Non-Directed Pd-Catalyzed Electrooxidative Olefination of Arenes

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Electronic Supporting Information

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1. General Considerations

1.1. Reagent Information: Unless otherwise stated, all the catalytic reactions were carried out in undivided electrochemical cells using pre-dried sample vowl glassware with magnetic stirring under air atmosphere. All the palladium salts were purchased from Alfa Aesar and used directly for the reaction. Remaining all the other chemicals were purchased from commercial source and used as received. For column chromatography silica gel (100-200 mesh) was supplied from SRL Co. During elution petrolium ether and ethyl acetate mixture was used. Thin layer chromatography was performed on EMD Chemicals Si 60 F_{254}. TLC plates (silica gel $60F_{254}$) were supplied from MERCK. AXIOMET AX-3003P potentiostat is used for electrocatalysis with constant current mode. Graphite felt (GF) electrodes (25 mm × 10 mm × 3 mm) and platinum electrodes (10 mm × 10 mm × 0.5 mm) were connected with commercially available copper wire. And the platinum electrode was obtained from Chempur and GF is from sglcarbon (SIGRACELL®).

1.2. Analytical Information: ^{1}H NMR, ^{13}C NMR spectroscopy, IR and HRMS were used to characterize all the isolated compounds. Copies of the ^{1}H NMR, ^{13}C NMR spectra were attached in this supporting information. All NMR spectra were recorded on a BRUKER 400 MHz or BRUKER 500 MHz instrument. All ^{1}H NMR spectra are reported in parts per million (ppm) downfield of TMS and were measured relative to residual $CHCl_3$ (7.26 ppm). All ^{13}C NMR spectra were reported in ppm relative to $CDCl_3$ (77.16 ppm) and were obtained with proton decoupling. Coupling constants, J, are reported in Hertz. All NMR analysis for optimization were performed with 1,3,5-trimthoxybenzene as the internal standard. All ^{13}C NMR spectra were obtained with ^{1}H decoupling. High-resolution mass spectra (HRMS) were recorded on a micro-mass ESI TOF (time of flight) mass spectrometer. UV/vis absorption spectra were recorded on Agilent Cary 8454 UV-vis spectrophotometer, equipped with a temperature control unit at 25 °C. The samples were measured in quartz cuvettes (chamber volume = 3.0 mL) fitted with a stopper.

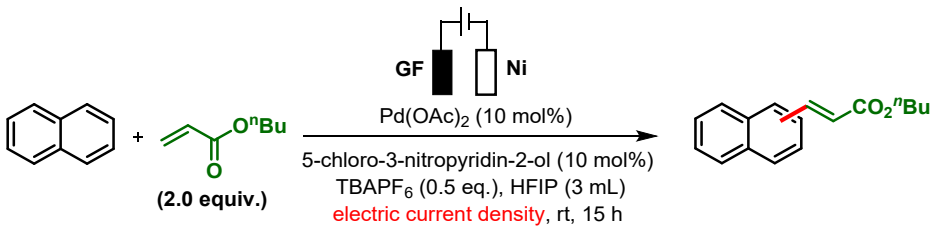
2. Preparation of Starting Materials for Non-Directed C-H Electroolefination

All the starting materials like ester, phenol, olefins and thiophene derivatives are prepared based on the following literature reports.[1-4]

3. Optimization Details for Non-Directed Electroolefination

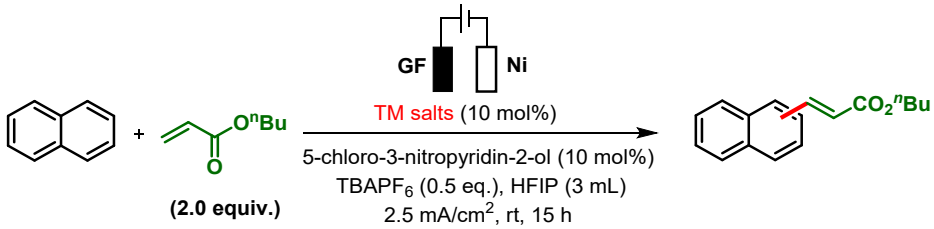
Yield and selectivity were determined by ^1H NMR analysis of the crude product using TMB as the internal standard.

Table S1: Current Optimization.



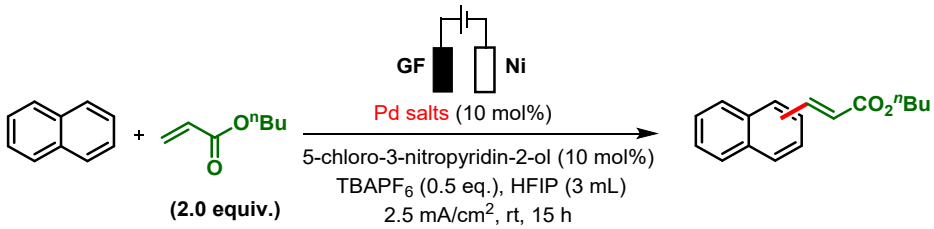
Entry	Electric Current (mA/cm^2)	Yield (%)	Selectivity (β:α)
1	0.2	15	2:1
2	0.4	13	2:1
3	0.6	17	2:1
4	0.8	34	7:1
5	1	19	2:1
6	1.2	16	2:1
7	1.4	51	7:1
8	1.6	42	6:1
9	1.8	49	7:1
10	2.0	51	8:1
11	2.2	46	7:1
12	**2.5**	**54**	**9:1**
13	2.6	50	7:1
14	2.8	43	6:1
15	3.0	45	7:1
16	3.2	47	7:1
17	3.4	26	4:1
18	3.6	21	4:1
19	3.8	27	4:1
20	4.0	31	5:1
21	6.0	19	4:1
22	8.0	15	3:1
23	10	Trace	-
24	No current	NR	-

Table S2: Transition Metal Salts Optimization.



Entry	Metal Salts (10 mol%)	Yield (%)	Selectivity (β:α)
1	No catalyst	NR	-
2	$Co(II)(OAc)_2.4H_2O$	9	1:1
3	$[Ru(p\text{-cymene})Cl_2]_2$	NR	-
4	$[Cp^*RhCl_2]_2$	NR	-
5	**$Pd(OAc)_2$**	**54**	**9:1**

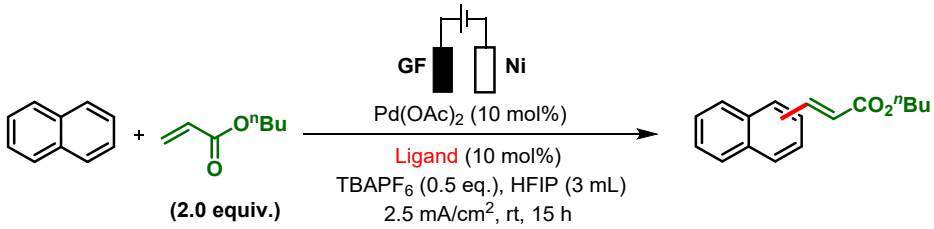
Table S3: Pd Salts Optimization.



Entry	Pd Salts (10 mol%)	Yield (%)	Selectivity (β:α)
1	**$Pd(OAc)_2$**	**54**	**9:1**
2	$Pd(acac)_2$	37	7:1
3	$Pd(PPh_3)_2Cl_2$	NR	-
4	$Pd(PhCN)_2Cl_2$	NR	-
5	$Pd(CH_3CN)_2Cl_2$	21	4:1
6	$Pd(OPiv)_2$	38	5:1
7[a]	$Pd(OAc)_2$	48	8:1
8[b]	$Pd(OAc)_2$	56	8:1
9[c]	**$Pd(OAc)_2$**	**59**	**9:1**

[a]5 mol%, [b]15 mol%, [c]20 mol%

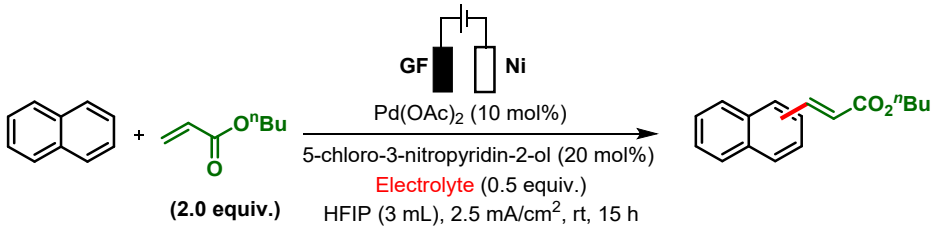
Table S4: Ligands Optimization.



Entry	Ligands (10 mol%)	Yield (%)	Selectivity (β:α)
1	**5-chloro-3-nitropyridin-2-ol**	**54**	**9:1**
2	3-nitropyridin-2-ol	46	8:1
3	3,5-dichloropyridin-2(1H)-one	51	9:1
4	5-methyl-3-nitropyridin-2-ol	34	5:1
5	5-nitropyridin-2-ol	25	5:1
6	2-hydroxy-4,6-dimethylnicotinonitrile	NR	NR
7	pyridin-2-ol	40	6:1
8	5-(trifluoromethyl)pyridin-2-ol	35	5:1
9	6-chloropyridin-2-ol	20	2:1
10	6-methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile	NR	NR
11	3-aminopyridin-2-ol	9	1:1
12	4-(benzyloxy)pyridin-2(1H)-one	15	5:1
13	Picolinonitrile	30	6:1
14	pyridine-2-sulfonic acid	32	3:1
15	1-(pyridin-2-yl)ethan-1-one	20	3:1
16	Quinoline	10	2:1
17	*N*-Acetyl-Glycine	Trace	-
18	*N*-Ac-L-Leucine	Trace	-
19	*N*-Boc-L-*tert*-Leucine	7	1:1
20[a]	5-chloro-3-nitropyridin-2-ol	59	9:1
21[b]	**5-chloro-3-nitropyridin-2-ol**	**62**	**11:1**
22	5-chloro-3-nitropyridin-2-ol + *N*-Acetyl-Glycine	60	11:1
22[c]	5-chloro-3-nitropyridin-2-ol	51	9:1
23[d]	5-chloro-3-nitropyridin-2-ol	38	7:1
24	No ligand	29	1:1

[a]15 mol%, [b]20 mol%, [c]30 mol%, [d]5 mol%

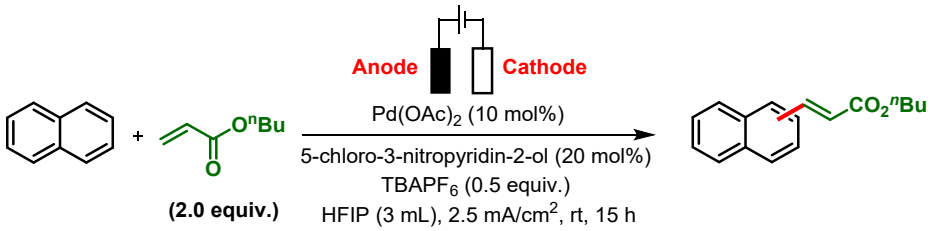
Table S5: Electrolyte Optimization.



Entry	Electrolyte (0.5 equiv.)	Yield (%)	Selectivity (β:α)
1	**$TBAPF_6$**	**62**	**11:1**
2	$TBABF_4$	32	9:1
3	Choline chloride	NR	-
4	$TBAClO_4$	46	9:1
5	TBABr	NR	-
6	TBACl	45	7:1
7	TBAI	NR	-
8	$TBABH_4$	22	6:1
9	TBAOAc	50	9:1
10	$TBAHSO_4$	49	9:1
11	TEAP	41	11:1
12	NH_4Cl	NR	-
13	NH_4PF_6	33	5:1
14	$LiClO_4$	NR	-
15	Et_4NBF_4	NR	-
16	LiOAc	NR	-
17	$NaClO_4$	NR	NR
18[a]	$TBAPF_6$	53	11:1
19[b]	$TBAPF_6$	41	11:1

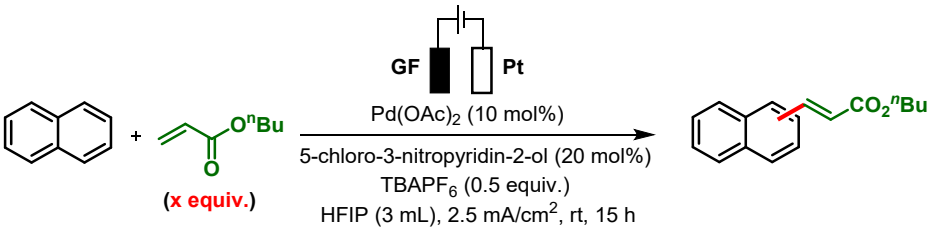
[a]1.0 equiv., [b] 0.25 equiv.

Table S6: Electrode Optimization.



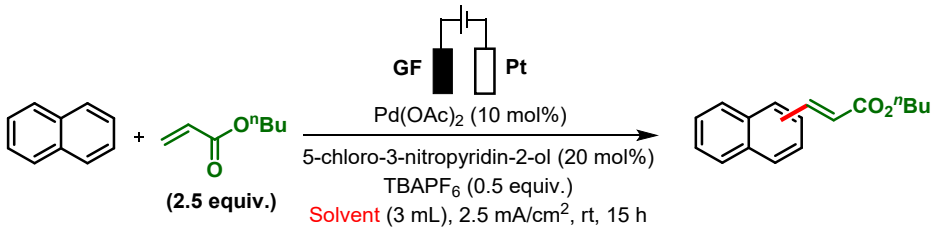
Entry	Electrode (Anode vs Cathode)	Yield (%)	Selectivity (β:α)
1	GF-Ni	62	11:1
2	Ni-GF	47	6:1
3	C-Pt (wire)	59	11:1
4	**GF-Pt (wire gauze)**	**70**	**11:1**
5	GF-Pt (wire ring)	31	5:1
6	GF-Steel	24	4:1
7	GF-GF	48	5:1
8	Ni-Ni	53	7:1
9	Ni-Pt (wire gauze)	58	6:1
10	GF-Pt (plate)	21	6:1
11	Pt (wire gauze)-GF	49	8:1
12	Pt (plate)-Ni	41	8:1
13	Pt (plate)-Pt (plate)	52	10:1
14	Pt (plate)-Pt (wire gauze)	48	9:1

Table S7: Olefin Amount Optimization.



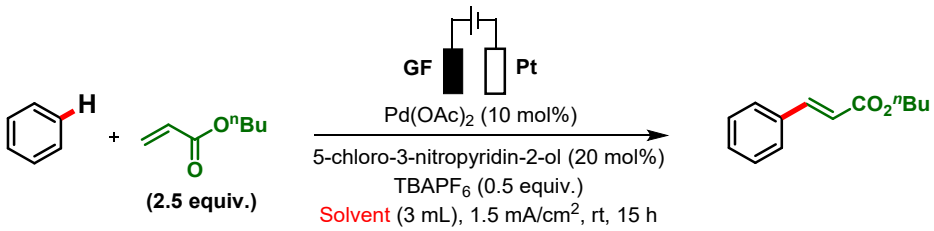
Entry	Olefin amount (equiv.)	Yield (%)	Selectivity (β:α)
1	1.0	37	8:1
2	1.5	49	10:1
3	2.0	70	11:1
4	**2.5**	**74**	**11:1**
5	3.0	72	11:1
6	3.5	62	10:1

Table S8: Solvent Optimization.

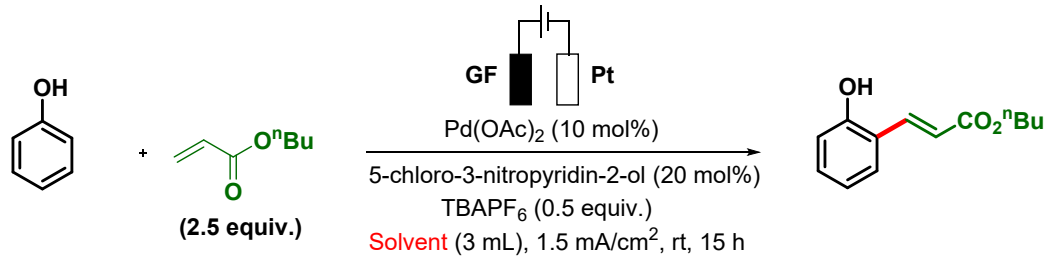


Entry	Solvents	Yield (%)	Selectivity (β:α)
1	**HFIP**	**74**	**11:1**
2	DMA	NR	-
3	AcCN	NR	-
4	MeOH	15	3:1
5	*gamma*-valerolactone	NR	-
6	*gamma*-butyrolactone	10	3:1
7	**DCE**	**70**	**>25:1**
8	HFIP:DCE (1:5)	70	18:1

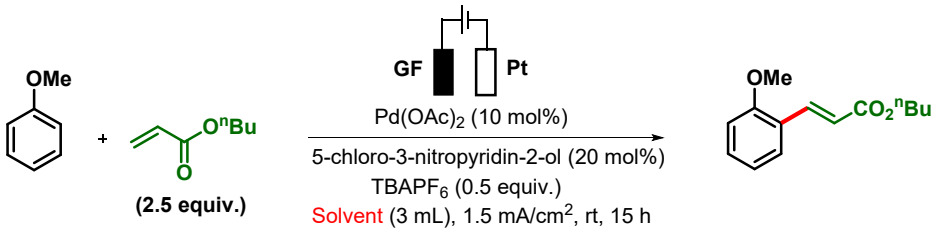
Table S9: Solvent Optimization for Arenes Other than Naphthalene.



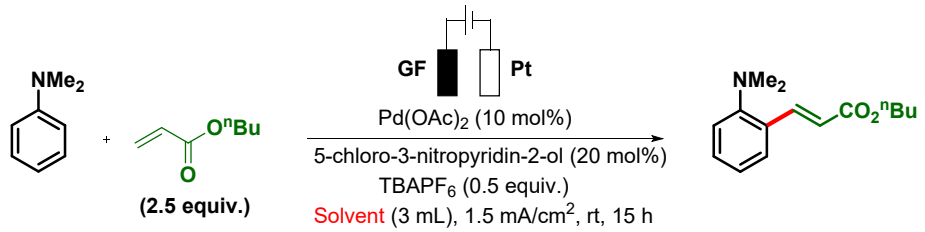
Entry	Solvents (DCE:HFIP)	Yield (%)	Selectivity (β:α)
1	DCE	25	-
2	10:1	33	-
4	9:1	36	-
5	8:1	37	-
6	7:1	41	-
7	6:1	49	-
8	5:1	63	-
9	4:1	62	-
10	3:1	60	-
11	2:1	52	-
12	1:1	47	-
13	HFIP	41	-



Entry	Solvents (DCE:HFIP)	Yield (%)	Selectivity (β:α)
1	DCE	n.r	n.r
2	10:1	n.r	n.r
3	9:1	25	5:1
4	8:1	25	5:1
5	7:1	38	6:1
6	6:1	41	7:1
7	**5:1**	**61**	**9:1**
8	4:1	57	9:1
9	3:1	58	9:1
10	2:1	58	9:1
11	1:1	49	9:1
12	HFIP	48	6:1



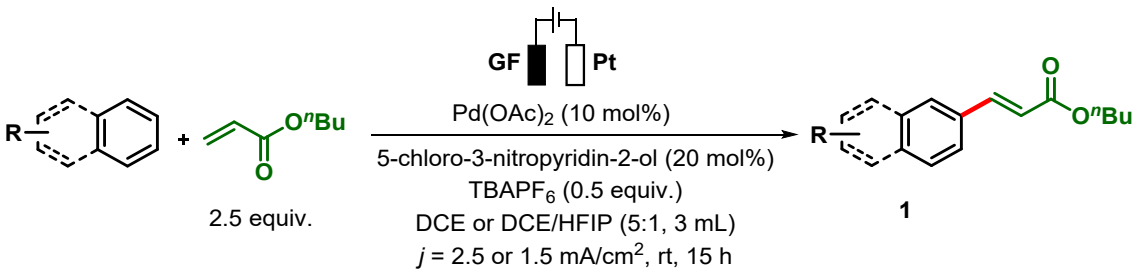
Entry	Solvents (DCE:HFIP)	Yield (%)	Selectivity (β:α)
1	DCE	25	6:1
2	10:1	17	3:1
3	9:1	31	5:1
4	8:1	44	9:1
5	7:1	37	8:1
6	6:1	61	12:1
7	5:1	72	15:1
8	4:1	72	15:1
9	3:1	74	15:1
10	**2:1**	**75**	**15:1**
11	1:1	53	10:1
12	HFIP	35	8:1



Entry	Solvents (DCE:HFIP)	Yield (%)	Selectivity (*o*:*others*)
1	DCE	25	3:1
2	10:1	28	3:1
3	9:1	38	4:1
4	8:1	52	6:1
5	7:1	57	7:1
6	6:1	50	7:1
7	**5:1**	**70**	**8:1**
8	4:1	58	7:1
9	3:1	66	8:1
10	2:1	64	8:1
11	1:1	43	4:1
12	HFIP	40	4:1

4. Experimental Details

4.1. General Procedure for Non-Directed Electroolefination



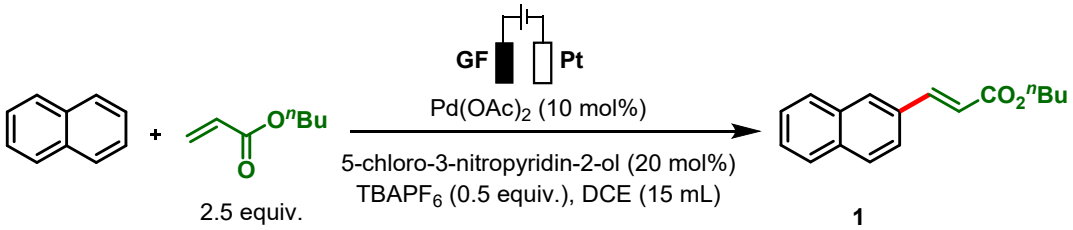
The electrooxidative reaction was carried out in an undivided cell under air, with a graphite felt (GF) anode and a platinum cathode. In an oven-dried sample vial was charged with magnetic stir-bar, corresponding arene (0.2 mmol), $Pd(OAc)_2$ (10 mol%), ligand 5-chloro-3-nitropyridin-2-ol (20 mol%), electrolyte tetra-*n*-butyl ammonium hexafluorophosphate ($TBAPF_6$) (0.5 equiv.), and olefinic coupling partner (0.5 mmol) in 3 mL of dichloroethane (DCE) or 5:1 ratio

of dichloroethane (DCE) and 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP), were added. The reaction mixutre was electrolyzed at a constant current = 1.225 mA or 0.735 mA and current density j = 2.5 mA/ cm^2 or 1.5 mA/ cm^2 with the surface area of electrodes dipped in solution = 0.7 cm x 0.7 cm, with stirring (380 rpm) at room temperature for 15 h. Upon completion, the GF anode was washed with ethyl acetate (10 ml) and filtered through a celite pad. The filtrate was evaporated under reduced pressure and the crude mixture was purified to get the corresponding products 1 by column chromatography using silica (100-200 mesh size) and petroleum ether / ethyl acetate as the eluent. The selectivity was monitored using ^{1}H NMR signal in presence of 1,3,5-trimethoxybenzene as internal standard. The regio-selectivity was determined from ^{1}H NMR spectra of olefinated product.

(**a**) (**b**) (**c**)

Figure S1: Photograph of the electro set up (**a**) Electrode used in the reaction (**b**) Before reaction set up (**c**) Set up during reaction time.

4.2. Gram Scale Experiment:

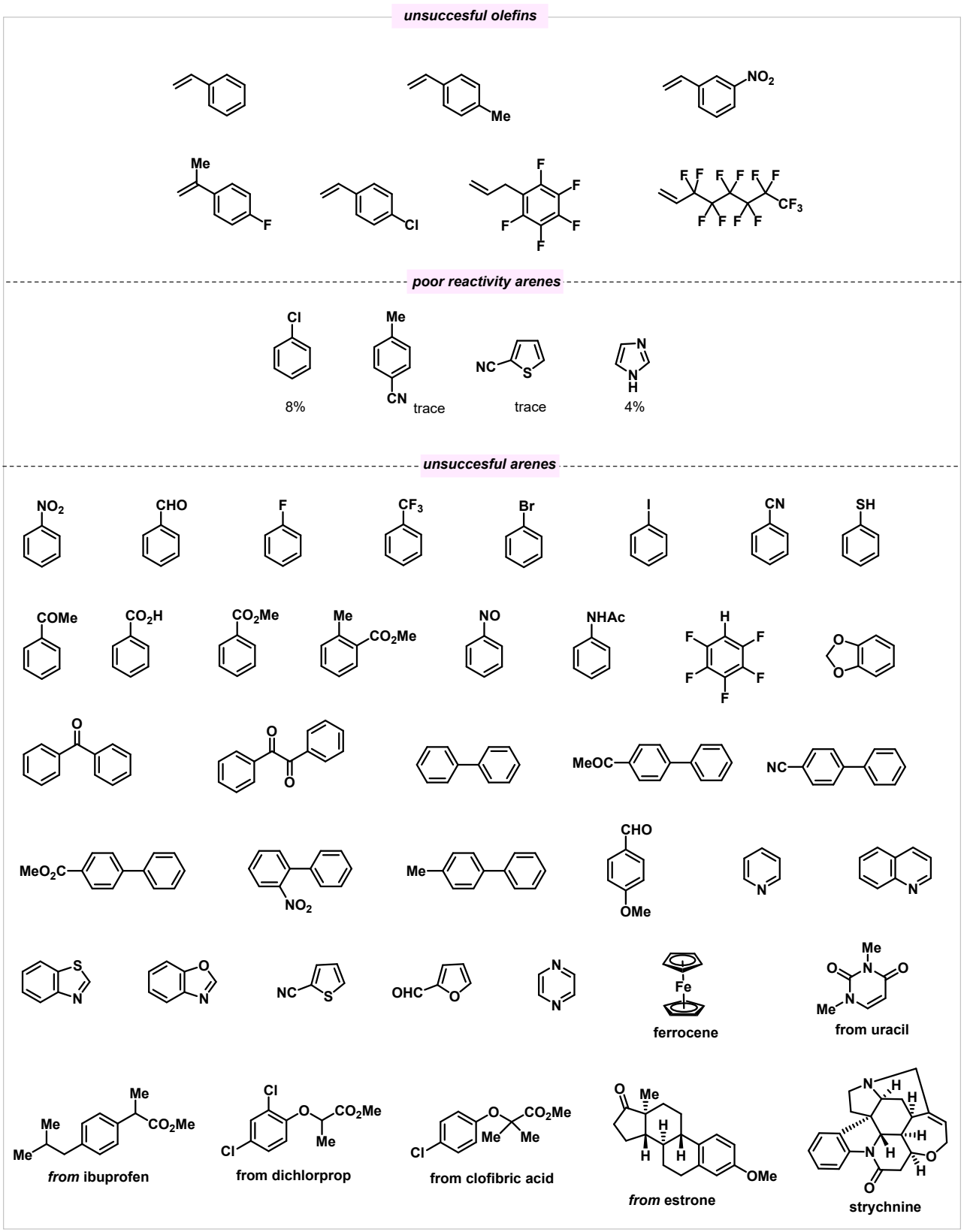


j = 6.0 mA/cm^2, rt, 30 h; 4 mmol, 0.504 g : 46%, (β:α = 7:1)
j = 12.0 mA/cm^2, rt, 30 h; 8 mmol, 1.080 g : 41%, (β:α = 7:1)

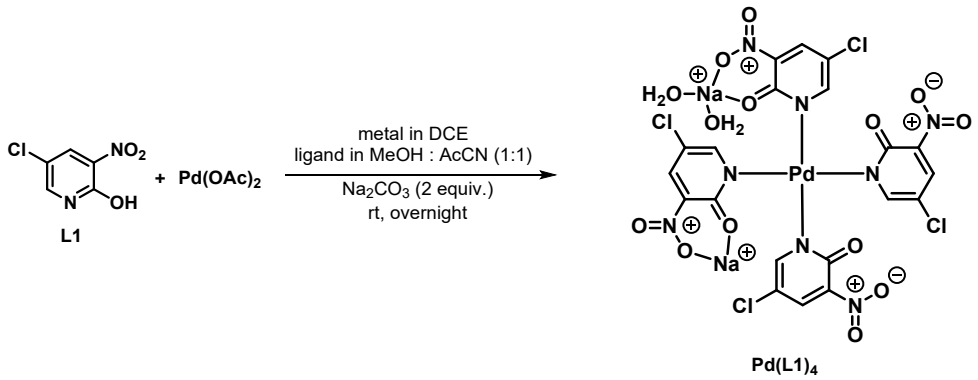
In an oven-dried 25 mL round-bottom flask equipped with a magnetic stir-bar, naphthalene (4.0/8.0 mmol), Pd(OAc)$_2$ (10 mol%), ligand 5-chloro-3-nitropyridin-2-ol (20 mol%), electrolyte *tetra-n*-butyl ammonium hexafluorophosphate (TBAPF$_6$) (0.5 equiv.), and olefinic coupling partner (2.5 equiv.) were dissolved in 15 mL of dichloroethane (DCE) and the mixture was stirred at 380 rpm. The reacton mixture was electrolzyed at a constant current of

j = 6.0 mA/cm^2 or 12.0 mA/cm^2 at a GF anode and a Pt cathode at room temperature for 30 h. Upon completion the GF anode was washed with ethyl acetate (50 mL) and the mixture was filtered through a celite pad. The ethyl acetate layer was dried over anhydrous Na_2SO_4 and the volatiles were removed under vacuum. The crude reaction mixture was purified by column chromatography to obtain the olefinated product (1) 46% with β:α selectivity 7:1 (for 0.504 g) and 41% with selectivity 7:1 (for 1.08 g).

