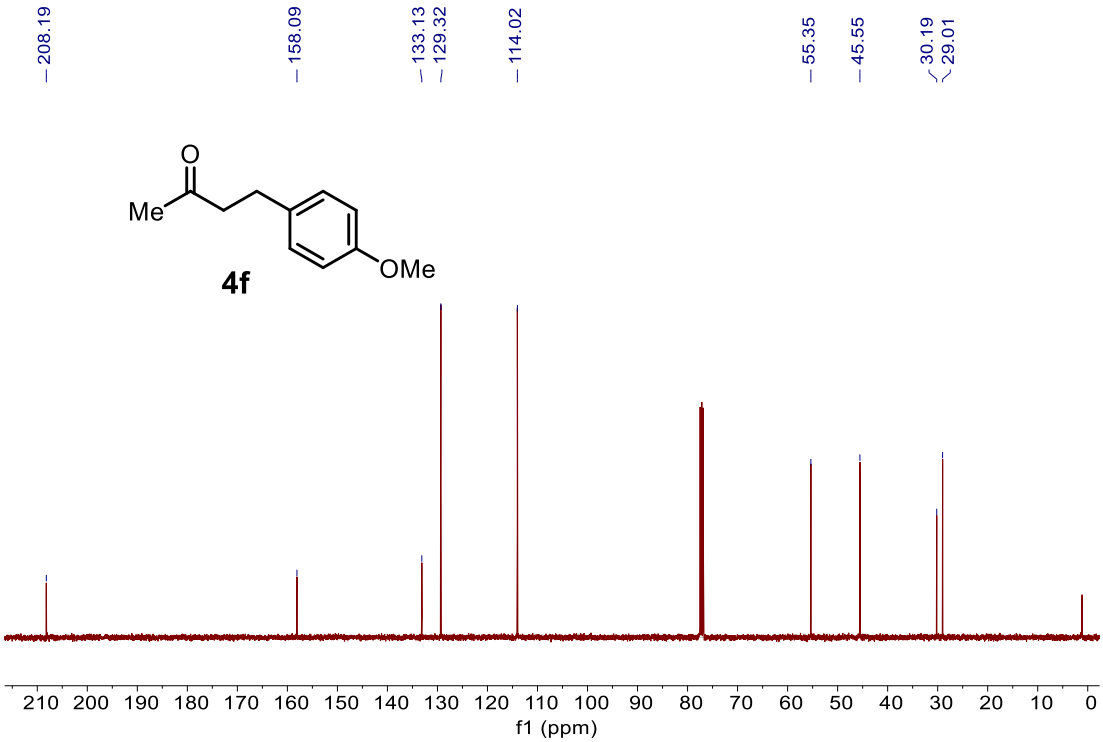
^{1}H NMR (600 MHz, Chloroform-*d*)



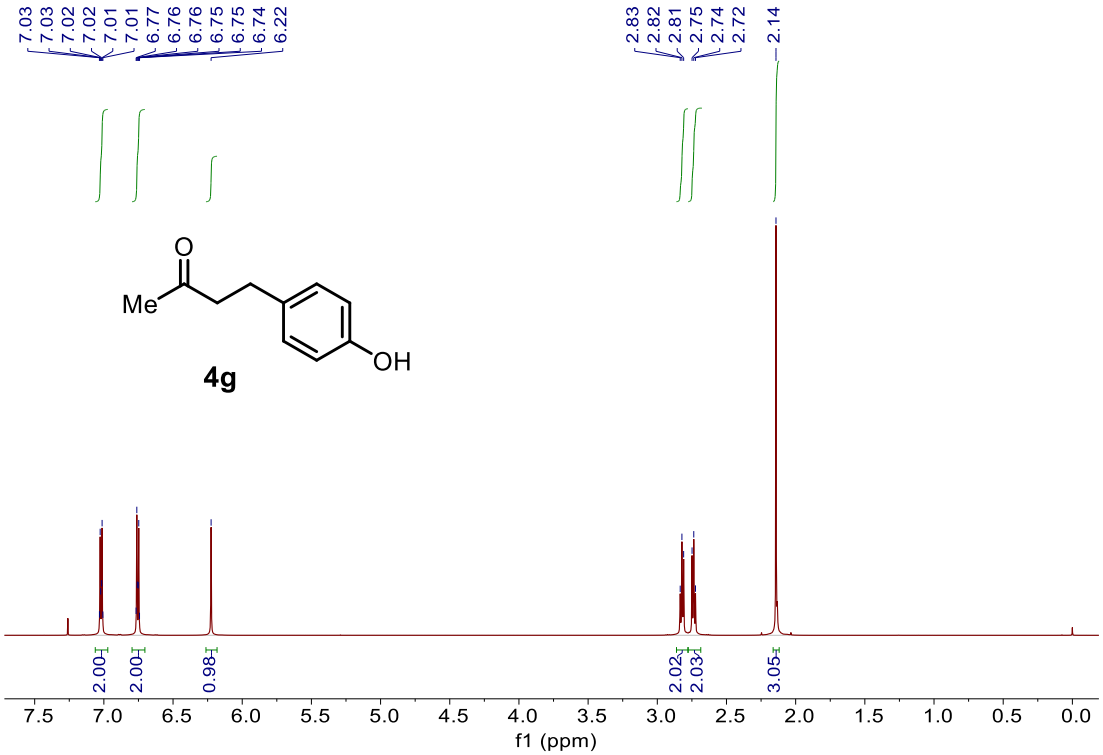
^{13}C NMR (151 MHz, Chloroform-*d*)



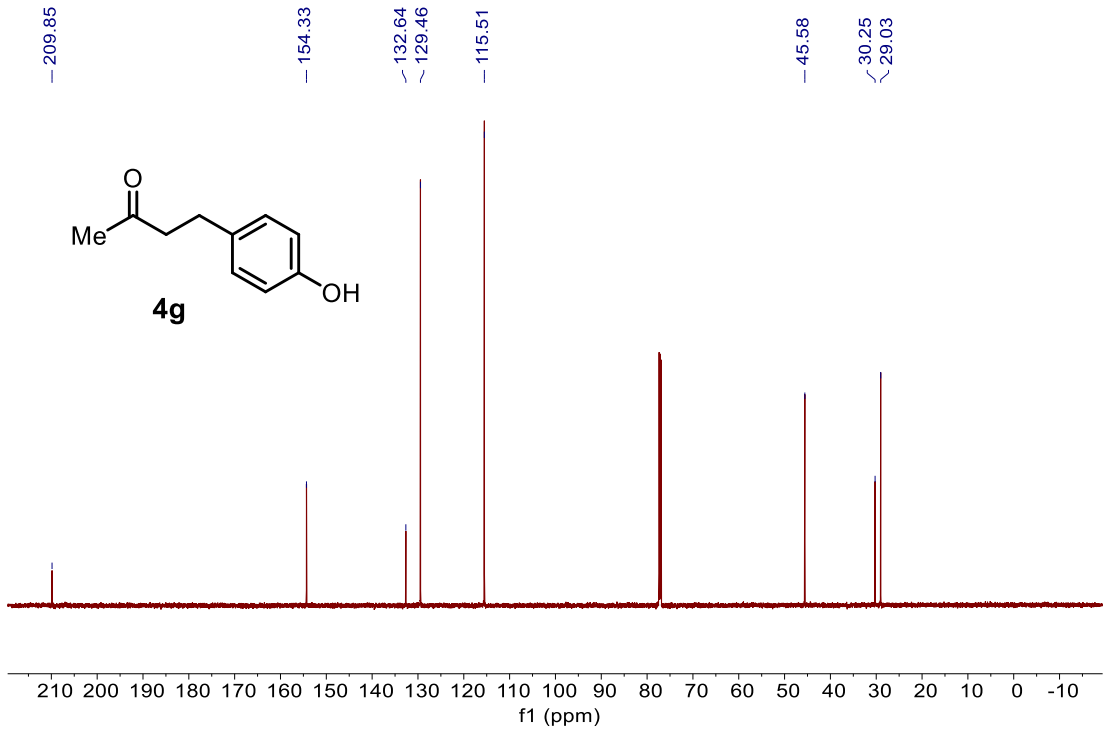
^{1}H NMR (400 MHz, Chloroform-*d*)



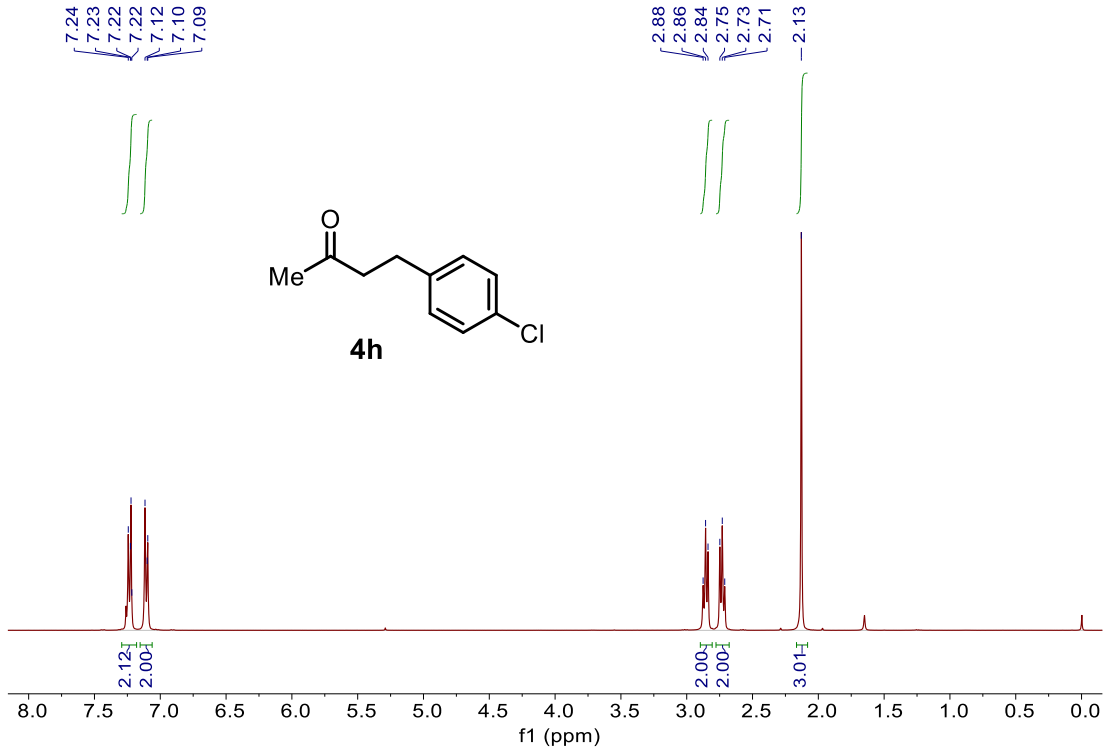
^{13}C NMR (101 MHz, Chloroform-*d*)



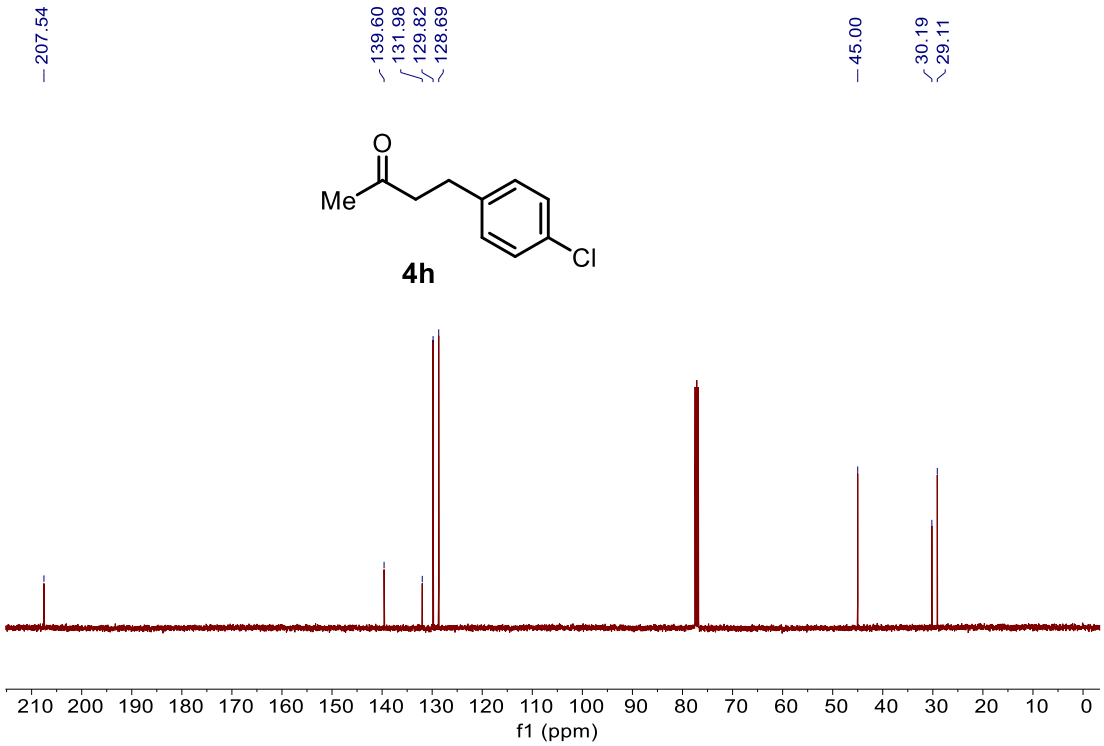
^{1}H NMR (600 MHz, Chloroform-*d*)



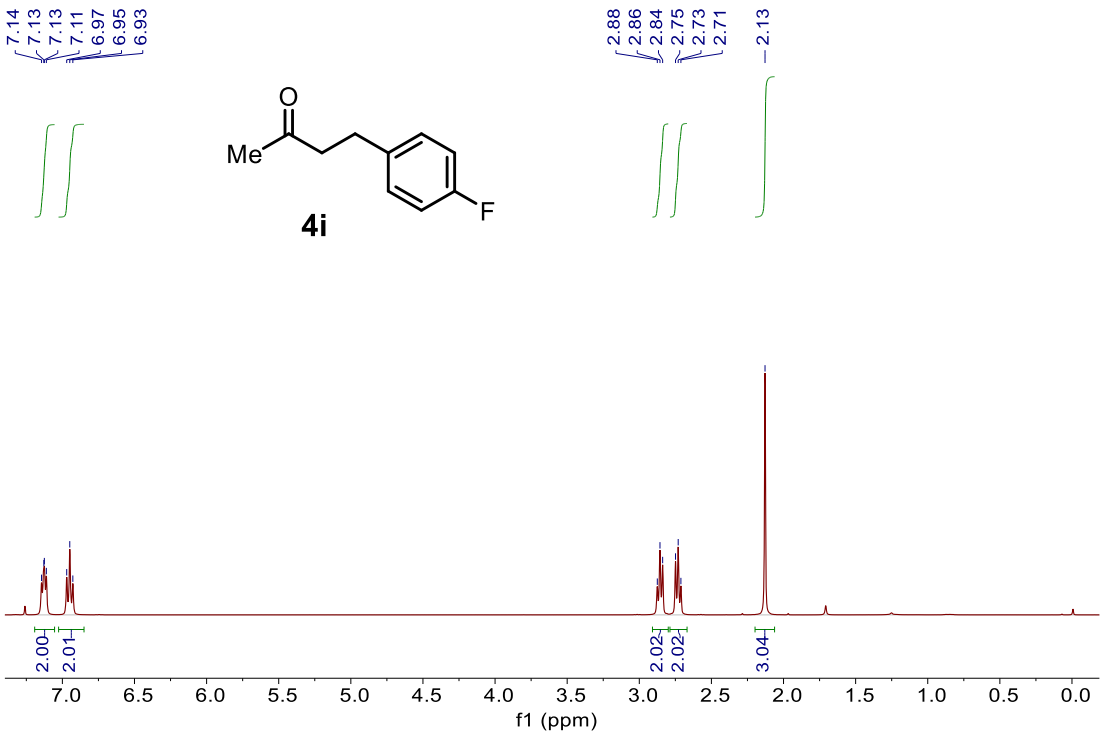
^{13}C NMR (151 MHz, Chloroform-*d*)



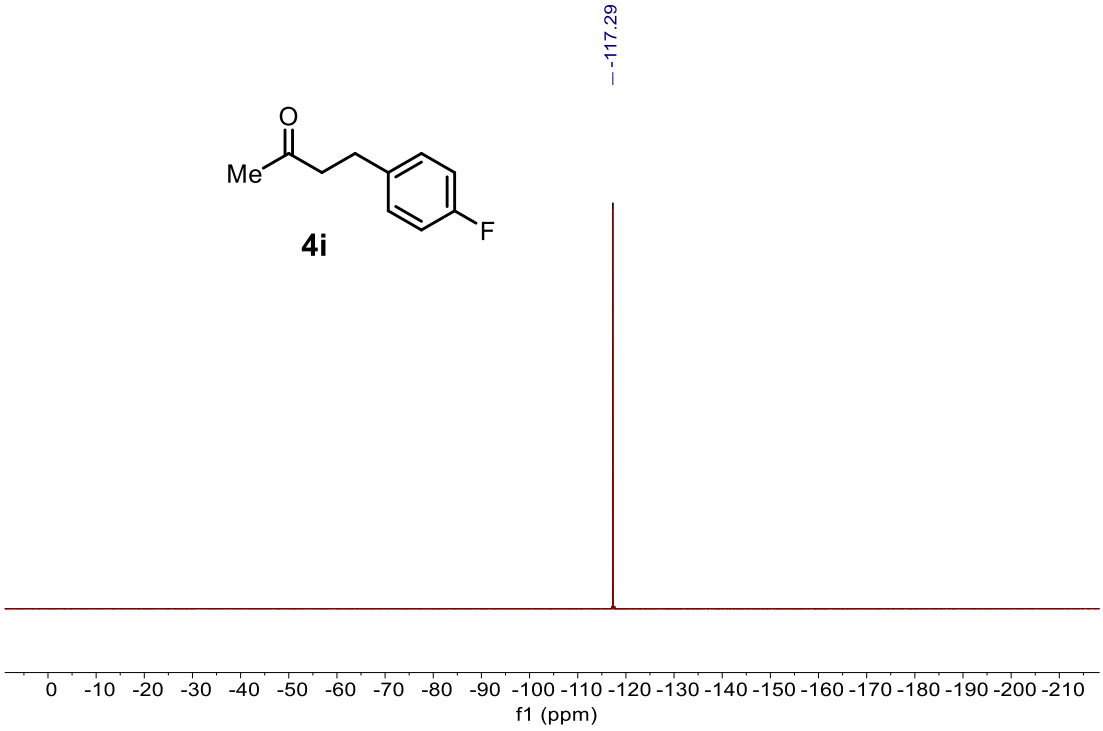
^{1}H NMR (400 MHz, Chloroform-*d*)



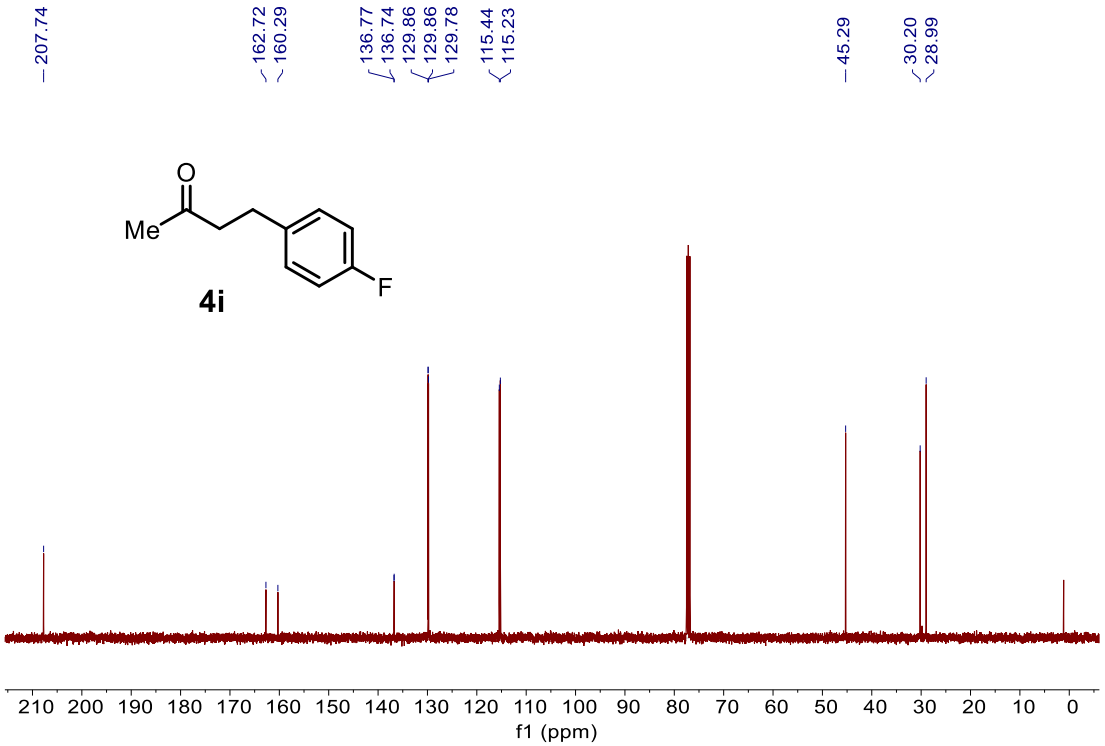
^{13}C NMR (101 MHz, Chloroform-*d*)



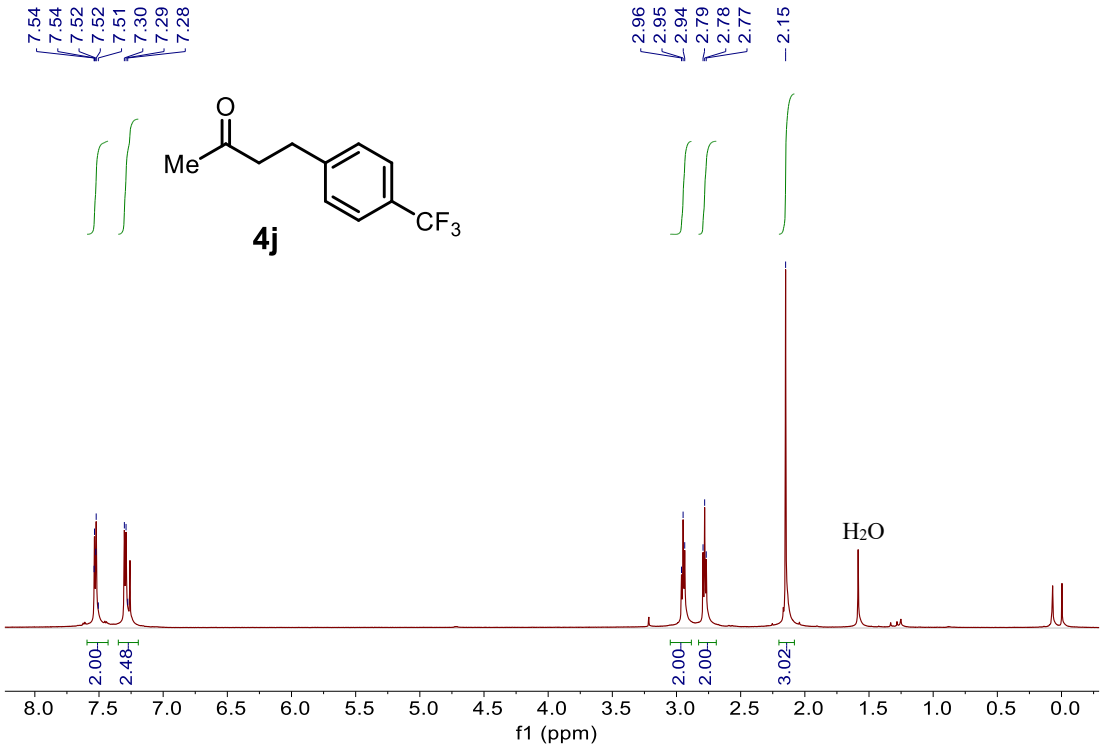
^{1}H NMR (400 MHz, Chloroform-*d*)



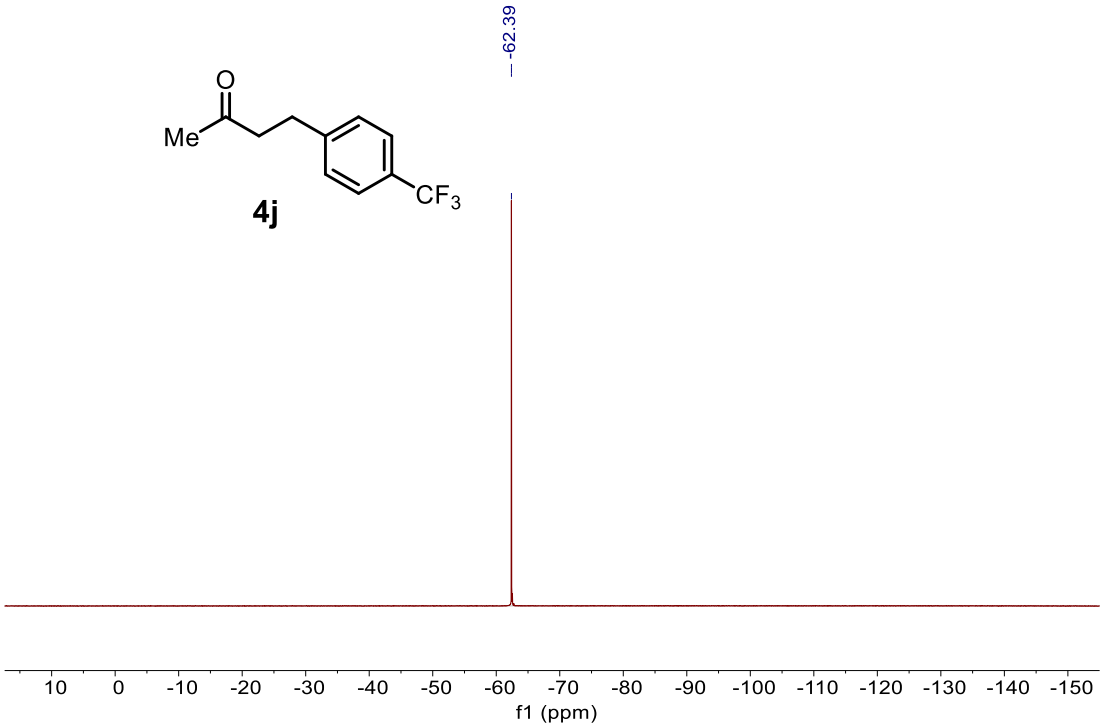
^{19}F NMR (377 MHz, Chloroform-*d*)