4.3 Unsuccesful substrates (Arenes and olefins).

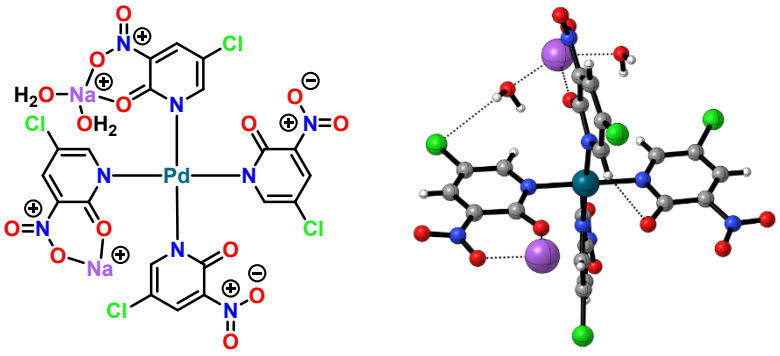


5. Procedure for the Preparation of [Pd(L1)$_4$] Complex:



General procedure: To the solution of $Pd(OAc)_2$ (112.25 mg, 0.5 mmol) in DCE (3 mL), the solution of **L1** (174.54 mg, 1.0 mmol, 2.0 equiv.) in mixture of solvent MeOH and AcCN in the ratio of 1:1 (7.0 mL) was added dropwise with stirring and then solid Na_2CO_3 (106 mg, 2.0 equiv.)was added to this mixture and stirred overnight. Next day, the suspension was allowed to settle and the supernatant was decanted. It was filtered and the clear solution was crystallised by vapour diffusing with diethyl ether.

6. X-Ray Structure of [Pd(L1)$_4$] Complex:



Crystallographic data

Identification code	DMAJ241
Formula	C20 H12 Cl4 N8 Na2 O14 Pd
Formula weight(g/mol)	882.56
Temperature/K	293 K
Crystal system	Orthorhombic
Space group	Pbca
a/Å	9.2426(6)
b/Å	18.4473(14)
c/Å	35.708(4)
$\alpha/^{o}$	90
$\beta/^{o}$	90
$\gamma/^{o}$	90
Volume/$Å^3$	6088.3(9)
Z	8
$P_{calc}g/cm^3$	1.926
μ/mm^{-1}	1.071
F(000)	3488.0
Crystal size/mm^3	0.2 x 0.15 x 0.15
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/^{o}	4.416 to 49.994
Index ranges	$-10 \leq h \leq 10$, $-21 \leq k \leq 21$, $-42 \leq l \leq 35$
Reflections collected	31132
Data / restraints / parameters	5284/108/459
Goodness-of-fit on F^2	1.114
Final R indices [I>2σ(I)]	R_1 = 0.1177, wR_2 = 0.2428
R indices (all data)	R_1 = 0.1709, wR_2 = 0.2701
Largest diff. peak and hole/ e $Å^{-3}$	2.23/-2.78

7. Spectroelectrochemical Study

Electrochemical experiments: Cyclic voltammetry experiments were carried out using a BioLogic SP-300 potentiostat (BioLogic, France) in a three-electrode setup at room temperature (25 °C). Working, counter and reference electrodes were glassy carbon disc (3 mm in diameter), platinum wire, and standard calomel electrode (SCE) electrode, respectively.

Redox potential values were converted to NHE following the equation: $E_{NHE} = (E_{SCE} + 0.241)$ V.

Experimental details for UV-Vis Spectroscopy: UV-vis absorption spectra were recorded using Cary 5000 Spectrophotometer from Agilent Technologies using 1 cm path length quartz cuvette.

Experimental details for spectroelectrochemistry: UV-Vis absorption spectra were recorded using an HR 4000 spectrophotometer developed by Ocean Optics along with a 1 cm cuvette holder and DT-MINI-2-GS UV-Vis-NIR light source. Samples were held at desired potentials using a CH Instruments potentiostat (CHI 760D). Working, counter and reference electrodes were Pt mesh, Pt wire, and Ag/AgCl (3M KCl), respectively. Redox potentials were converted to NHE following the equation: $E_{NHE} = (E_{Ag/AgCl} + 0.210)$ V.

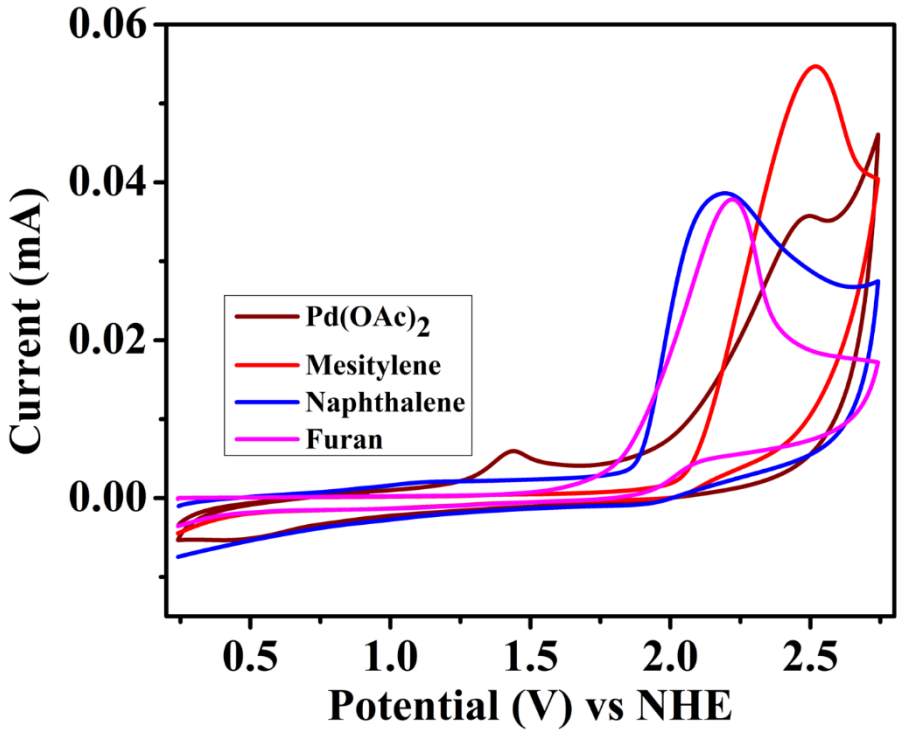


Figure S2: Cyclic voltammogram of various substrates (1 mM) at 100 mVs^{-1} scan rate on glassy carbon electrode in DCE containing 0.1 M NBu_4PF_6.

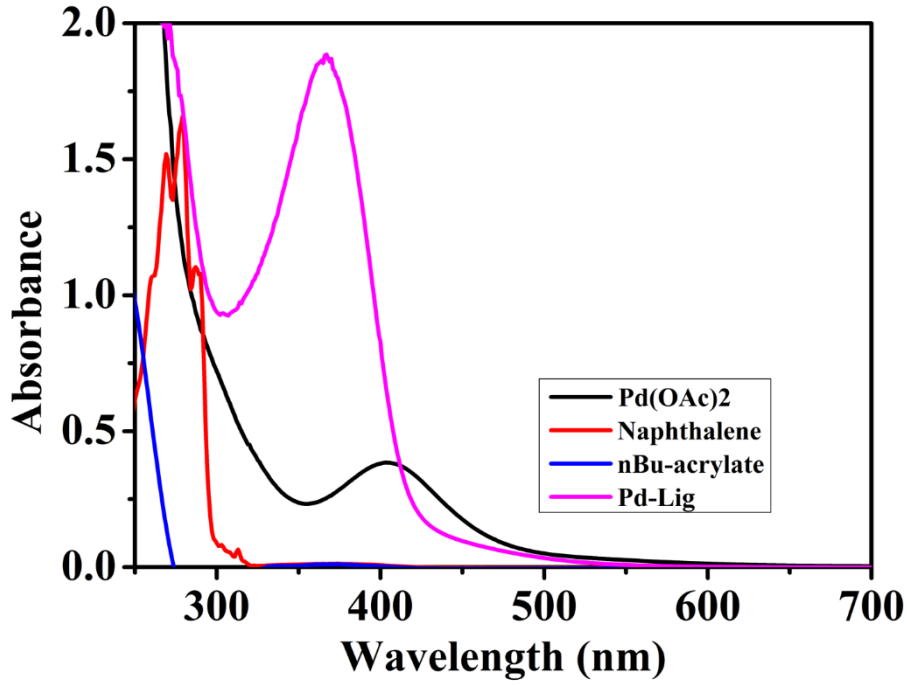


Figure S3: UV-vis spectra of reactants.

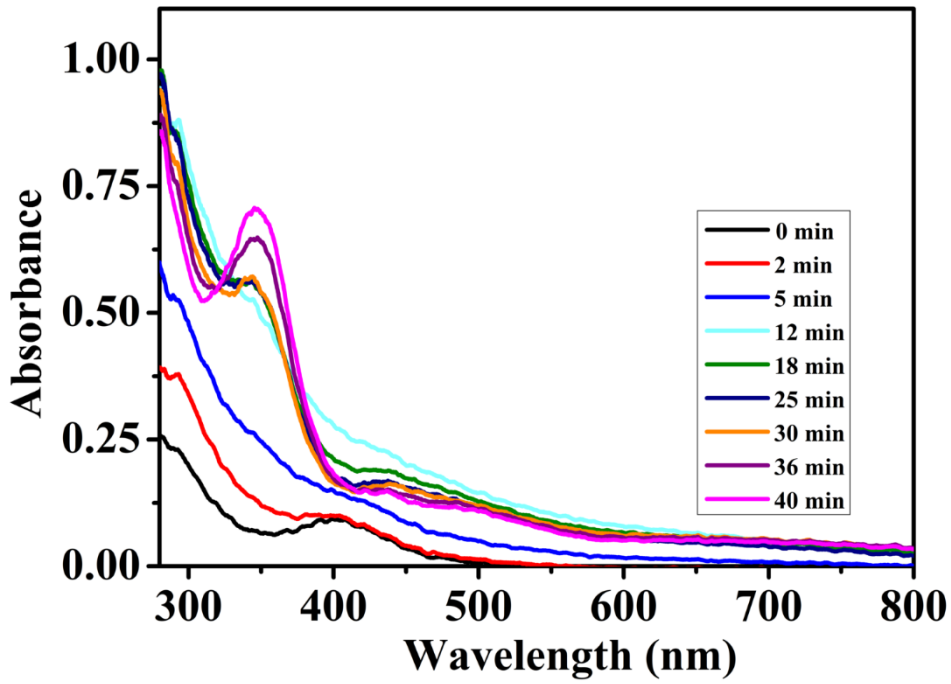


Figure S4: In-situ UV-vis spectroelectrochemical spectra of $Pd(OAc)_2$ undergoing bulk electrolysis at 2.61 V vs. NHE.

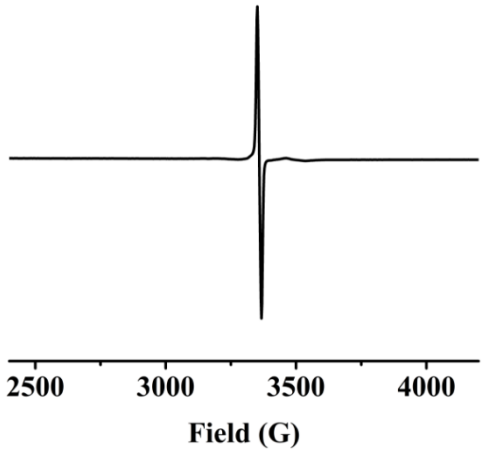


Figure S5: X-band EPR (ν = 9.43 GHz) spectrum of reaction mixture (in absence of *n*-butyl acrylate) after 2 h of experiment.

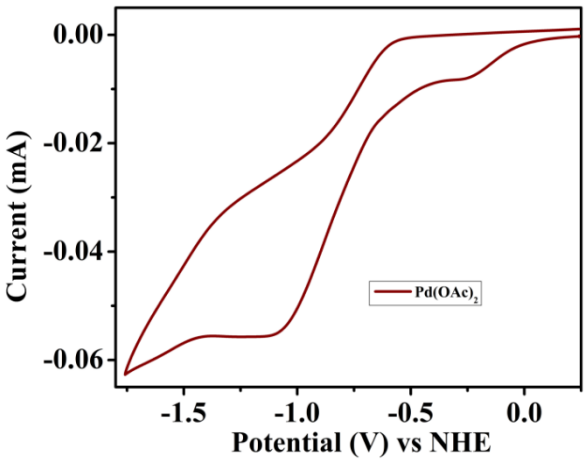


Figure S6: Reduction potential CV of $Pd(OAc)_2$.

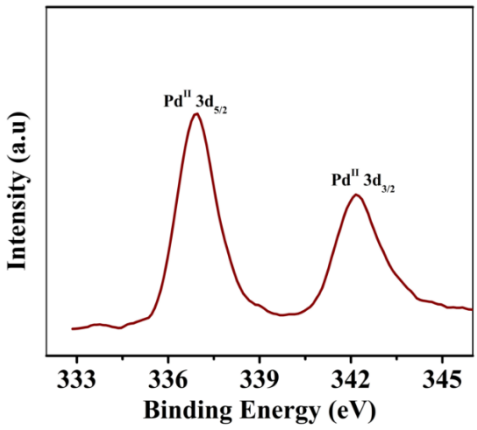


Figure S7. XPS investigation of cathode electrode.

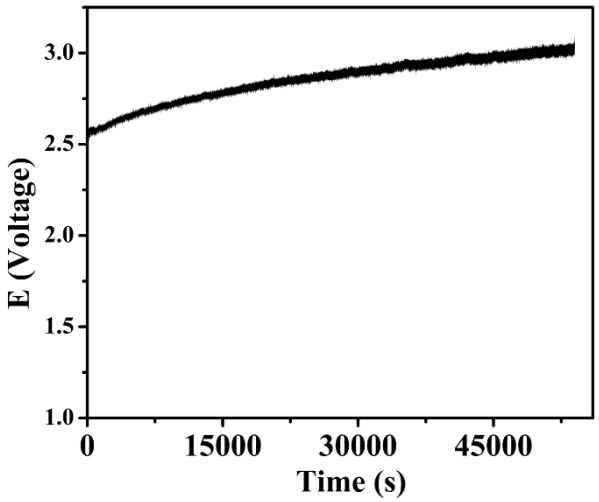


Figure S8. Voltage-time graph of the reaction system

An electrochemical study by Budnikova and co-workers reported approximate ranges of redox potentials for Pd oxidation states. The oxidation of **Pd(IV/II) is usually observed in the range of +1.00 – 2.00 V (vs. $Fc^{+/0}$)** or **1.63 – 2.63 V (vs. NHE)** and the oxidation of **Pd(II/0) is observed in the range of -0.50 - 0.00 V (vs. $Fc^{+/0}$) or 0.13 – 0.63 V (vs. NHE)**.[5a] Furthermore, literature reports from Mirica group have also proved the formation of Pd(III) and Pd(IV) intermediates with the help of exhaustive studies of CV and EPR experiments in the Pd-catalyzed C-C coupling reaction.[5b-c]

When we carried out CV experiments of $Pd(OAc)_2$, the CV profile of $Pd(OAc)_2$ in the negative range revealed two reduction waves, one at -0.23 V and the other at -1.06 V (vs. NHE) which might refer to the redox conversion of Pd(II/I) and Pd(I/0) **(Figure S6)**. The CV profile of $Pd(OAc)_2$ in the positive range resulted in two oxidation waves at +1.42 V and +2.47 V (vs. NHE) which could refer to the redox conversion of Pd(II/III) and Pd(III/IV) **(Figure S2)**. However, when we recorded the anodic voltage of the reaction system, the voltage readings varied between 2.6 – 3.0 V **(Figure S8)** that are close to the oxidation potential in the mechanistic study.[5a] This applied potential should be able to generate a higher oxidation state of Pd according to the electrochemical study. To check for a Pd(0)/Pd(II) pathway, experimentally we have used other Pd(0) catalysts [Pd on carbon, tris(dibenzylideneacetonyl)bis palladium, $Pd(PPh_3)_4$] instead of $Pd(OAc)_2$ and we have observed that all the Pd(0) catalysts remained silent in our protocol. In addition, we have included the following articles as references **23d** and **23e** in the main manuscript.

Additionally, EPR experiments were also performed, which suggested the generation of a Pd(III) intermediate as mentioned in the main manuscript. We performed the reaction and it

was observed by naked eye that slight electrode passivation was happening. However, X-ray photoelectron spectroscopy (XPS) investigations of cathode surface material provided binding energy values of 336.9 and 342.2 eV for the Pd $3d_{5/2}$ and Pd $3d_{3/2}$ levels, respectively, confirming the presence of Pd(II) **(Figure S7)**.[5d-e]

This study confirmed that Pd(IV) was reduced on cathode surface to generate Pd(II).

All these evidences and the comparison with literature reports strongly support the involvement of a Pd(II/IV) cycle. Thus, a Pd(II/0) path seems unlikely since it requires negative potential to carry out the reaction.

we have included the CV of L-Pd$(OAc)_2$ in the main manuscript **(Figure 1a)** and the control experiment with stoichiometric [LPdII] without current suggests that the desired product is not formed under the reaction conditions which corroborates the crucial role of Pd complexes with a higher oxidation state than +II in the C-H activation process.

Electrochemical surface area calculation:

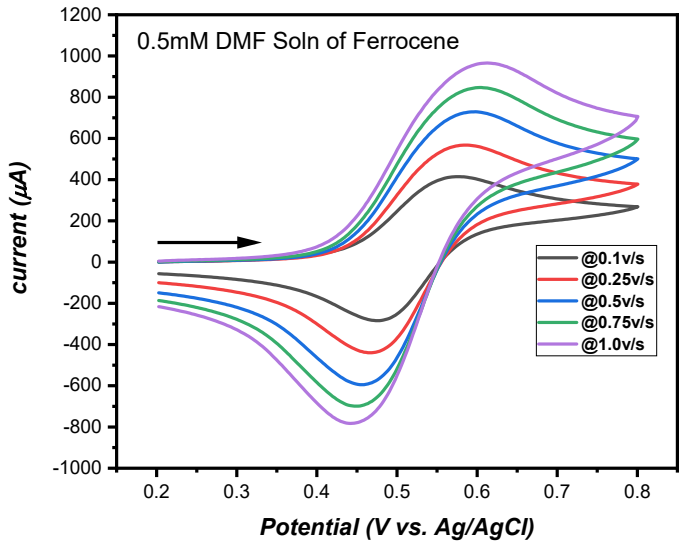


Figure S8a: Cyclic Voltammetry (CV) study with different scan rate (0.1 V/s to 10 V/s) in Ar atmosphere for 0.5 mM ferrocene in DMF. Data were recorded at a temperature of 289 K and Pt was used as a counter electrode. The arrow describes the origin and the direction of the scan.

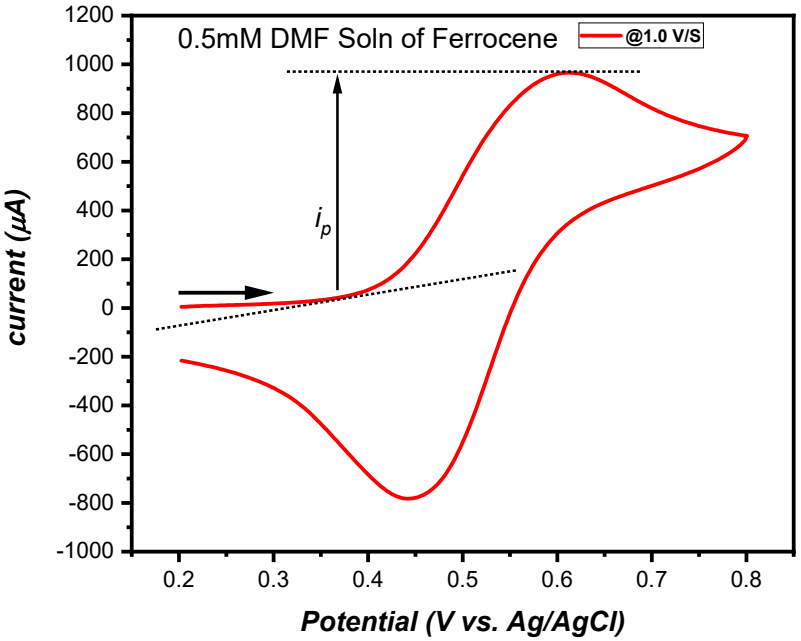


Figure S8b: This figure indicates calculation of i_p current.

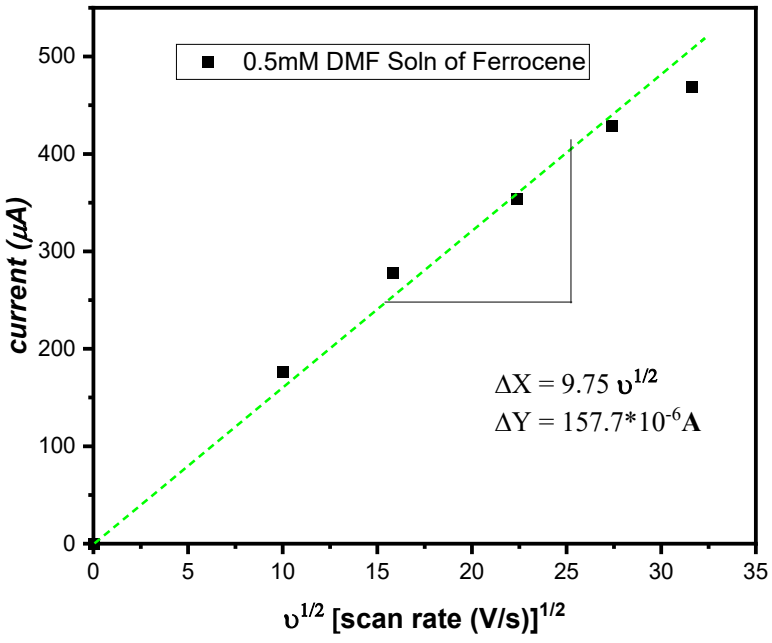


Figure S8c: $\nu^{1/2}$ vs i_p(current) plots of different scan rate.

Randles–Sevcik equation

$i_p = 0.4463*nFAC(nFD\nu/RT)^{1/2}$

$= 0.4463*nFAC(nFD/RT)^{1/2} * \nu^{1/2}$----------equation 1

Now if we compare the equation-1 with y = mx, then the slope(m) will be

$m = 0.4463*nFAC(nFD/RT)^{1/2}$

- i_p = current maximum in amps
- n = number of electrons transferred in the redox event (usually 1)
- A = electrode area in cm^2
- F = Faraday Constant in C mol^{-1}
- D = diffusion coefficient in cm^2/s
- C = concentration in mol/cm^3
- ν = scan rate in V/s
- R = gas constant in J K^{-1} mol^{-1}
- T = temperature in K
- The constant with a value of $2.69x10^5$ has units of C mol^{-1} $V^{-1/2}$

Putting all the values

n = 1, F = 96500, D = $2.4*10^{-5}$, R = 8.314, C = $0.5*10^{-6}$, T = 298 all units are remaining same as mentioned above.

Slope (m) = (ΔY/ΔX) = (0.0001577/9.75) = **$16.2*10^{-4}$**

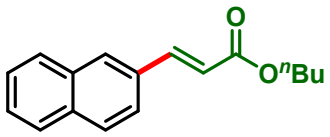
So, **$0.4463*nFAC(nFD/RT)^{1/2} = 16.2*10^{-4}$**

Or, A = 80636/65542.6

= 1.23 cm^2

Therefore, the surface area of the working electrode is 1.23 cm^2

8. Characterization Data of Non-Directed Olefinated Products:



Butyl (*E*)-3-(naphthalen-2-yl)acrylate (1):[6]

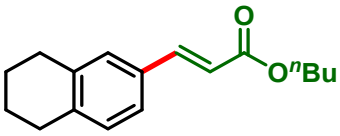
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 68% (*β*:α = >25:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 1.7 Hz, 1H), 7.88 – 7.79 (m, 4H), 7.67 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.55 – 7.48 (m, 2H), 6.56 (d, *J* = 16.0 Hz, 1H), 4.24 (t, *J* = 6.7 Hz, 2H), 1.76 – 1.65 (m, 2H), 1.50 – 1.40 (m, 2H), 0.98 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.27, 144.71, 134.32, 133.41, 132.09, 130.00, 128.77, 128.67, 127.89, 127.31, 126.80, 123.62, 118.55, 64.57, 30.95, 19.37, 13.91.



Butyl (*E*)-3-(5,6,7,8-tetrahydronaphthalen-2-yl)acrylate (2):[7]

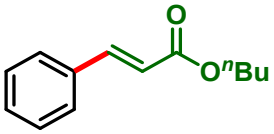
Physical appearance: Colorless gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 52% (*β*:α = 11:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 16.0 Hz, 1H), 7.19 – 7.15 (m, 1H), 7.14 (d, *J* = 1.8 Hz, 1H), 6.98 (d, *J* = 7.9 Hz, 1H), 6.30 (d, *J* = 16.0 Hz, 1H), 4.11 (t, *J* = 6.7 Hz, 2H), 2.68 (dd, *J* = 4.5, 2.5 Hz, 4H), 1.71 (t, *J* = 3.3 Hz, 4H), 1.60 (ddt, *J* = 8.8, 7.9, 6.5 Hz, 2H), 1.42 – 1.30 (m, 2H), 0.88 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.51, 144.97, 140.16, 137.78, 131.87, 129.78, 129.16, 125.14, 117.10, 64.42, 30.93, 29.57, 29.43, 23.13, 23.10, 19.34, 13.88.



Butyl cinnamate (3):[6]

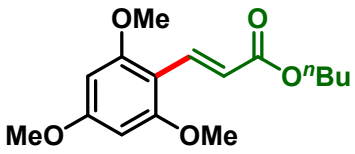
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 63%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (d, *J* = 16.1 Hz, 1H), 7.55 – 7.51 (m, 2H), 7.41 – 7.35 (m, 3H), 6.44 (d, *J* = 16.0 Hz, 1H), 4.21 (t, *J* = 6.7 Hz, 2H), 1.69 (dq, *J* = 8.6, 6.8 Hz, 2H), 1.50 – 1.36 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.31, 144.75, 134.66, 130.40, 129.06, 128.24, 118.48, 64.63, 30.96, 19.39, 13.33.



Butyl (*E*)-3-(2,4,6-trimethoxyphenyl)acrylate (4):[8]

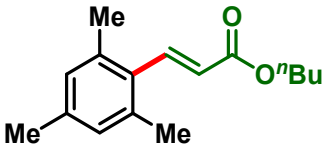
Physical appearance: Colorless gummy liquid. .

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (95/5, v/v).

Yield: 65%

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 16.2 Hz, 1H), 6.72 (d, *J* = 16.2 Hz, 1H), 6.08 (s, 2H), 4.17 (t, *J* = 6.7 Hz, 2H), 3.84 (s, 6H), 3.81 (s, 3H), 1.66 (dq, *J* = 8.4, 6.8 Hz, 2H), 1.50 – 1.32 (m, 2H), 0.94 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.28, 162.84, 161.34, 135.56, 117.61, 105.91, 90.49, 64.00, 55.79, 55.44, 31.06, 19.36, 13.91.



Butyl (*E*)-3-mesitylacrylate (5):[9]

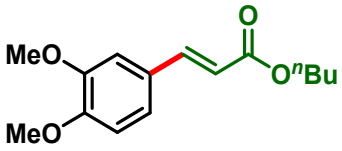
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 59%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (d, *J* = 16.4 Hz, 1H), 6.90 (s, 2H), 6.06 (d, *J* = 16.4 Hz, 1H), 4.22 (t, *J* = 6.8 Hz, 2H), 2.34 (s, 6H), 2.29 (s, 3H), 1.70 (p, *J* = 7.1 Hz, 2H), 1.45 (h, *J* = 7.5 Hz, 2H), 0.98 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.36, 143.37, 138.48, 137.03, 131.17, 129.35, 123.39, 64.64, 30.97, 21.29, 21.24, 19.43, 13.98.



Butyl (*E*)-3-(3,4-dimethoxyphenyl)acrylate (6):[10]

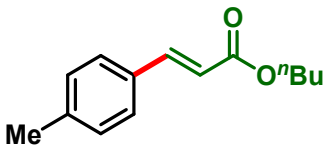
Physical appearance: Colorless gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/4, v/v).

Yield: 59% (*β*:α = 7:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (d, *J* = 15.9 Hz, 1H), 7.08 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.04 (d, *J* = 2.0 Hz, 1H), 6.84 (d, *J* = 8.2 Hz, 1H), 6.30 (d, *J* = 16.0 Hz, 1H), 4.18 (t, *J* = 6.7 Hz, 2H), 3.89 (d, *J* = 1.3 Hz, 6H), 1.73 – 1.60 (m, 2H), 1.42 (ddt, *J* = 14.5, 9.7, 7.3 Hz, 2H), 0.95 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.42, 151.14, 149.27, 144.55, 127.53, 122.67, 116.05, 111.09, 109.63, 64.38, 56.03, 55.94, 30.90, 20.00, 13.84.



Butyl (*E*)-3-(p-tolyl)acrylate (7):[6]

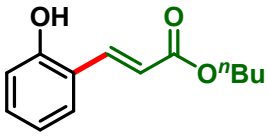
Physical appearance: Colorless gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 41% (*p:others*= 7:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 16.0 Hz, 1H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 6.39 (d, *J* = 16.0 Hz, 1H), 4.20 (t, *J* = 6.7 Hz, 2H), 2.37 (s, 3H), 1.74 – 1.63 (m, 2H), 1.49 – 1.37 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.54, 144.77, 140.83, 132.00, 129.82, 128.27, 117.45, 64.57, 31.03, 29.92, 19.43, 13.97.



Butyl (*E*)-3-(2-hydroxyphenyl)acrylate (8):[10]

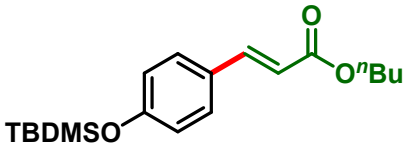
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (95/5, v/v).

Yield: 61% (*o*:*others* = 9:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 16.1 Hz, 1H), 7.48 (dd, *J* = 7.8, 1.7 Hz, 1H), 7.22 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.94 (td, *J* = 7.6, 1.1 Hz, 1H), 6.81 (dd, *J* = 8.2, 1.2 Hz, 1H), 6.56 (d, *J* = 16.1 Hz, 1H), 4.21 (t, *J* = 6.7 Hz, 2H), 1.75 – 1.65 (m, 2H), 1.49 – 1.37 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.88, 154.99, 139.86, 131.48, 129.40, 122.04, 121.28, 119.34, 116.52, 64.62, 31.02, 22.91, 13.98.



Butyl (*E*)-3-(4-((*tert*-butyldimethylsilyl)oxy)phenyl)acrylate (9):

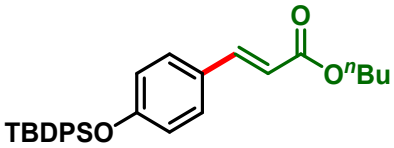
Physical appearance: Colorless gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (96/4, v/v).

Yield: 66% (*p*:*others* = 8:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 15.9 Hz, 1H), 7.41 (d, *J* = 8.6 Hz, 2H), 6.83 (d, *J* = 8.5 Hz, 2H), 6.30 (d, *J* = 15.9 Hz, 1H), 4.19 (t, *J* = 6.7 Hz, 2H), 1.77 – 1.63 (m, 2H), 1.49 – 1.36 (m, 3H), 0.98 (s, 9H), 0.21 (s, 6H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 167.68, 158.01, 144.51, 129.86, 128.00, 120.71, 116.17, 64.50, 31.03, 25.84, 19.43, 13.98, 1.24, -4.17. **IR** (thin film, cm^{-1}) 2958, 2859, 1717, 1602, 1510, 1167, 1024, 913, cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{30}NaO_3Si$ 357.1895; Found 357.1926.



Butyl (*E*)-3-(4-((*tert*-butyldiphenylsilyl)oxy)phenyl)acrylate (10):

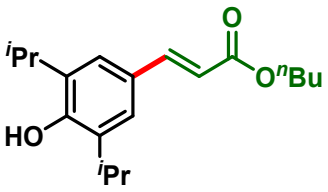
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (96/4, v/v).

Yield: 68% (*p*:*others* = 100:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.70 (dd, *J* = 8.0, 1.5 Hz, 4H), 7.55 (d, *J* = 15.9 Hz, 1H), 7.44 (td, *J* = 6.2, 5.0, 3.2 Hz, 2H), 7.40 – 7.35 (m, 4H), 7.30 – 7.27 (m, 2H), 6.75 (d, *J* = 8.7 Hz, 2H), 6.23 (d, *J* = 15.9 Hz, 1H), 4.17 (t, *J* = 6.6 Hz, 2H), 1.66 (dq, *J* = 8.5, 6.8 Hz, 2H), 1.41 (td, *J* = 9.3, 8.5, 6.5 Hz, 2H), 1.10 (s, 9H), 0.95 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.64, 157.81, 144.47, 135.71, 135.65, 132.61, 130.28, 129.67, 128.11, 128.08, 127.75, 120.38, 116.06, 64.45, 31.00, 26.63, 19.66, 19.40, 13.96. **IR** (thin film, cm^{-1}) 3027, 2929, 2857, 1713, 1600, 1215, 1169, 1114, 919 cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{29}H_{34}NaO_3Si$ 481.2175; Found 481.2167.



Butyl (*E*)-3-(4-hydroxy-3,5-diisopropylphenyl)acrylate (11):

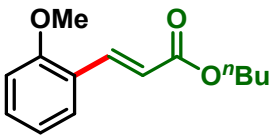
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (95/5 v/v).

Yield: 67% (*p*:*others* > 99:1). With the desired olefinated product little amount of phenol dimerization product (18%) is formed in the reaction medium.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 16.0 Hz, 1H), 7.25 (s, 2H), 6.32 (d, *J* = 15.9 Hz, 1H), 5.08 (s, 1H), 4.20 (t, *J* = 6.7 Hz, 2H), 3.14 (p, *J* = 6.8 Hz, 2H), 1.69 (dd, *J* = 8.7, 6.3 Hz, 2H), 1.50 – 1.39 (m, 2H), 1.29 (s, 6H), 1.27 (s, 6H), 0.97 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.77, 152.38, 145.44, 134.26, 127.16, 124.08, 115.29, 64.37, 31.01, 29.85, 22.76, 19.38, 13.91. **IR** (thin film, cm^{-1}) 3463, 2962, 1689, 1631, 1465, 1384, 1273, 1170 cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{28}NaO_3$ 327.1936; Found 327.1920.



Butyl (*E*)-3-(2-methoxyphenyl)acrylate (12):[10]

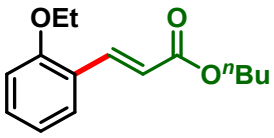
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (96/4, v/v).

Yield: 72% (*o*:*others* = 15:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 16.2 Hz, 1H), 7.51 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.39 – 7.31 (m, 1H), 6.96 (td, *J* = 7.5, 1.1 Hz, 1H), 6.91 (dd, *J* = 8.4, 1.1 Hz, 1H), 6.53 (d, *J* = 16.2 Hz, 1H), 4.20 (t, *J* = 6.7 Hz, 2H), 3.89 (s, 3H), 1.76 – 1.65 (m, 2H), 1.49 – 1.38 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.88, 140.23, 131.60, 129.16, 124.9, 123.70, 120.90, 119.04, 111.34, 64.51, 55.69, 31.04, 19.43, 13.98.



Butyl (*E*)-3-(2-ethoxyphenyl)acrylate (13):

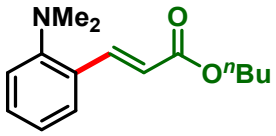
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (96/4, v/v).

Yield: 70% (*o*:*others* = 10:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (d, *J* = 16.2 Hz, 1H), 7.51 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.35 – 7.28 (m, 1H), 6.94 (t, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 8.4 Hz, 1H), 6.53 (d, *J* = 16.1 Hz, 1H), 4.20 (t, *J* = 6.7 Hz, 2H), 4.10 (q, *J* = 7.0 Hz, 2H), 1.74 – 1.64 (m, 2H), 1.48 (t, *J* = 6.9 Hz, 3H), 1.47 – 1.39 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 167.94, 157.94, 140.37, 131.56, 129.08, 123.73, 120.76, 118.81, 112.30, 64.47, 64.23, 31.03, 19.44, 14.99, 13.98. **IR** (thin film, cm^{-1}) 3471, 2928, 1714, 1632, 1457, 1389, 1317, 1246 cm^{-1}; **HRMS** (ESI-TOF) m/z: [M + Na]+ Calcd for $C_{15}H_{20}NaO_3$ 271.1310; Found 271.1340.



Butyl (*E*)-3-(2-(dimethylamino)phenyl)acrylate (14):[11]

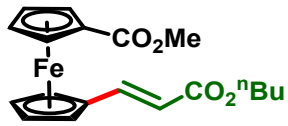
Physical appearance: Greenish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (95/5, v/v).

Yield: 70% (*o*:*others* = 8:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 16.1 Hz, 1H), 7.50 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.32 (ddd, *J* = 8.8, 7.4, 1.6 Hz, 1H), 7.07 – 6.93 (m, 2H), 6.40 (d, *J* = 16.1 Hz, 1H), 4.21 (t, *J* = 6.7 Hz, 2H), 2.76 (s, 6H), 1.77 – 1.65 (m, 2H), 1.50 – 1.38 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.77, 153.72, 142.94, 130.76, 128.31, 128.29, 122.22, 118.44, 117.58, 64.41, 45.07, 30.96, 19.37, 13.92.



1-(Methoxycarbonyl)-7-[(1*E*)-3-butoxy-3-oxo-1-propenyl]-ferrocene (15):

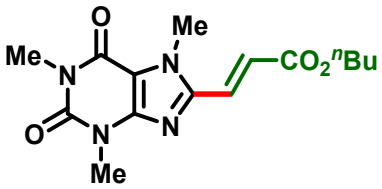
Physical appearance: Reddish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (90/10, v/v).

Yield: 71% (exclusive)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (d, *J* = 15.7 Hz, 1H), 6.03 (d, *J* = 15.8 Hz, 1H), 4.80 (s, 2H), 4.51 (s, 2H), 4.42 (s, 2H), 4.37 (s, 2H), 4.17 (t, *J* = 6.7 Hz, 2H), 3.77 (s, 3H), 1.75 – 1.63 (m, 2H), 1.51 – 1.37 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.91, 167.23, 143.40, 117.08, 80.49, 72.74, 72.31, 71.60, 69.98, 64.46, 51.86, 31.02, 19.42, 13.98. **IR** (thin film, cm^{-1}) 2959, 1715, 1634, 1466, 1280, 1192 cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{22}FeNaO_4$ 393.0765; Found 393.0853.



Butyl (*E*)-3-(1,3,7-trimethyl-2,6-dioxo-2,3,6,7-tetrahydro-1H-purin-8-yl)acrylate (16):[4]

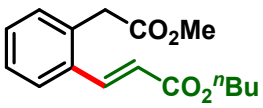
Physical appearance: White solid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (85/15, v/v).

Yield: 48%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49 (d, *J* = 15.3 Hz, 1H), 7.01 (d, *J* = 15.4 Hz, 1H), 4.22 (t, *J* = 6.6 Hz, 2H), 4.07 (s, 3H), 3.56 (s, 3H), 3.39 (s, 3H), 1.75 – 1.63 (m, 2H), 1.46 – 1.37 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.18, 155.48, 151.71, 148.53, 146.96, 126.59, 126.57, 109.41, 65.27, 32.04, 30.82, 29.92, 28.22, 19.31, 13.88.



Butyl (*E*)-3-(2-(2-methoxy-2-oxoethyl)phenyl)acrylate (17):

Physical appearance: Yellowish gummy liquid.

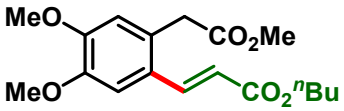
Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (95/5, v/v).

Yield: 51% (*o:others* = 7:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 (d, *J* = 15.7 Hz, 1H), 7.53 (d, *J* = 7.5 Hz, 1H), 7.30 – 7.24 (m, 2H), 7.24 – 7.18 (m, 1H), 6.30 (d, *J* = 15.8 Hz, 1H), 4.14 (t, *J* = 6.7 Hz, 2H), 3.71 (s, 2H), 3.62 (s, 3H), 1.61 (d, *J* = 7.4 Hz, 2H), 1.37 (q, *J* = 7.5 Hz, 2H), 0.90 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.52, 166.98, 141.67, 134.20, 133.65, 131.23, 130.24, 128.02, 127.04, 120.70, 64.63, 52.34, 38.77, 30.88, 19.33, 13.88. **IR** (thin film, cm^{-1}) 2927, 1714, 1636,

1455, 1314, 1274 cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{21}O_4$ 277.1440; Found 277.1472.



Butyl (*E*)-3-(4,5-dimethoxy-2-(2-methoxy-2-oxoethyl)phenyl)acrylate (18):

Physical appearance: Yellowish gummy liquid.

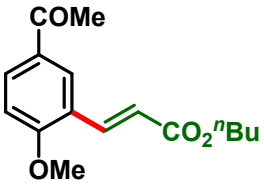
Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (95/5, v/v).

Yield: 48% (*o:others* = 15:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.88 (d, *J* = 15.6 Hz, 1H), 7.09 (s, 1H), 6.75 (s, 1H), 6.28 (d, *J* = 15.6 Hz, 1H), 4.20 (t, *J* = 6.7 Hz, 2H), 3.90 (s, 6H), 3.73 (s, 2H), 3.69 (s, 3H), 1.68 (dq, *J* = 8.5, 6.7 Hz, 2H), 1.49 – 1.37 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.69, 167.26, 150.90, 148.60, 141.26, 127.38, 126.24, 117.99, 113.61, 108.99, 64.51, 56.10, 52.35, 38.17, 30.92, 19.33, 13.88. **IR** (thin film, cm^{-1}) 2959, 1737, 1603, 1518, 1464, 1272 cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{24}NaO_6$ 359.1471; Found 359.1497.



Butyl (*E*)-3-(5-acetyl-2-methoxyphenyl)acrylate (19):

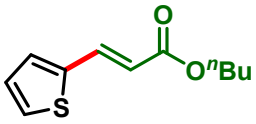
Physical appearance: Reddish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (95/5, v/v).

Yield: 59% (*m:others* = 7:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.13 (d, *J* = 1.7 Hz, 1H), 8.06 – 7.89 (m, 2H), 6.96 (d, *J* = 8.7 Hz, 1H), 6.60 (dd, *J* = 16.2, 1.4 Hz, 1H), 4.22 (td, *J* = 6.7, 1.4 Hz, 2H), 3.96 (s, 3H), 2.58 (s, 3H), 1.74 – 1.60 (m, 2H), 1.44 (d, *J* = 7.5 Hz, 2H), 0.97 (td, *J* = 7.3, 1.3 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 196.51, 167.39, 161.89, 139.11, 132.02, 130.40, 129.63, 123.70, 120.37, 110.92, 64.61, 56.05, 30.95, 26.45, 19.35, 13.88. **IR** (thin film, cm^{-1}) 2959, 1715, 1634, 1466, 1280, 1192 cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{16}H_{21}O_4$ 277.1440; Found 277.1261.



Butyl (*E*)-3-(thien-2-yl)acrylate (20): [6]

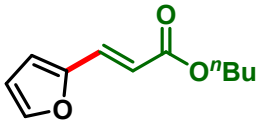
Physical appearance: Colorless gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 64% (*C2:others* = 18:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 15.7 Hz, 1H), 7.37 (d, *J* = 5.0 Hz, 1H), 7.25 (s, 1H), 7.05 (dd, *J* = 5.1, 3.6 Hz, 1H), 6.24 (d, *J* = 15.7 Hz, 1H), 4.19 (t, *J* = 6.7 Hz, 2H), 1.73 – 1.66 (m, 2H), 1.47 – 1.35 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 167.21, 139.84, 137.23, 131.03, 128.55, 128.27, 126.86, 124.61, 124.42, 117.29, 64.66, 30.99, 19.41, 13.96.



Butyl (*E*)-3-(fur-3-yl)acrylate (21):[3]

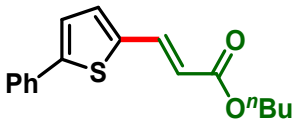
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 68% (*C2:others* = 19:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (d, *J* = 1.8 Hz, 1H), 7.42 (d, *J* = 15.8 Hz, 1H), 6.60 (d, *J* = 3.4 Hz, 1H), 6.51 – 6.43 (m, 1H), 6.31 (d, *J* = 15.7 Hz, 1H), 4.19 (t, *J* = 6.7 Hz, 2H), 1.67 (dq, *J* = 8.4, 6.7 Hz, 2H), 1.49 – 1.36 (m, 2H), 0.95 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 167.34, 151.11, 144.80, 131.08, 116.12, 114.75, 112.38, 64.53, 30.90, 19.33, 13.89.



Butyl (*E*)-3-(5-phenylthien-2-yl)acrylate (22):[12]

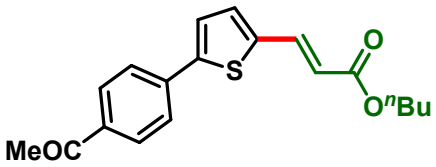
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 76% (*C5:others* = >25:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 15.7 Hz, 1H), 7.63 – 7.56 (m, 2H), 7.41 – 7.34 (m, 2H), 7.34 – 7.27 (m, 1H), 7.24 (d, *J* = 3.7 Hz, 1H), 7.20 (d, *J* = 3.8 Hz, 1H), 6.22 (d, *J* = 15.6 Hz, 1H), 4.19 (t, *J* = 6.7 Hz, 2H), 1.67 (dq, *J* = 8.6, 6.8 Hz, 2H), 1.42 (h, *J* = 7.4 Hz, 2H), 0.95 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.23, 147.49, 138.97, 137.26, 133.80, 132.42, 129.24, 128.59, 126.17, 124.14, 116.83, 64.64, 31.00, 19.41, 13.96.



Butyl (*E*)-3-(5-(*p*-acetylphenyl)thien-2-yl)acrylate (23):

Physical appearance: Yellow solid.

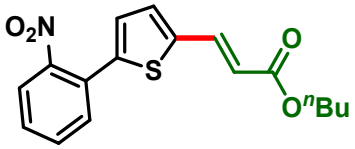
m.p.: 102-107 ^oC

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (96/4, v/v).

Yield: 73% (*C5:others* = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.5 Hz, 2H), 7.80 – 7.71 (m, 1H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.36 (d, *J* = 3.8 Hz, 1H), 7.24 (d, *J* = 3.9 Hz, 1H), 6.26 (d, *J* = 15.6 Hz, 1H), 4.20 (t, *J* = 6.7 Hz, 2H), 2.61 (s, 3H), 1.75 – 1.61 (m, 2H), 1.53 – 1.35 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 197.42, 167.02, 145.59, 140.46, 138.05, 136.84, 136.56, 132.36, 129.38, 125.99, 125.61, 117.77, 64.75, 30.96, 26.81, 19.39, 13.95. IR (thin film, cm^{-1}) 1702, 1622, 1454, 1163, 968.810, 804, 711cm^{-1}; HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{20}NaO_3S$ 351.1031; Found 351.1019.



Butyl (*E*)-3-(5-(2-nitrophenyl)thien-2-yl)acrylate (24):

Physical appearance: Yellowish gummy liquid.

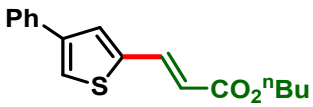
Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (94/6, v/v).

Yield: 64% (*C5 exlusive*)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.2 Hz, 1H), 7.73 (d, *J* = 15.7 Hz, 1H), 7.61 (dd, *J* = 8.2, 6.9 Hz, 1H), 7.57 – 7.53 (m, 1H), 7.51 (td, *J* = 7.7, 1.5 Hz, 1H), 7.20 (d, *J* = 3.8 Hz, 1H),

7.01 (d, *J* = 3.7 Hz, 1H), 6.24 (d, *J* = 15.7 Hz, 1H), 4.20 (t, *J* = 6.7 Hz, 2H), 1.73 – 1.65 (m, 2H), 1.43 (h, *J* = 7.4 Hz, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); **^{13}C** NMR (101 MHz, CDCl3) δ 166.93, 149.42, 141.33, 140.04, 136.66, 132.38, 132.28, 131.51, 129.47, 128.29, 127.97, 124.37, 118.08, 64.75, 30.96, 19.39, 13.94. **IR** (thin film, cm^{-1}) 2961, 1705, 1625, 1529, 1352, 1269, 1215 cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{17}NNaO_4S$ 354.0776; Found 354.0810.



Butyl (*E*)-3-(4-phenylthien-2-yl)acrylate (25):

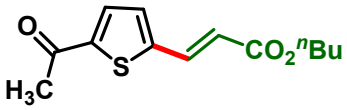
Physical appearance: Greenish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 72% (*C5:others* = 6:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, *J* = 15.7 Hz, 1H), 7.61 – 7.54 (m, 2H), 7.52 (d, *J* = 1.5 Hz, 1H), 7.47 (d, *J* = 1.4 Hz, 1H), 7.41 (ddt, *J* = 8.3, 6.1, 1.5 Hz, 2H), 7.35 – 7.29 (m, 1H), 6.28 (d, *J* = 15.7 Hz, 1H), 4.21 (t, *J* = 6.7 Hz, 2H), 1.76 – 1.64 (m, 2H), 1.51 – 1.37 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.12, 143.44, 140.38, 137.18, 135.20, 129.90, 129.49, 129.15, 128.90, 127.85, 126.49, 123.23, 117.59, 64.72, 30.99, 19.41, 13.97. **IR** (thin film, cm^{-1}) 2960, 1710, 1627, 1455, 1271, 1166 cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{18}NaO_2S$ 309.0925; Found 309.0897.



Butyl (*E*)-3-(5-acetylthien-2-yl)acrylate (26):[13]

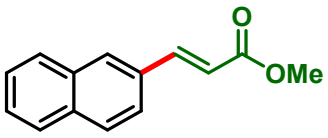
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 60% (*C5:others* = 8:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 15.8 Hz, 1H), 7.59 (dd, *J* = 3.9, 1.0 Hz, 1H), 7.23 (d, *J* = 3.9 Hz, 1H), 6.37 (dd, *J* = 15.8, 1.1 Hz, 1H), 4.24 – 4.13 (m, 2H), 2.55 (s, 3H), 1.71 – 1.62 (m, 2H), 1.47 – 1.35 (m, 2H), 0.95 (td, *J* = 7.4, 1.1 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 190.58, 166.38, 146.66, 145.53, 136.14, 132.84, 130.80, 120.66, 64.90, 30.82, 26.88, 19.28, 13.83.



Methyl (*E*)-3-(naphthalen-2-yl)acrylate (27):[14]

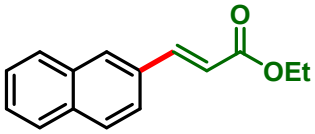
Physical appearance: White solid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 60% (*β*:α = 20:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 – 7.86 (m, 1H), 7.86 – 7.78 (m, 4H), 7.64 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.54 – 7.45 (m, 2H), 6.55 (d, *J* = 16.1 Hz, 1H), 3.84 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.64, 145.07, 134.37, 133.41, 132.00, 130.11, 128.83, 128.71, 127.92, 127.38, 126.85, 123.60, 118.05, 51.87.



Ethyl (*E*)-3-(naphthalen-2-yl)acrylate (28):[15]

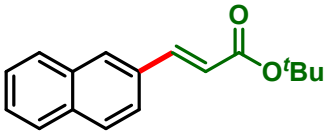
Physical appearance: Yellowish solid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 57% (*β*:α = >25:1)

^{1}H NMR (400 MHz, CDCl*3*) δ 7.93 (s, 1H), 7.90 – 7.77 (m, 4H), 7.67 (d, *J* = 8.5 Hz, 1H), 7.55 – 7.41 (m, 2H), 6.55 (d, *J* = 16.0 Hz, 1H), 4.28 (dd, *J* = 8.7, 5.5 Hz, 2H), 1.36 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.31, 144.87, 134.43, 133.51, 132.19, 130.10, 128.90, 128.77, 127.99, 127.42, 126.92, 123.73, 118.66, 60.76, 14.57.



***tert*-Butyl (*E*)-3-(naphthalen-2-yl)acrylate (29):**[16]

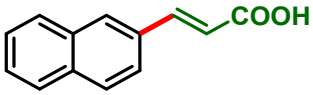
Physical appearance: Yellowish solid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 60% (*β*:α = 22:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.89 (m, 1H), 7.87 – 7.79 (m, 3H), 7.75 (d, *J* = 16.0 Hz, 1H), 7.66 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.53 – 7.46 (m, 2H), 6.49 (d, *J* = 15.9 Hz, 1H), 1.56 (s, 9H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 166.63, 143.81, 134.32, 133.54, 132.40, 129.81, 128.81, 128.72, 127.97, 127.27, 126.85, 123.82, 120.61, 80.77, 28.45.



(*E*)-3-(Naphthalen-2-yl)acrylic acid (30):[17]

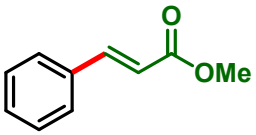
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (90/10, v/v).

Yield: 54% (*β*:*α* = 3:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (s, 1H), 7.96 – 7.77 (m, 4H), 7.70 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.64 – 7.47 (m, 2H), 6.57 (dd, *J* = 15.8, 3.5 Hz, 1H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.95, 147.17, 134.65, 133.48, 131.83, 131.18, 130.65, 129.03, 129.01, 128.89, 128.03, 127.69, 127.26, 127.03, 126.53, 125.69, 125.56, 123.76, 123.50, 117.44.



Methyl cinnamate (31):[18]

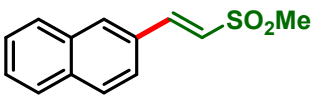
Physical appearance: Colorless gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 62%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 (d, *J* = 16.0 Hz, 1H), 7.50 (dd, *J* = 6.6, 3.1 Hz, 2H), 7.40 – 7.25 (m, 3H), 6.44 (d, *J* = 16.0 Hz, 1H), 3.79 (s, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 167.48, 144.94, 134.44, 130.38, 128.96, 128.16, 117.86, 51.74.



(*E*)-2-(2-(Methylsulfonyl)vinyl)naphthalene (32):[19]

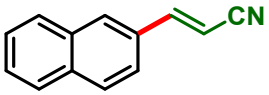
Physical appearance: Yellowish solid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (92/8, v/v).

Yield: 49% (*β*:α = 18:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 1.8 Hz, 1H), 7.86 (dt, *J* = 11.3, 4.7 Hz, 3H), 7.78 (d, *J* = 15.4 Hz, 1H), 7.60 (dd, *J* = 8.6, 1.8 Hz, 1H), 7.58 – 7.50 (m, 2H), 7.03 (d, *J* = 15.4 Hz, 1H), 3.07 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.19, 134.73, 133.24, 131.21, 129.63, 129.22, 128.87, 128.04, 127.98, 127.19, 126.29, 123.43, 43.50.



(*E*)-3-(Naphthalen-2-yl)acrylonitrile (33):[20]

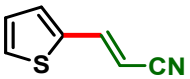
Physical appearance: Colorless gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (97/3, v/v).