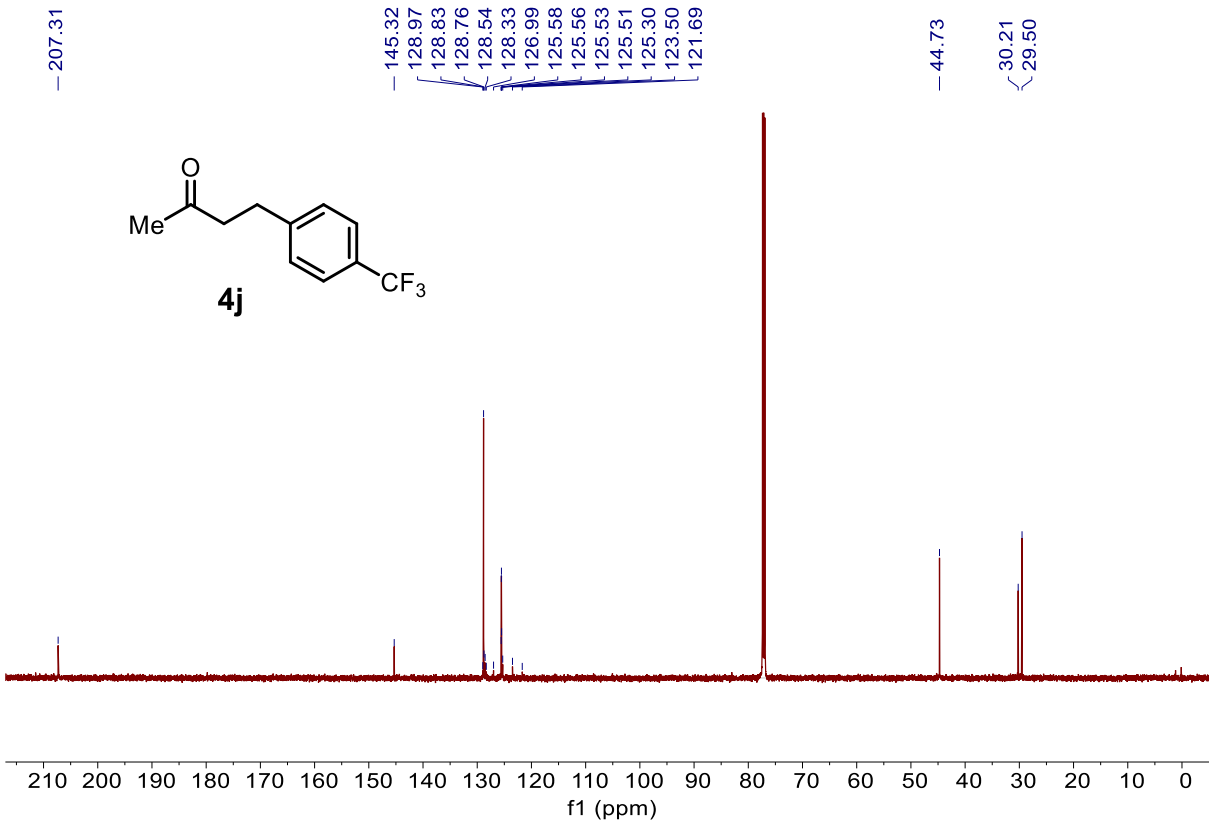
^{13}C NMR (101 MHz, Chloroform-*d*)



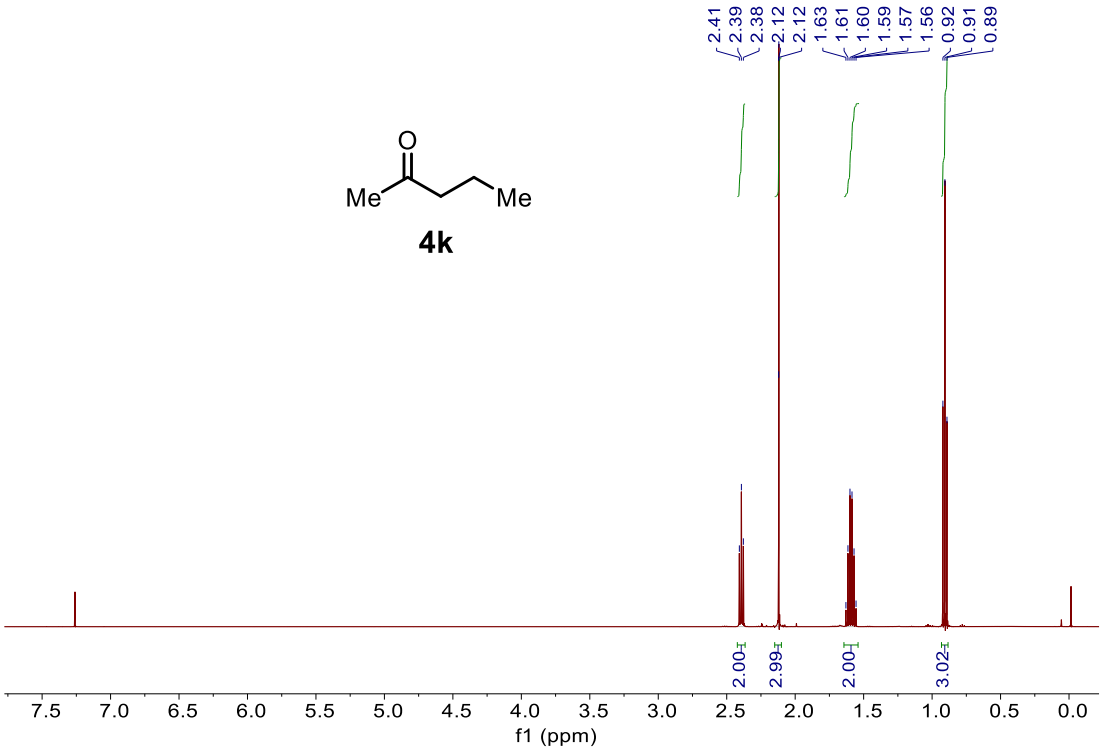
^{1}H NMR (600 MHz, Chloroform-*d*)



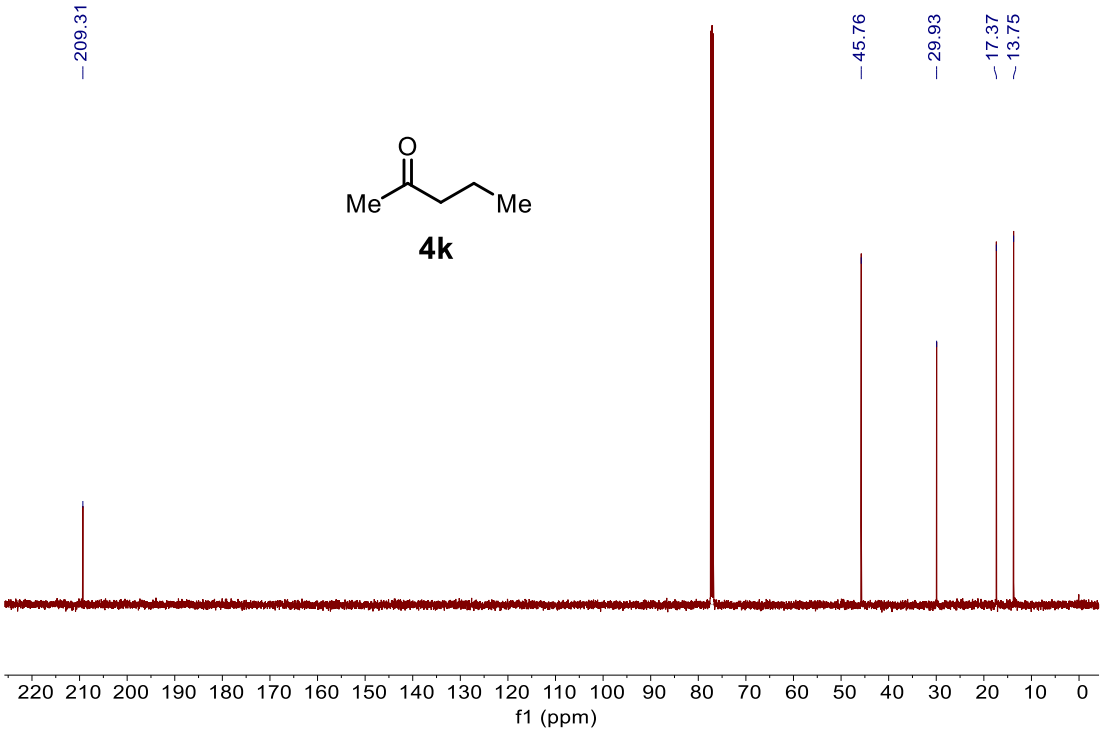
^{19}F NMR (565 MHz, Chloroform-*d*)



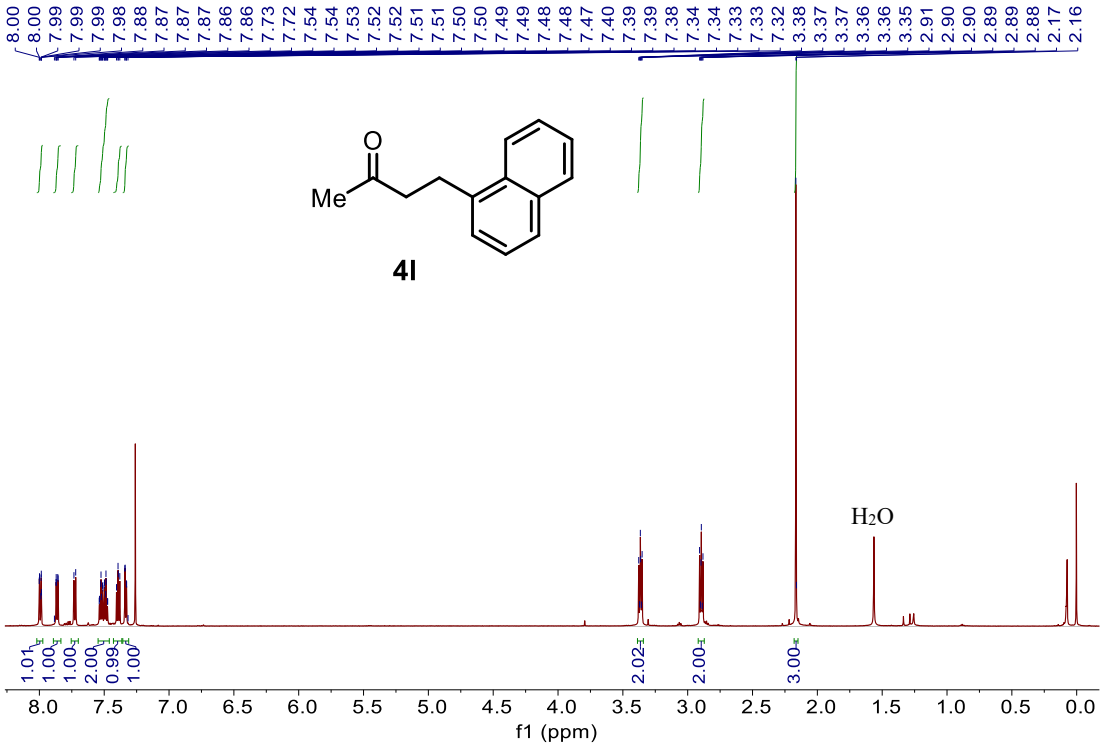
^{13}C NMR (151 MHz, Chloroform-*d*)



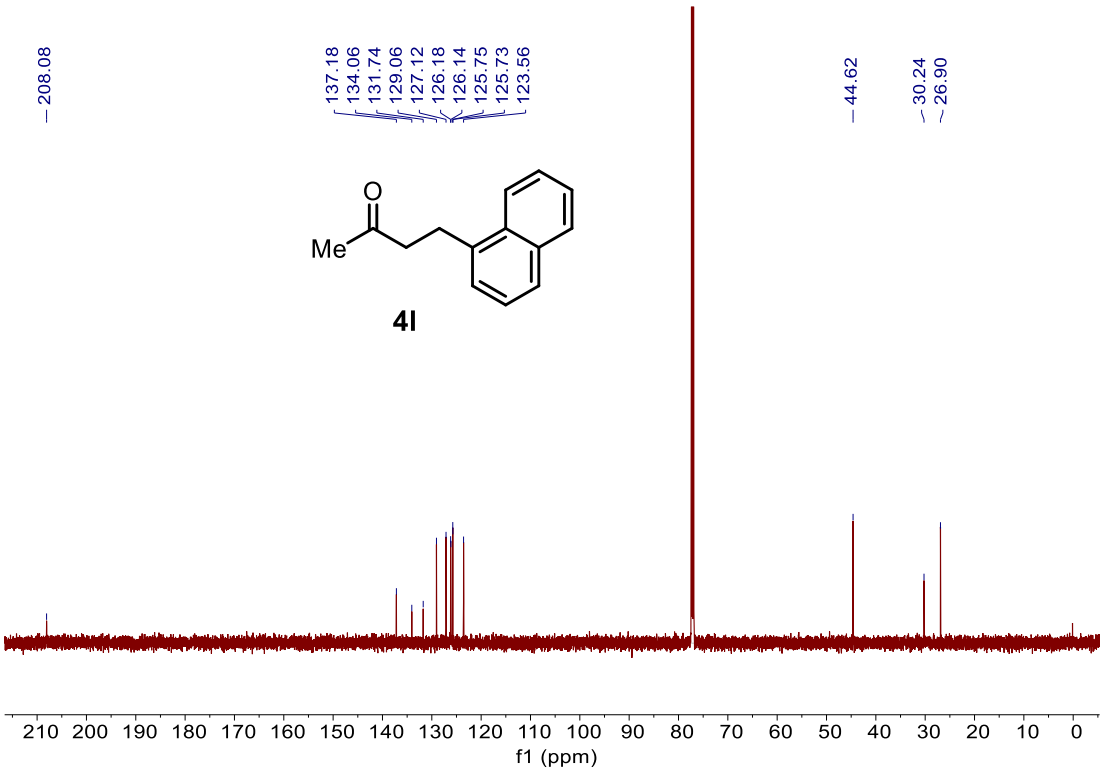
^{1}H NMR (500 MHz, Chloroform-*d*)



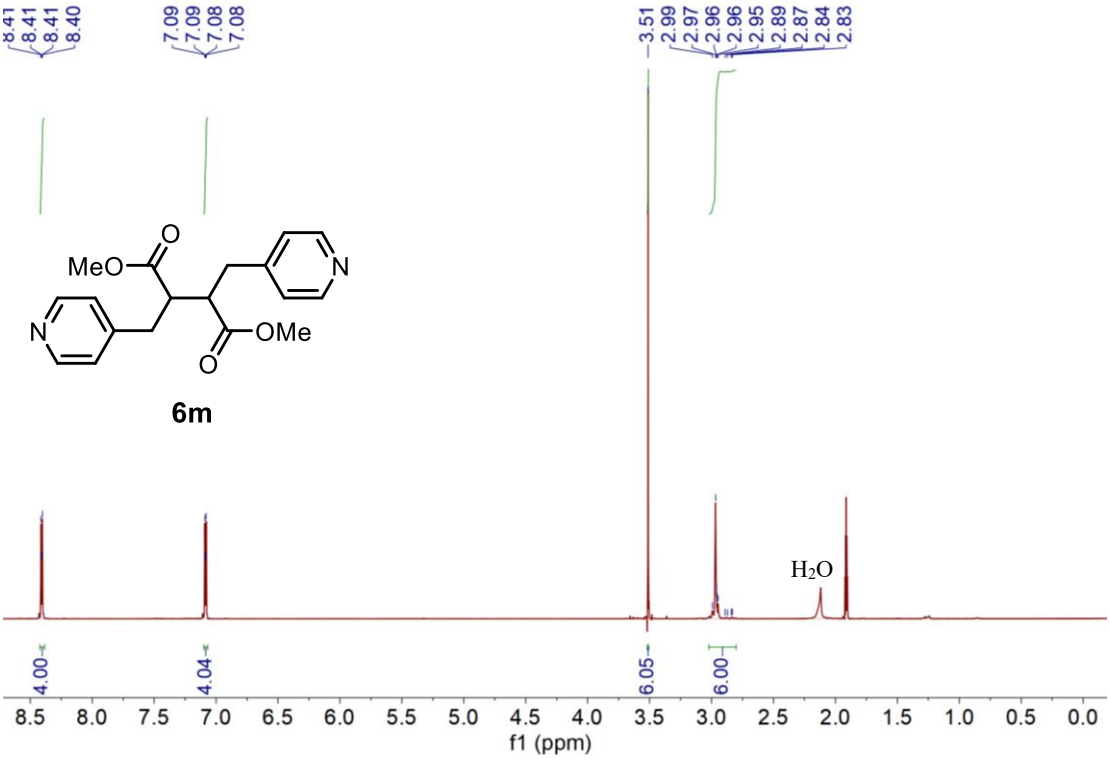
^{13}C NMR (126 MHz, Chloroform-*d*)



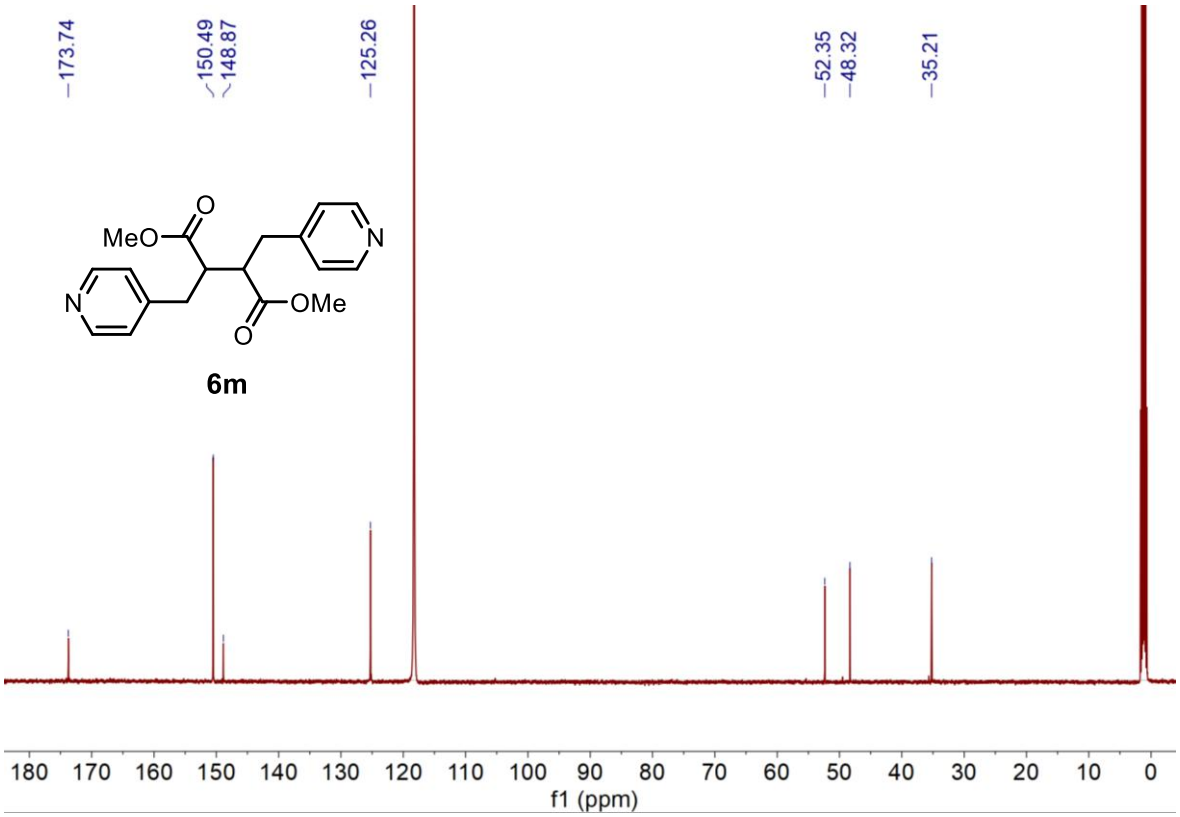
^{1}H NMR (600 MHz, Chloroform-*d*)



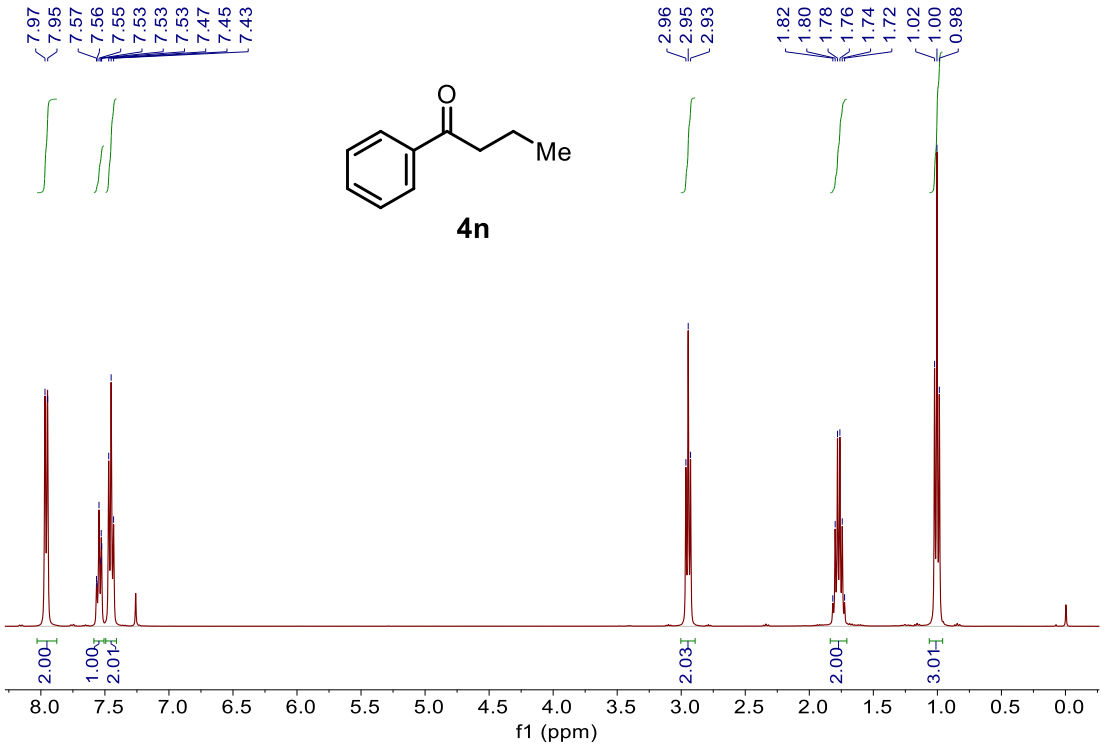
^{13}C NMR (151 MHz, Chloroform-*d*)



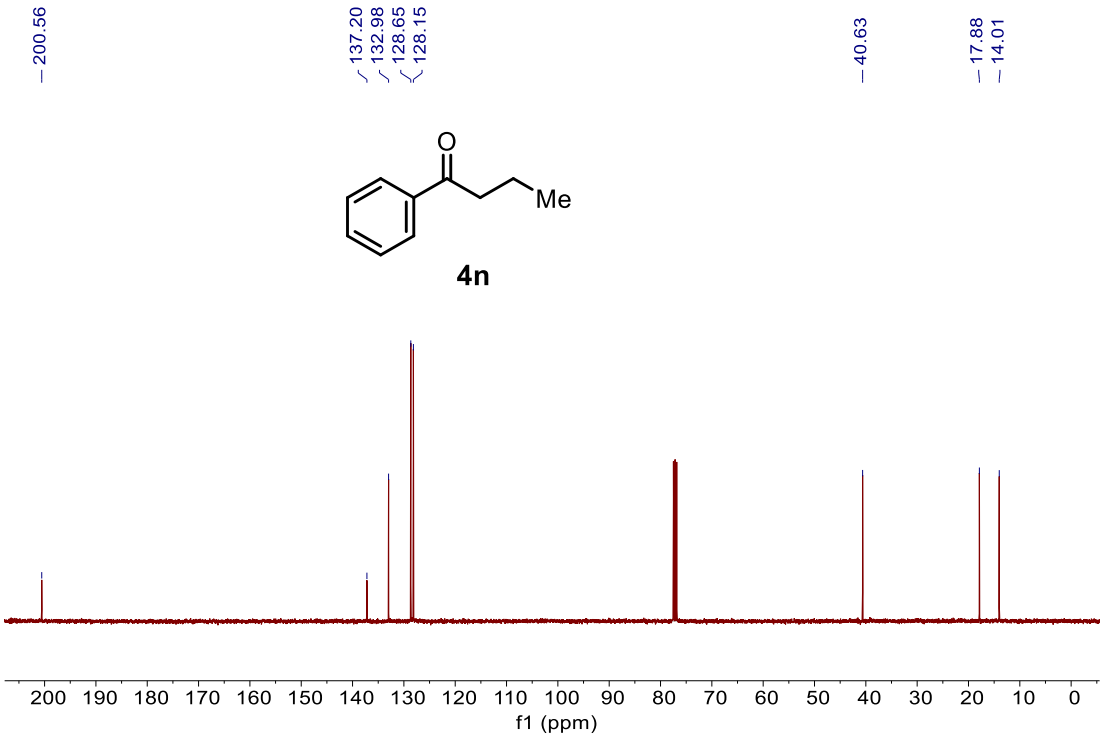
^{1}H NMR (500 MHz, Acetonitrile-d_3)



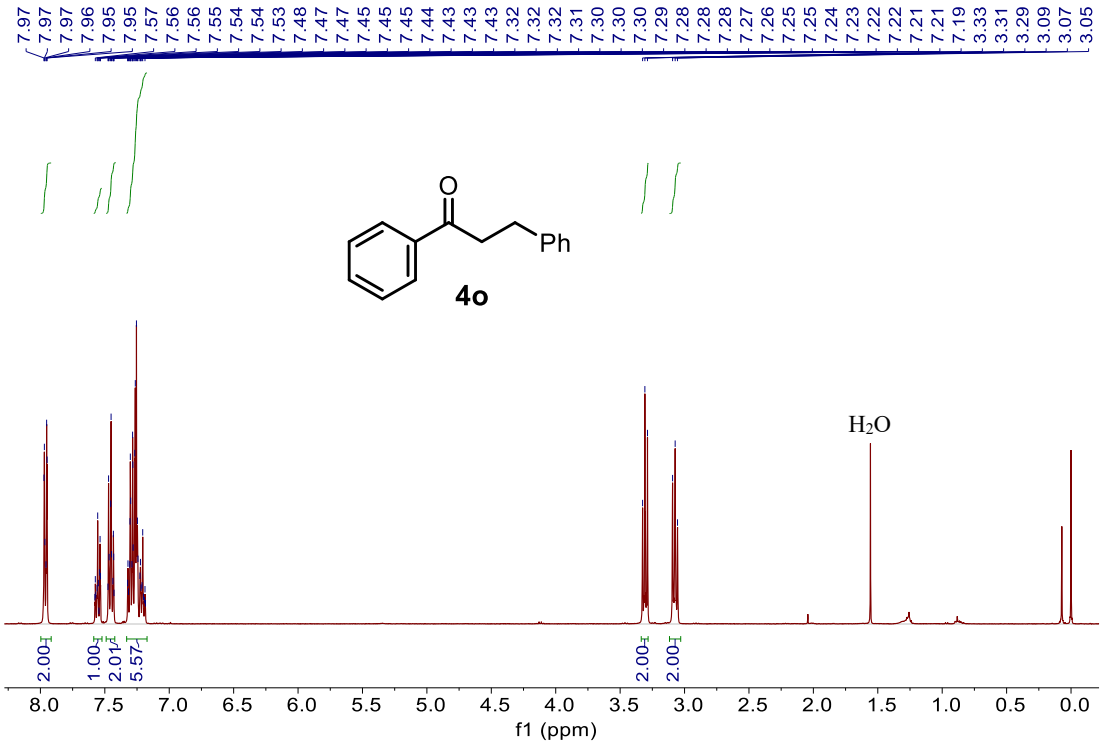
^{13}C NMR (126 MHz, Acetonitrile-d_3)



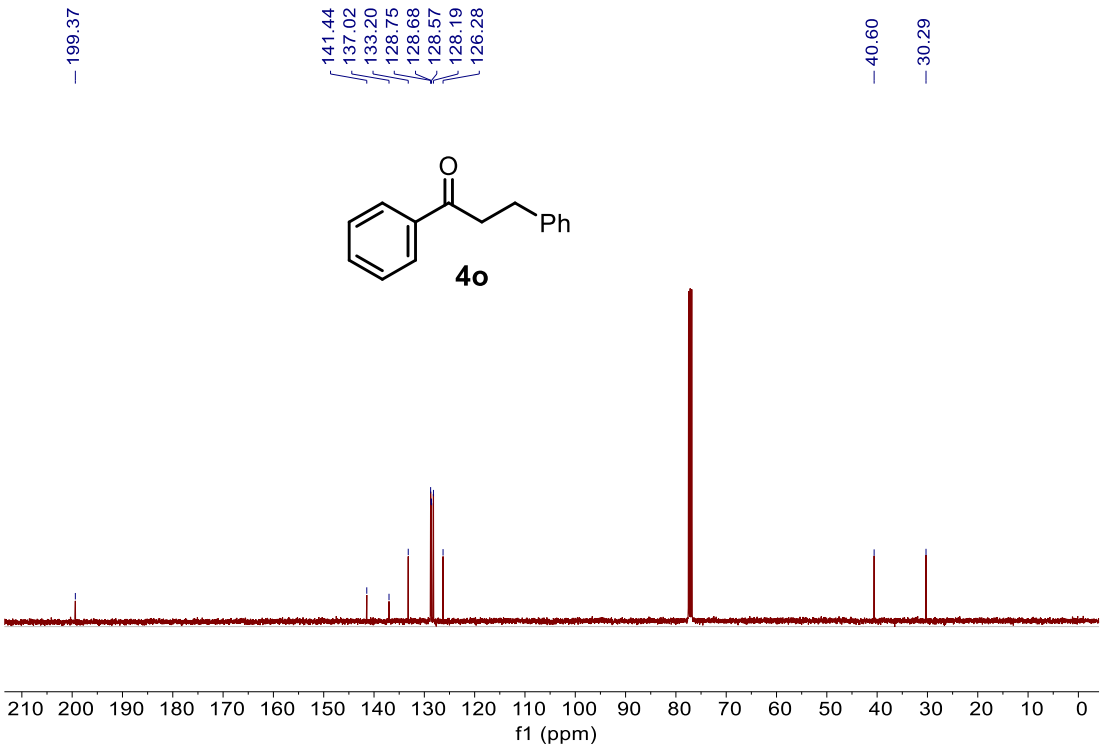
^{1}H NMR (400 MHz, Chloroform-*d*)



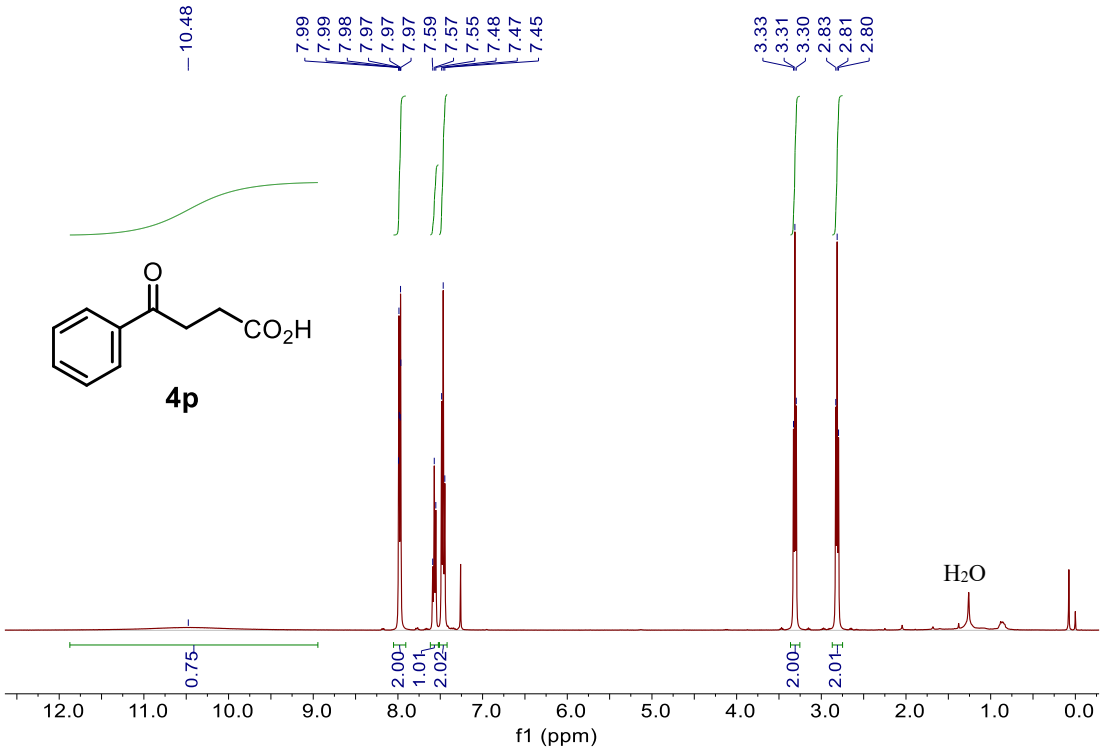
^{13}C NMR (101 MHz, Chloroform-*d*)



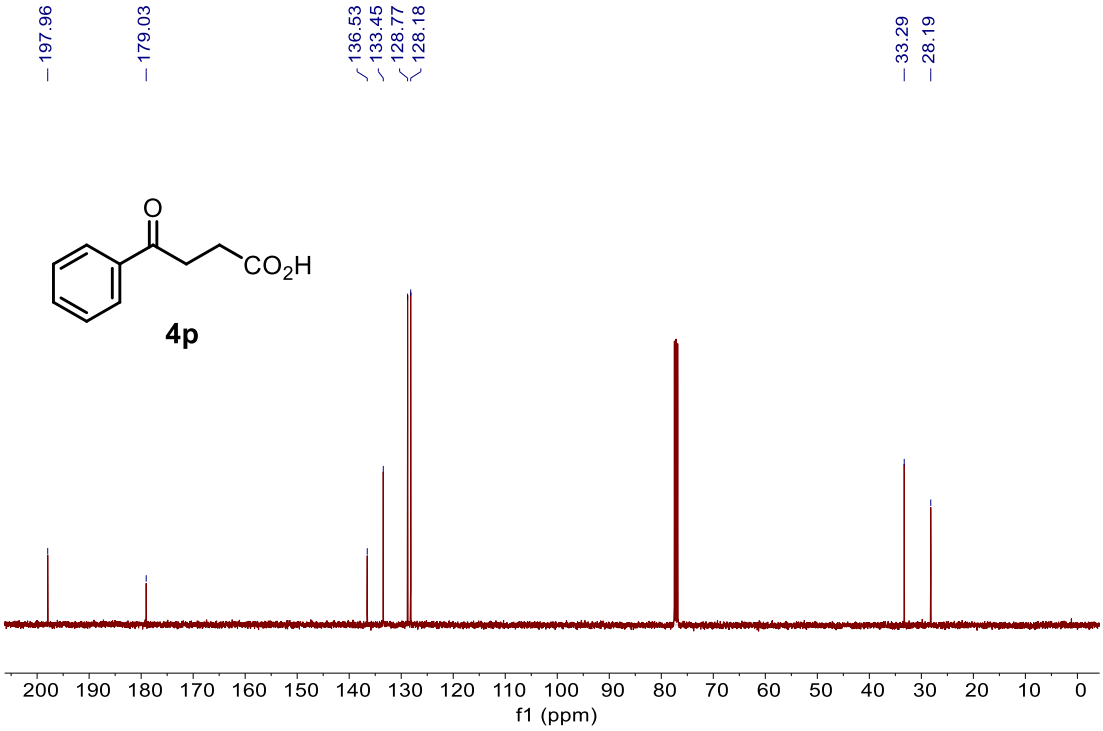
^{1}H NMR (400 MHz, Chloroform-*d*)



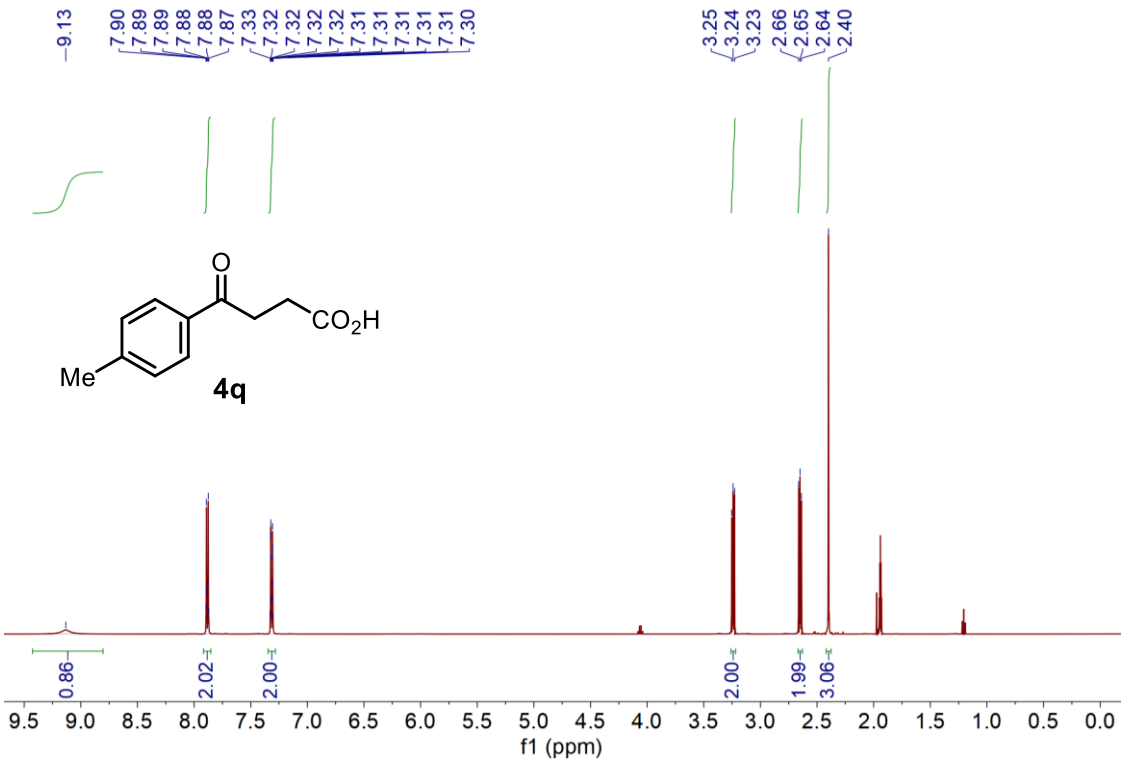
^{13}C NMR (101 MHz, Chloroform-*d*)



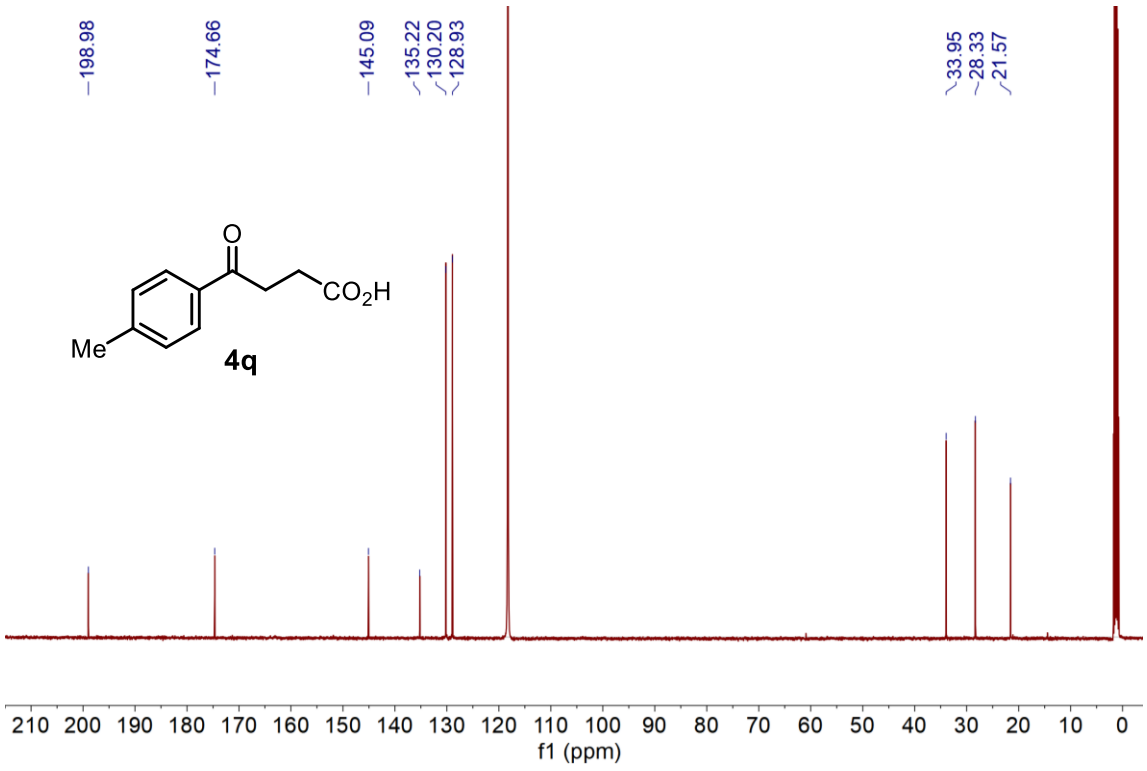
^{1}H NMR (400 MHz, Chloroform-*d*)



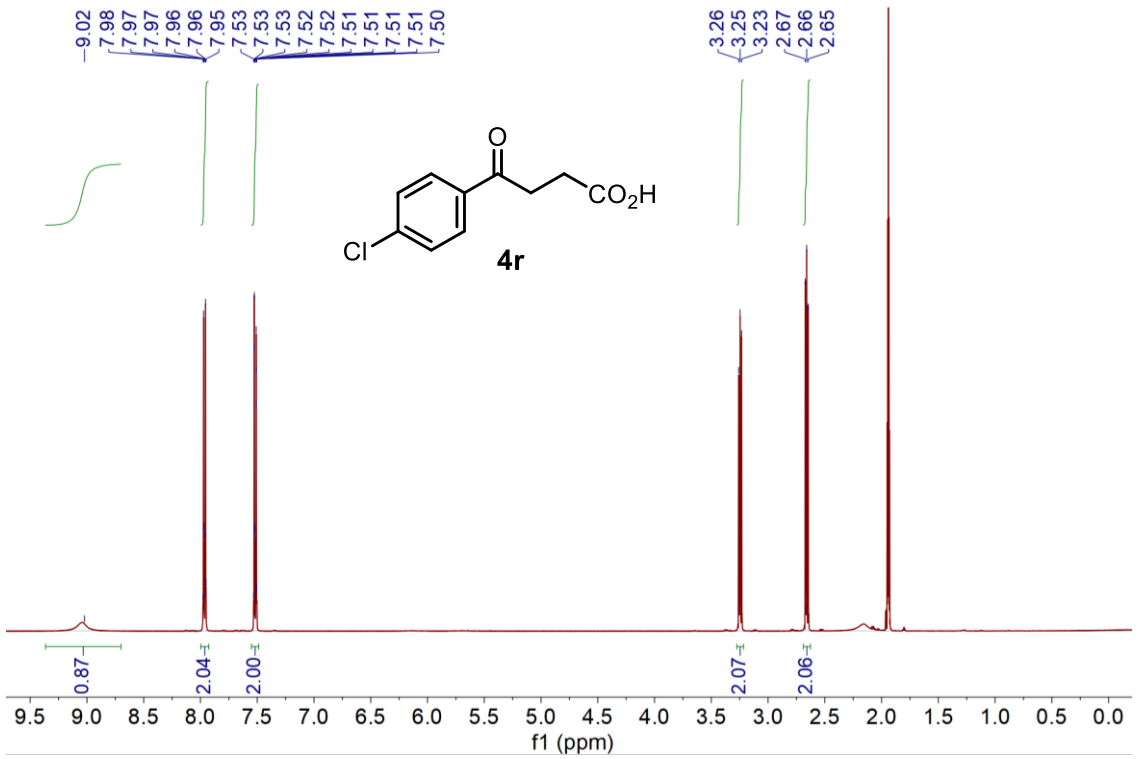
^{13}C NMR (101 MHz, Chloroform-*d*)



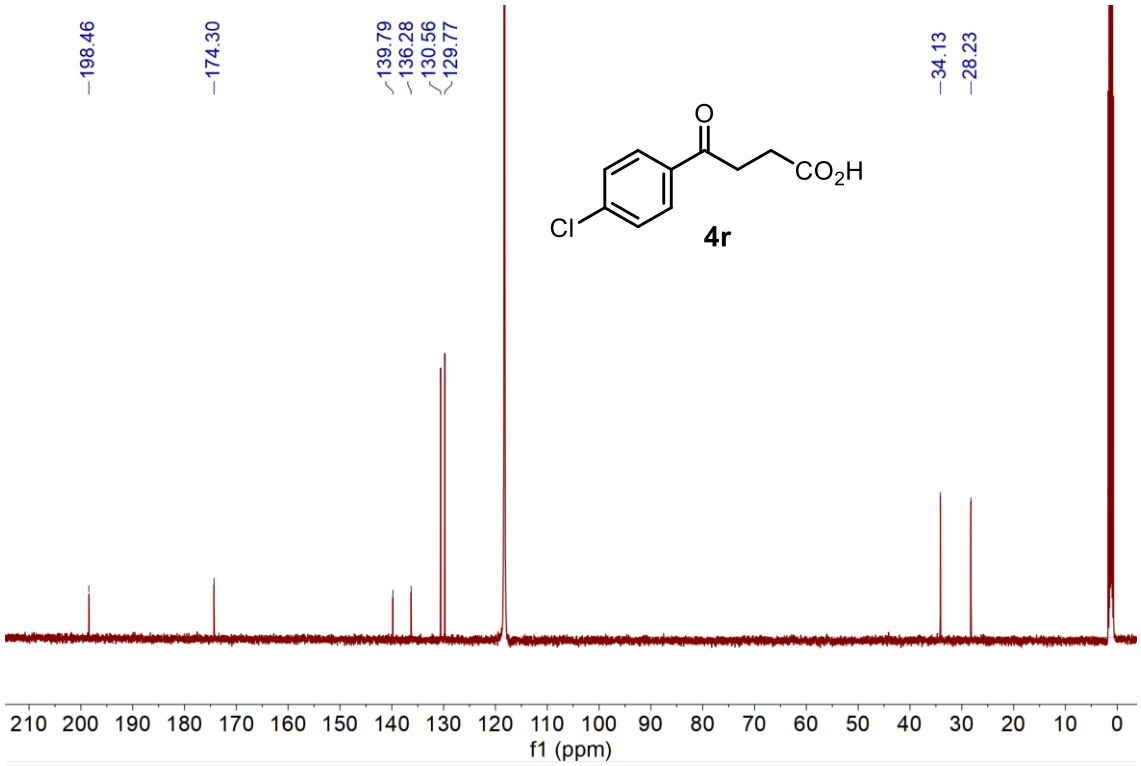
^{1}H NMR (500 MHz, CD_3CN)



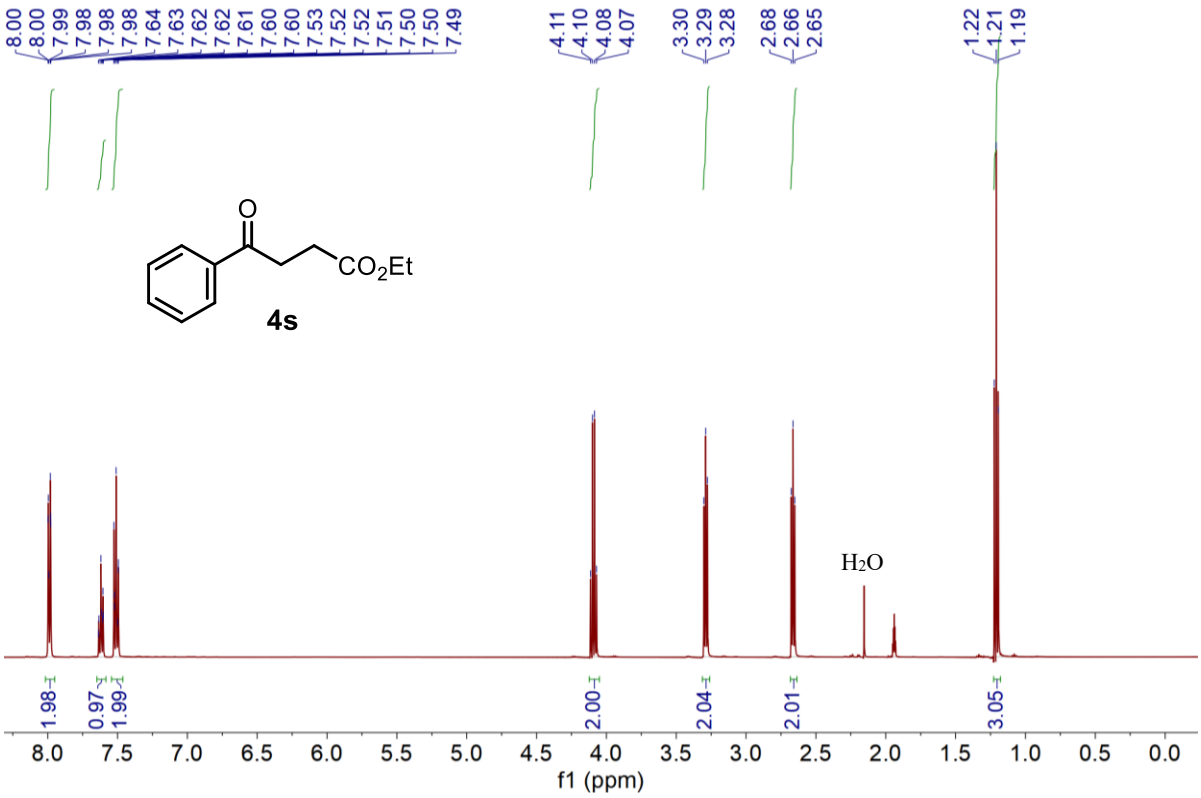
^{13}C NMR (126 MHz, CD_3CN)