Yield: 57% (*β*:α = 8:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (q, *J* = 10.1, 8.7 Hz, 5H), 7.62 – 7.44 (m, 4H), 5.95 (d, *J* = 16.6 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 150.58, 134.56, 133.11, 131.01, 129.73, 129.09, 128.78, 127.92, 127.88, 127.16, 122.25, 118.41, 96.32.



Butyl (*E*)-3-(naphthalen-2-yl)acrylate (34):[20]

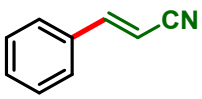
Physical appearance: Colorless gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/3, v/v).

Yield: 61% (*C2:others*= 7:1)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 16.3 Hz, 1H), 7.42 (dd, *J* = 5.1, 1.1 Hz, 1H), 7.27 – 7.22 (m, 1H), 7.08 (dd, *J* = 5.1, 3.6 Hz, 1H), 5.65 (d, *J* = 16.3 Hz, 1H).



Cinnamonitrile (35):[19]

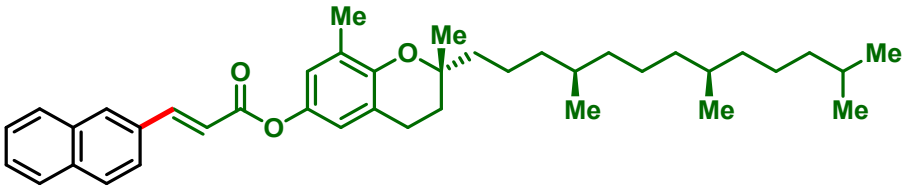
Physical appearance: Reddish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (90/10, v/v).

Yield: 54%

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 6.7, 2.9 Hz, 2H), 7.52 – 7.40 (m, 3H), 7.13 (d, *J* = 12.1 Hz, 1H), 5.46 (d, J = 12.1 Hz, 1H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ 148.95, 133.79, 131.21, 129.24, 129.16, 117.57, 95.29.



(*R*)-2,8-Dimethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl (*E*)-3-(naphthalen-2-yl)acrylate (36):

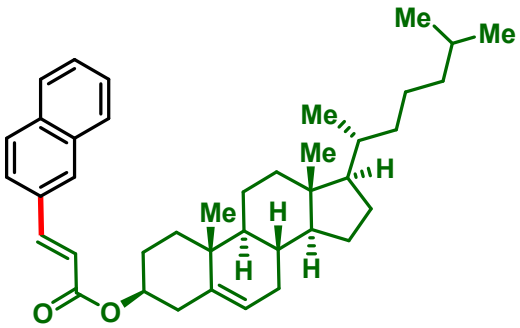
Physical appearance: Yellowish gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 57% (*β*:α = 5:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 – 7.94 (m, 2H), 7.87 (q, *J* = 8.7, 7.6 Hz, 3H), 7.75 – 7.66 (m, 1H), 7.55 – 7.49 (m, 2H), 6.83 – 6.70 (m, 3H), 2.76 (q, *J* = 6.3 Hz, 2H), 2.34 – 2.07 (m, 5H), 1.86 – 1.72 (m, 4H), 1.55 – 1.49 (m, 4H), 1.33 (s, 6H), 1.26 (s, 6H), 0.92 – 0.85 (m, 15H); **^{13}C NMR** (126 MHz, CDCl3) δ 166.43, 150.03, 147.29, 146.30, 142.76, 138.74, 134.57, 133.50, 132.05, 130.50, 129.00, 128.86, 128.03, 127.60, 127.56, 126.99, 124.68, 124.20, 123.74, 121.40, 121.21, 119.33, 119.30, 118.02, 76.37, 40.39, 39.59, 37.67, 37.65, 37.50, 33.02, 32.92, 31.65, 31.21, 30.40, 29.92, 28.20, 25.02, 24.68, 24.48, 22.94, 22.85, 22.71, 21.20, 19.97, 19.88, 16.40. **IR** (thin film, cm^{-1}) 2927, 1728, 1466, 1214, 1150, 751cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{40}H_{54}NaO_3$ 605.3971; Found 605.4012.



(3*S*,8*S*,9*S*,10*R*,13*R*,14*S*,17*R*)-10,13-dimethyl-17-((*R*)-6-methylheptan-2-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl (*E*)-3-(naphthalen-2-yl)acrylate (37):

Physical appearance: Colourless gummy liquid.

Column material: 100-200 mesh silica.

Eluent: petroleum ether/ethyl acetate (98/2, v/v).

Yield: 37% (β:α = 3:1)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (s, 1H), 7.88 – 7.81 (m, 3H), 7.67 (d, *J* = 8.5 Hz, 1H), 7.55 – 7.47 (m, 3H), 6.54 (d, *J* = 16.0 Hz, 1H), 5.42 (d, *J* = 4.9 Hz, 1H), 4.84 – 4.71 (m, 1H), 2.43 (d, *J* = 8.1 Hz, 2H), 2.09 – 1.94 (m, 6H), 1.54 – 1.43 (m, 4H), 1.34 (t, *J* = 15.4 Hz, 8H), 1.15 – 0.97 (m, 8H), 0.95 – 0.79 (m, 15H). **^{13}C NMR** (126 MHz, CDCl3) δ 166.63, 165.51, 147.21, 144.62, 140.30, 139.79, 136.13, 134.33, 133.47, 132.21, 131.49, 130.43, 129.21, 128.81, 128.70, 128.60, 127.93, 127.32, 126.84, 126.06, 124.62, 124.13, 123.70, 122.89, 122.13, 121.94, 119.06, 114.01, 88.02, 79.91, 74.30, 56.87, 56.31, 50.23, 42.49, 39.91, 39.68, 38.42, 38.26, 37.25, 37.21, 37.15, 36.81, 36.35, 35.95, 35.04, 34.73, 32.87, 32.08, 32.05, 31.59, 30.35, 29.85, 29.51, 28.39, 28.17, 28.08, 27.93, 27.39, 24.45, 23.99, 22.97, 22.84, 22.71, 21.21, 19.52, 19.48, 18.52, 14.26, 12.02. **IR** (thin film, cm^{-1}) 2943, 1735, 1470, 1246, 1166 cm^{-1}; **HRMS** (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{40}H_{54}NaO_2$ 589.4022; Found 589.4006.

9. References

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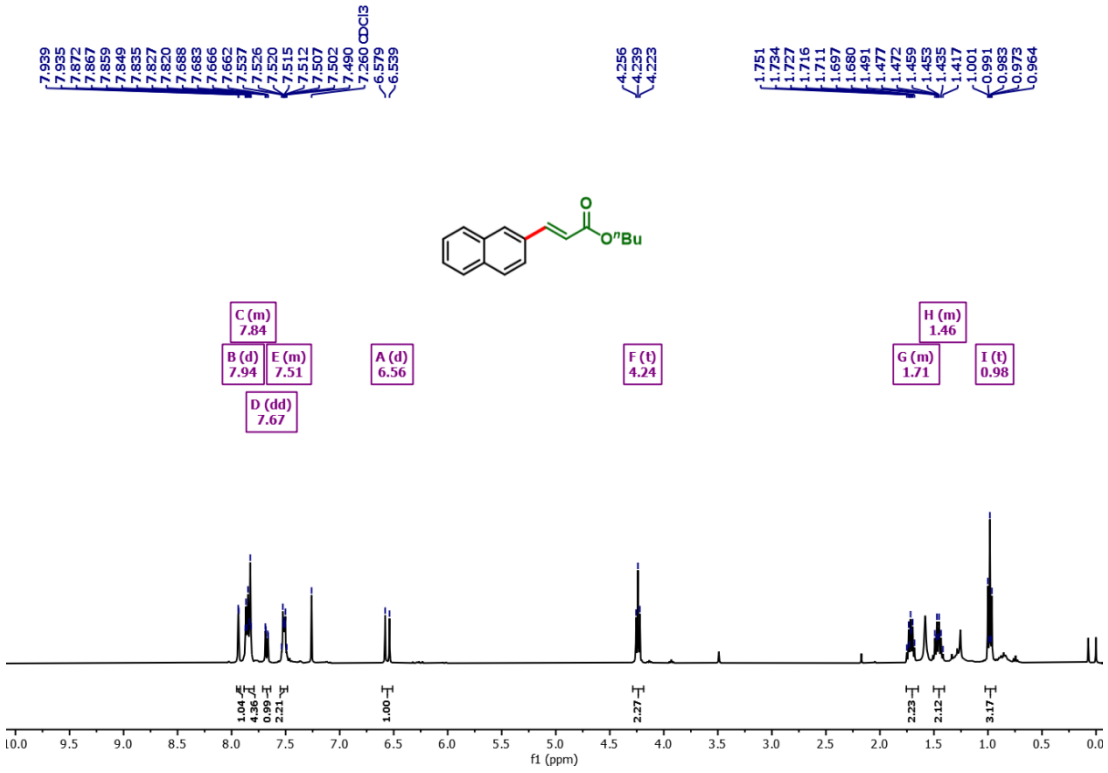
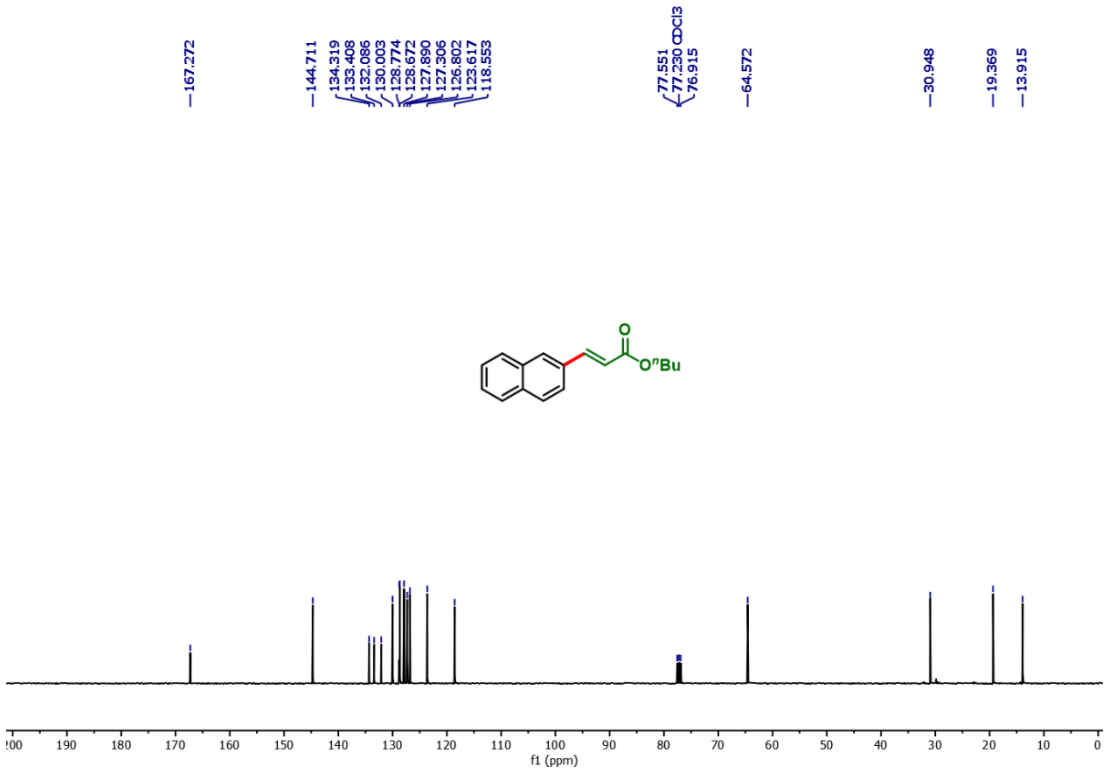
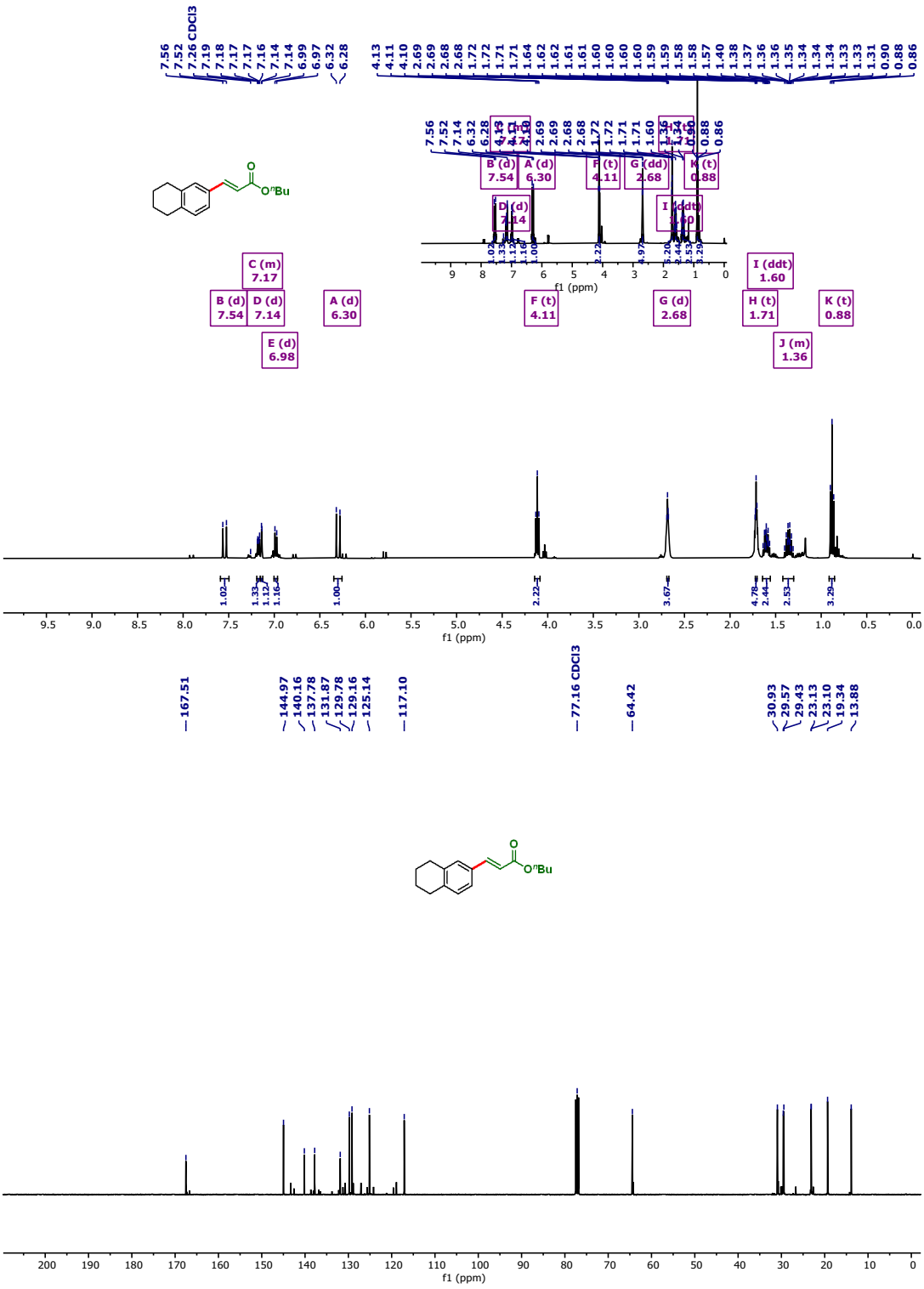
NMR Spectra of the Synthesized Compounds



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O
O nBu
C (m) 7.17
B (d) 7.54
D (d) 7.14
A (d) 6.30
E (d) 6.98
F (t) 4.11
G (dd) 2.68
I (ddt) 1.60
H (t) 1.71
K (t) 0.88
J (m) 1.36
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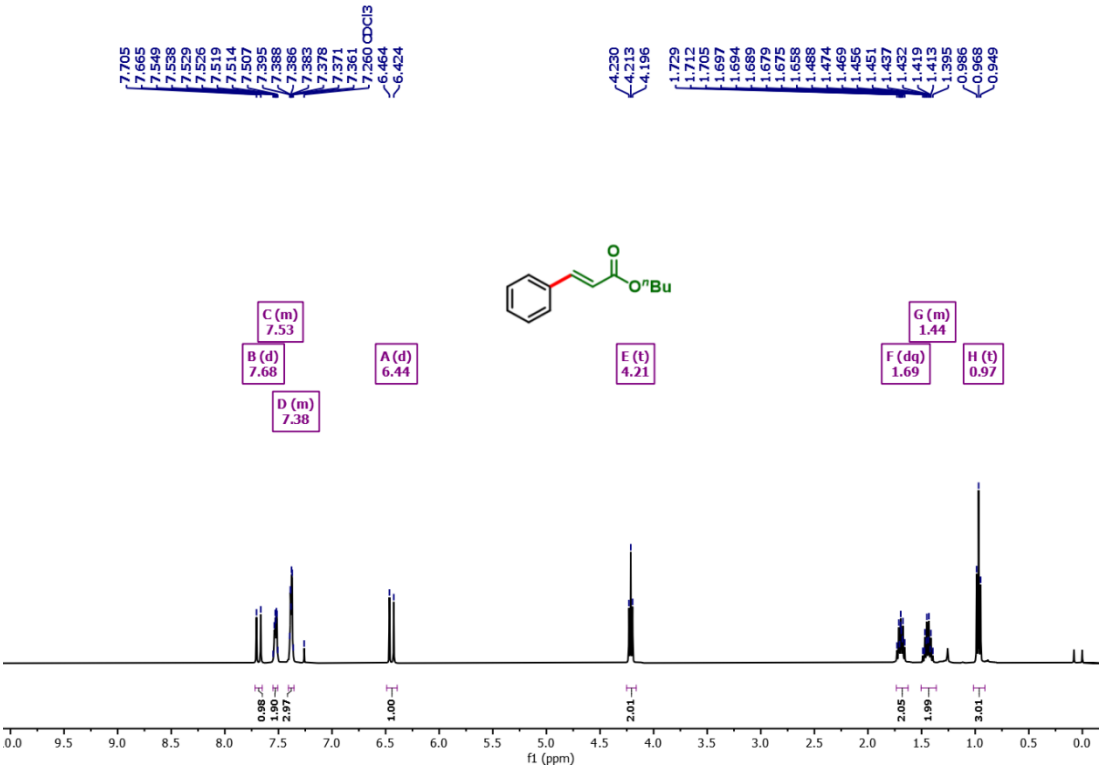
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6.424
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4.196
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1.705
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1.694
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1.395
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B (d) 7.68
D (m) 7.38
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F (dq) 1.69
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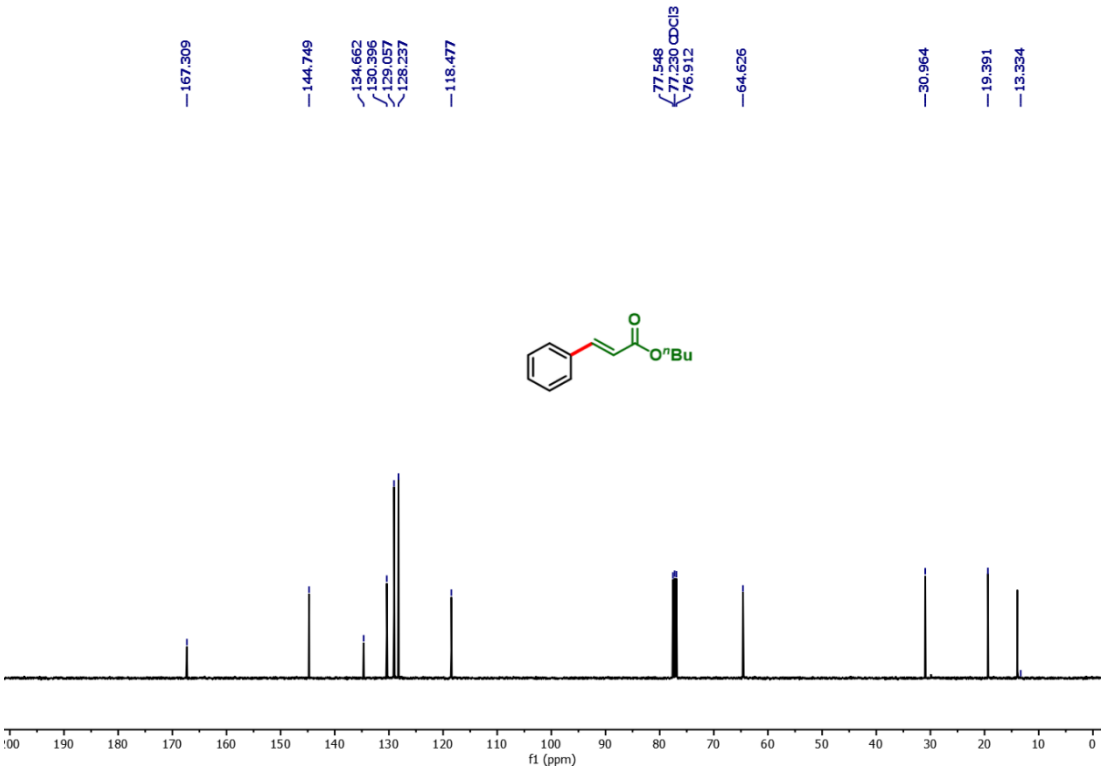
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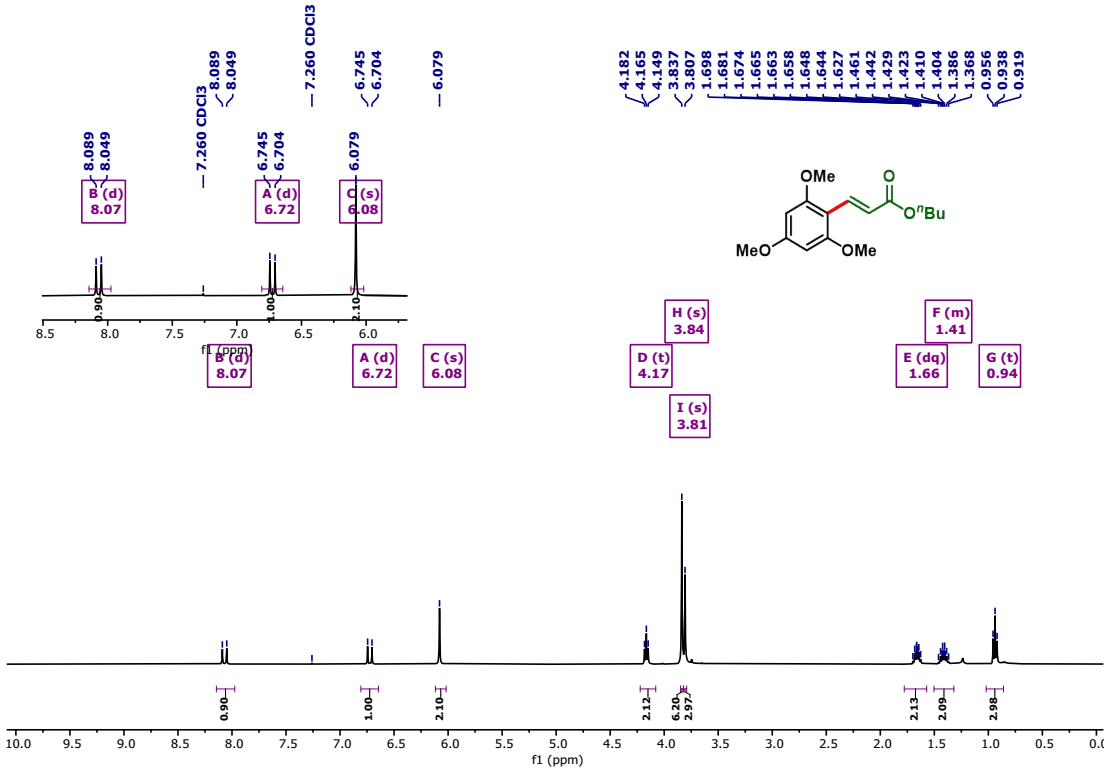
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OMe
H (s)
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D (t)
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I (s)
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F (m)
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1.66
G (t)
0.94
f1 (ppm)