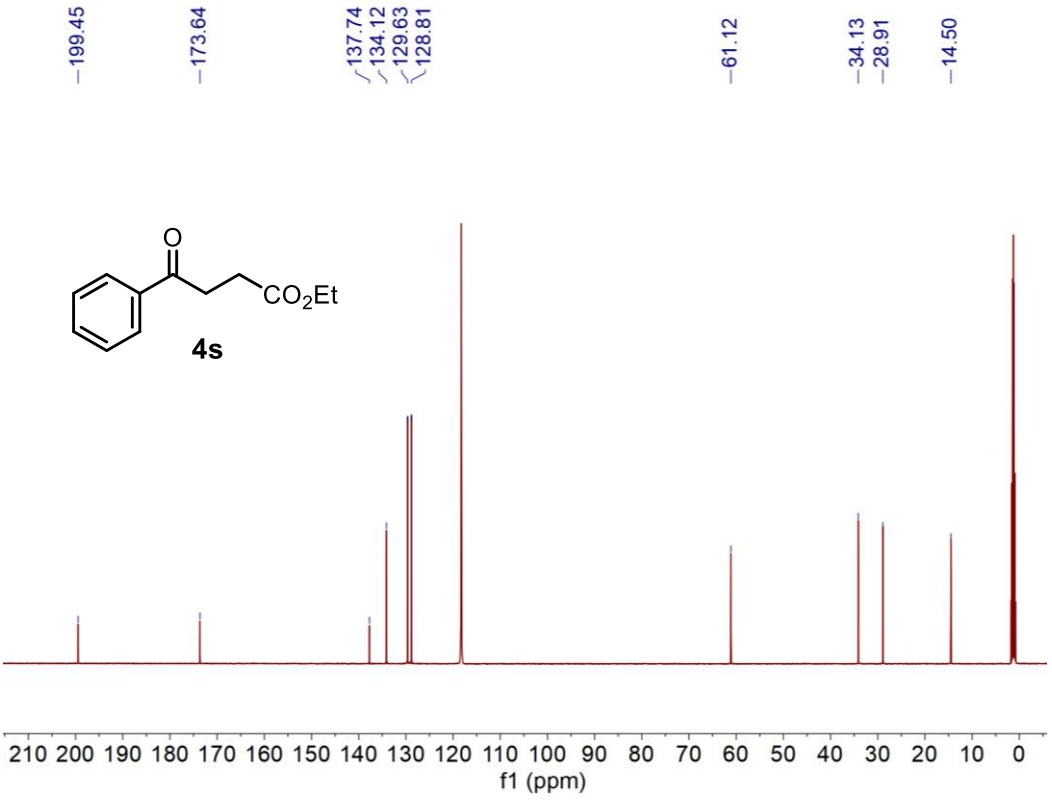
^{1}H NMR (500 MHz, CD_3CN)



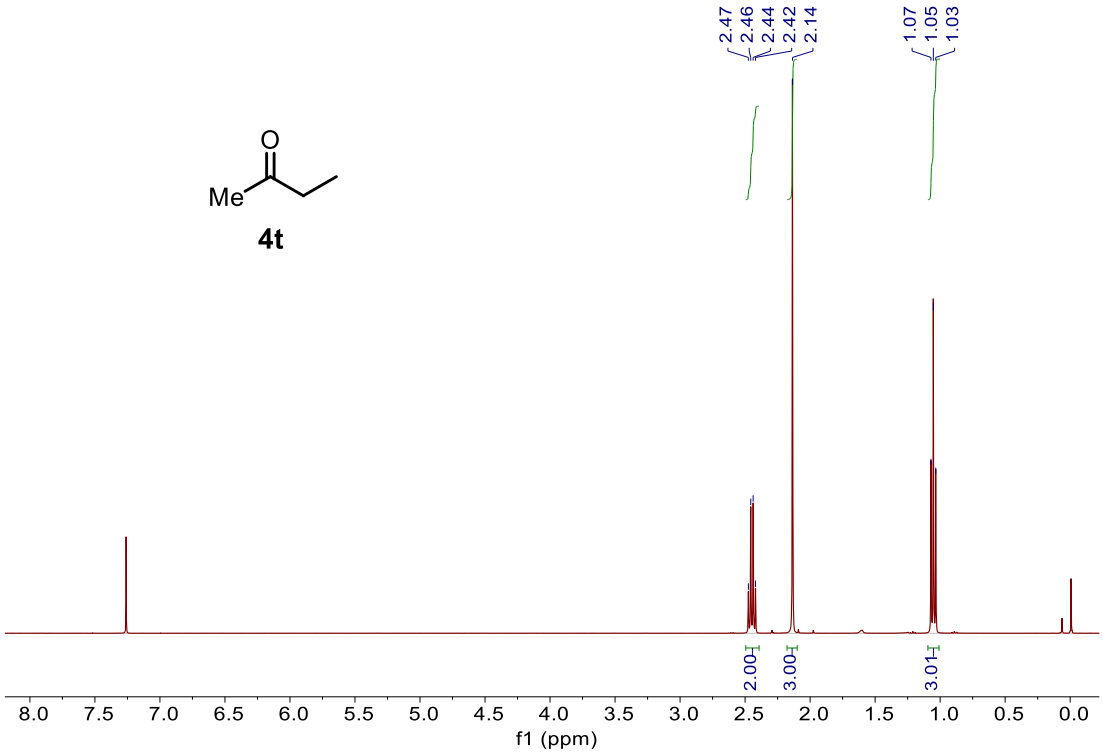
^{13}C NMR (126 MHz, CD_3CN)



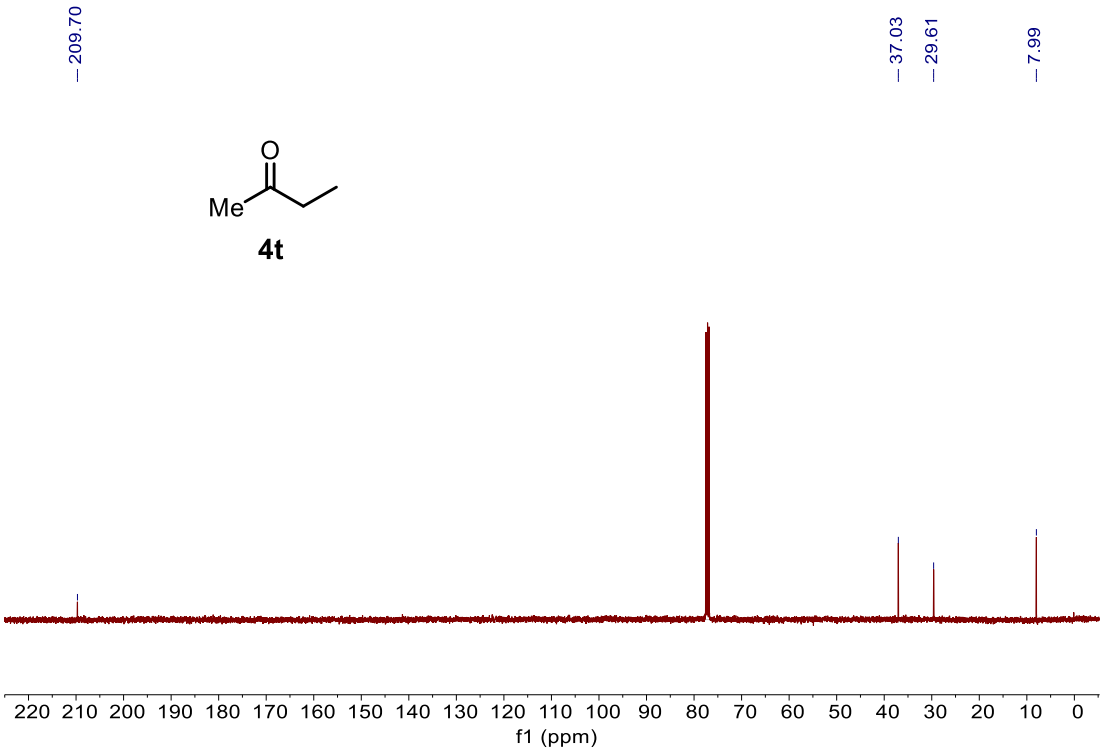
^{1}H NMR (500 MHz, CD_3CN)



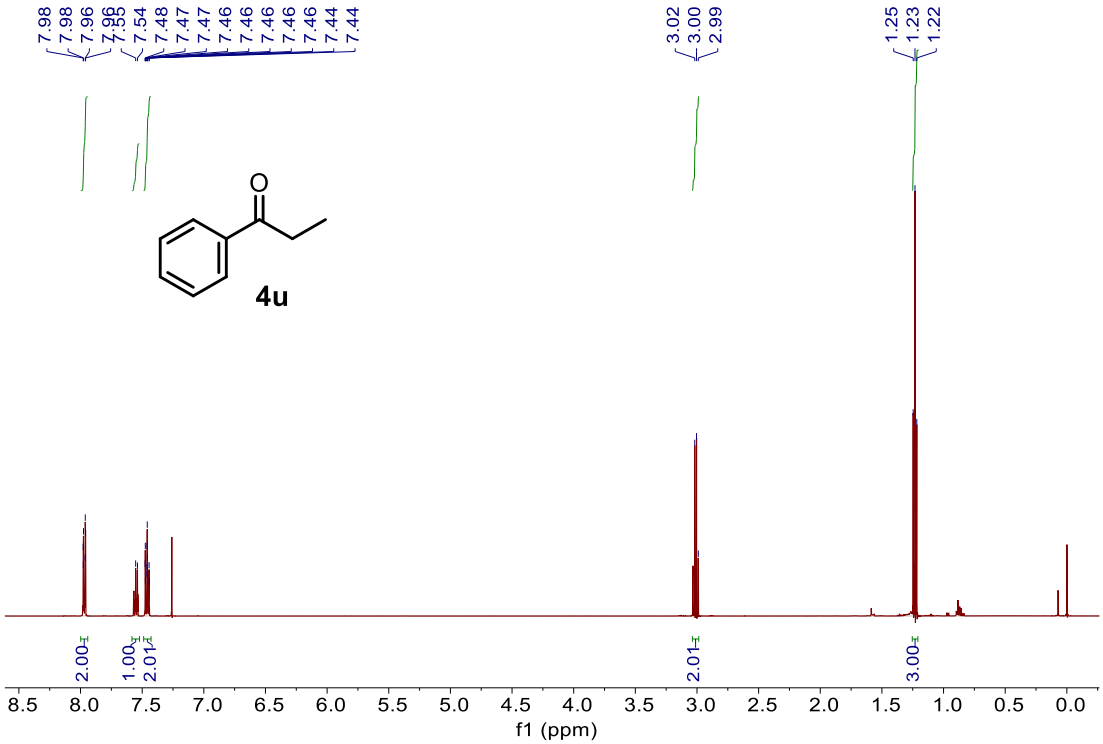
^{13}C NMR (126 MHz, CD_3CN)



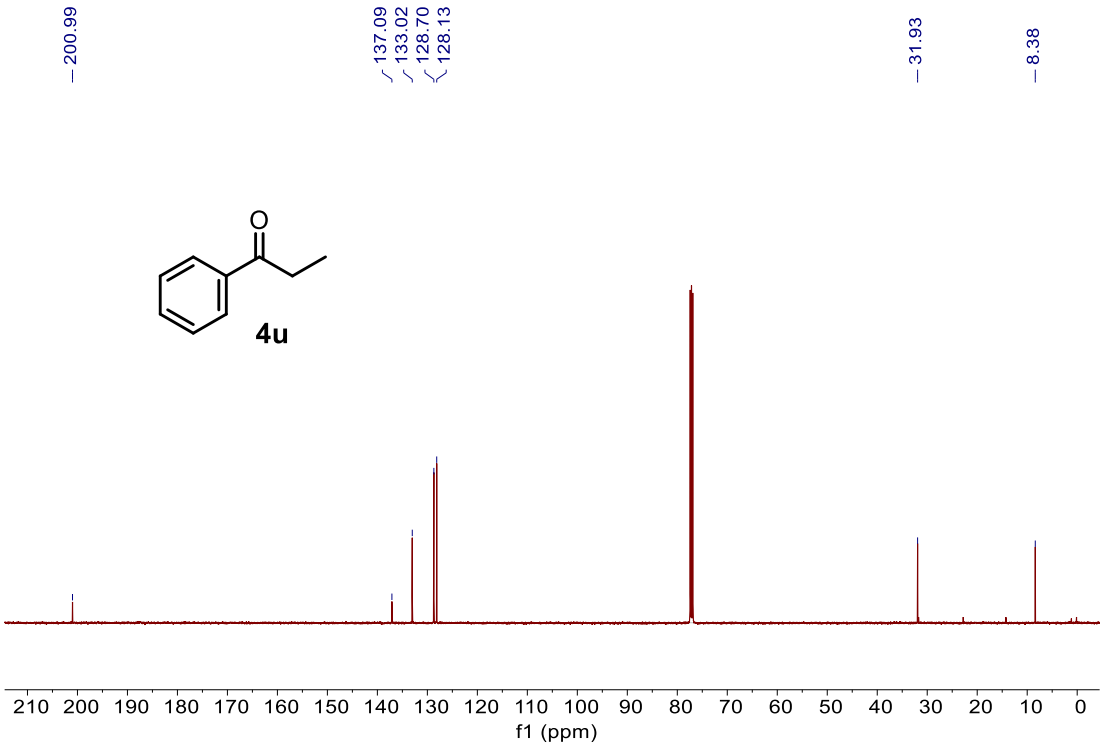
^{1}H NMR (400 MHz, Chloroform-*d*)



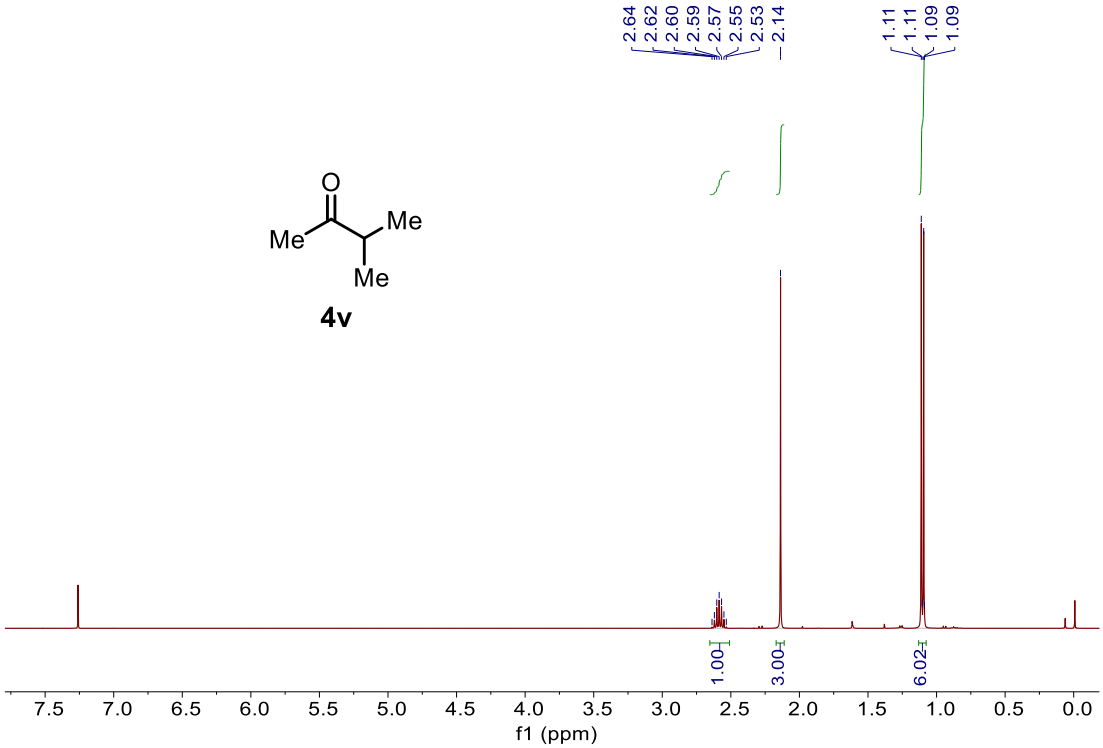
^{13}C NMR (101 MHz, Chloroform-*d*)



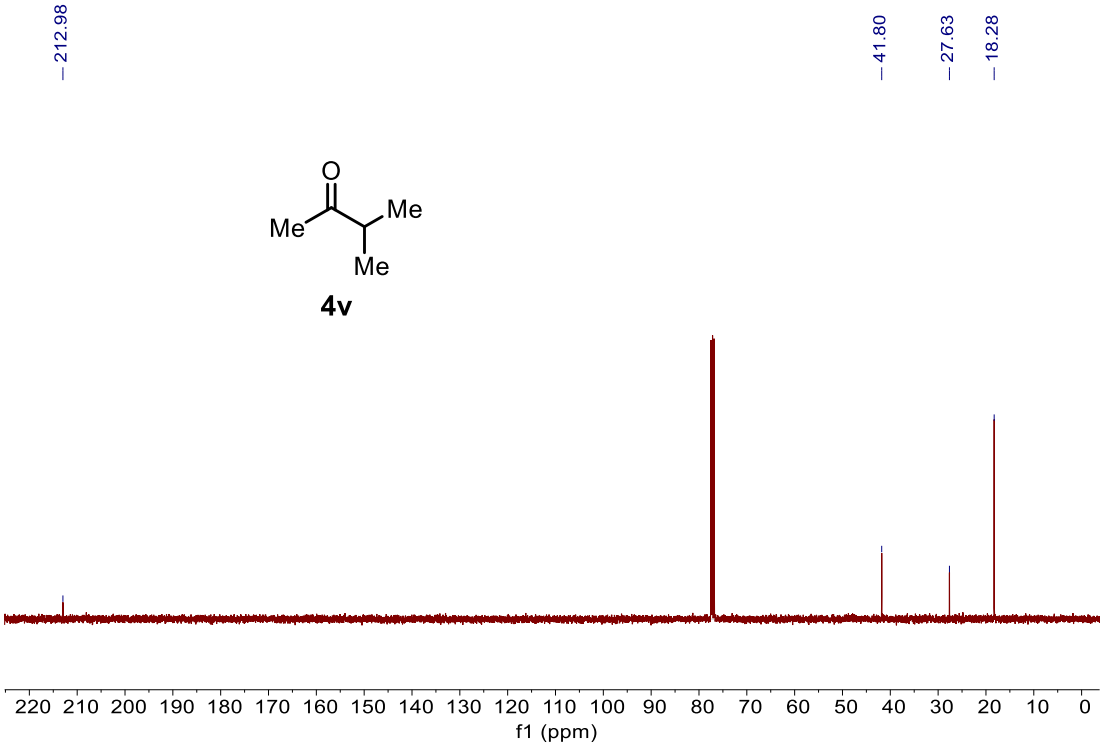
^{1}H NMR (500 MHz, Chloroform-*d*)



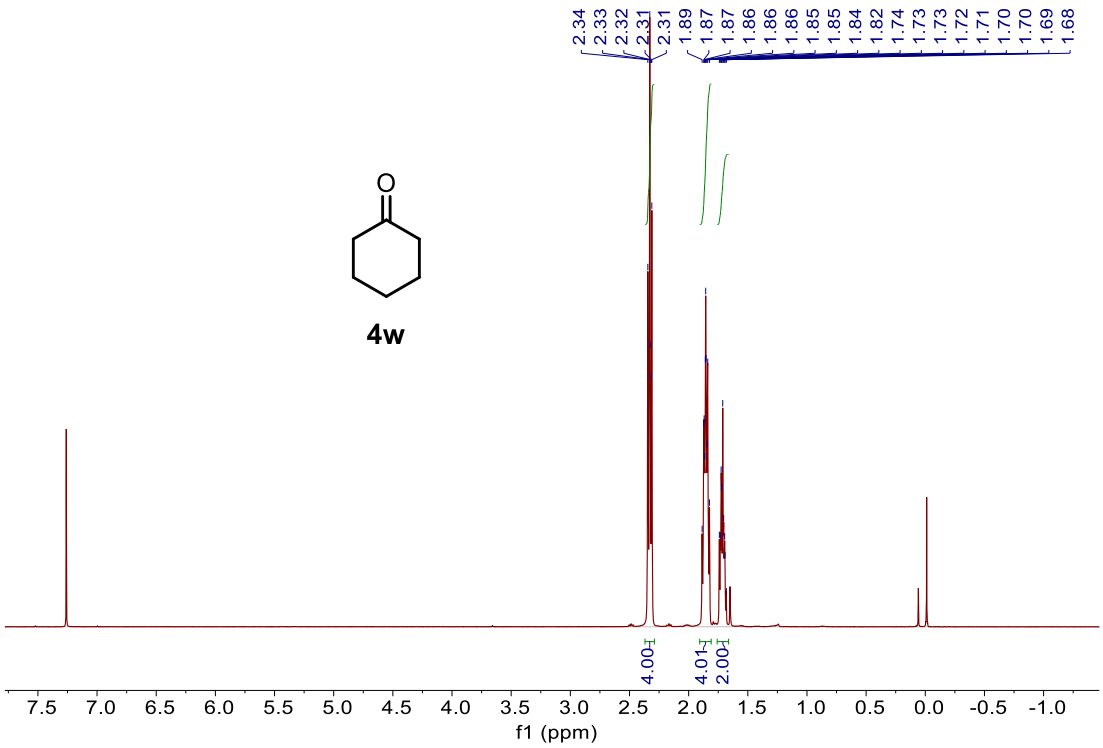
^{13}C NMR (126 MHz, Chloroform-*d*)



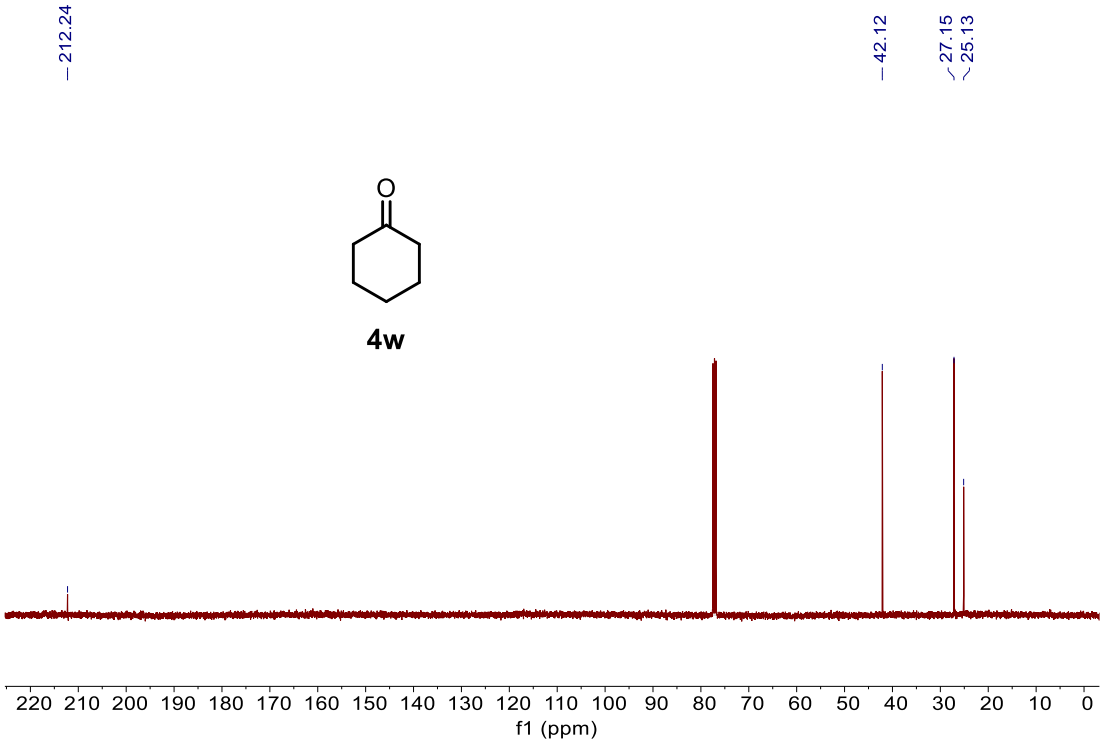
^{1}H NMR (400 MHz, Chloroform-*d*)



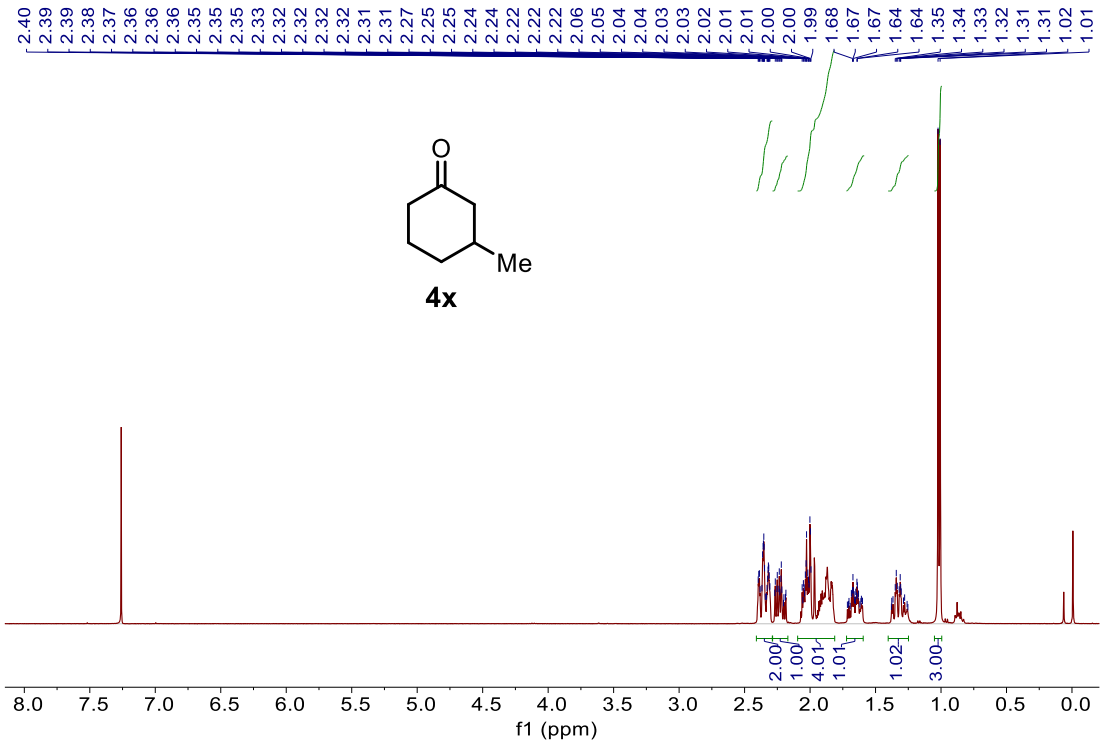
^{13}C NMR (101 MHz, Chloroform-*d*)



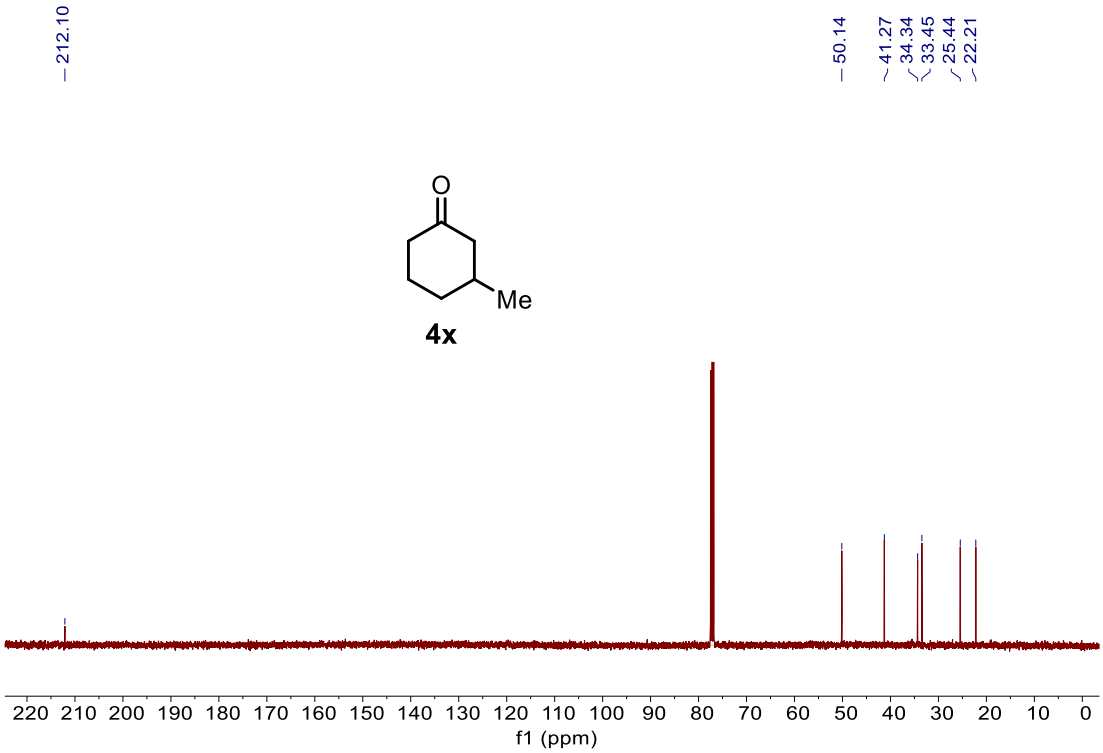
^{1}H NMR (400 MHz, Chloroform-*d*)



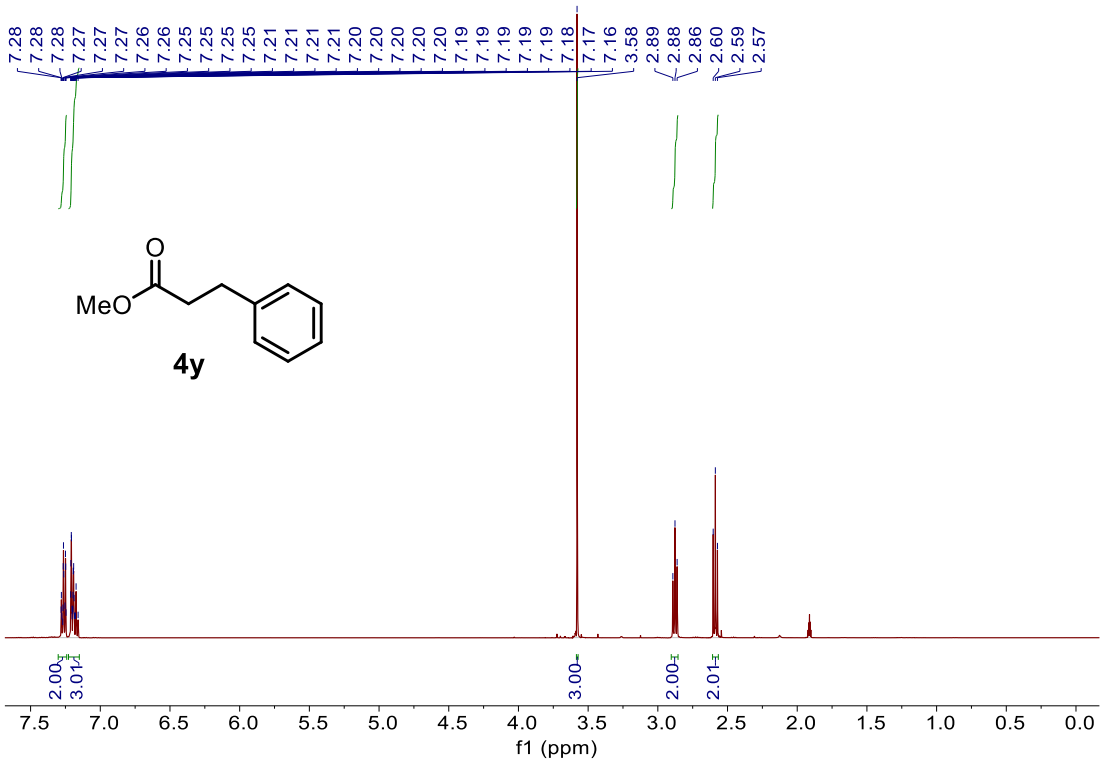
^{13}C NMR (101 MHz, Chloroform-*d*)



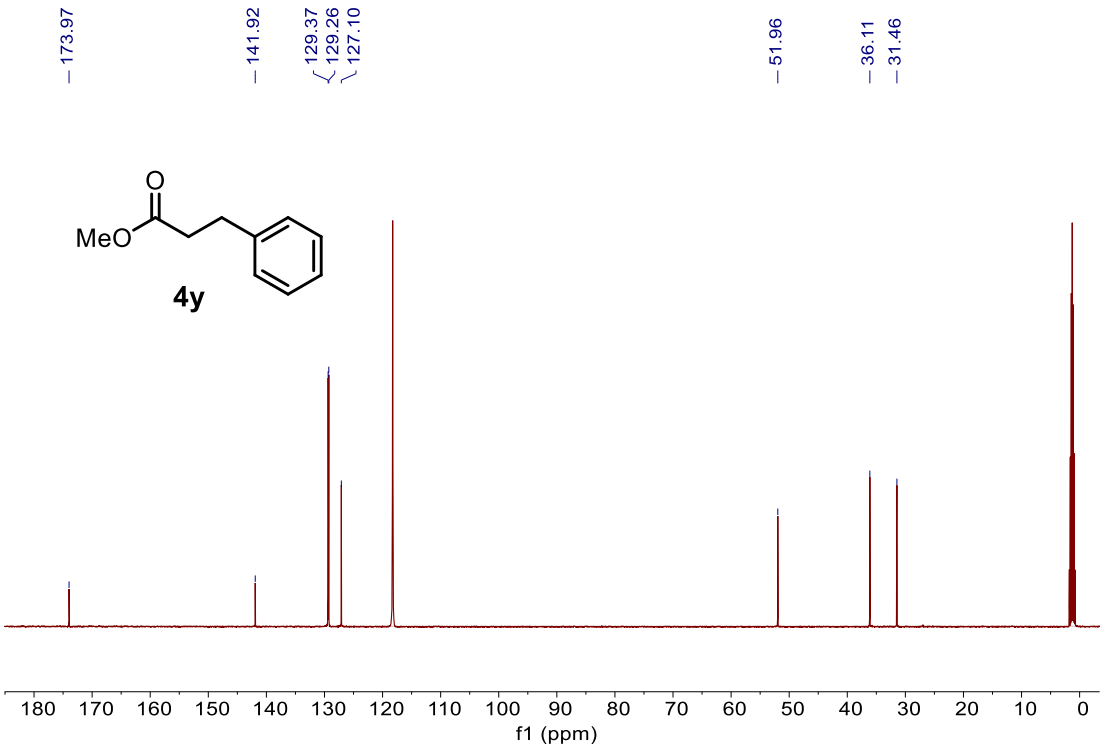
^{1}H NMR (400 MHz, Chloroform-*d*)



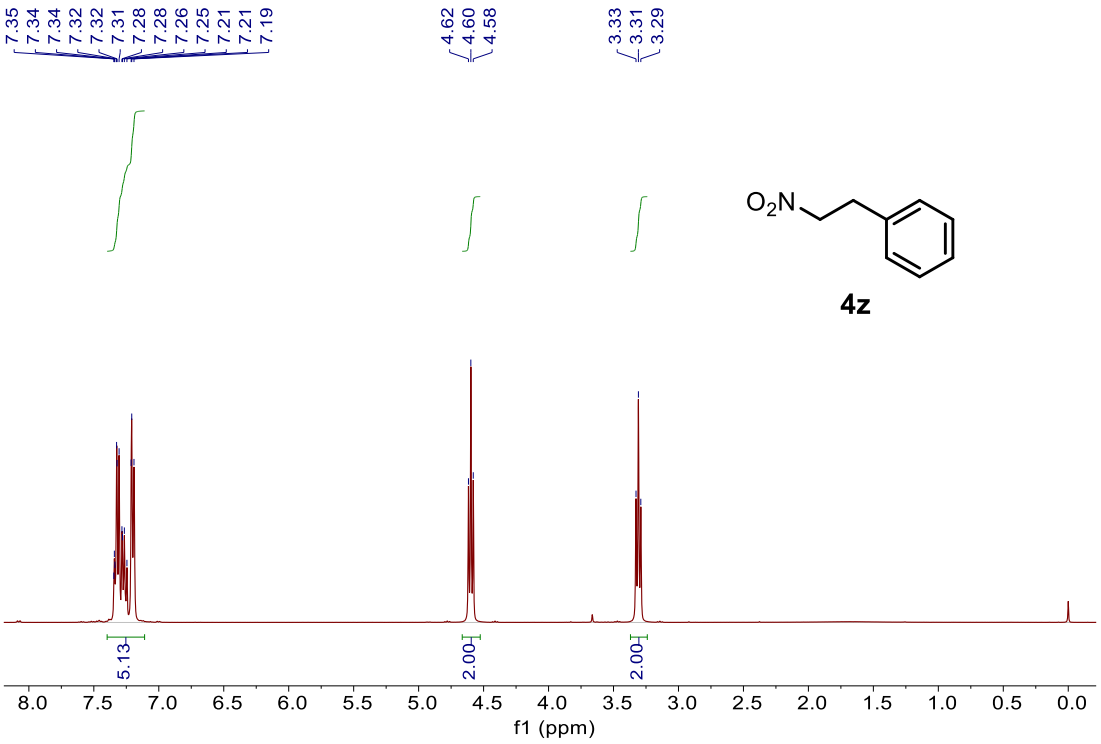
^{13}C NMR (101 MHz, Chloroform-*d*)



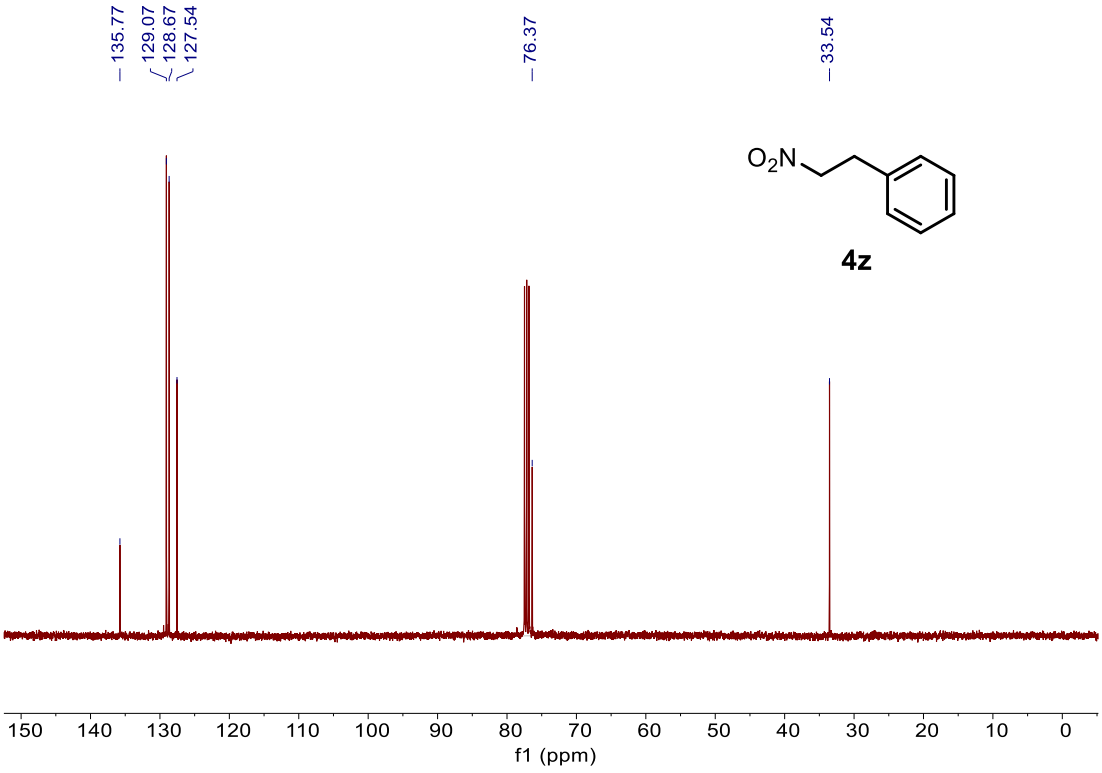
^{1}H NMR (500 MHz, Acetonitrile-d_3)



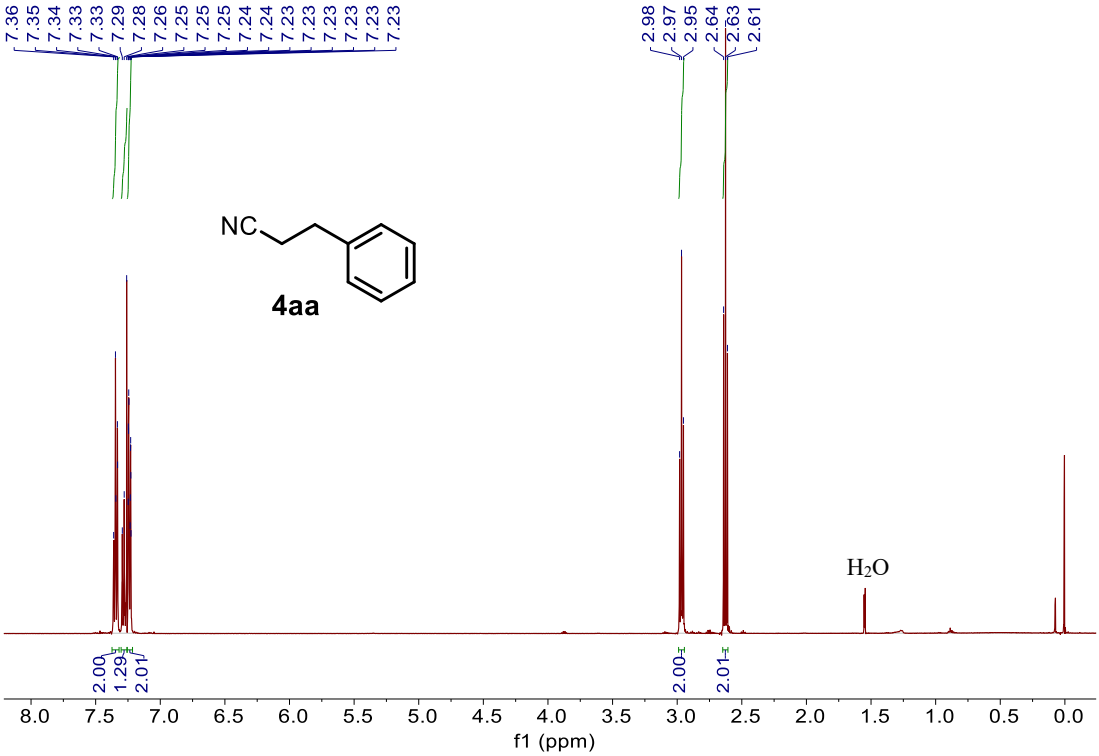
^{13}C NMR (126 MHz, Acetonitrile-d_3)



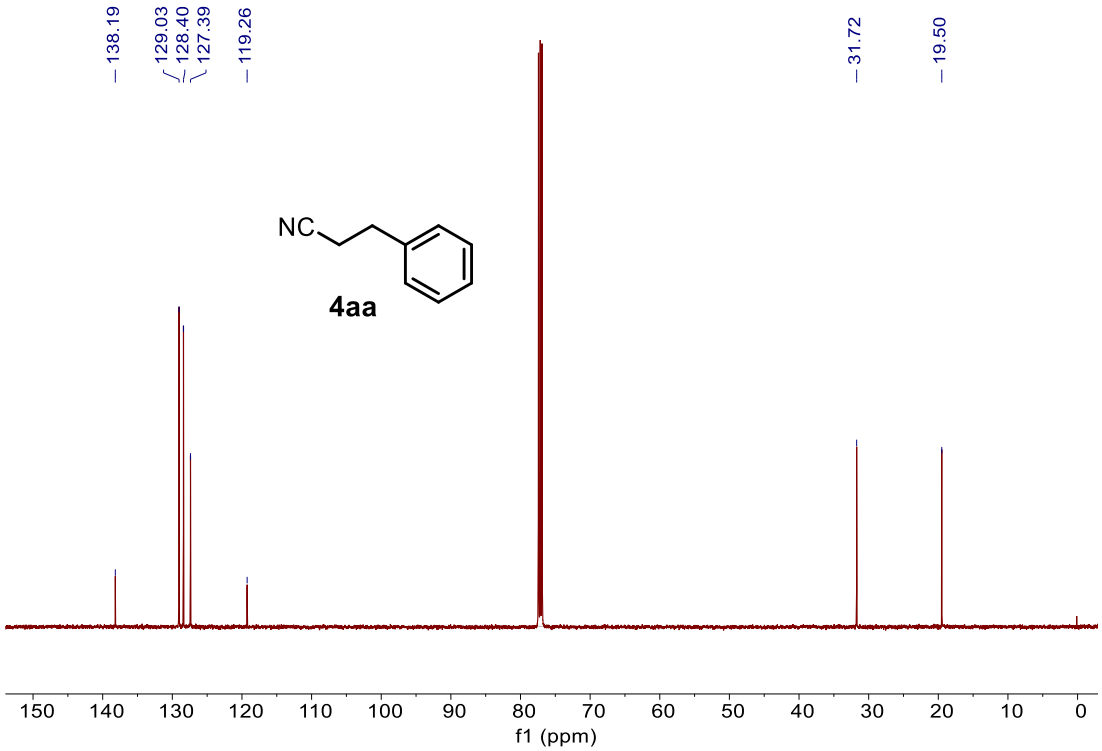
^{1}H NMR (400 MHz, Chloroform-*d*)



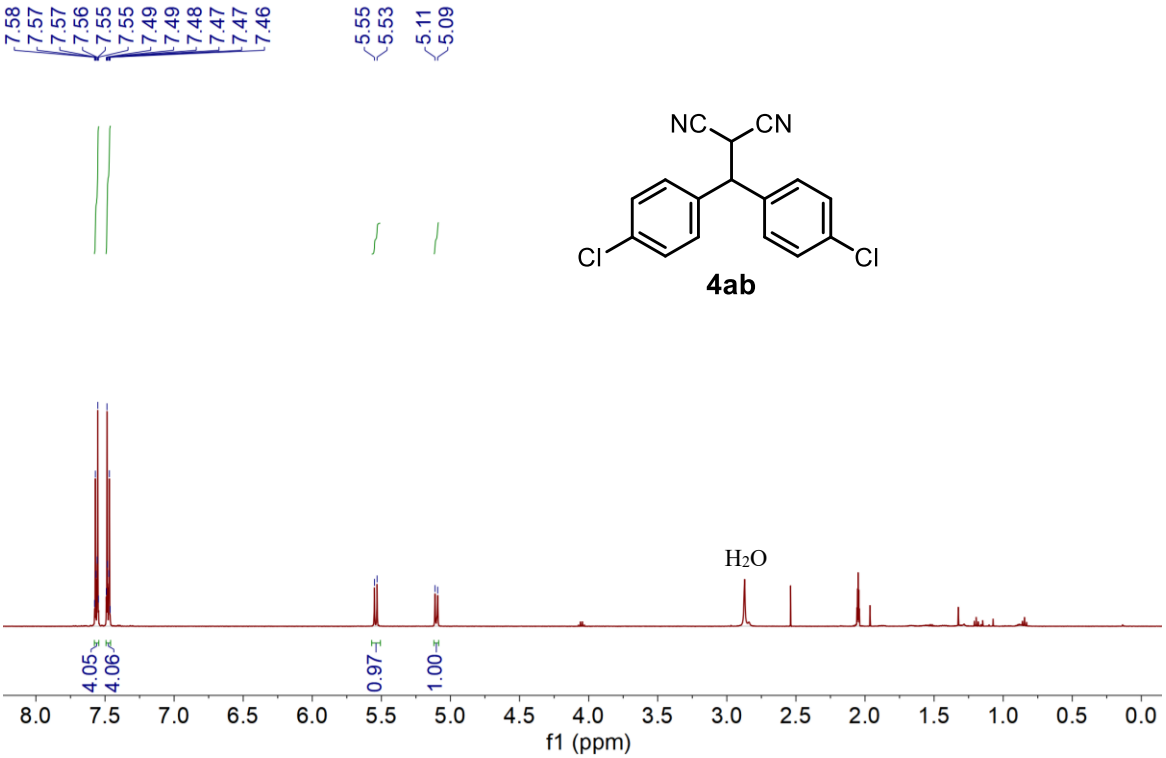
^{13}C NMR (101 MHz, Chloroform-*d*)



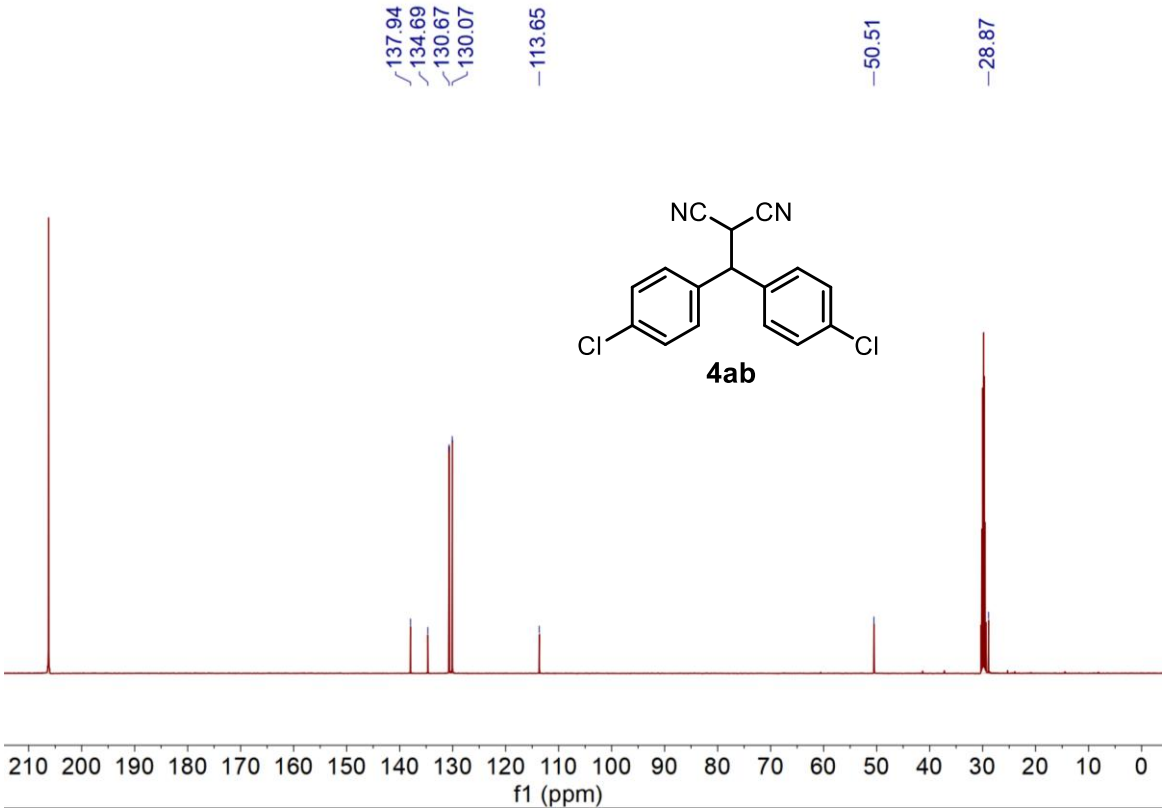
^{1}H NMR (500 MHz, Chloroform-*d*)