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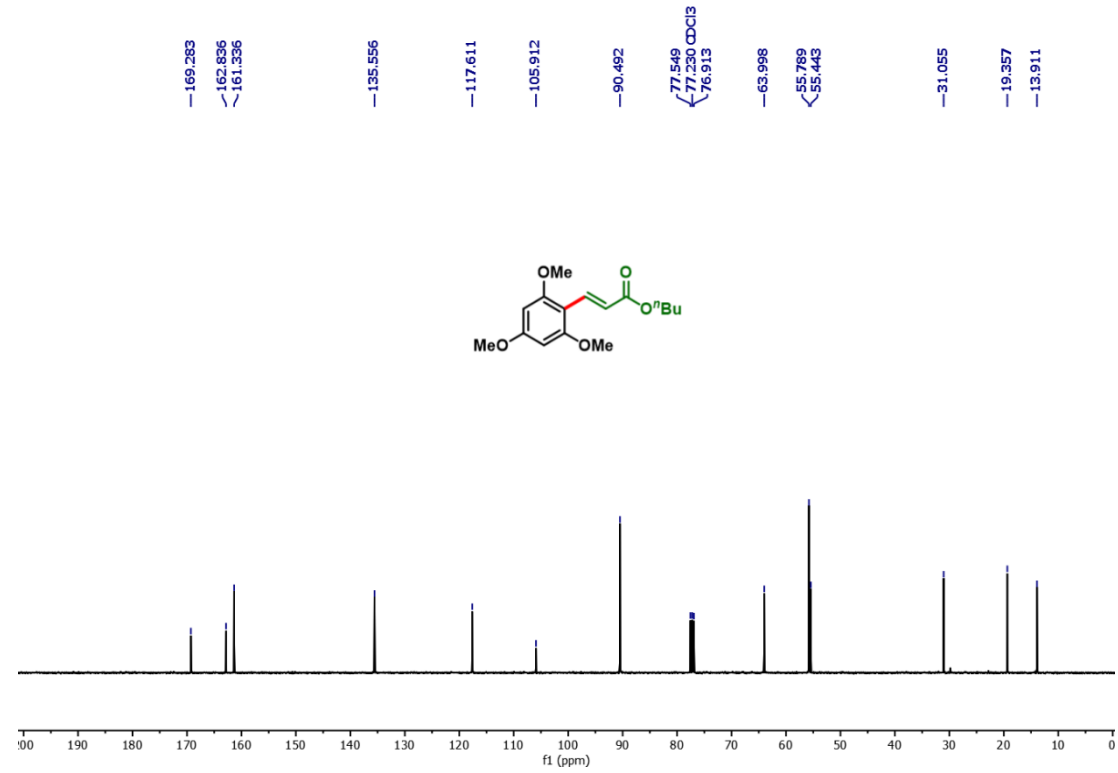
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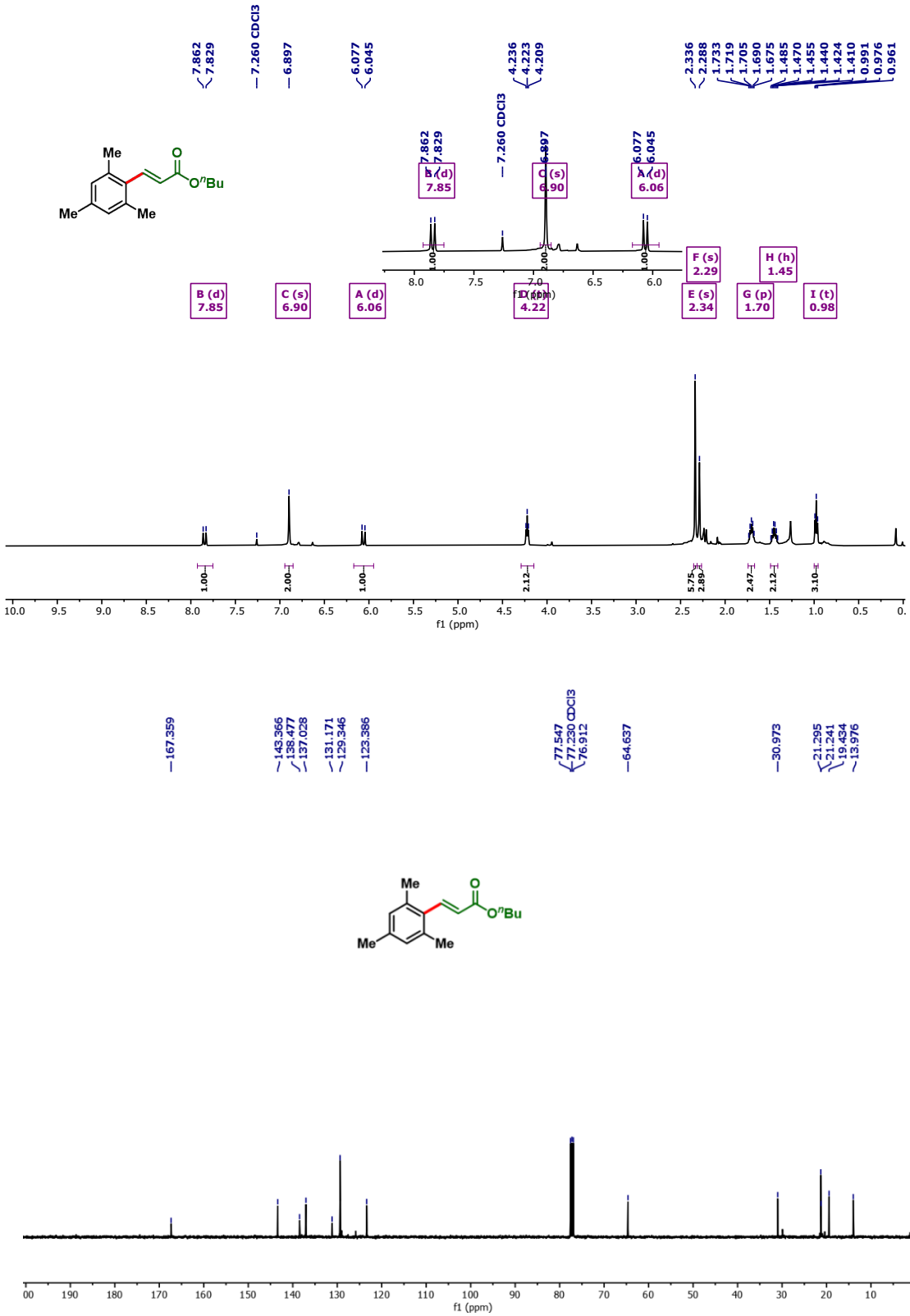
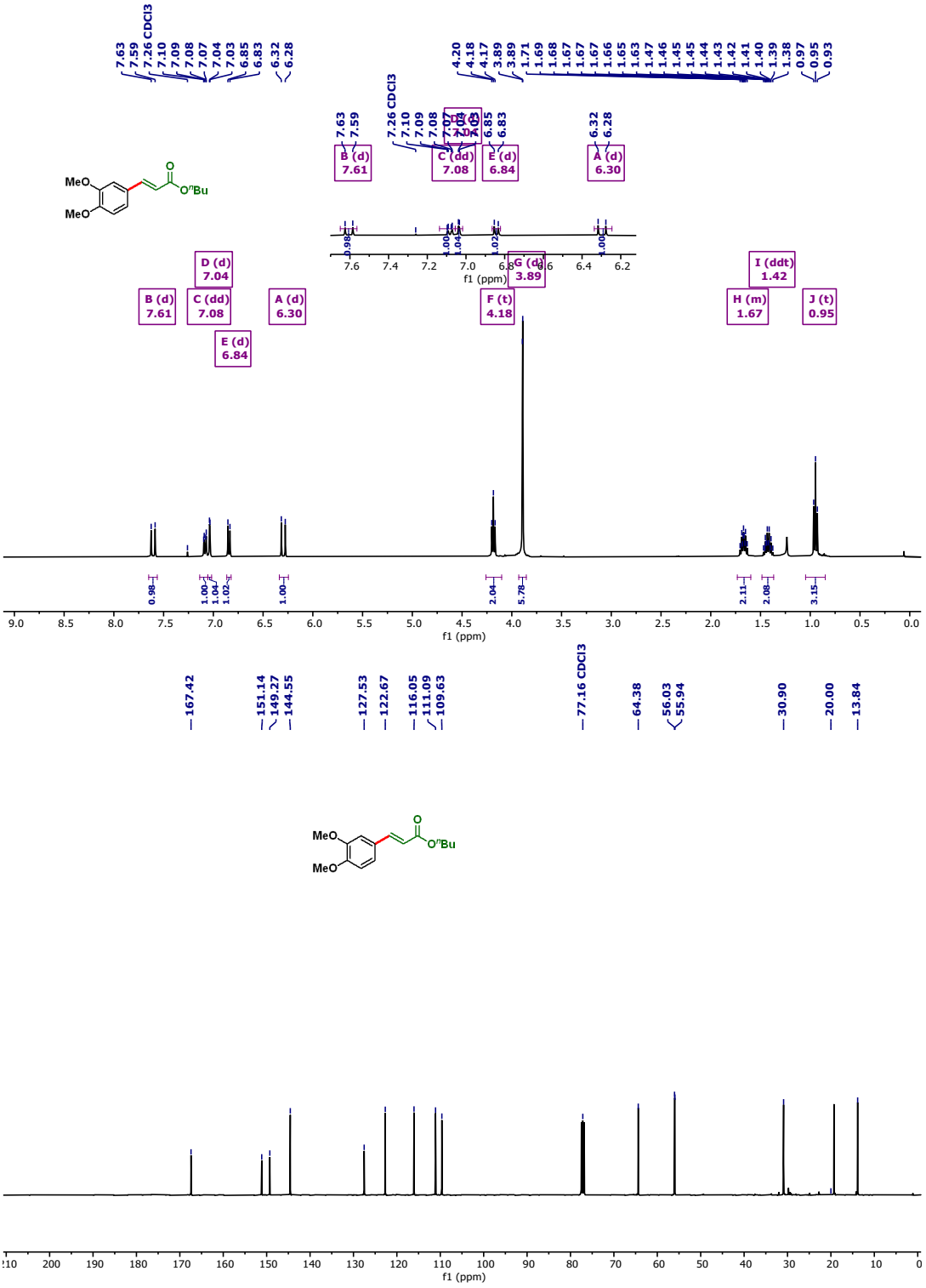
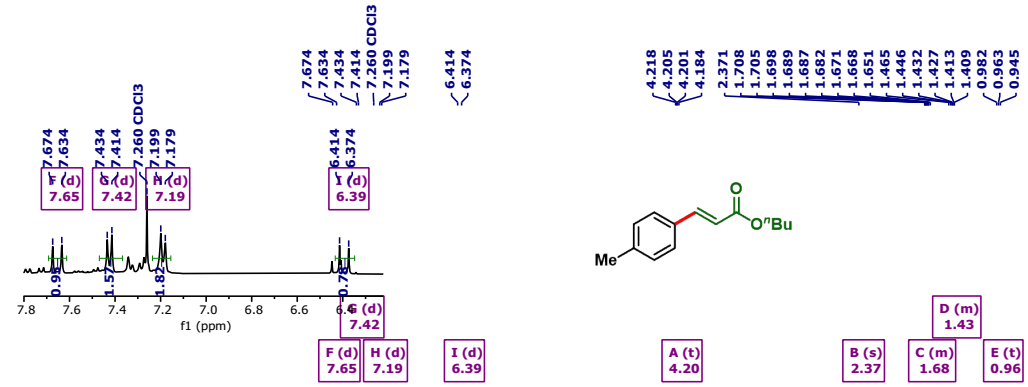
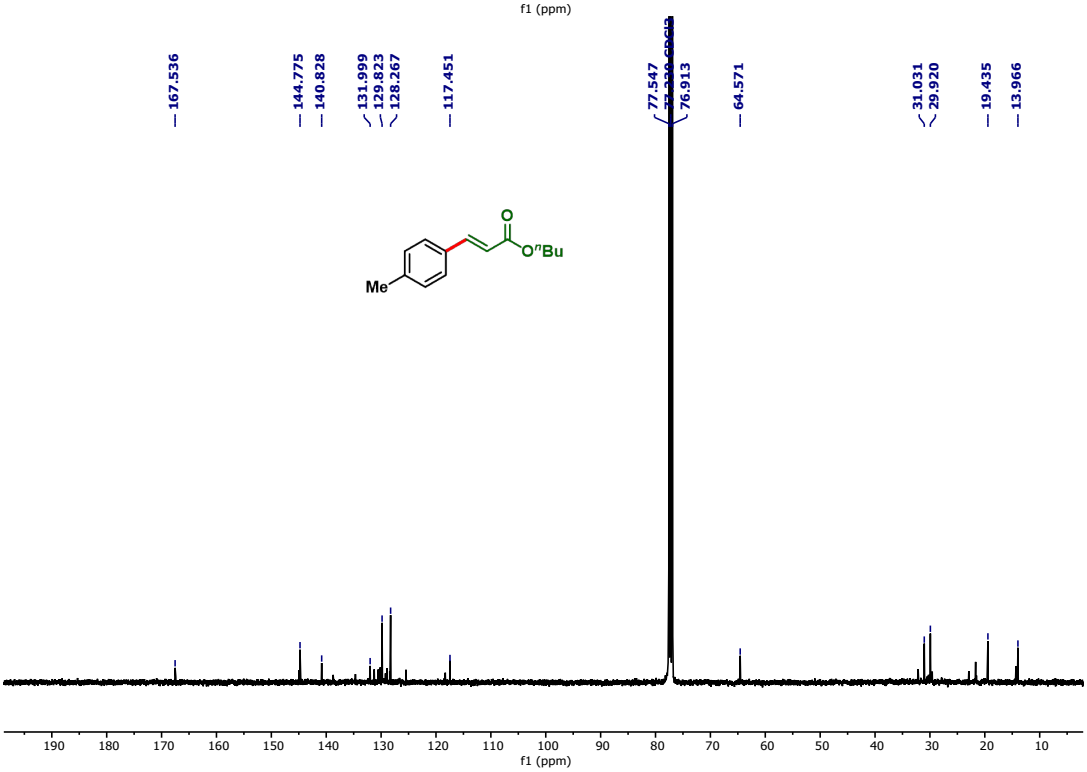
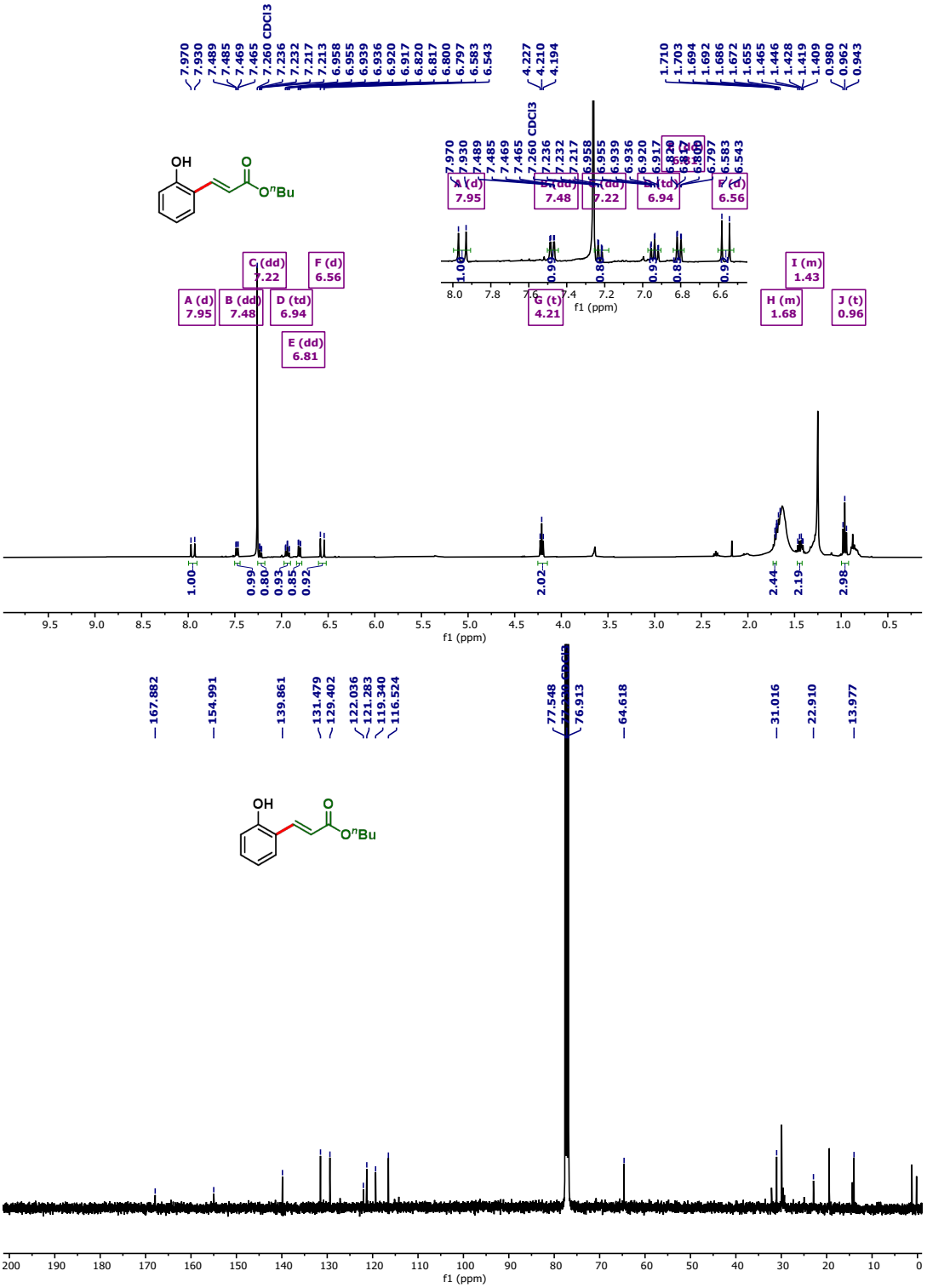
31.055
19.357
13.911
OMe
O
O^{n}Bu
MeO
OMe
f1 (ppm)

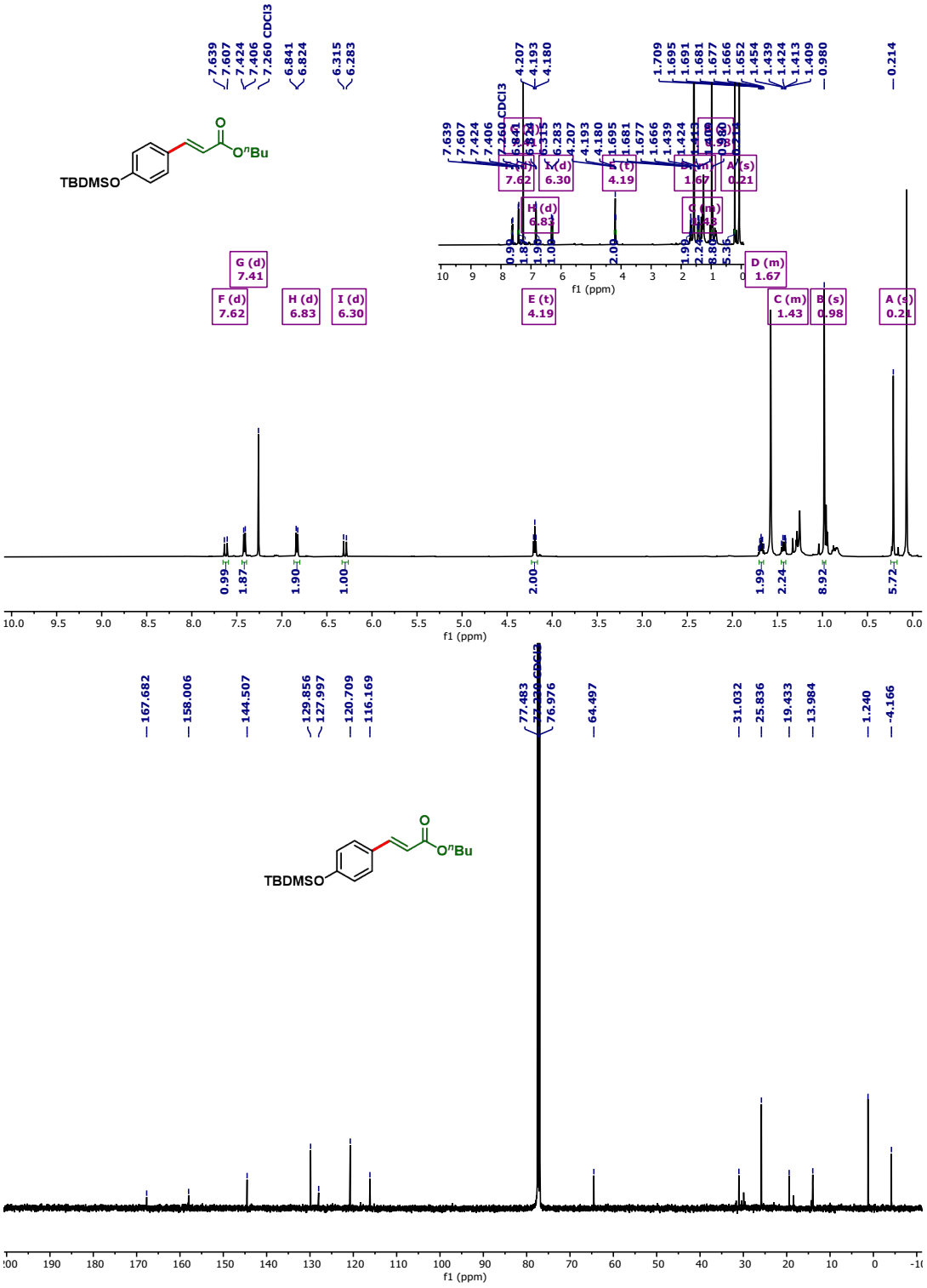

7.862
7.829
7.260 CDCl3
6.897
6.077
6.045
4.236
4.223
4.209
2.336
2.288
1.733
1.719
1.705
1.690
1.675
1.485
1.470
1.455
1.440
1.424
1.410
0.991
0.976
0.961
Me
O
O^{n}Bu
Me
Me
B (d) 7.85
C (s) 6.90
A (d) 6.06
F (s) 2.29
H (h) 1.45
E (s) 2.34
G (p) 1.70
I (t) 0.98
D (t) 4.22
1.00
2.00
1.00
2.12
5.75
2.89
2.47
2.12
3.10
f1 (ppm)
167.359
143.366
138.477
137.028
131.171
129.346
123.386
77.547
77.230 CDCl3
76.912
64.637
30.973
21.295
21.241
19.434
13.976

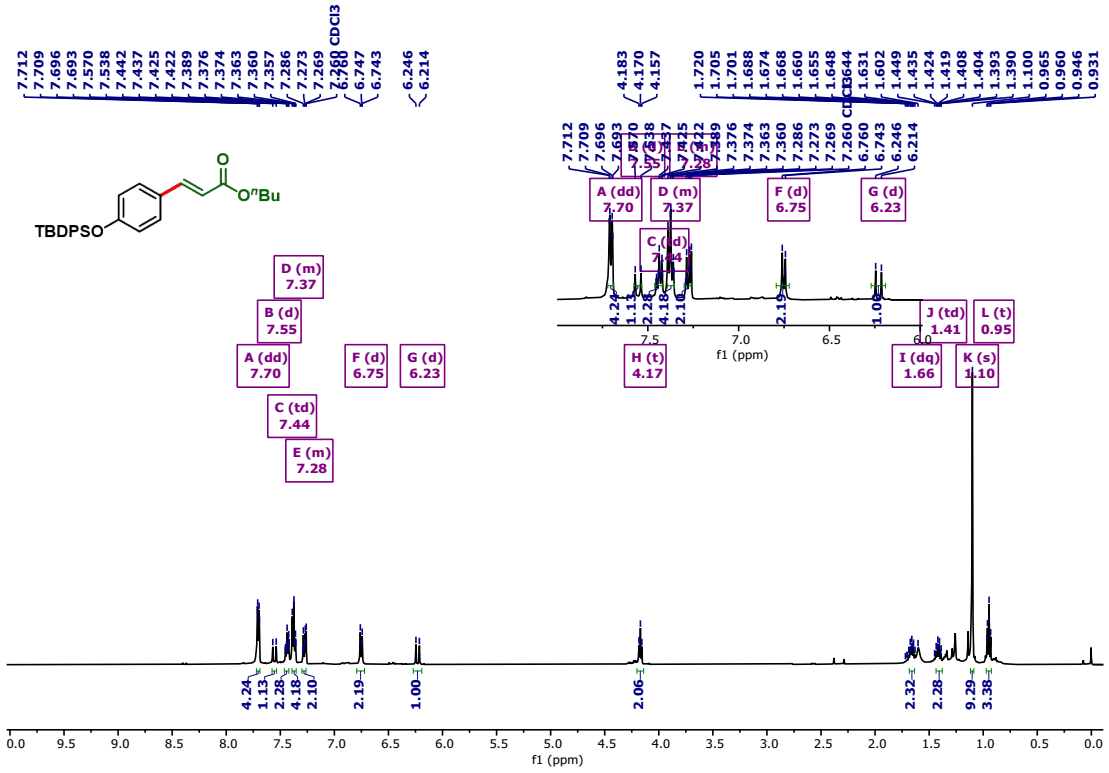

7.63
7.59
7.26 CDCl3
7.10
7.09
7.08
7.07
7.04
7.03
6.85
6.83
6.32
6.28
4.20
4.18
4.17
3.89
3.89
1.71
1.69
1.68
1.67
1.67
1.67
1.66
1.65
1.63
1.47
1.46
1.45
1.45
1.44
1.43
1.42
1.41
1.40
1.39
1.38
0.97
0.95
0.93
MeO
MeO
O
O"Bu
B (d) 7.61
C (dd) 7.08
E (d) 6.84
A (d) 6.30
D (d) 7.04
G (d) 3.89
F (t) 4.18
I (ddt) 1.42
H (m) 1.67
J (t) 0.95
f1 (ppm)
167.42
151.14
149.27
144.55
127.53
122.67
116.05
111.09
109.63
77.16 CDCl3
64.38
56.03
55.94
30.90
20.00
13.84

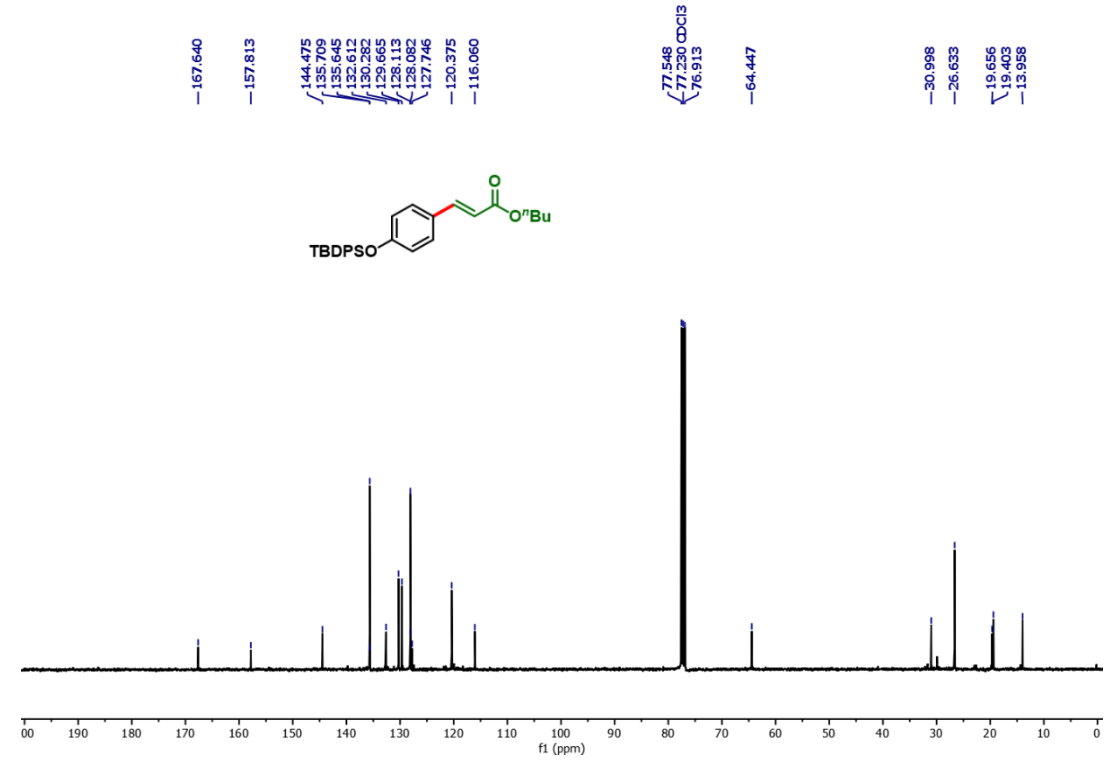

7.674
7.634
7.434
7.414
7.260 CDCl3
7.199
7.179
6.414
6.374
4.218
4.205
4.201
4.184
2.371
1.708
1.705
1.698
1.689
1.687
1.682
1.671
1.668
1.651
1.465
1.446
1.432
1.427
1.413
1.409
0.982
0.963
0.945
F (d) 7.65
G (d) 7.42
H (d) 7.19
I (d) 6.39
A (t) 4.20
B (s) 2.37
C (m) 1.68
D (m) 1.43
E (t) 0.96
0.95
1.57
1.82
0.78
2.00
3.01
2.27
2.35
3.81
f1 (ppm)
Me
O
O"Bu

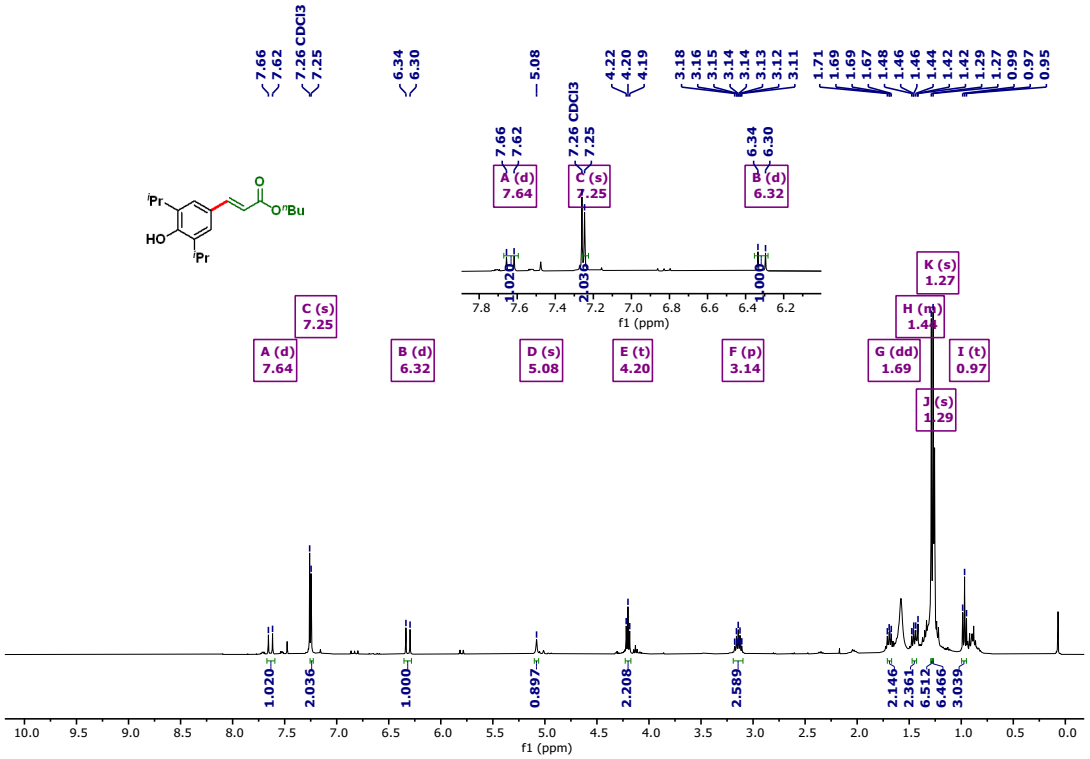

167.536
144.775
140.828
131.999
129.823
128.267
117.451
77.547
77.230 CDCl3
76.913
64.571
31.031
29.920
19.435
13.966
f1 (ppm)
Me
O
O"Bu

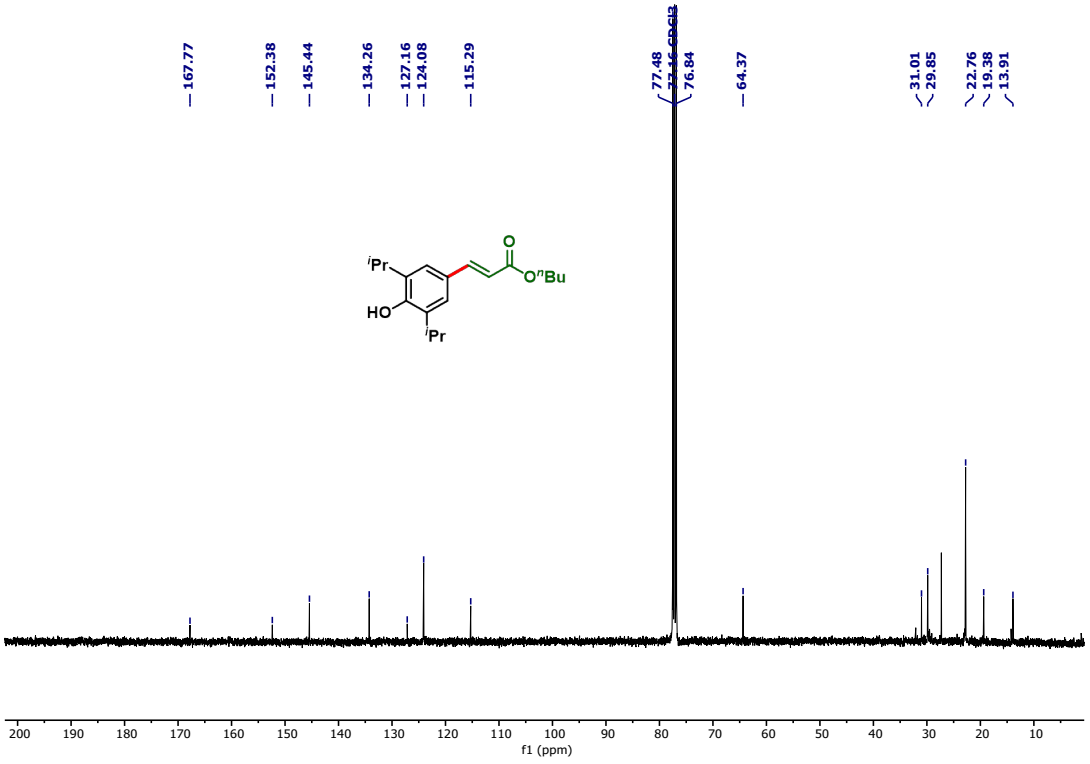

OH
O
O^{n}Bu
A (d) 7.95
B (dd) 7.48
C (dd) 7.22
D (td) 6.94
E (dd) 6.81
F (d) 6.56
G (t) 4.21
H (m) 1.68
I (m) 1.43
J (t) 0.96
f1 (ppm)
167.882
154.991
139.861
131.479
129.402
122.036
121.283
119.340
116.524
77.548
76.913
64.618
31.016
22.910
13.977

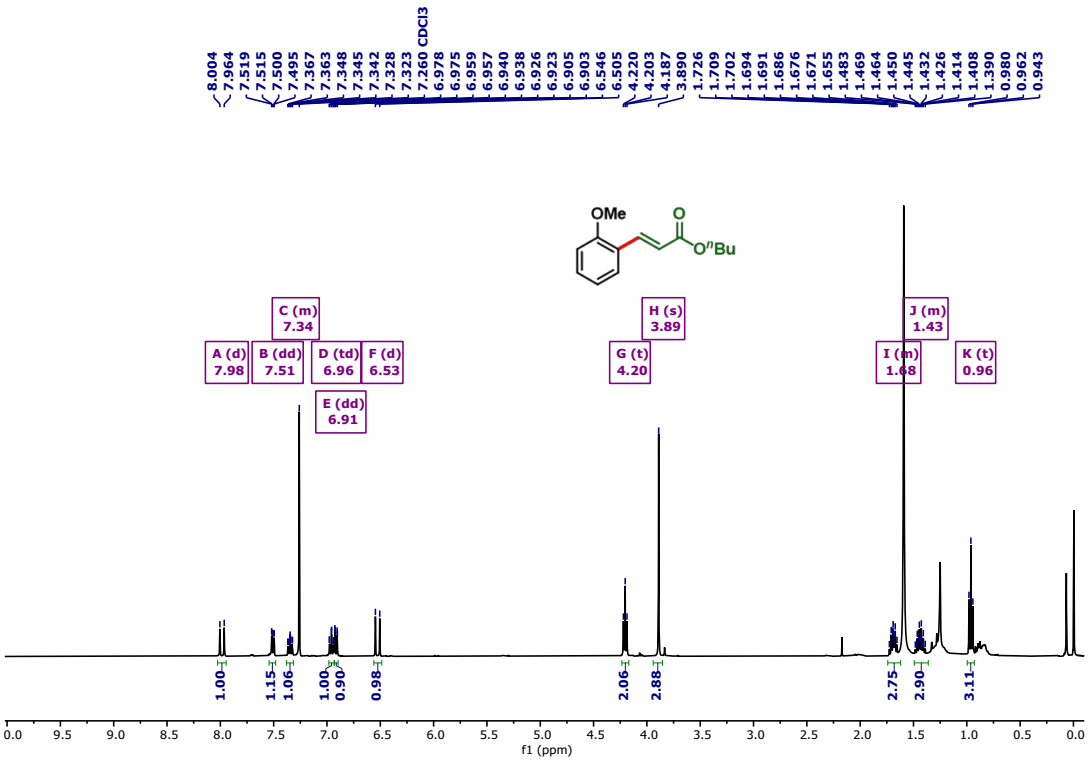

TBDMSO
O
O^{n}Bu
f1 (ppm)
TBDMSO
O
O^{n}Bu
f1 (ppm)

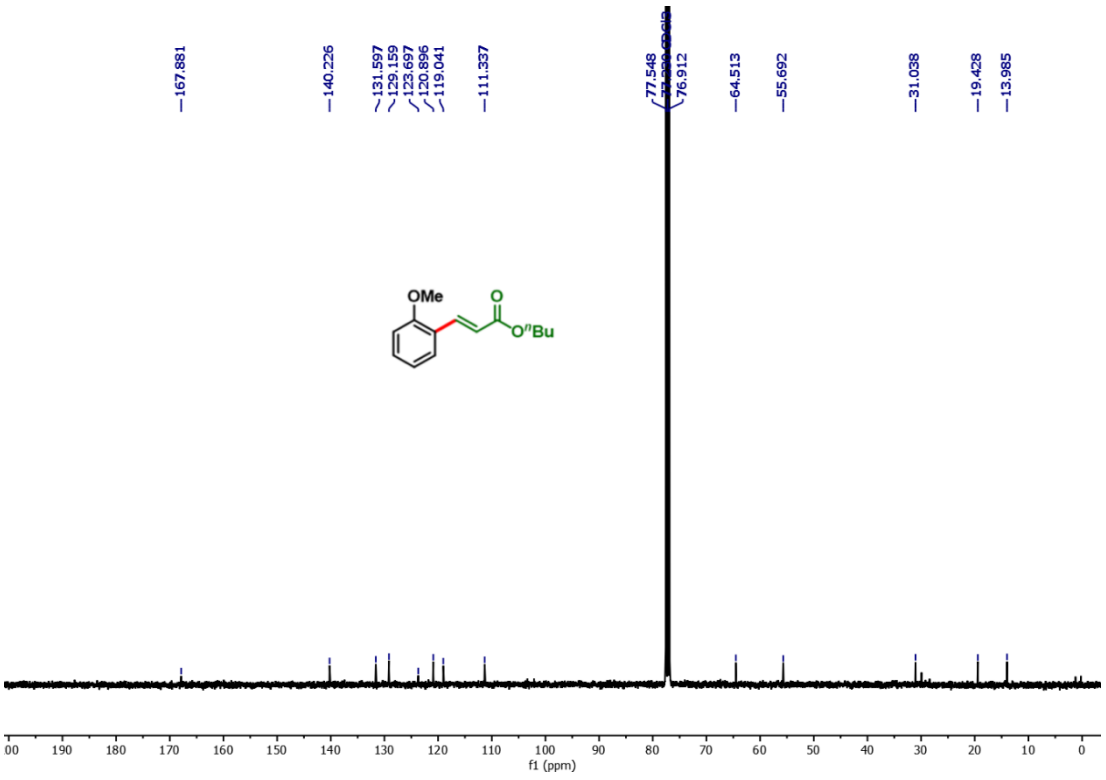

TBDPSO
O
O^{n}Bu
A (dd) 7.70
B (d) 7.55
C (td) 7.44
D (m) 7.37
E (m) 7.28
F (d) 6.75
G (d) 6.23
H (t) 4.17
I (dq) 1.66
J (td) 1.41
K (s) 1.10
L (t) 0.95
f1 (ppm)

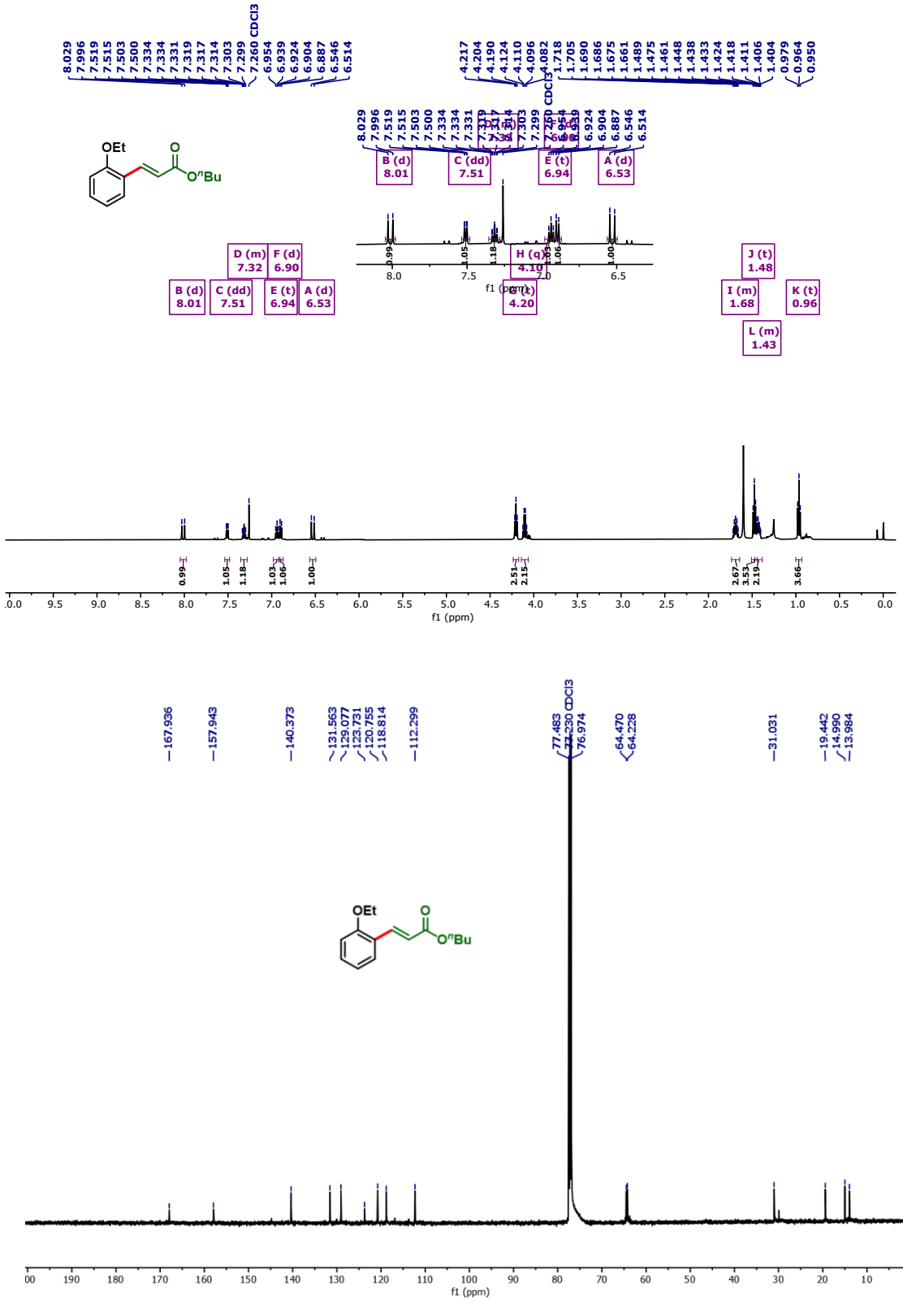

TBDPSO
O
O^{n}Bu
f1 (ppm)

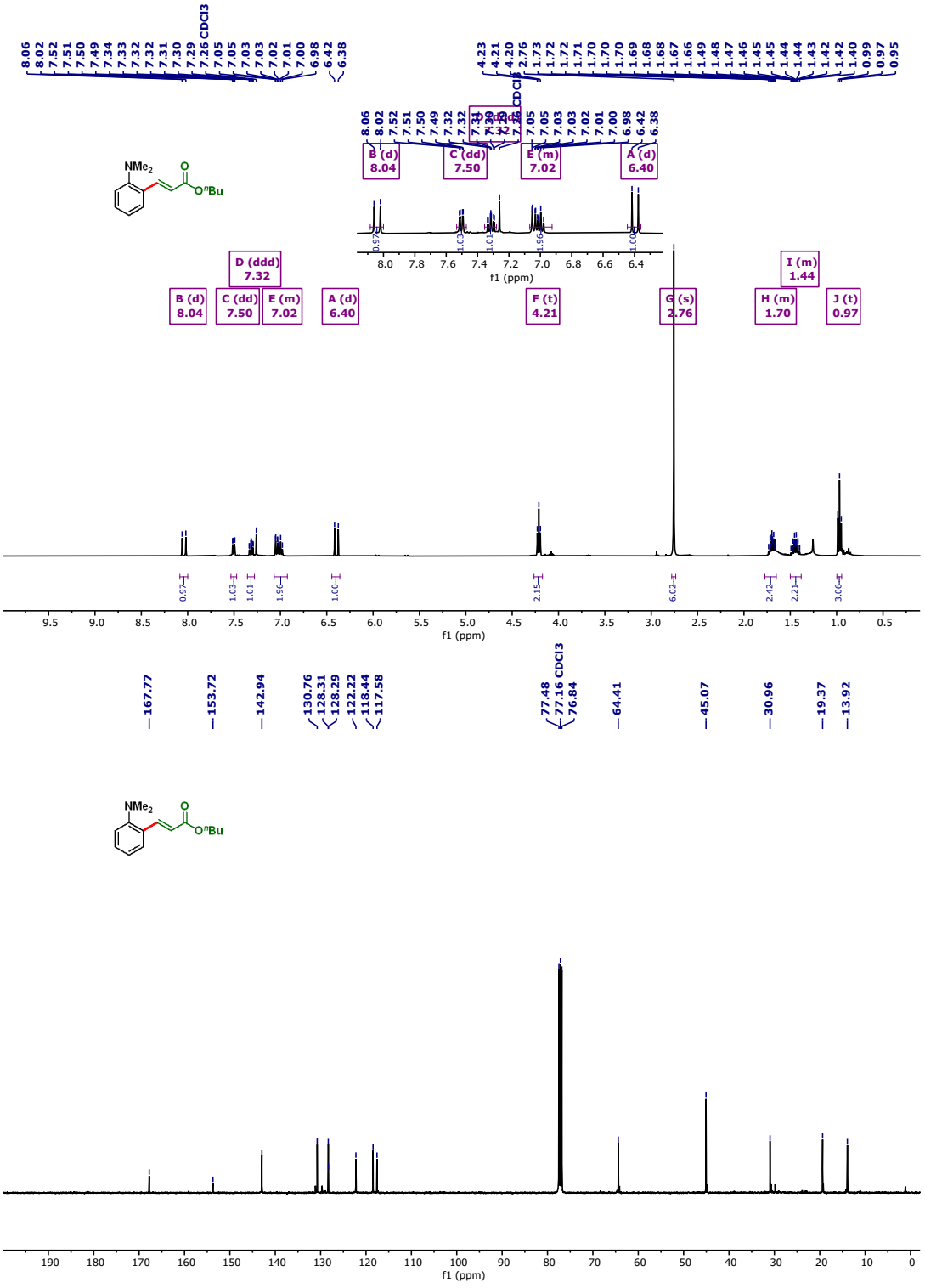

7.66
7.62
7.26 CDCl3
7.25
6.34
6.30
5.08
4.22
4.20
4.19
3.18
3.16
3.15
3.14
3.14
3.13
3.12
3.11
1.71
1.69
1.69
1.67
1.48
1.46
1.46
1.44
1.42
1.42
1.29
1.27
0.99
0.97
0.95
A (d) 7.64
C (s) 7.25
B (d) 6.32
D (s) 5.08
E (t) 4.20
F (p) 3.14
G (dd) 1.69
H (m) 1.44
I (t) 0.97
J (s) 1.29
K (s) 1.27
1.020
2.036
1.000
0.897
2.208
2.589
2.146
2.361
6.512
6.466
3.039
f1 (ppm)


167.77
152.38
145.44
134.26
127.16
124.08
115.29
77.48
77.16 CDCl3
76.84
64.37
31.01
29.85
22.76
19.38
13.91
f1 (ppm)

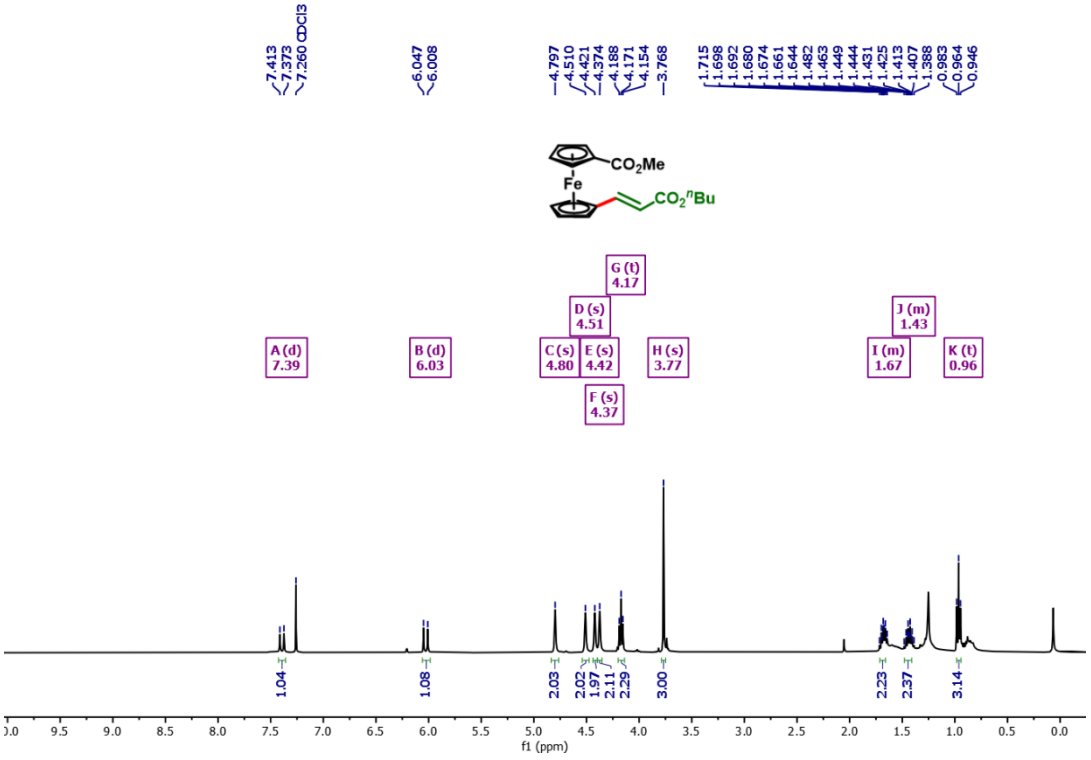

8.004
7.964
7.519
7.515
7.500
7.495
7.367
7.363
7.348
7.345
7.342
7.328
7.323
7.260 CDCl3
6.978
6.975
6.959
6.957
6.940
6.938
6.926
6.923
6.905
6.903
6.546
6.505
4.220
4.203
4.187
3.890
1.726
1.709
1.702
1.694
1.691
1.686
1.676
1.671
1.655
1.483
1.469
1.464
1.450
1.445
1.432
1.426
1.414
1.408
1.390
0.980
0.962
0.943
OMe
O
O^{n}Bu
A (d) 7.98
B (dd) 7.51
C (m) 7.34
D (td) 6.96
E (dd) 6.91
F (d) 6.53
G (t) 4.20
H (s) 3.89
I (m) 1.68
J (m) 1.43
K (t) 0.96
1.00
1.15
1.06
1.00
0.90
0.98
2.06
2.88
2.75
2.90
3.11
f1 (ppm)

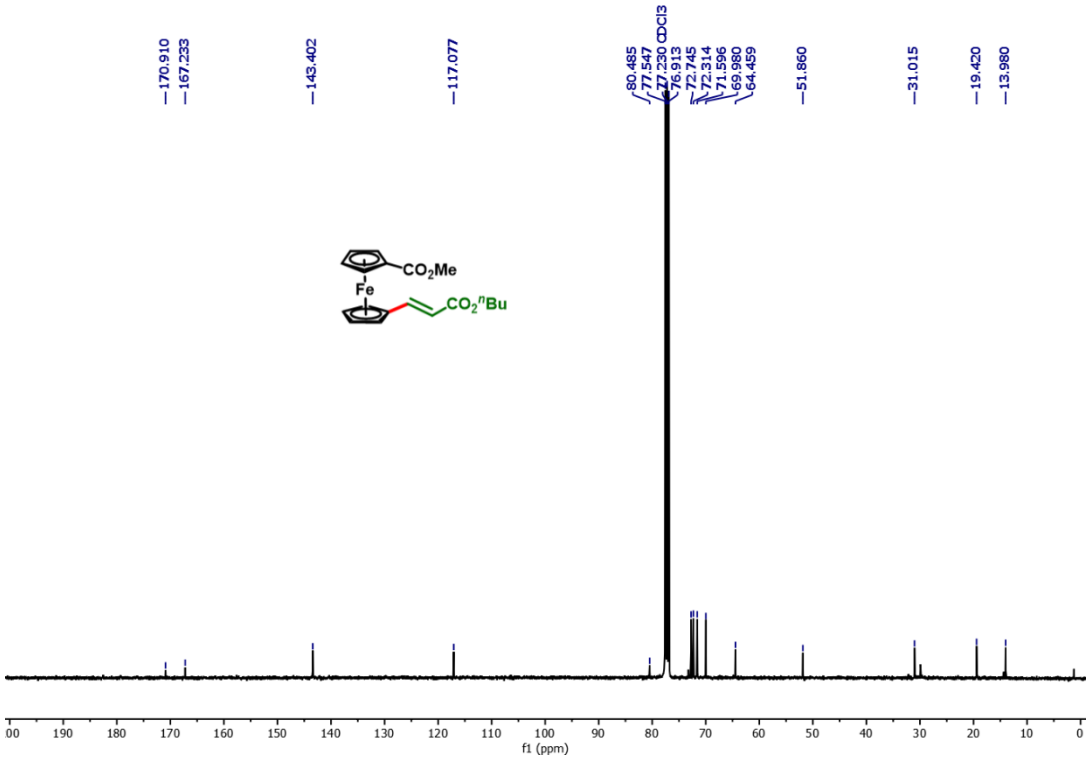

167.881
140.226
131.597
129.159
123.697
120.896
119.041
111.337
77.548
76.912
64.513
55.692
31.038
19.428
13.985
OMe
O
O^{n}Bu
f1 (ppm)

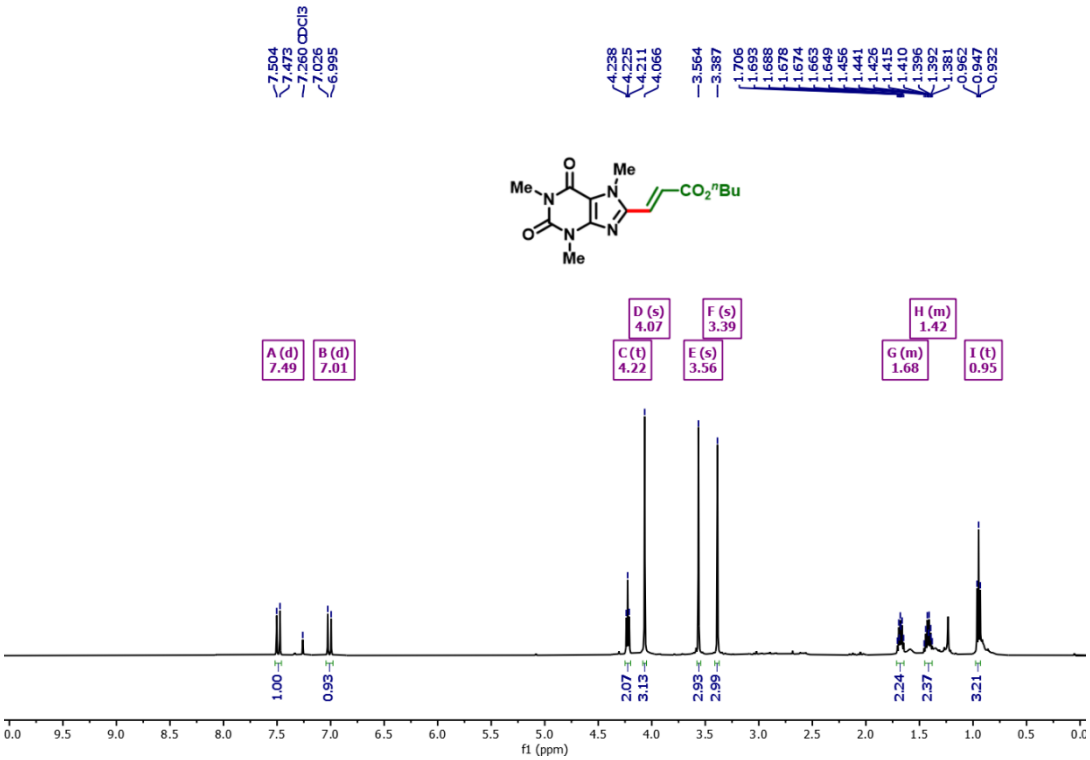

8.029
7.996
7.519
7.515
7.503
7.500
7.334
7.334
7.331
7.319
7.317
7.314
7.303
7.299
7.260 CDCl3
6.954
6.939
6.924
6.904
6.887
6.546
6.514
4.217
4.204
4.190
4.124
4.110
4.096
4.082
1.718
1.705
1.690
1.686
1.675
1.661
1.489
1.475
1.461
1.448
1.438
1.433
1.424
1.418
1.411
1.406
1.404
0.979
0.964
0.950
OEt
O
O^{n}Bu
B (d) 8.01
C (dd) 7.51
E (t) 6.94
A (d) 6.53
D (m) 7.32
F (d) 6.90
H (q) 4.10
G (t) 4.20
J (t) 1.48
I (m) 1.68
K (t) 0.96
L (m) 1.43
f1 (ppm)
167.936
157.943
140.373
131.563
129.077
123.731
120.755
118.814
112.299
77.483
77.230 CDCl3
76.974
64.470
64.228
31.031
19.442
14.990
13.984