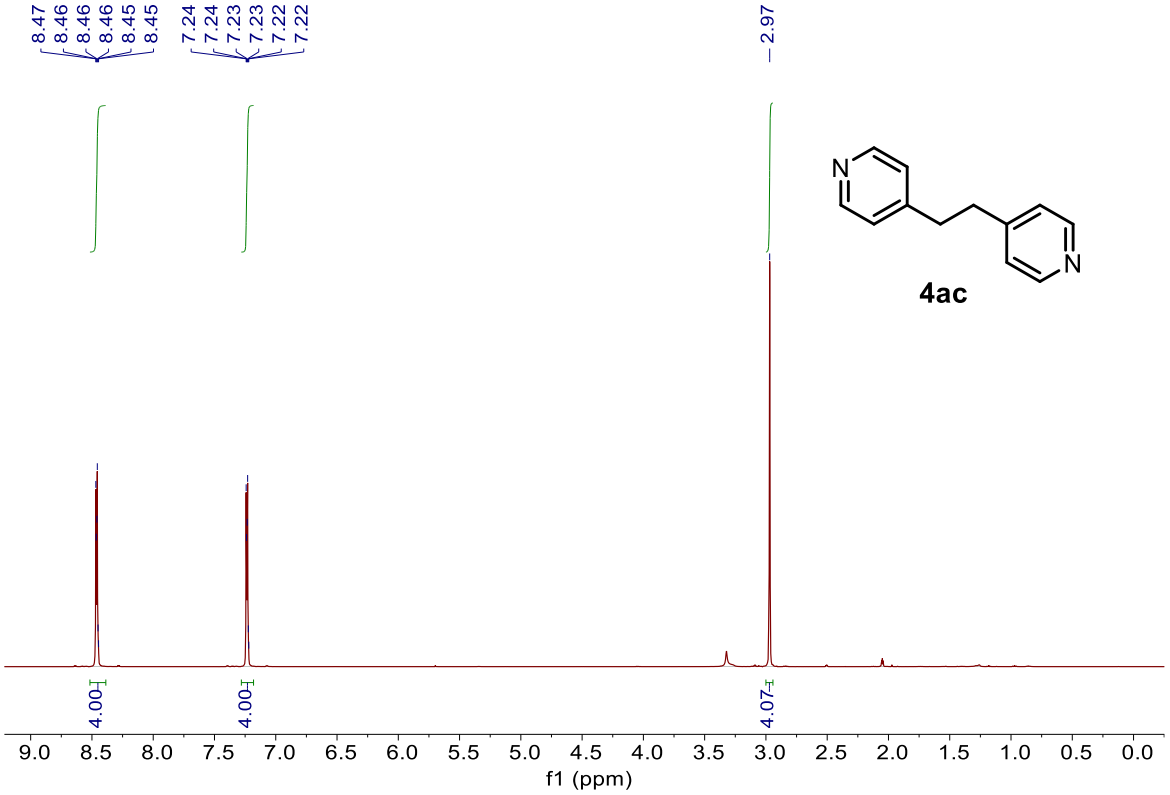
^{13}C NMR (126 MHz, Chloroform-*d*)



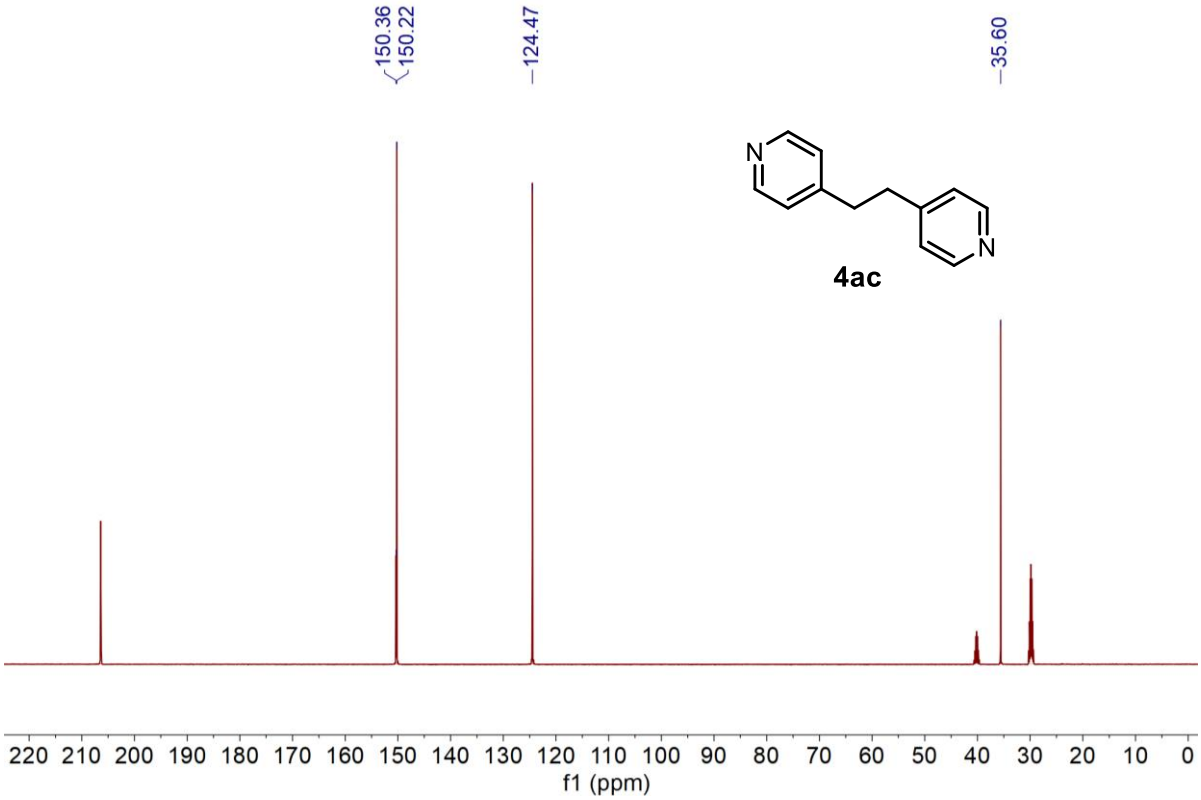
^{1}H NMR (500 MHz, Acetone-d_6)



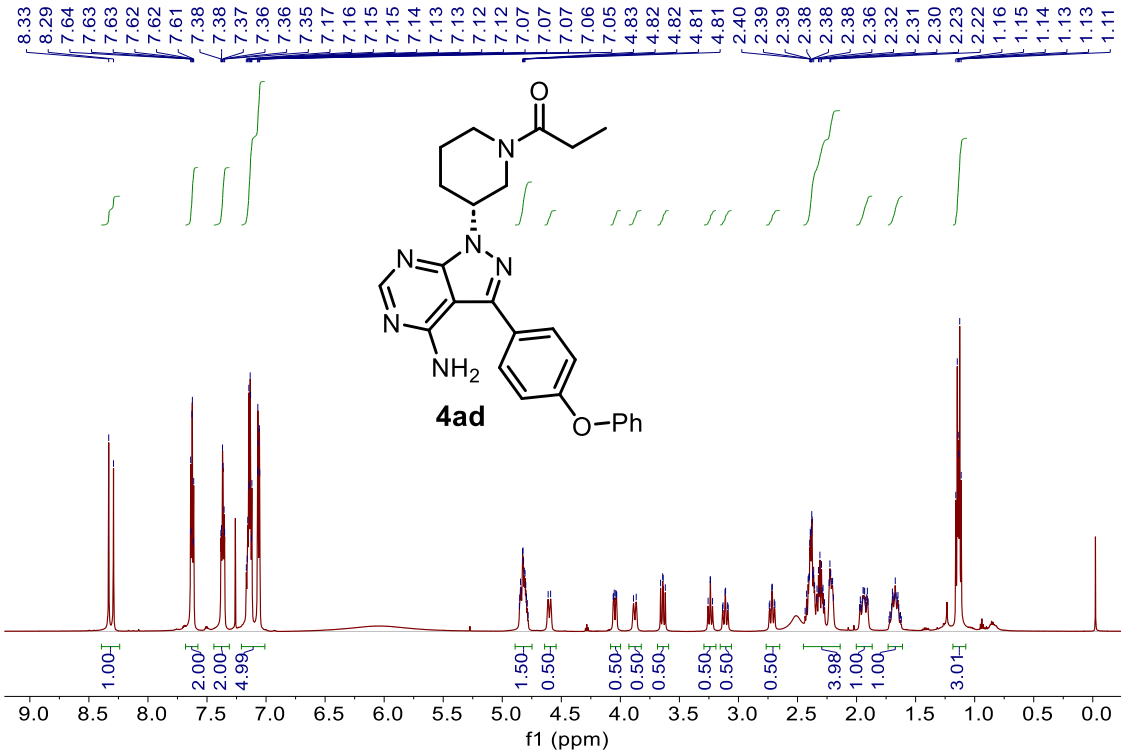
^{13}C NMR (126 MHz, Acetone-d_6)



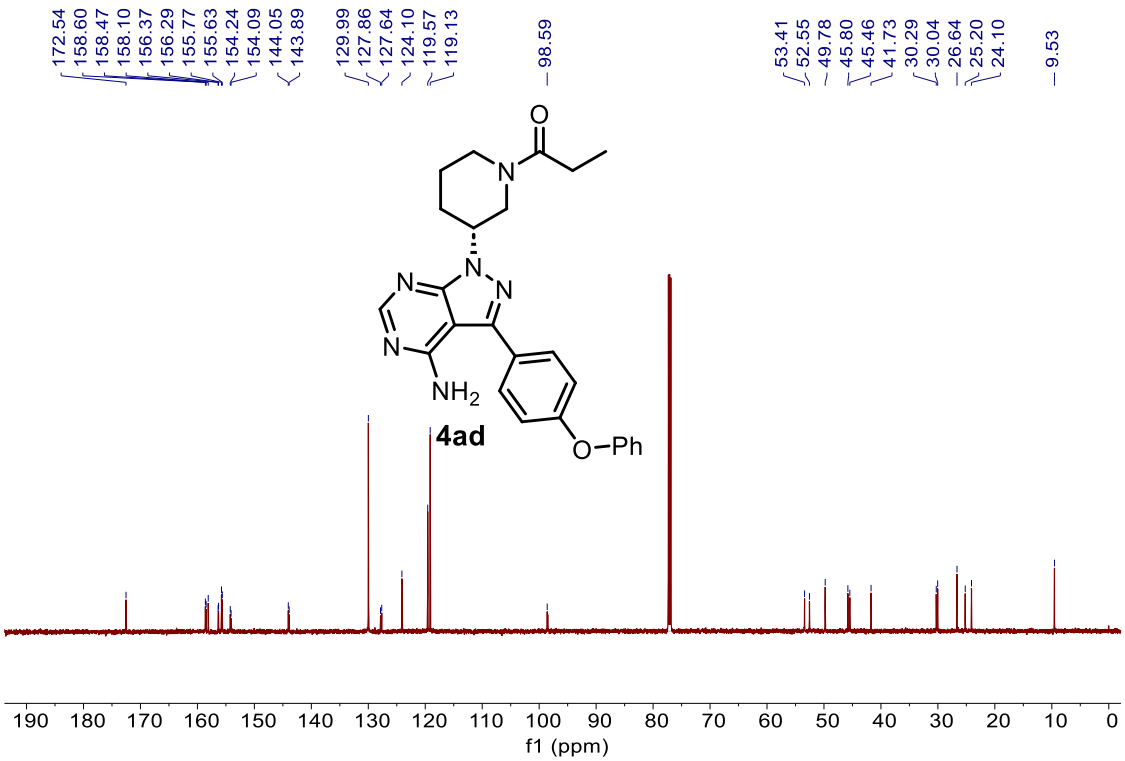
^{1}H NMR (500 MHz, Acetone-d_6 / DMSO-d_6)



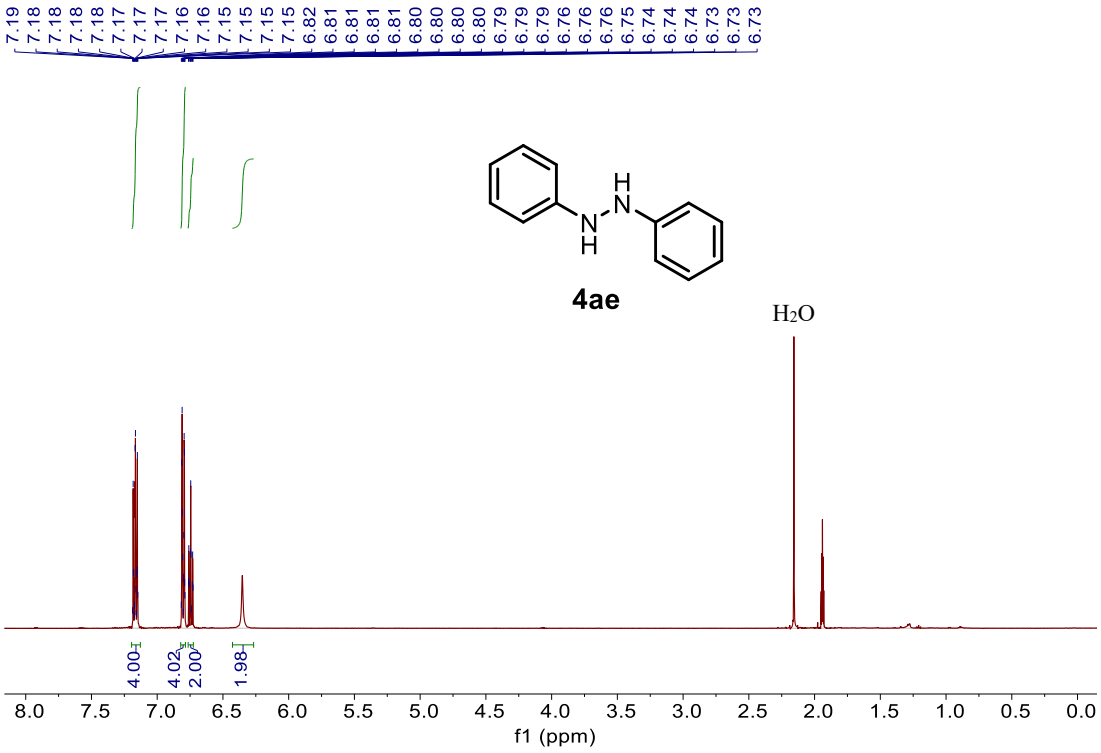
^{13}C NMR (126 MHz, Acetone-d_6 / DMSO-d_6)



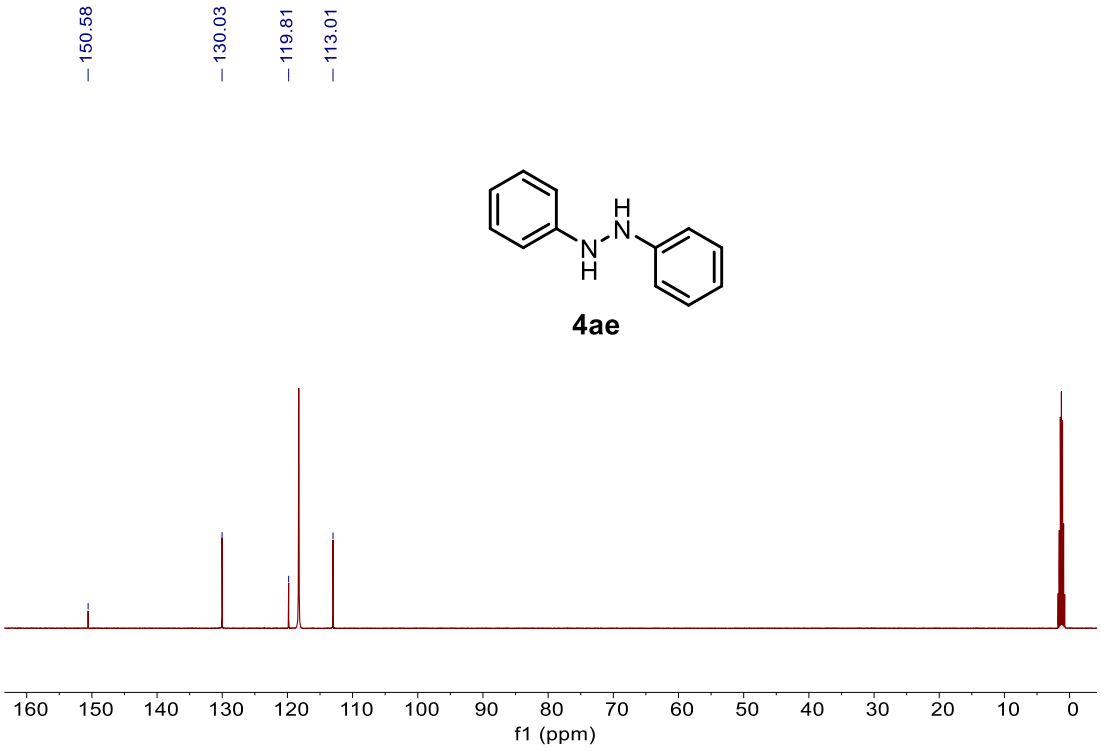
^{1}H NMR (600 MHz, Chloroform-*d*)



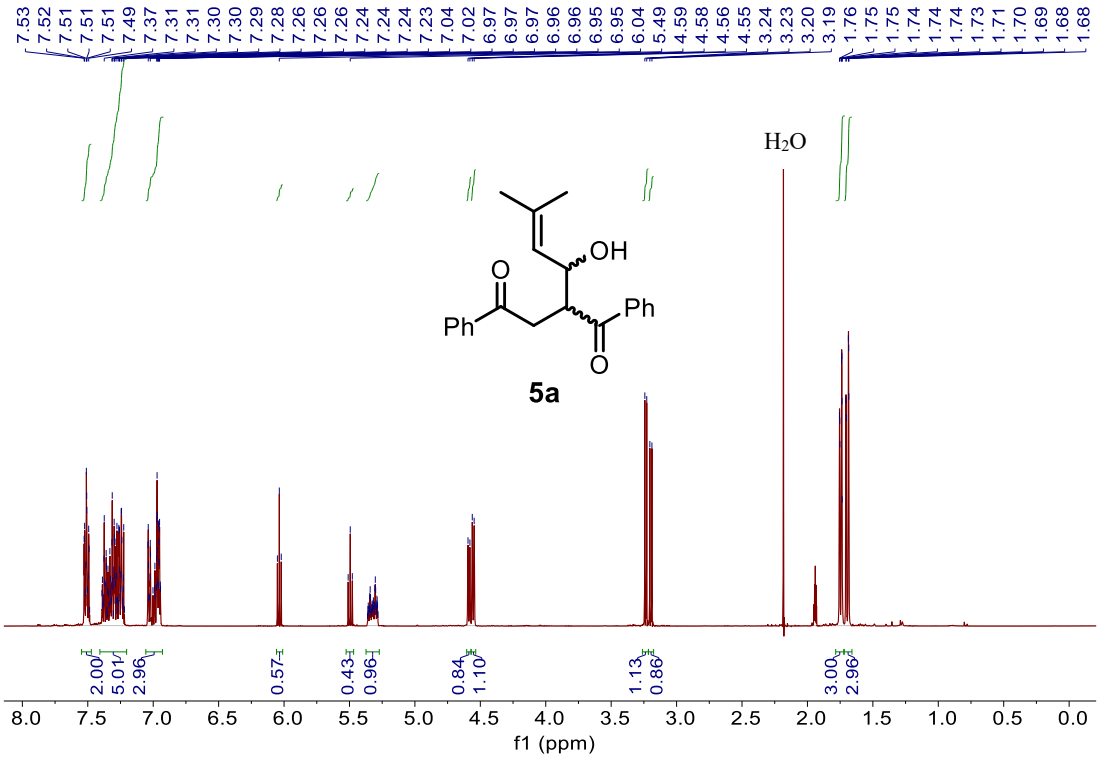
^{13}C NMR (151 MHz, Chloroform-*d*)



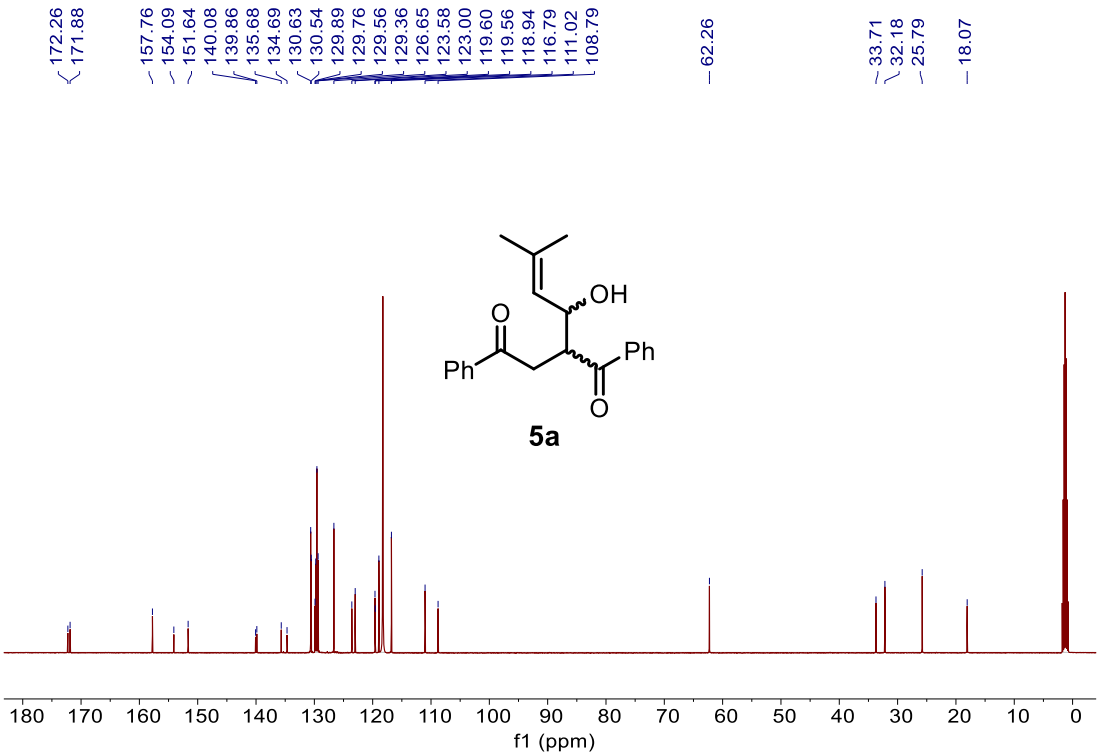
^{1}H NMR (500 MHz, CD_3CN)



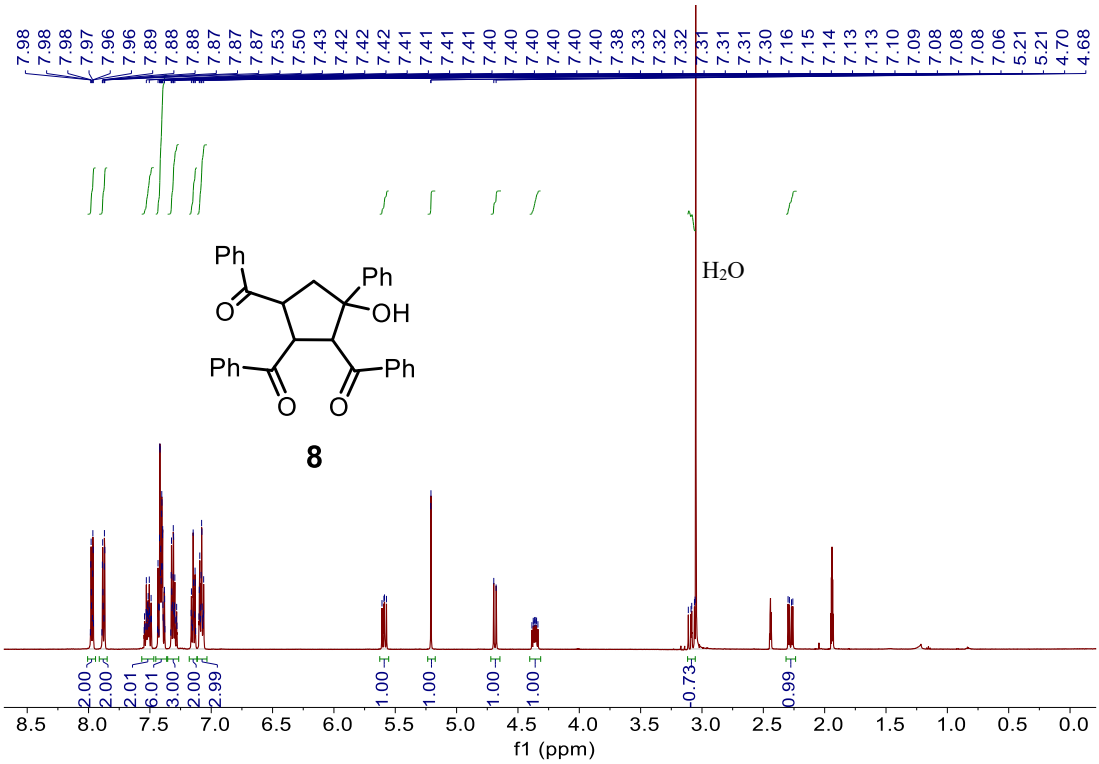
^{13}C NMR (126 MHz, CD_3CN)