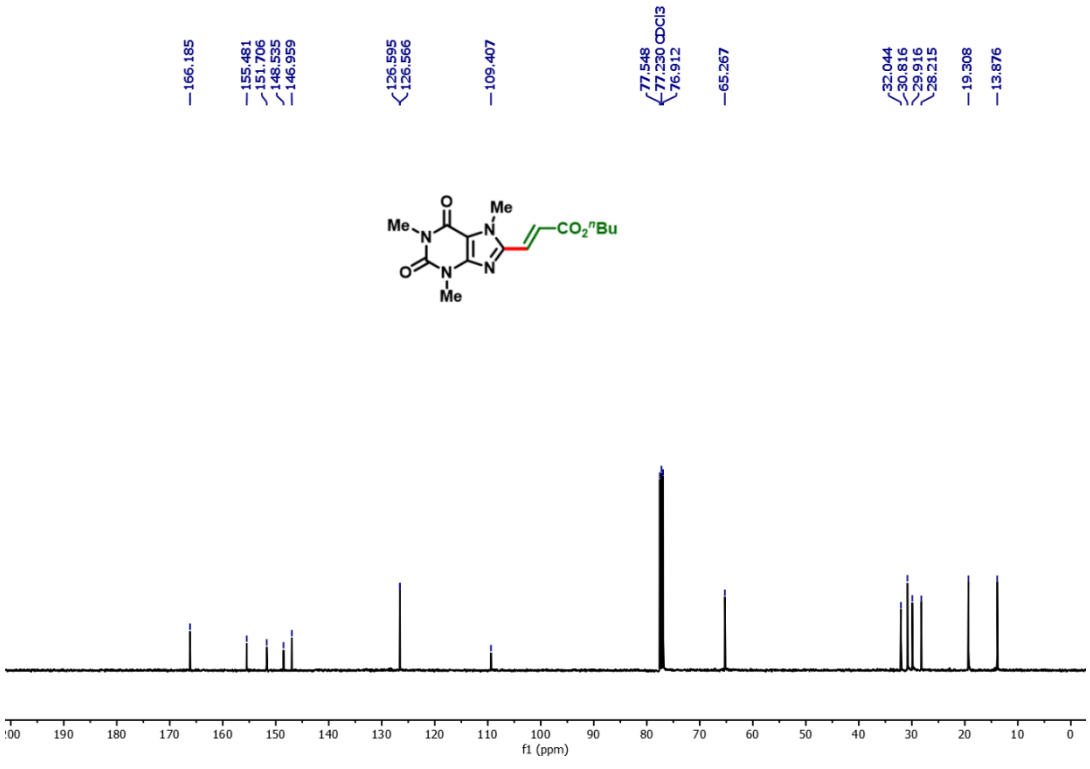

NMe2
O
O
nBu
B (d) 8.04
C (dd) 7.50
E (m) 7.02
A (d) 6.40
D (ddd) 7.32
F (t) 4.21
G (s) 2.76
H (m) 1.70
I (m) 1.44
J (t) 0.97
f1 (ppm)

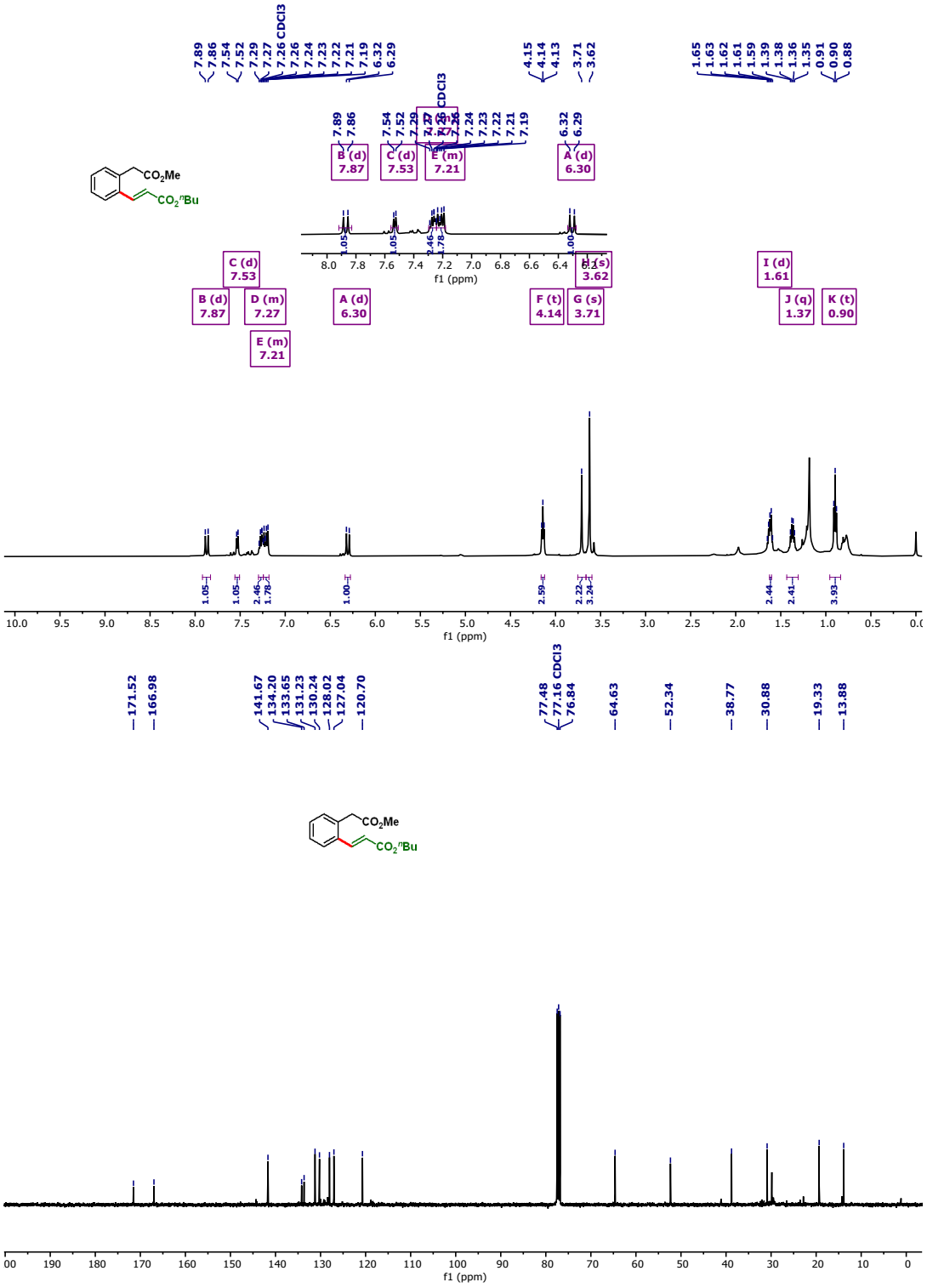
167.77
153.72
142.94
130.76
128.31
128.29
122.22
118.44
117.58
77.48
77.16 CDCl3
76.84
64.41
45.07
30.96
19.37
13.92
f1 (ppm)

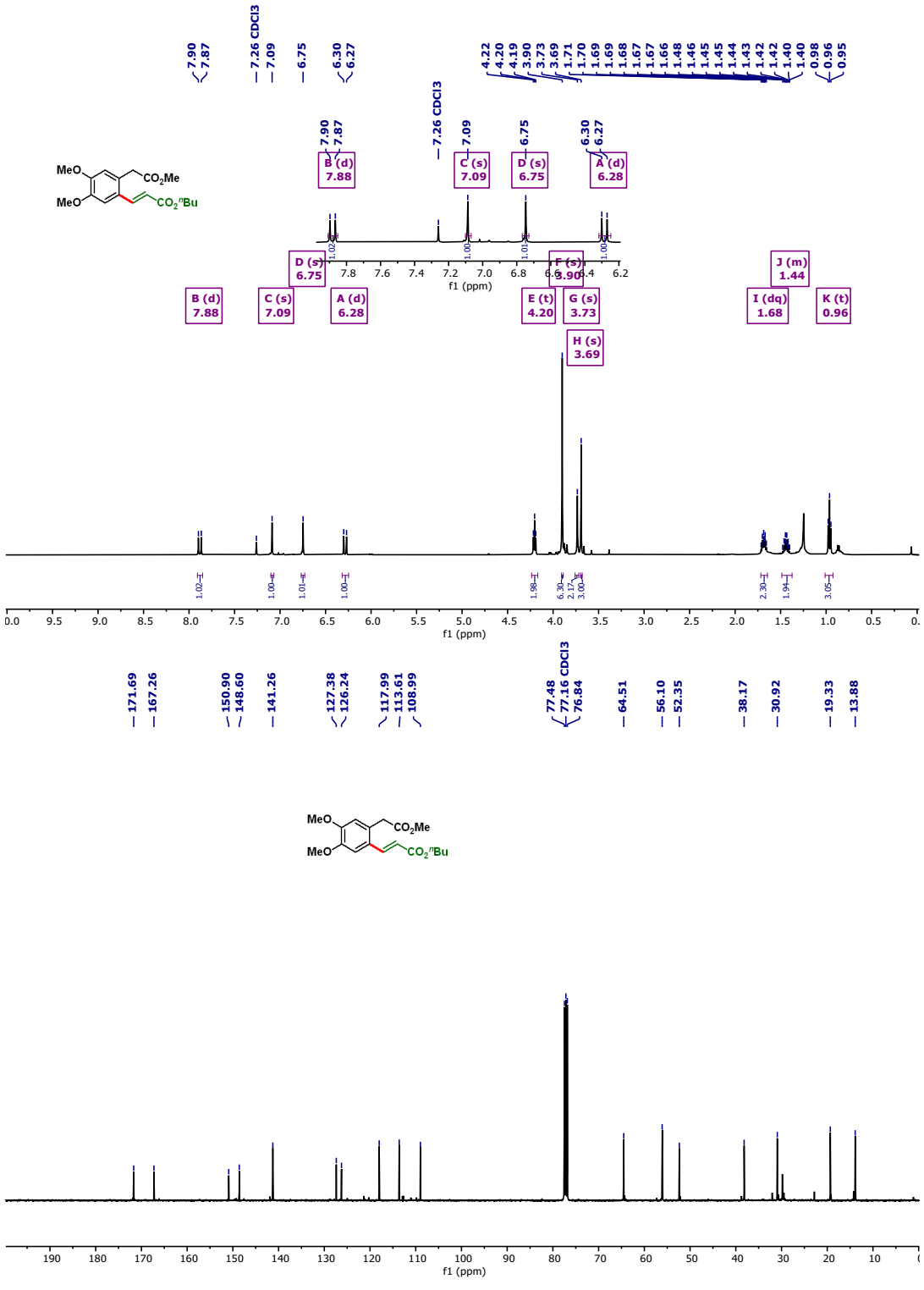

7.413
7.373
7.260 CDCl3
6.047
6.008
4.797
4.510
4.421
4.374
4.188
4.171
4.154
3.768
1.715
1.698
1.692
1.680
1.674
1.661
1.644
1.482
1.463
1.449
1.444
1.431
1.425
1.413
1.407
1.388
0.983
0.964
0.946
CO2Me
Fe
CO2nBu
A (d) 7.39
B (d) 6.03
C (s) 4.80
D (s) 4.51
E (s) 4.42
F (s) 4.37
G (t) 4.17
H (s) 3.77
I (m) 1.67
J (m) 1.43
K (t) 0.96
1.04
1.08
2.03
2.02
1.97
2.11
2.29
3.00
2.23
2.37
3.14
f1 (ppm)

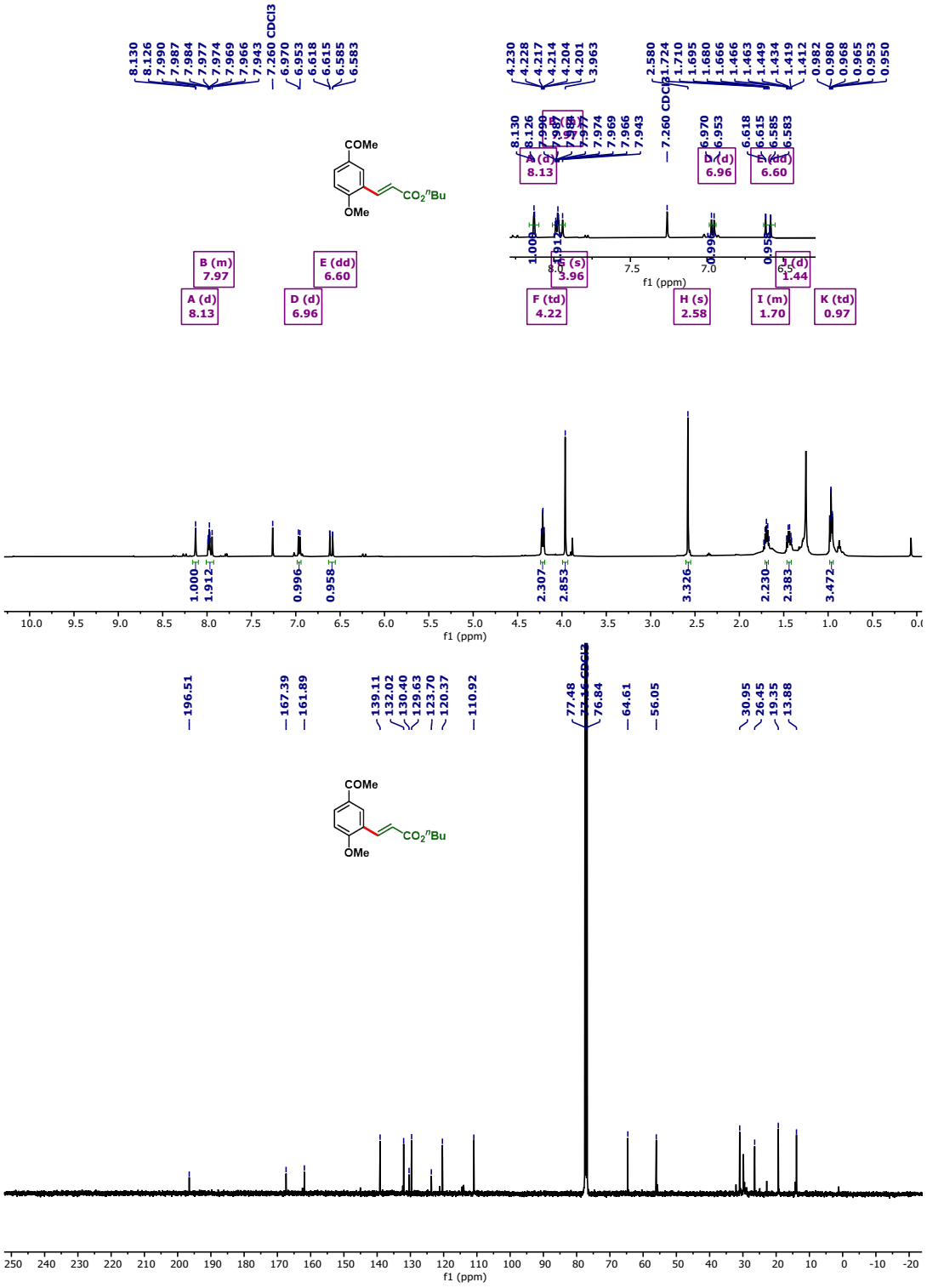

170.910
167.233
143.402
117.077
80.485
77.547
77.230 CDCl3
76.913
72.745
72.314
71.596
69.980
64.459
51.860
31.015
19.420
13.980
CO2Me
Fe
CO2nBu
f1 (ppm)

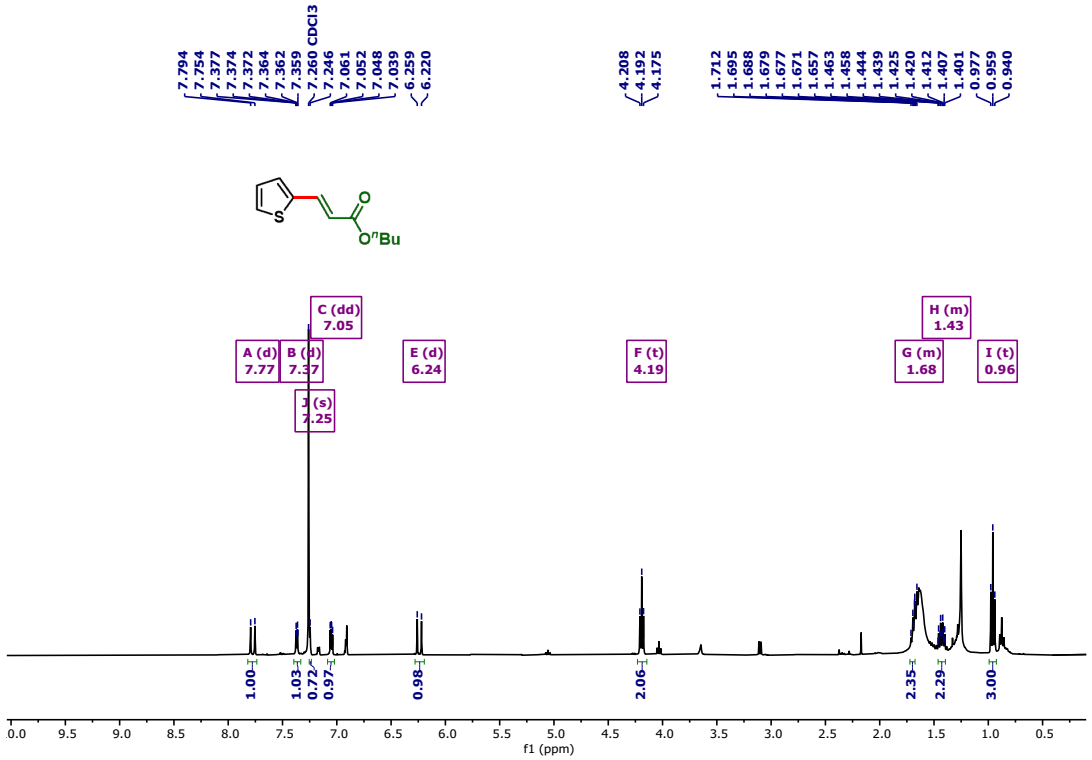

7.504
7.473
7.260 CDCl3
7.026
6.995
4.238
4.225
4.211
4.066
3.564
3.387
1.706
1.693
1.688
1.678
1.674
1.663
1.649
1.456
1.441
1.426
1.415
1.410
1.396
1.392
1.381
0.962
0.947
0.932
Me
Me
Me
CO2nBu
A (d) 7.49
B (d) 7.01
C (t) 4.22
D (s) 4.07
E (s) 3.56
F (s) 3.39
G (m) 1.68
H (m) 1.42
I (t) 0.95
1.00
0.93
2.07
3.13
2.93
2.99
2.24
2.37
3.21
f1 (ppm)

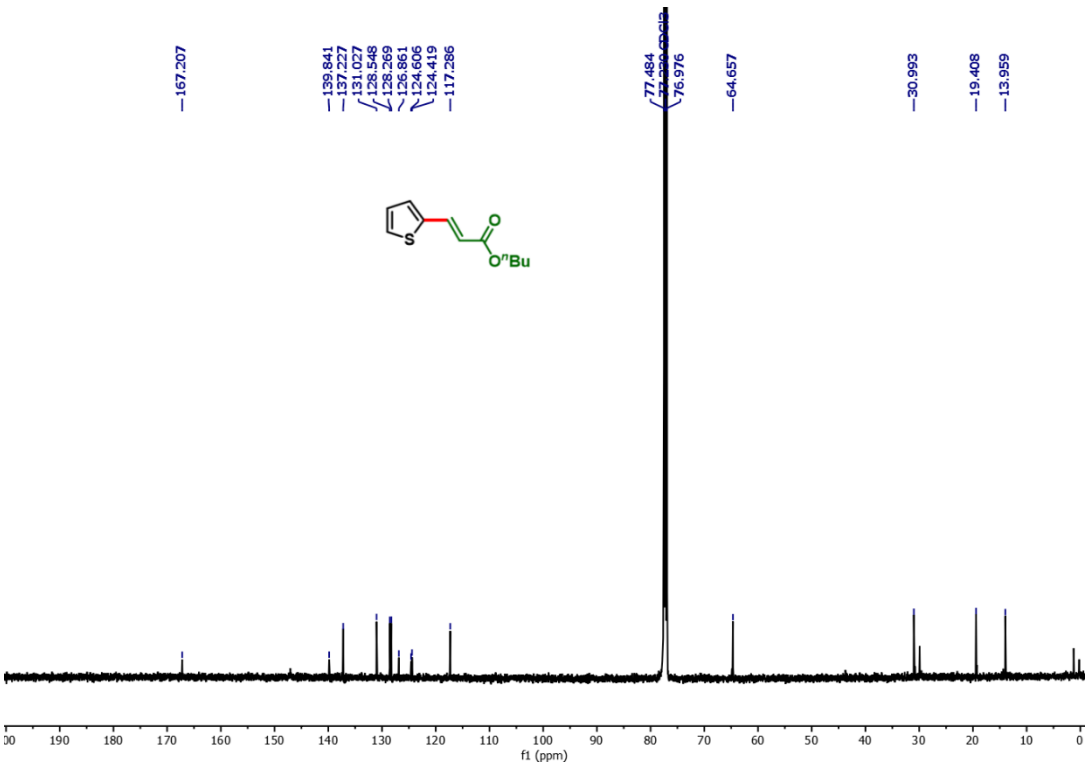

166.185
155.481
151.706
148.535
146.959
126.595
126.566
109.407
77.548
77.230 CDCl3
76.912
65.267
32.044
30.816
29.916
28.215
19.308
13.876
Me
Me
Me
CO2nBu
f1 (ppm)

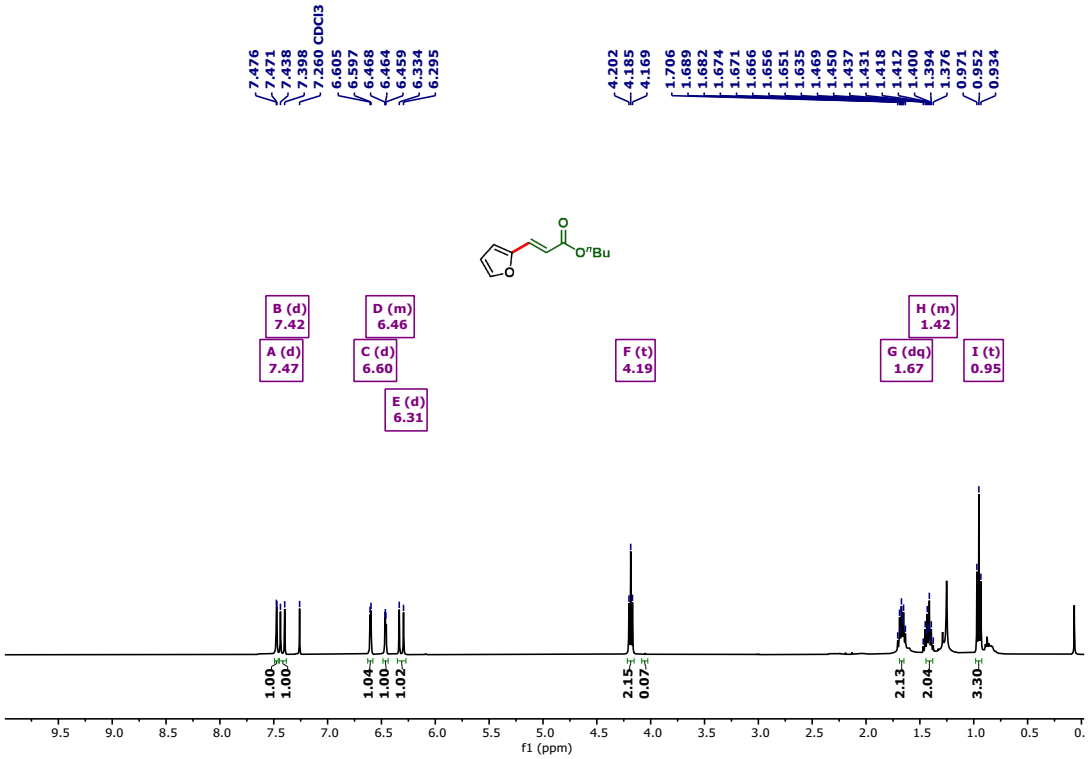

7.89
7.86
7.54
7.52
7.29
7.27
7.26 CDCl3
7.24
7.23
7.22
7.21
7.19
6.32
6.29
4.15
4.14
4.13
3.71
3.62
1.65
1.63
1.62
1.61
1.59
1.39
1.38
1.36
1.35
0.91
0.90
0.88
CO2Me
CO2nBu
B (d) 7.87
C (d) 7.53
E (m) 7.21
A (d) 6.30
f1 (ppm)
C (d) 7.53
B (d) 7.87
D (m) 7.27
E (m) 7.21
A (d) 6.30
F (t) 4.14
G (s) 3.71
I (d) 1.61
J (q) 1.37
K (t) 0.90
1.05
1.05
2.46
1.78
1.00
2.59
2.22
3.24
2.44
2.41
3.93
171.52
166.98
141.67
134.20
133.65
131.23
130.24
128.02
127.04
120.70
77.48
77.16 CDCl3
76.84
64.63
52.34
38.77
30.88
19.33
13.88

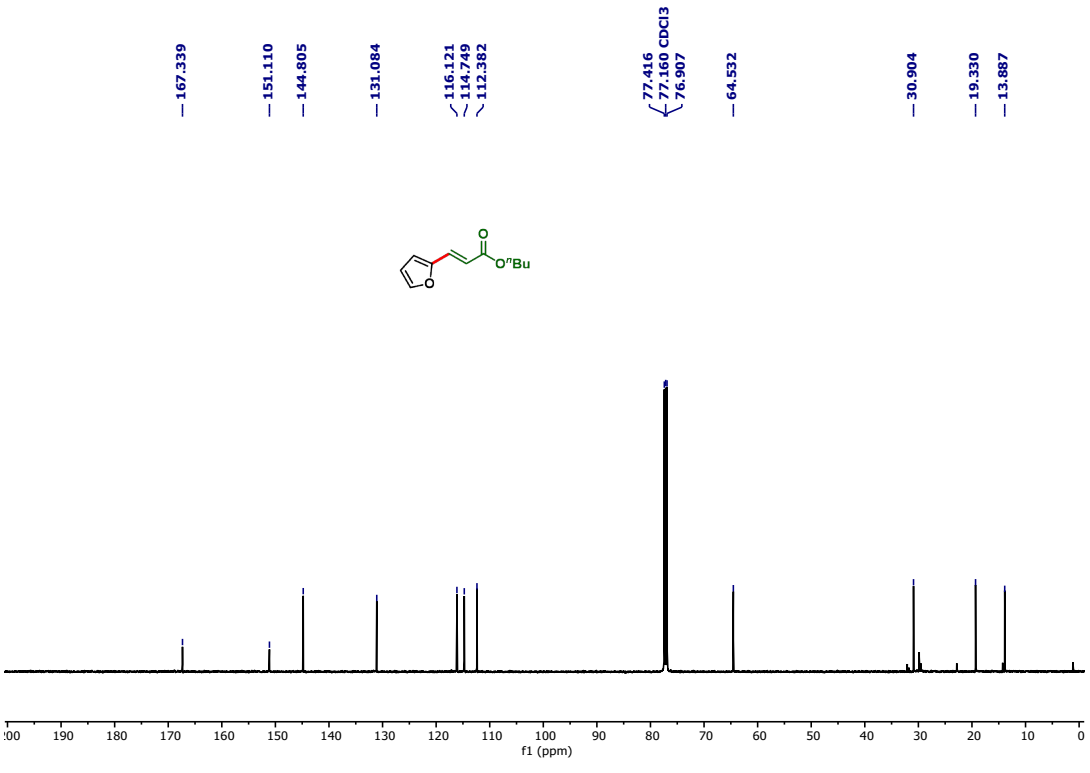

MeO
MeO
CO2Me
CO2tBu
7.90
7.87
7.26 CDCl3
7.09
6.75
6.30
6.27
4.22
4.20
4.19
3.90
3.73
3.69
1.71
1.70
1.69
1.69
1.68
1.67
1.67
1.66
1.48
1.46
1.45
1.45
1.44
1.43
1.42
1.42
1.40
1.40
0.98
0.96
0.95
B (d) 7.88
C (s) 7.09
D (s) 6.75
A (d) 6.28
F (s) 5.90
J (m) 1.44
E (t) 4.20
G (s) 3.73
I (dq) 1.68
K (t) 0.96
H (s) 3.69
f1 (ppm)
171.69
167.26
150.90
148.60
141.26
127.38
126.24
117.99
113.61
108.99
77.48
77.16 CDCl3
76.84
64.51
56.10
52.35
38.17
30.92
19.33
13.88