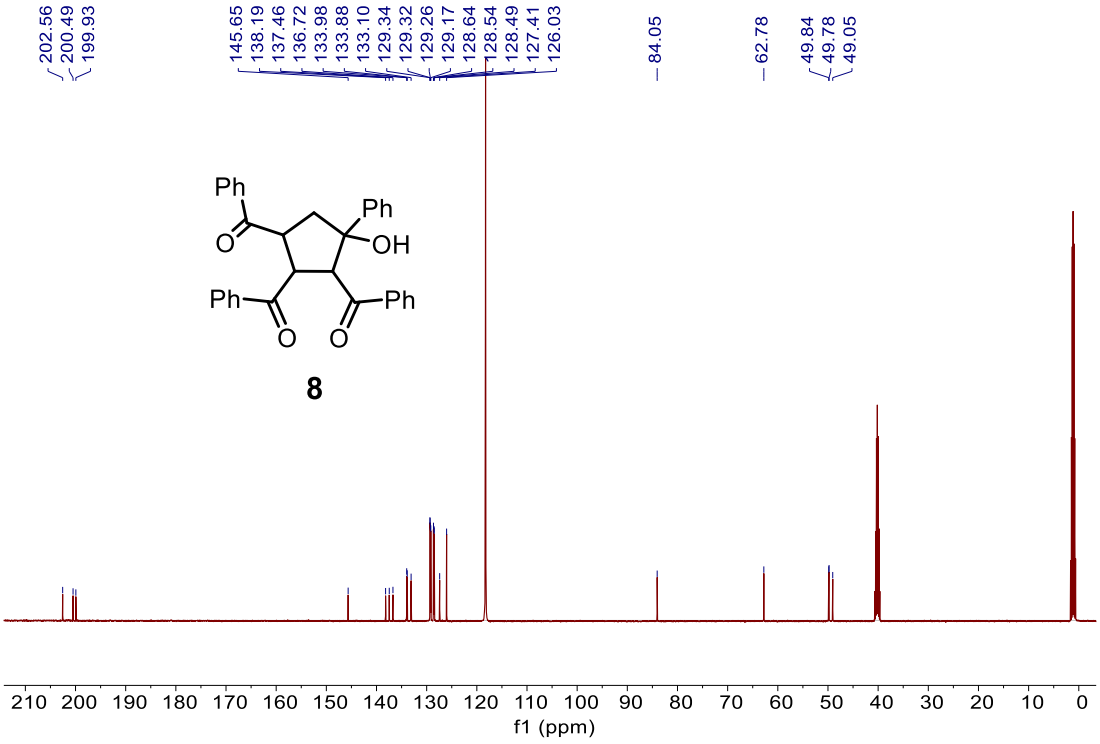
^{1}H NMR (500 MHz, Acetonitrile-d_3)



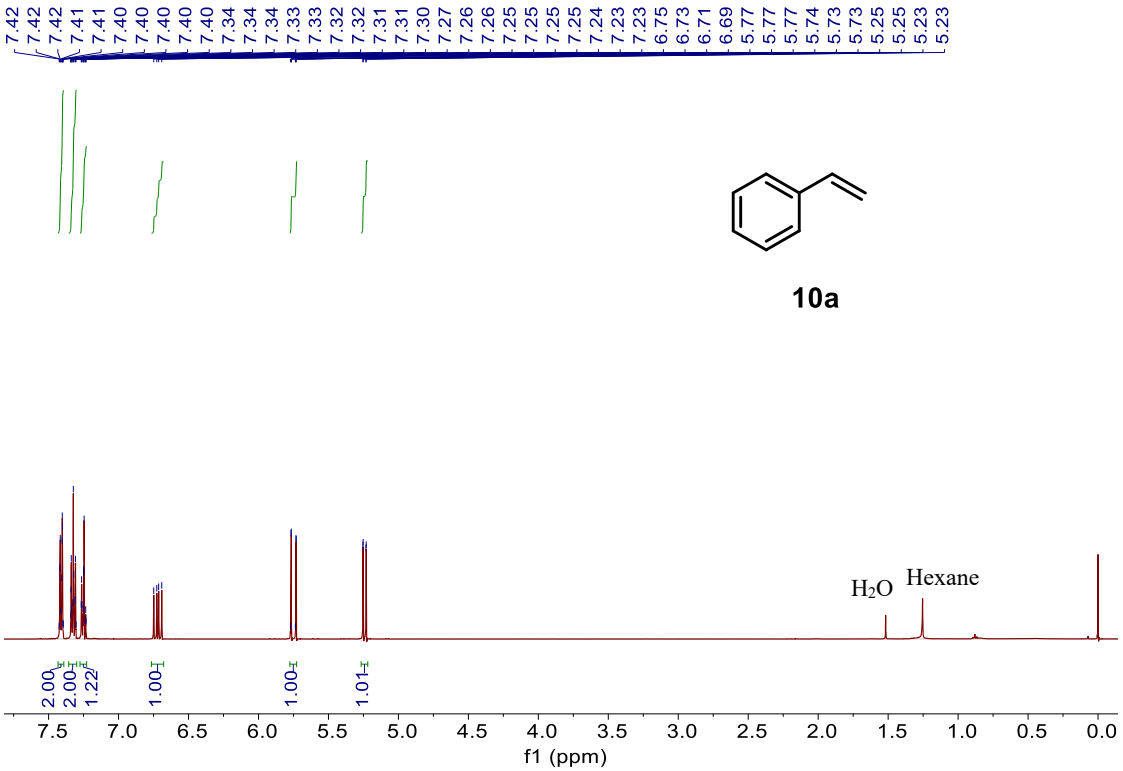
^{13}C NMR (126 MHz, Acetonitrile-d_3)



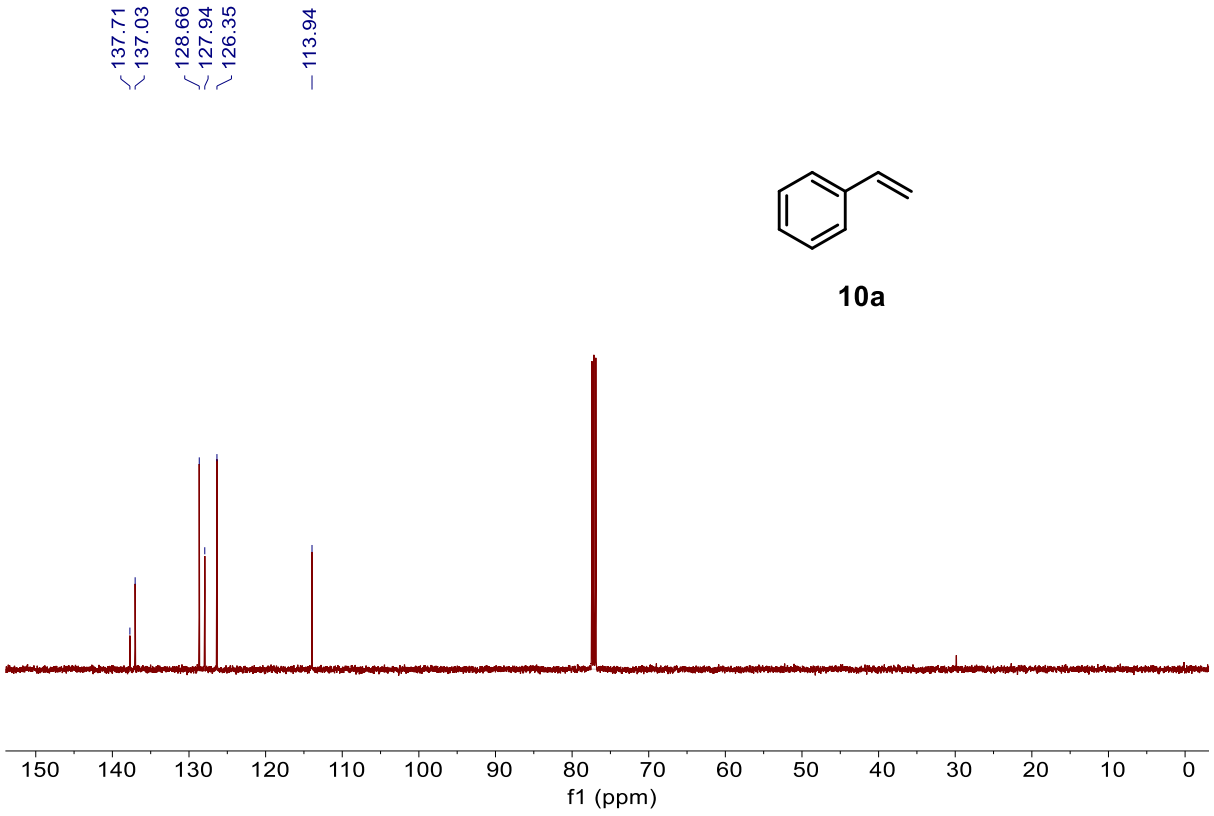
^{1}H NMR (500 MHz, Acetonitrile-d_3 / DMSO-d_6)



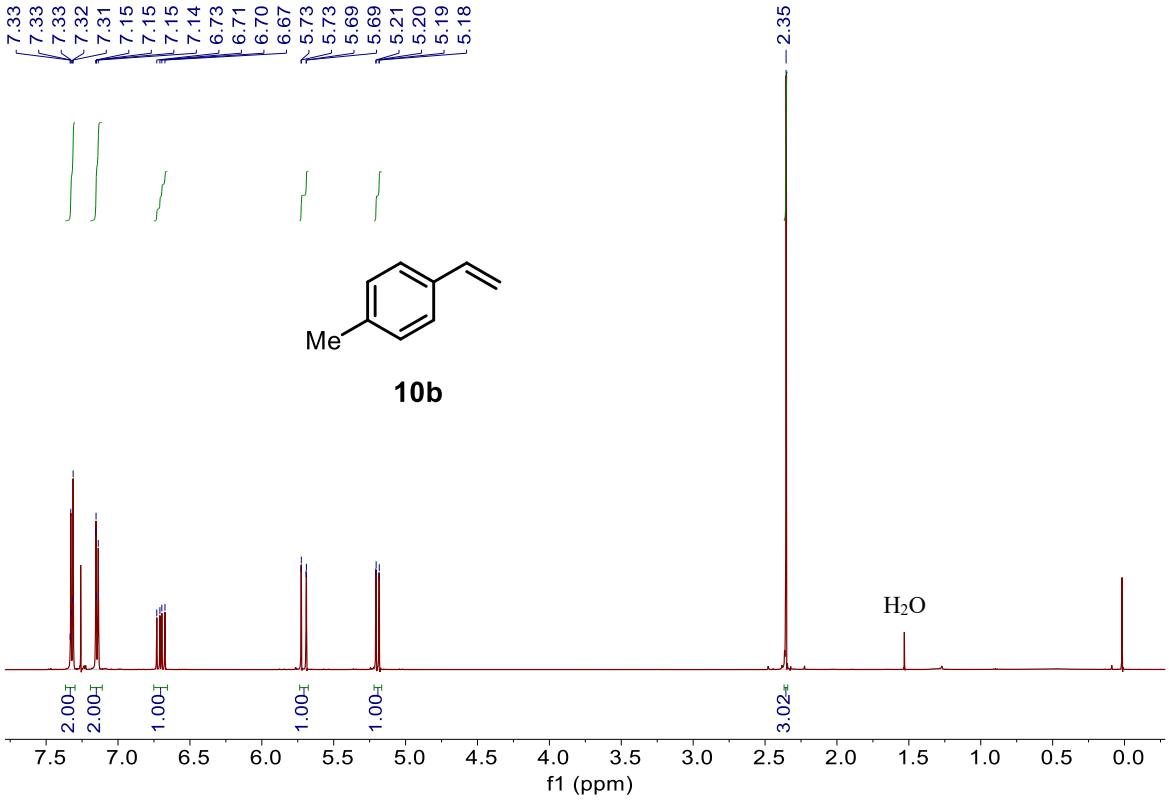
^{13}C NMR (126 MHz, Acetonitrile-d_3/ DMSO-d_6)



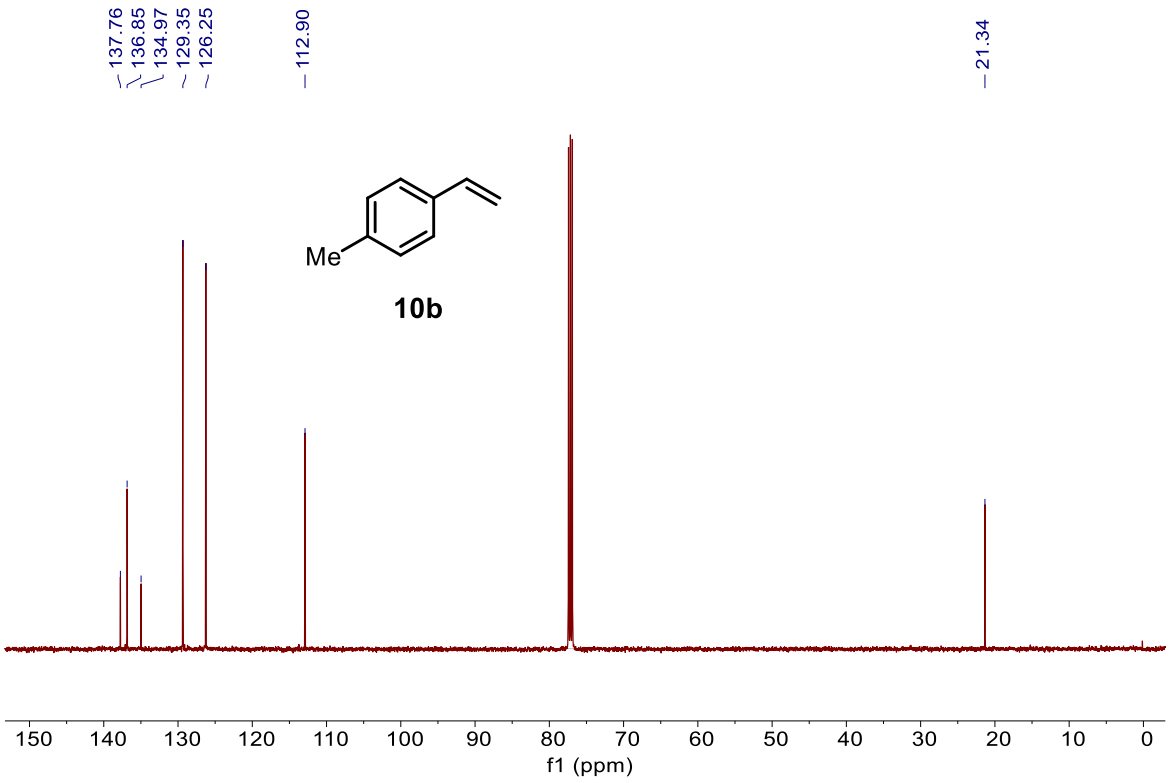
^{1}H NMR (500 MHz, Chloroform-*d*)



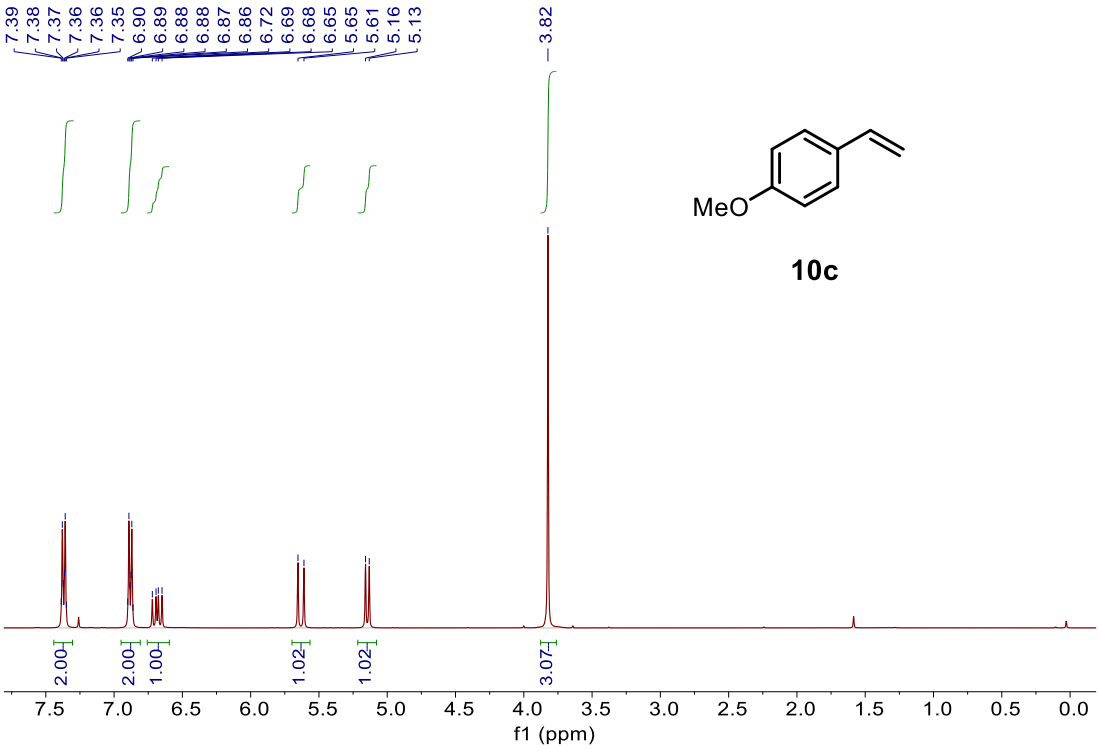
^{13}C NMR (126 MHz, Chloroform-*d*)



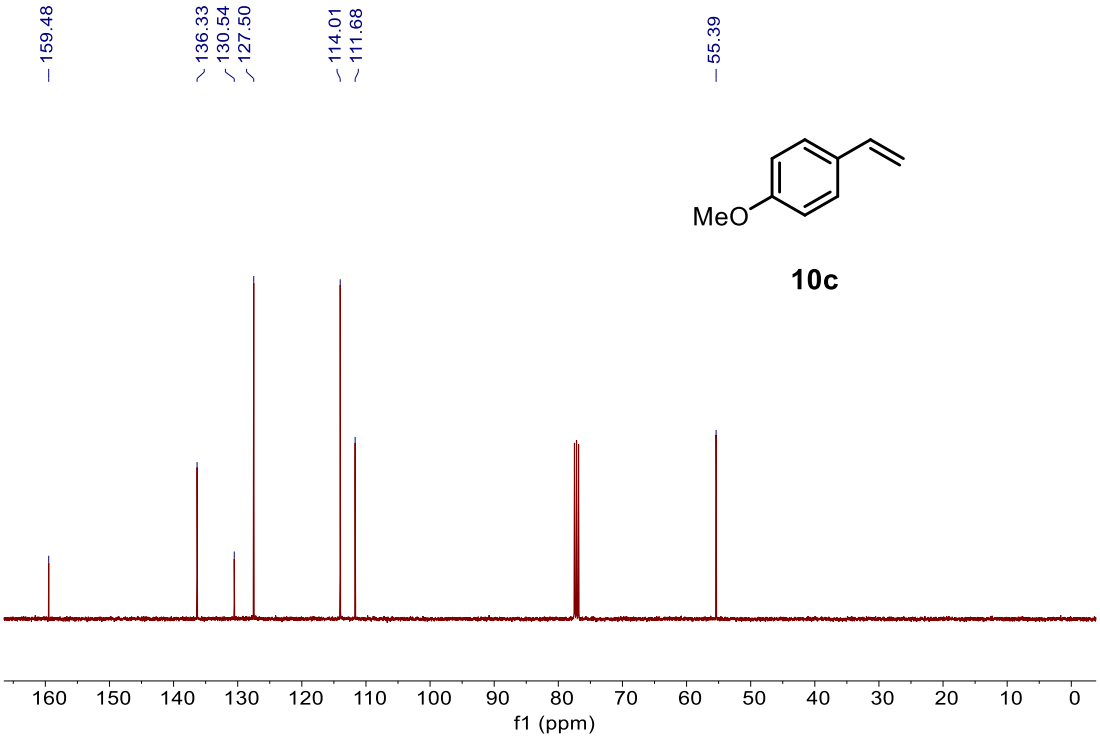
^{1}H NMR (500 MHz, Chloroform-*d*)



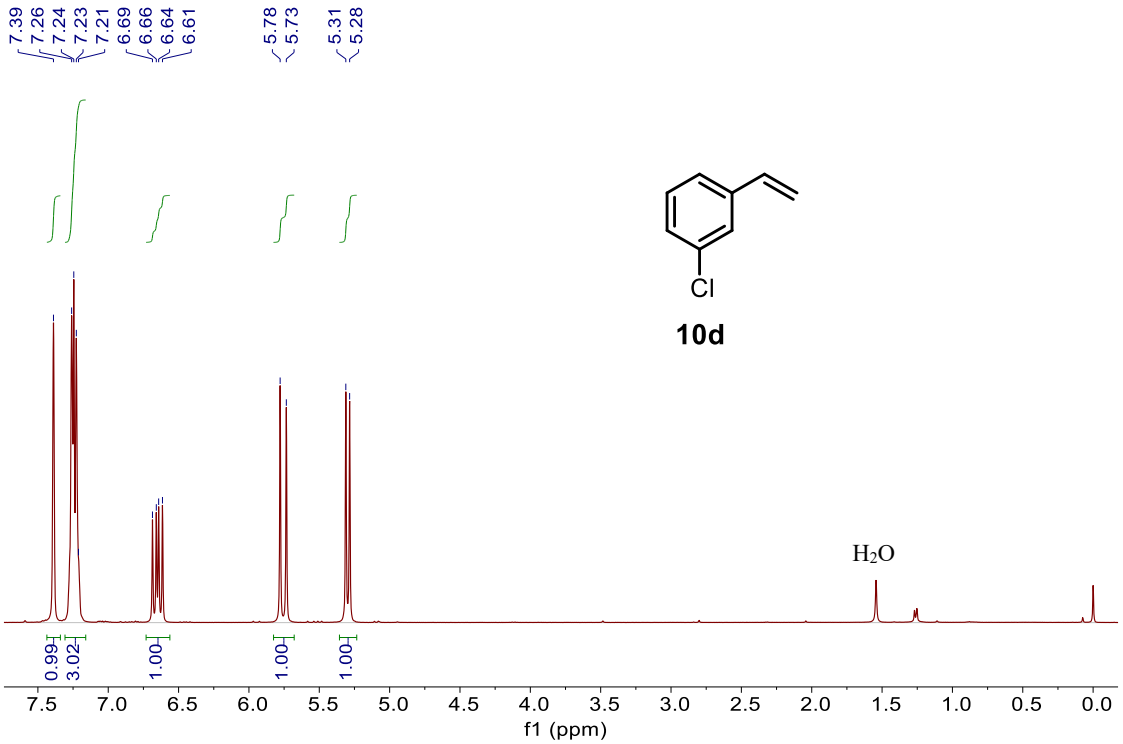
^{13}C NMR (126 MHz, Chloroform-*d*)



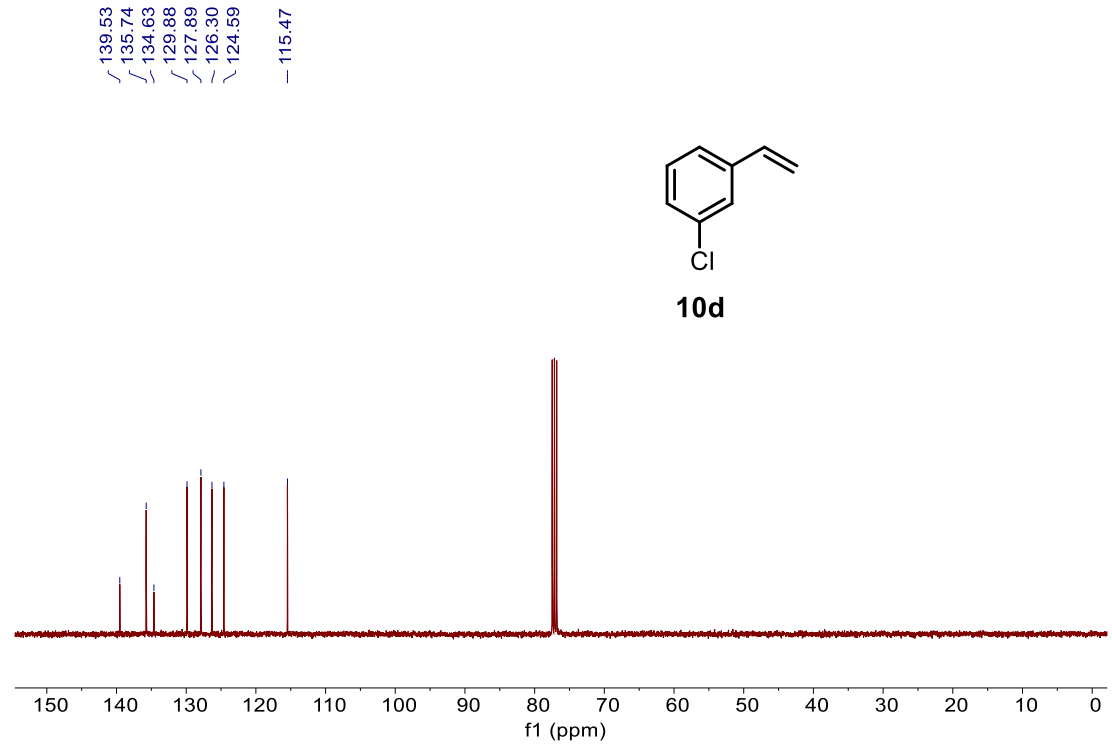
^{1}H NMR (400 MHz, Chloroform-*d*)



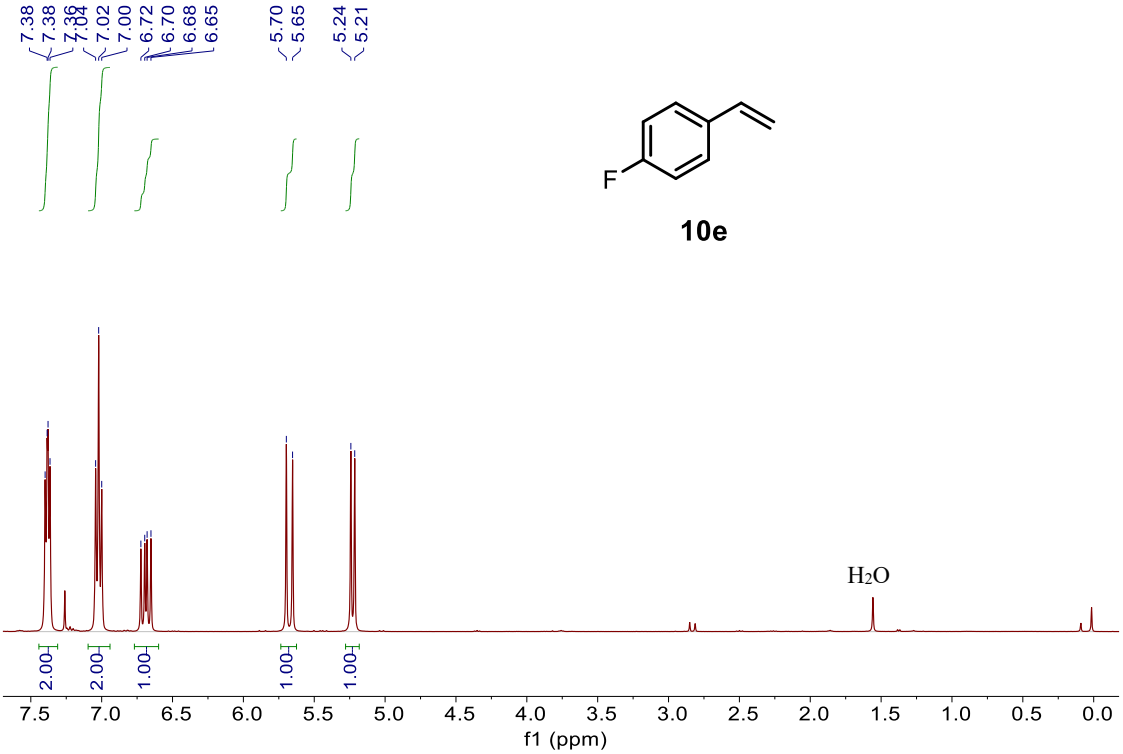
^{13}C NMR (101 MHz, Chloroform-*d*)



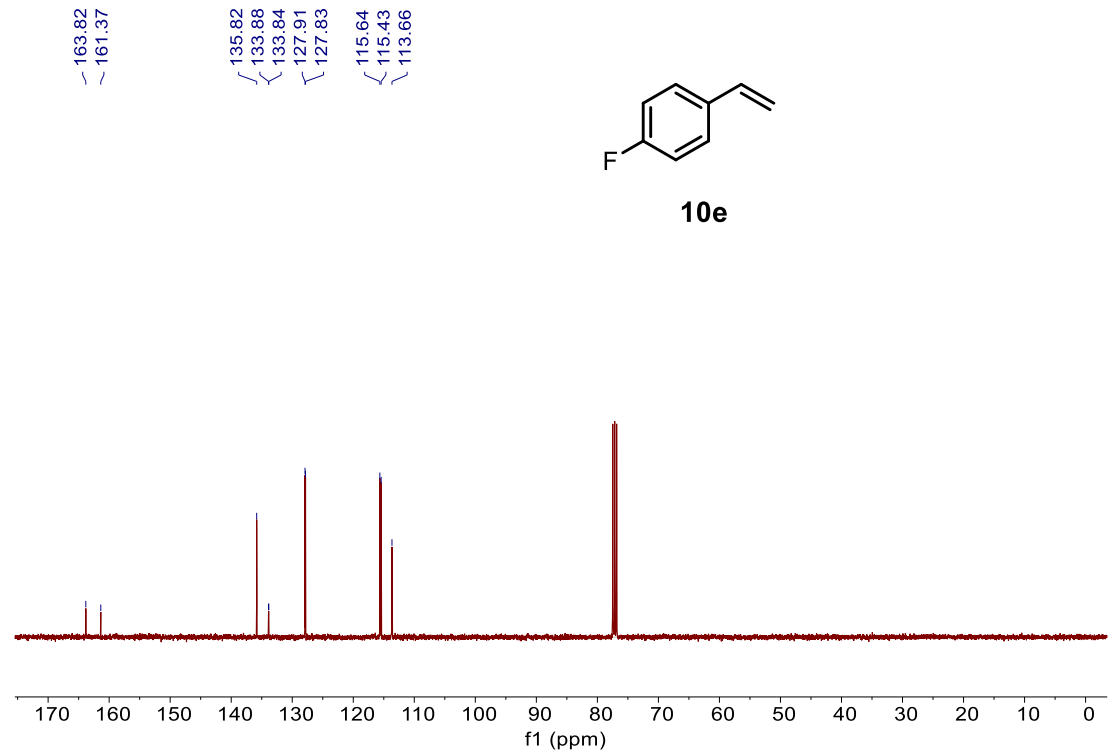
^{1}H NMR (400 MHz, Chloroform-*d*)



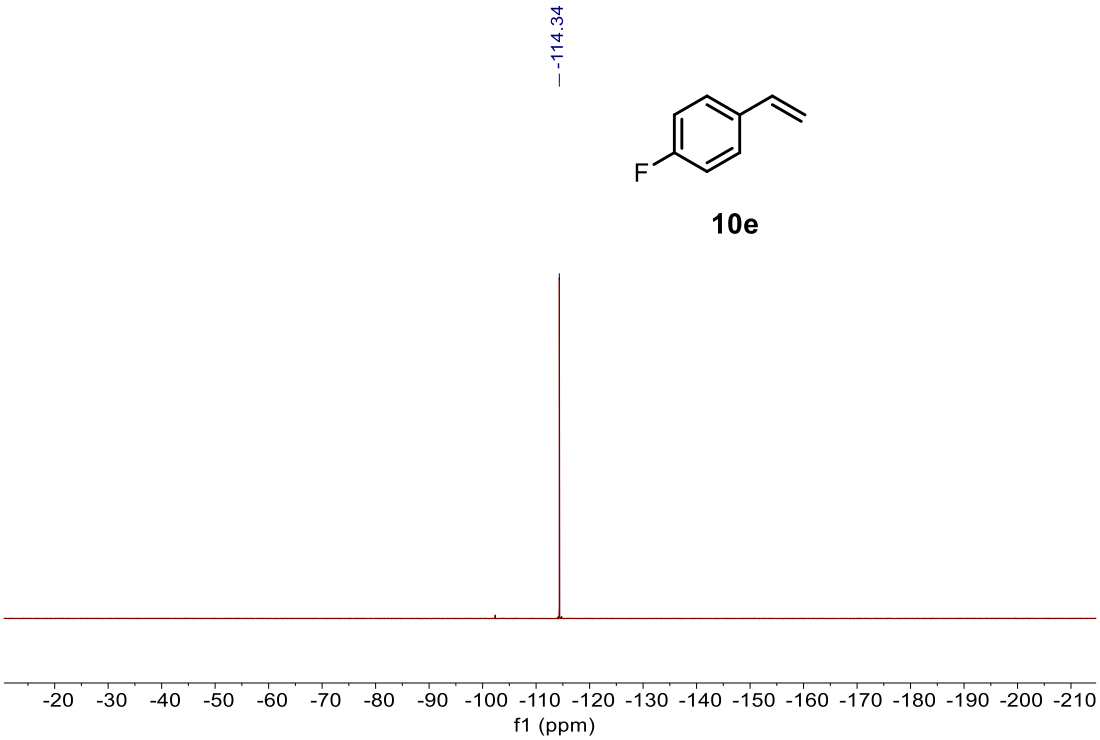
^{13}C NMR (101 MHz, Chloroform-*d*)



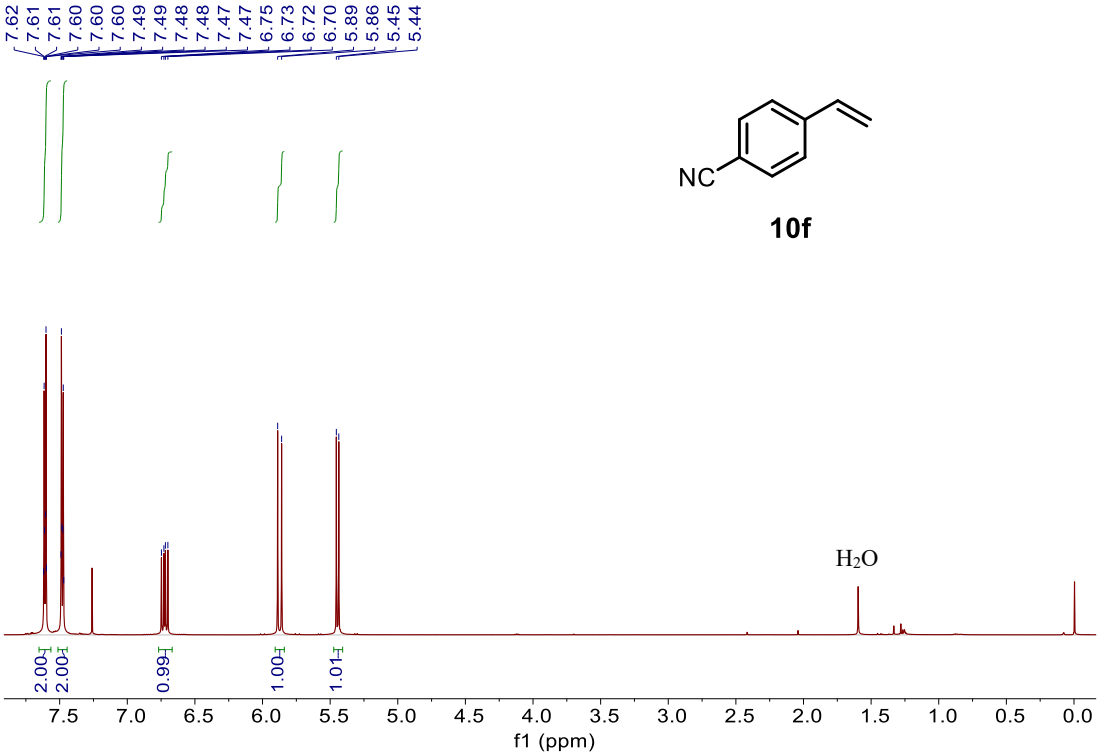
^{1}H NMR (400 MHz, Chloroform-*d*)



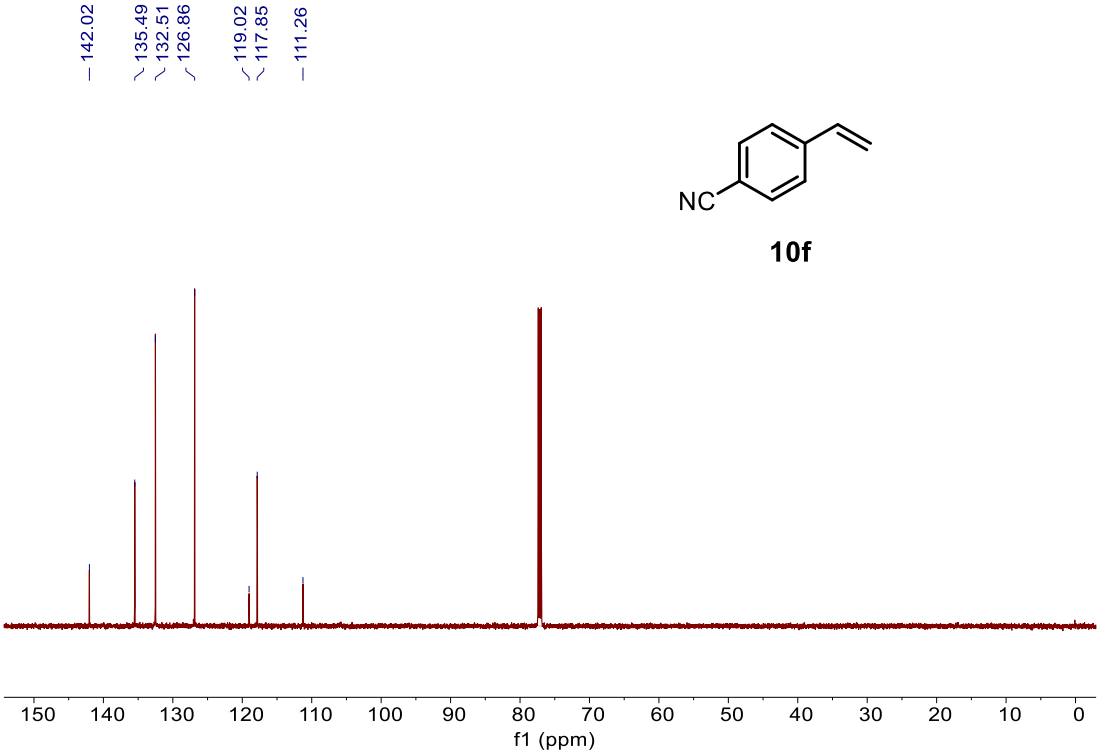
^{13}C NMR (101 MHz, Chloroform-*d*)



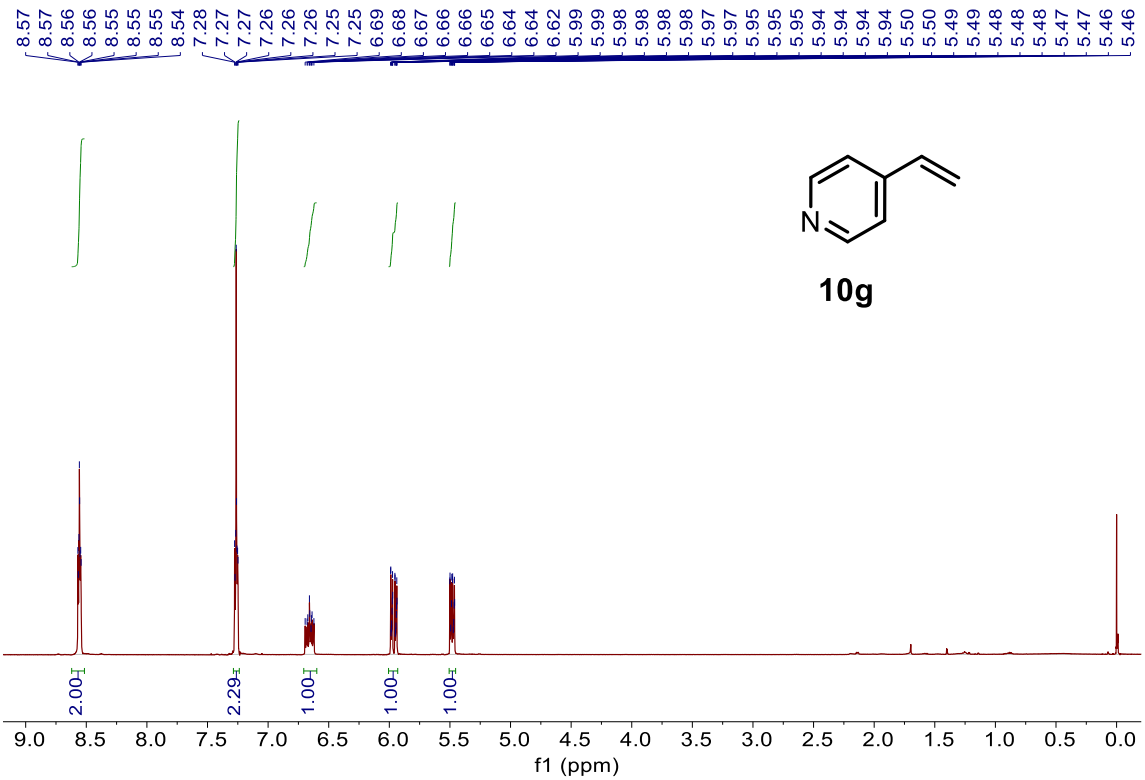
^{19}F NMR (377 MHz, Chloroform-*d*)



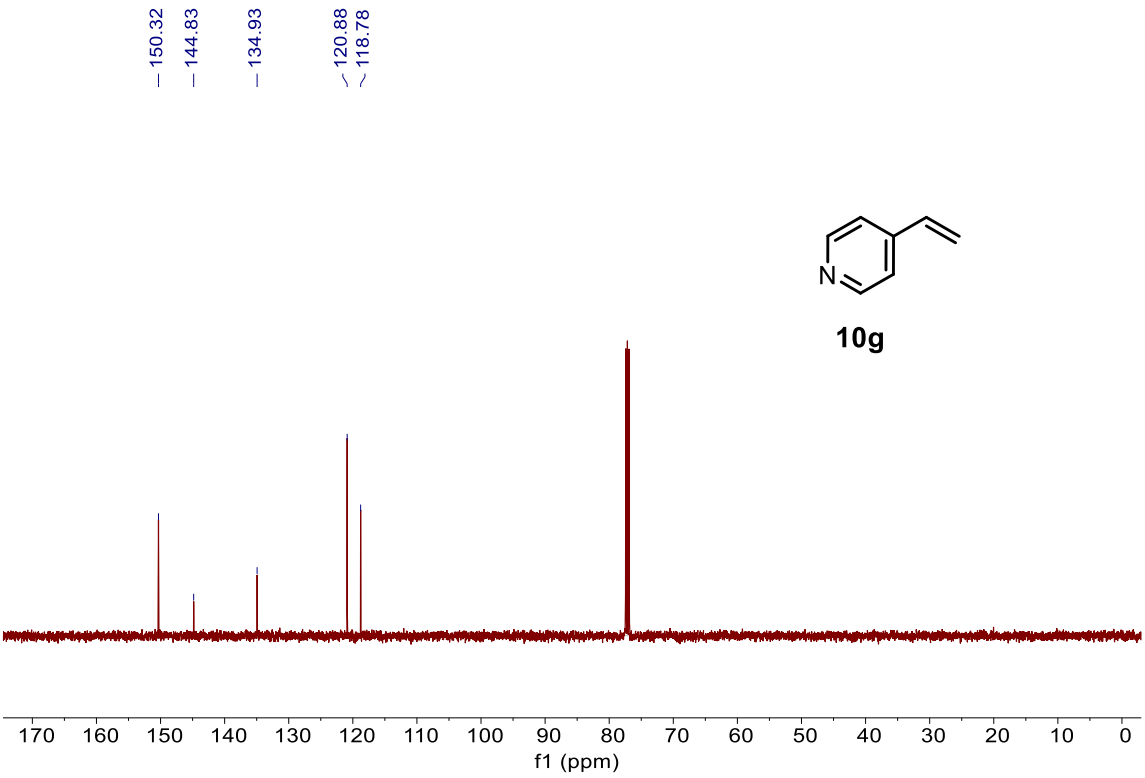
^{1}H NMR (600 MHz, Chloroform-*d*)



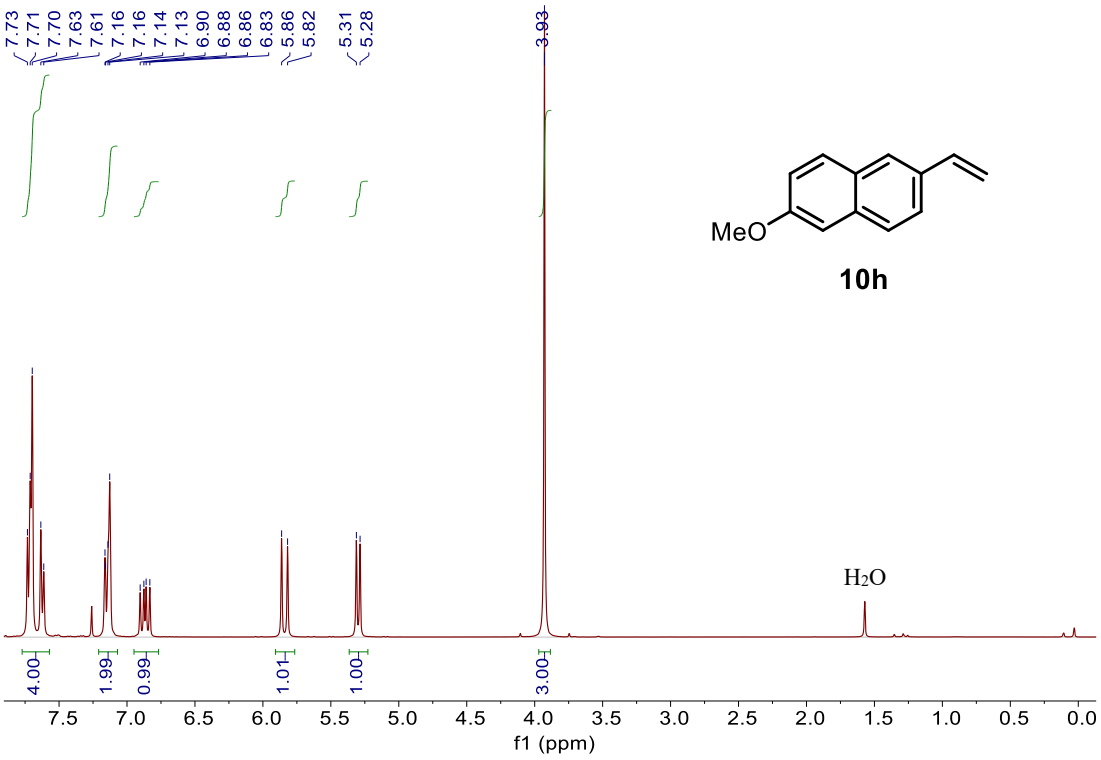
^{13}C NMR (151 MHz, Chloroform-*d*)



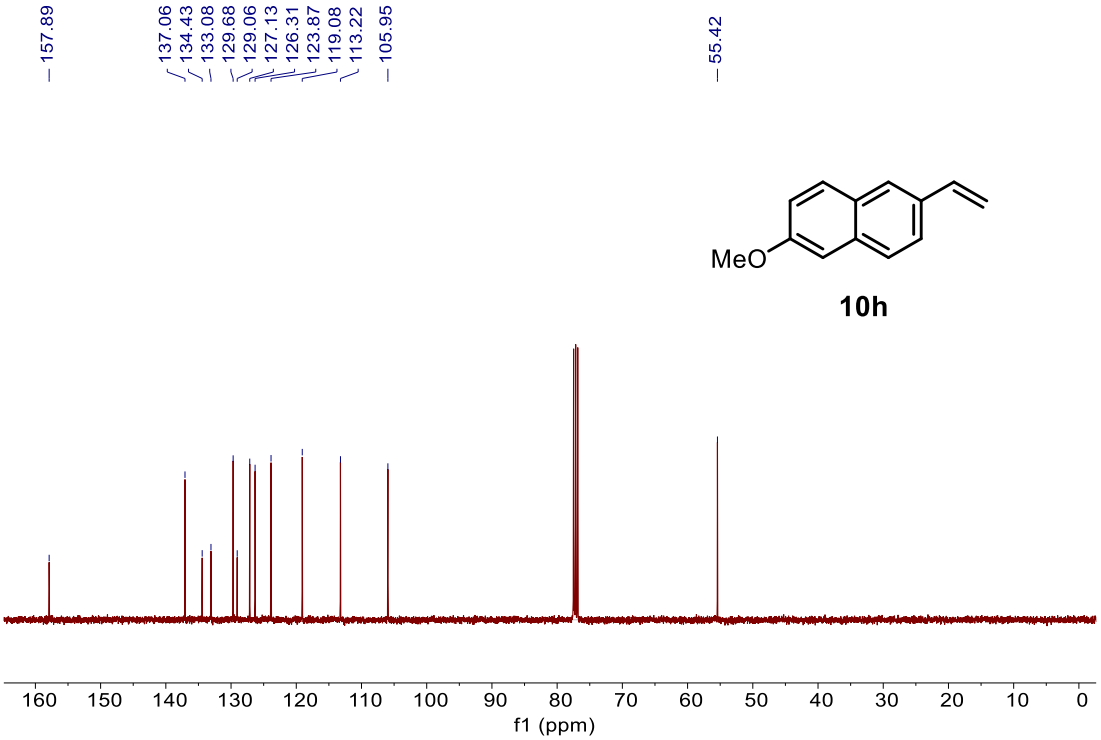
^{1}H NMR (500 MHz, Chloroform-*d*)



^{13}C NMR (126 MHz, Chloroform-*d*)



^{1}H NMR (400 MHz, Chloroform-*d*)



^{13}C NMR (101 MHz, Chloroform-*d*)

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