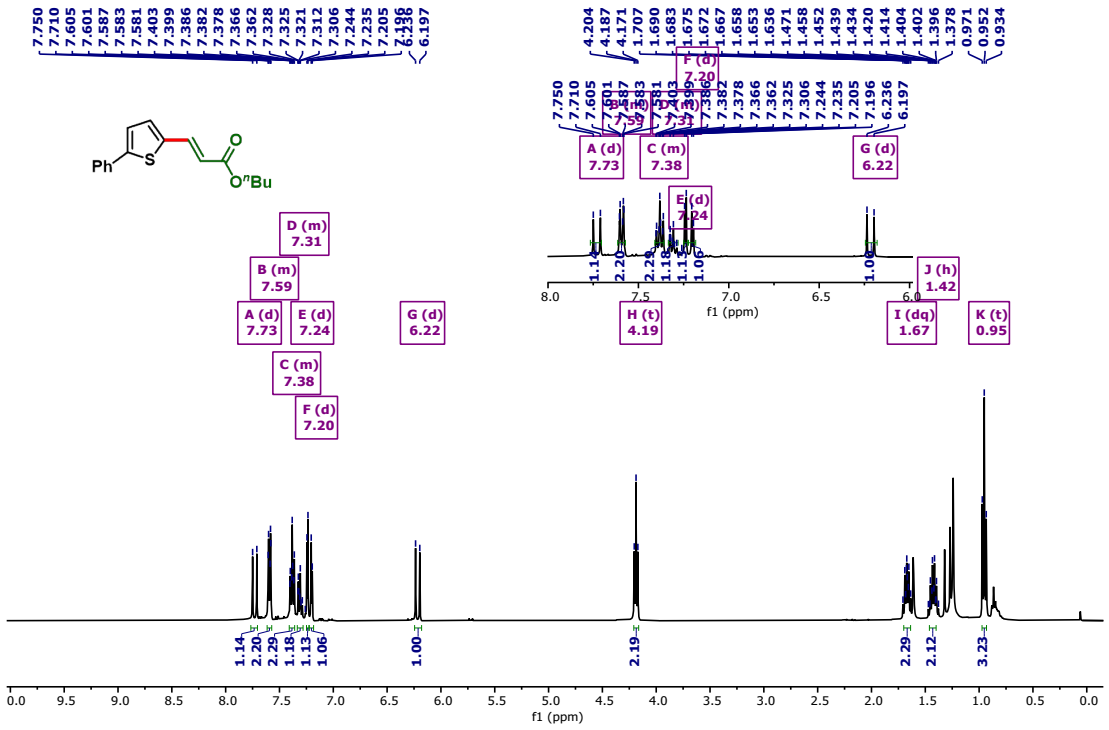

8.130
8.126
7.990
7.987
7.984
7.977
7.974
7.969
7.966
7.943
7.260 CDCl3
6.970
6.953
6.618
6.615
6.585
6.583
4.230
4.228
4.217
4.214
4.204
4.201
3.963
2.580
1.724
1.710
1.695
1.680
1.666
1.466
1.463
1.449
1.434
1.419
1.412
0.982
0.980
0.968
0.965
0.953
0.950
COMe
CO2nBu
OMe
A (d) 8.13
B (m) 7.97
D (d) 6.96
E (dd) 6.60
F (td) 4.22
G (s) 3.96
H (s) 2.58
I (m) 1.70
J (d) 1.44
K (td) 0.97
1.000
1.912
0.996
0.958
2.307
2.853
3.326
2.230
2.383
3.472
f1 (ppm)
196.51
167.39
161.89
139.11
132.02
130.40
129.63
123.70
120.37
110.92
77.48
77.16 CDCl3
76.84
64.61
56.05
30.95
26.45
19.35
13.88

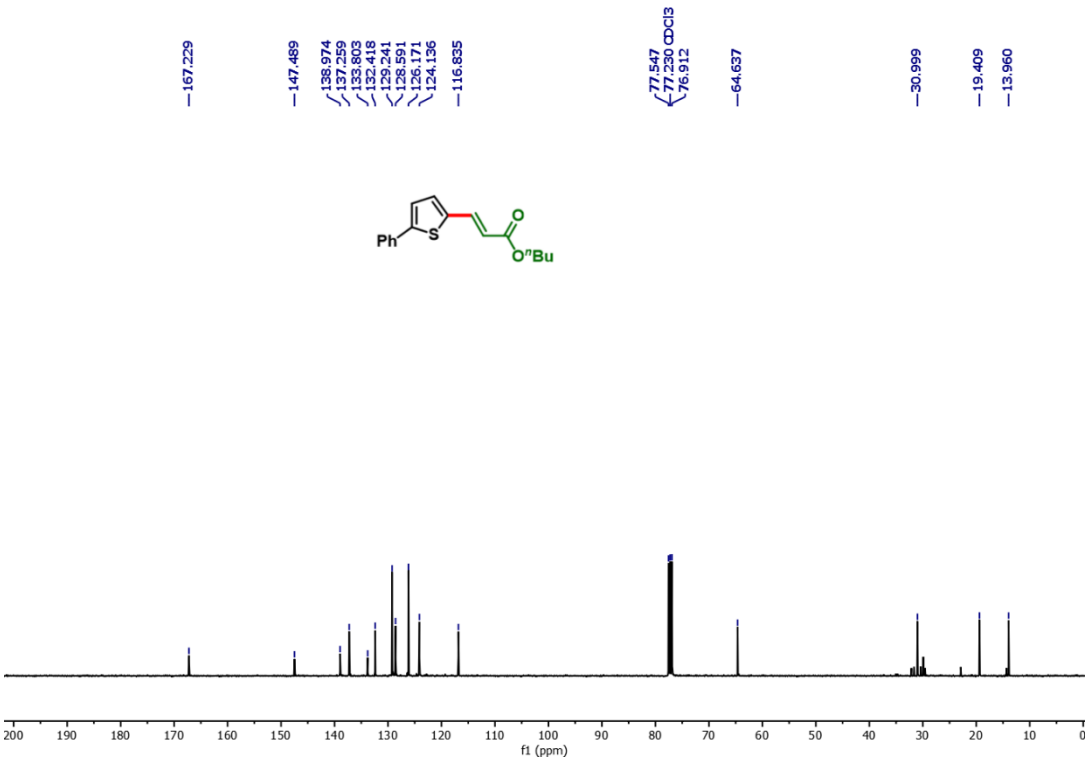

7.794
7.754
7.377
7.374
7.372
7.364
7.362
7.359
7.260 CDCl3
7.246
7.061
7.052
7.048
7.039
6.259
6.220
4.208
4.192
4.175
1.712
1.695
1.688
1.679
1.677
1.671
1.657
1.463
1.458
1.444
1.439
1.425
1.420
1.412
1.407
1.401
0.977
0.959
0.940
O
O
nBu
A (d) 7.77
B (d) 7.37
C (dd) 7.05
J (s) 7.25
E (d) 6.24
F (t) 4.19
G (m) 1.68
H (m) 1.43
I (t) 0.96
1.00
1.03
0.72
0.97
0.98
2.06
2.35
2.29
3.00
f1 (ppm)

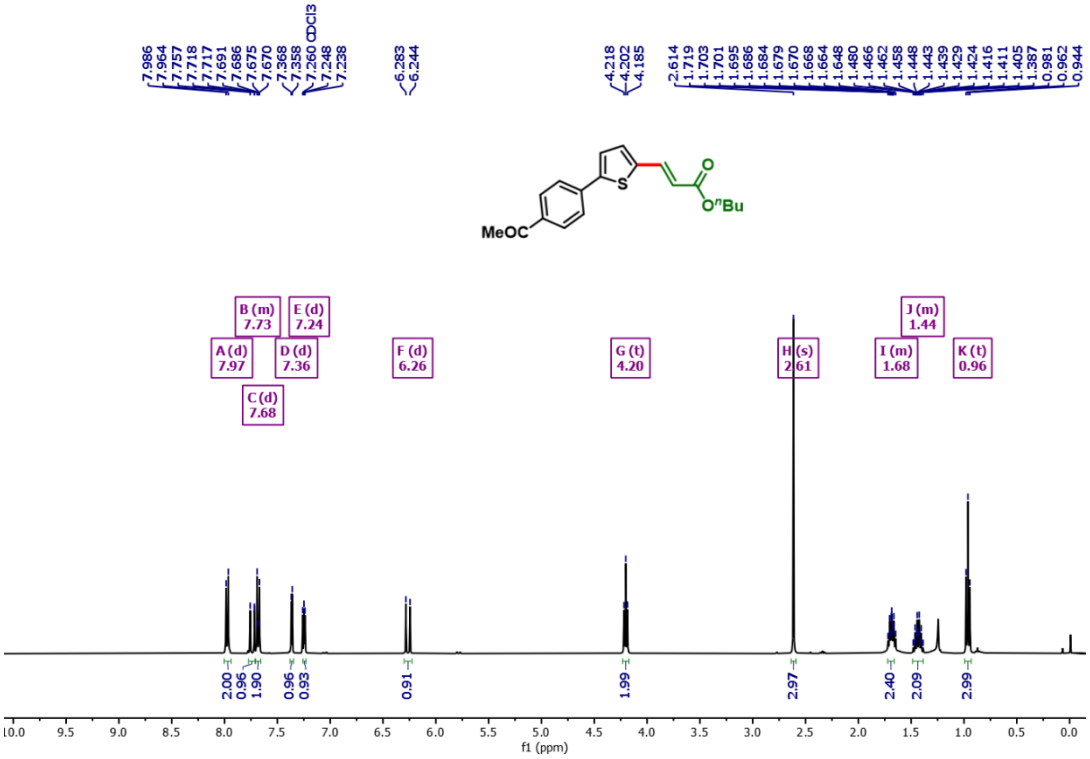

167.207
139.841
137.227
131.027
128.548
128.269
126.861
124.606
124.419
117.286
77.484
77.260 CDCl3
76.976
64.657
30.993
19.408
13.959
O
O
nBu
f1 (ppm)

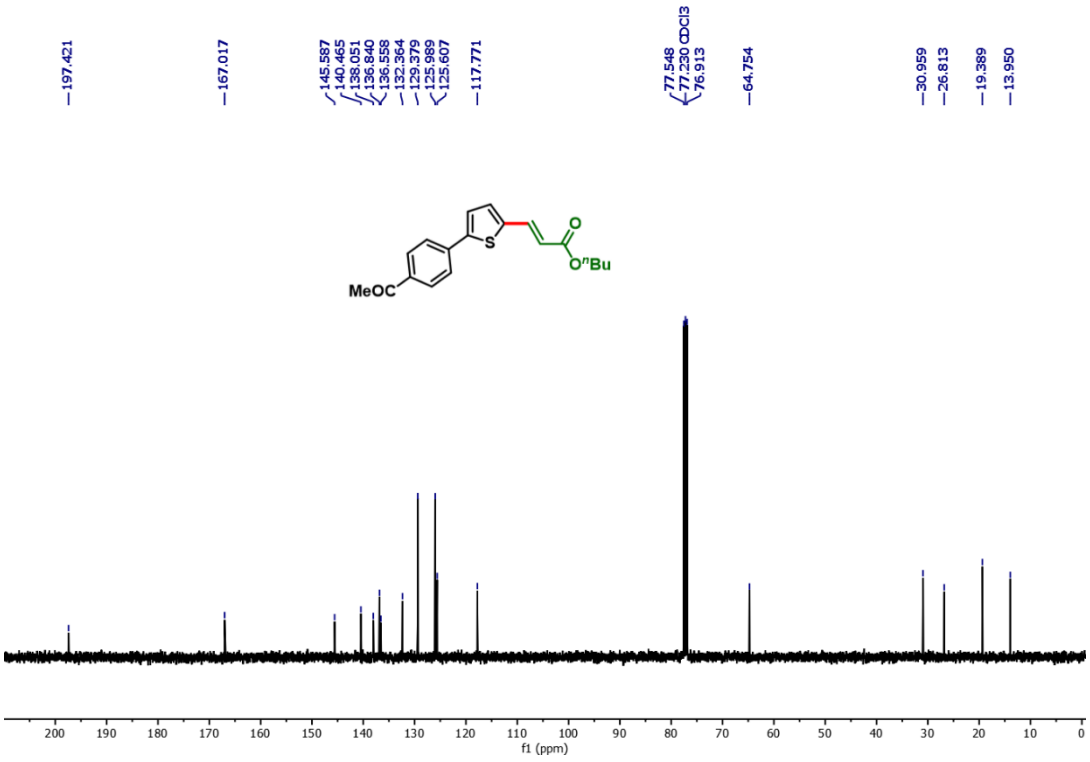

7.476
7.471
7.438
7.398
7.260 CDCl3
6.605
6.597
6.468
6.464
6.459
6.334
6.295
4.202
4.185
4.169
1.706
1.689
1.682
1.674
1.671
1.666
1.656
1.651
1.635
1.469
1.450
1.437
1.431
1.418
1.412
1.400
1.394
1.376
0.971
0.952
0.934
O
O
nBu
B (d) 7.42
A (d) 7.47
D (m) 6.46
C (d) 6.60
E (d) 6.31
F (t) 4.19
H (m) 1.42
G (dq) 1.67
I (t) 0.95
1.00
1.00
1.04
1.00
1.02
2.15
0.07
2.13
2.04
3.30
f1 (ppm)

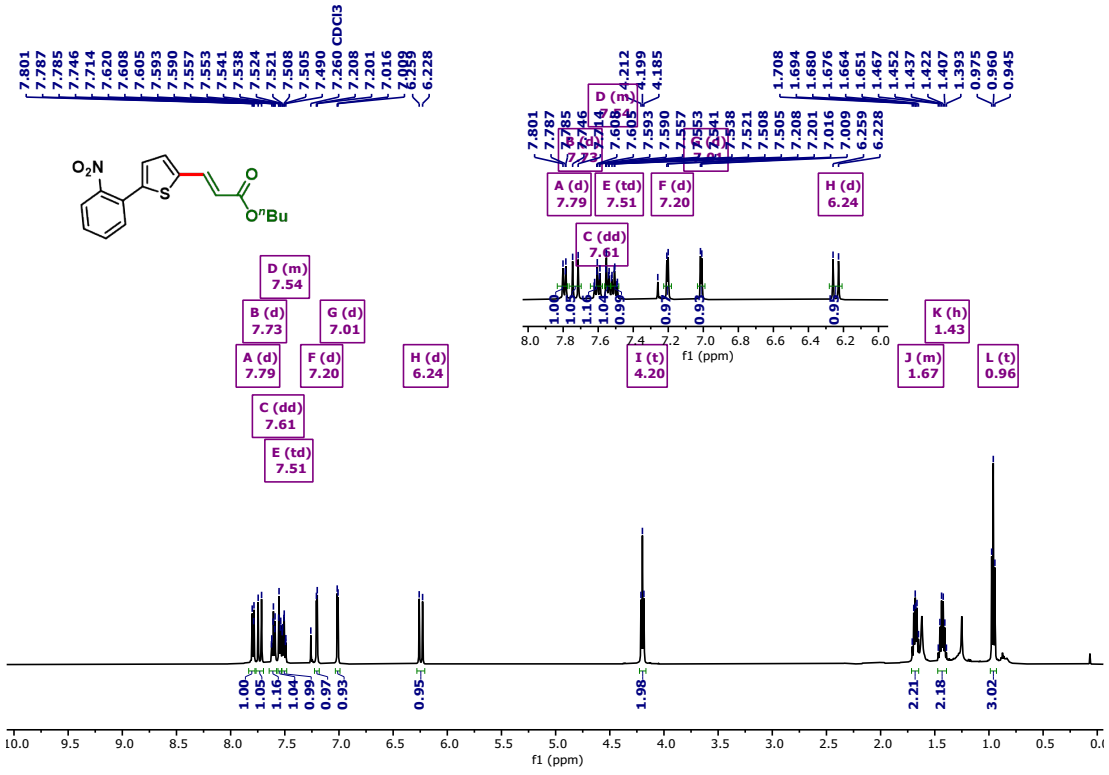

167.339
151.110
144.805
131.084
116.121
114.749
112.382
77.416
77.160 CDCl3
76.907
64.532
30.904
19.330
13.887
O
O
nBu
f1 (ppm)

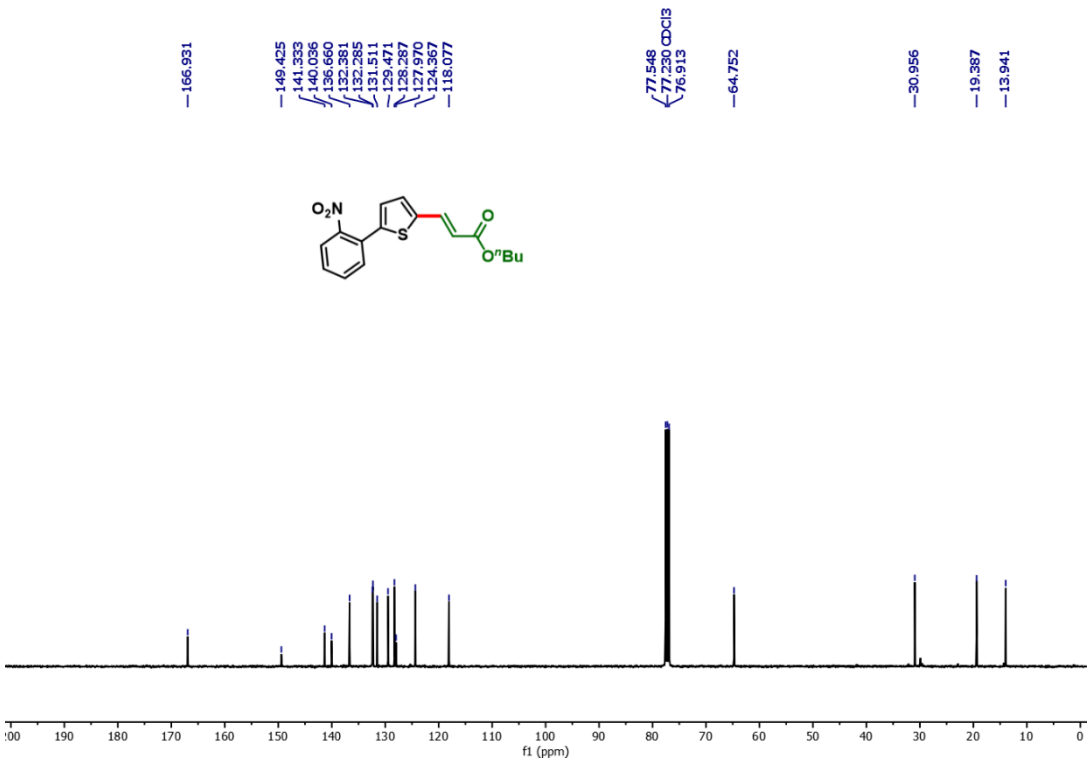

Ph
S
O
O^{n}Bu
D (m) 7.31
B (m) 7.59
A (d) 7.73
E (d) 7.24
G (d) 6.22
C (m) 7.38
F (d) 7.20
H (t) 4.19
I (dq) 1.67
J (h) 1.42
K (t) 0.95
f1 (ppm)

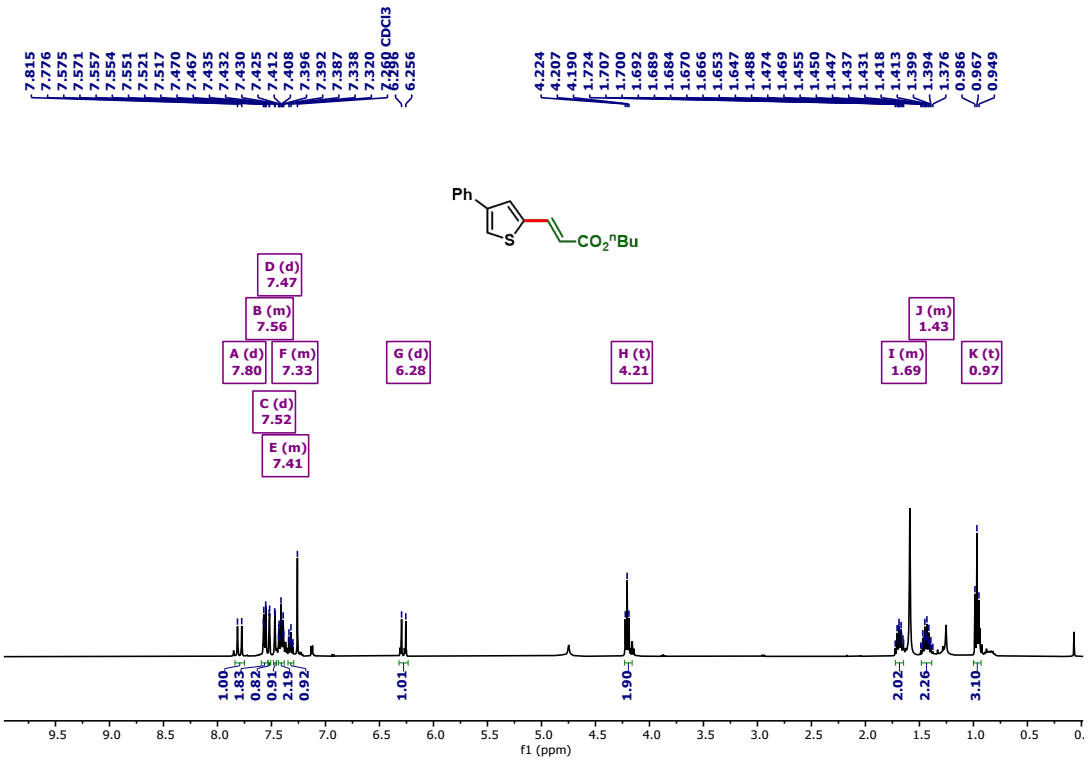

167.229
147.489
138.974
137.259
133.803
132.418
129.241
128.591
126.171
124.136
116.835
77.547
77.230 CDCl3
76.912
64.637
30.999
19.409
13.960
Ph
S
O
O^{n}Bu
f1 (ppm)

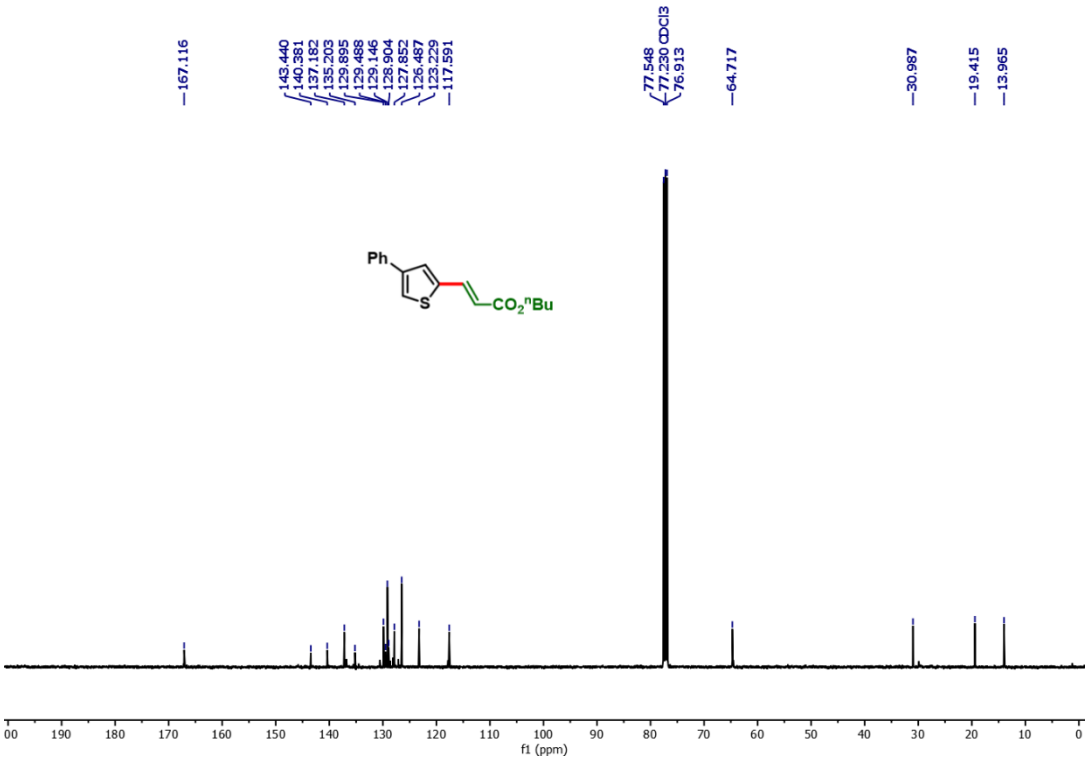

7.986
7.964
7.757
7.718
7.717
7.691
7.686
7.675
7.670
7.368
7.358
7.260 CDCl3
7.248
7.238
6.283
6.244
4.218
4.202
4.185
2.614
1.719
1.703
1.701
1.695
1.686
1.684
1.679
1.670
1.668
1.664
1.648
1.480
1.466
1.462
1.458
1.448
1.443
1.439
1.429
1.424
1.416
1.411
1.405
1.387
0.981
0.962
0.944
MeOC
O
O"Bu
A (d) 7.97
B (m) 7.73
C (d) 7.68
D (d) 7.36
E (d) 7.24
F (d) 6.26
G (t) 4.20
H (s) 2.61
I (m) 1.68
J (m) 1.44
K (t) 0.96
2.00
0.96
1.90
0.96
0.93
0.91
1.99
2.97
2.40
2.09
2.99
f1 (ppm)

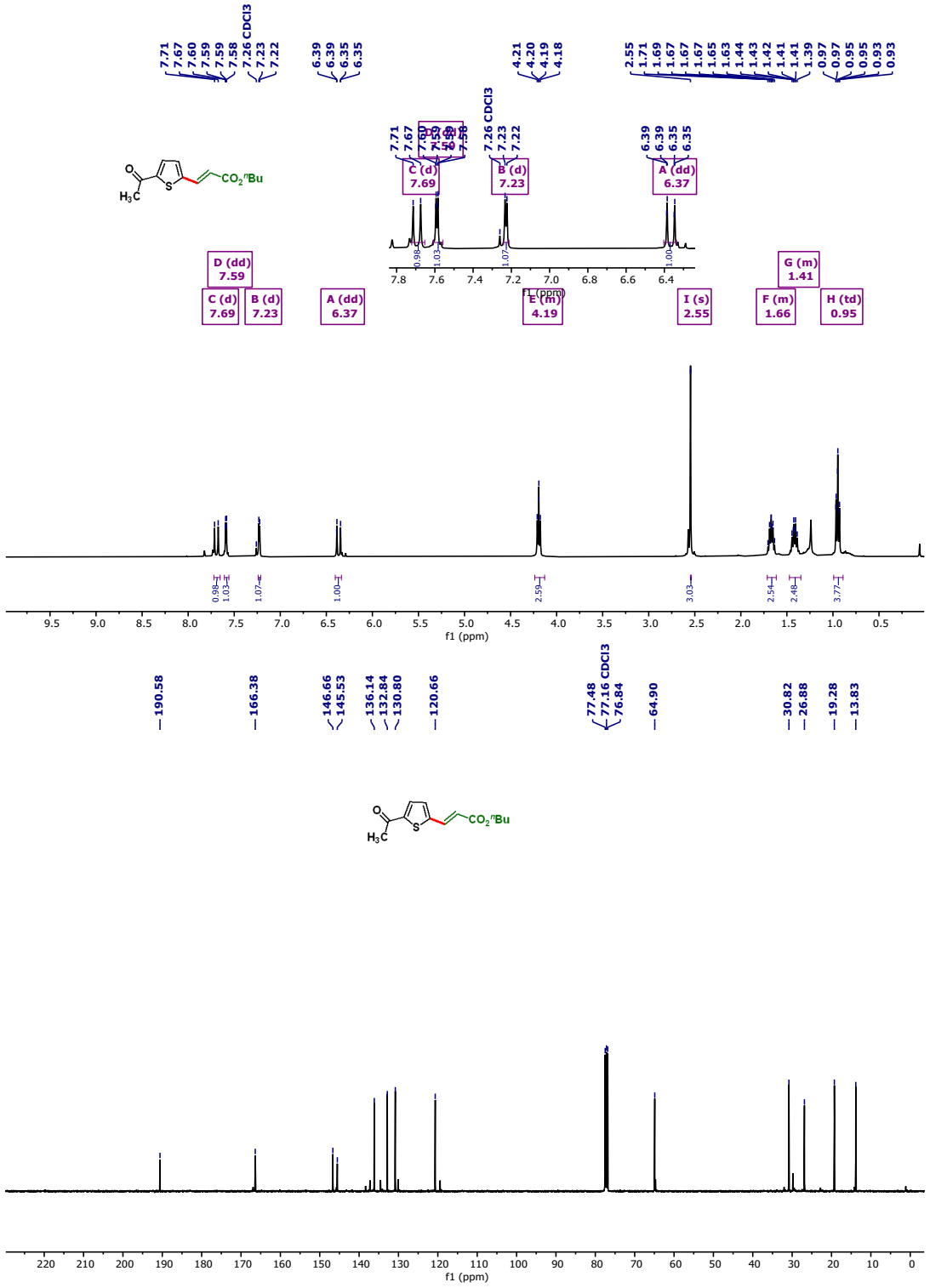

197.421
167.017
145.587
140.465
138.051
136.840
136.558
132.364
129.379
125.989
125.607
117.771
77.548
77.230 CDCl3
76.913
64.754
30.959
26.813
19.389
13.950
MeOC
O
O"Bu
f1 (ppm)

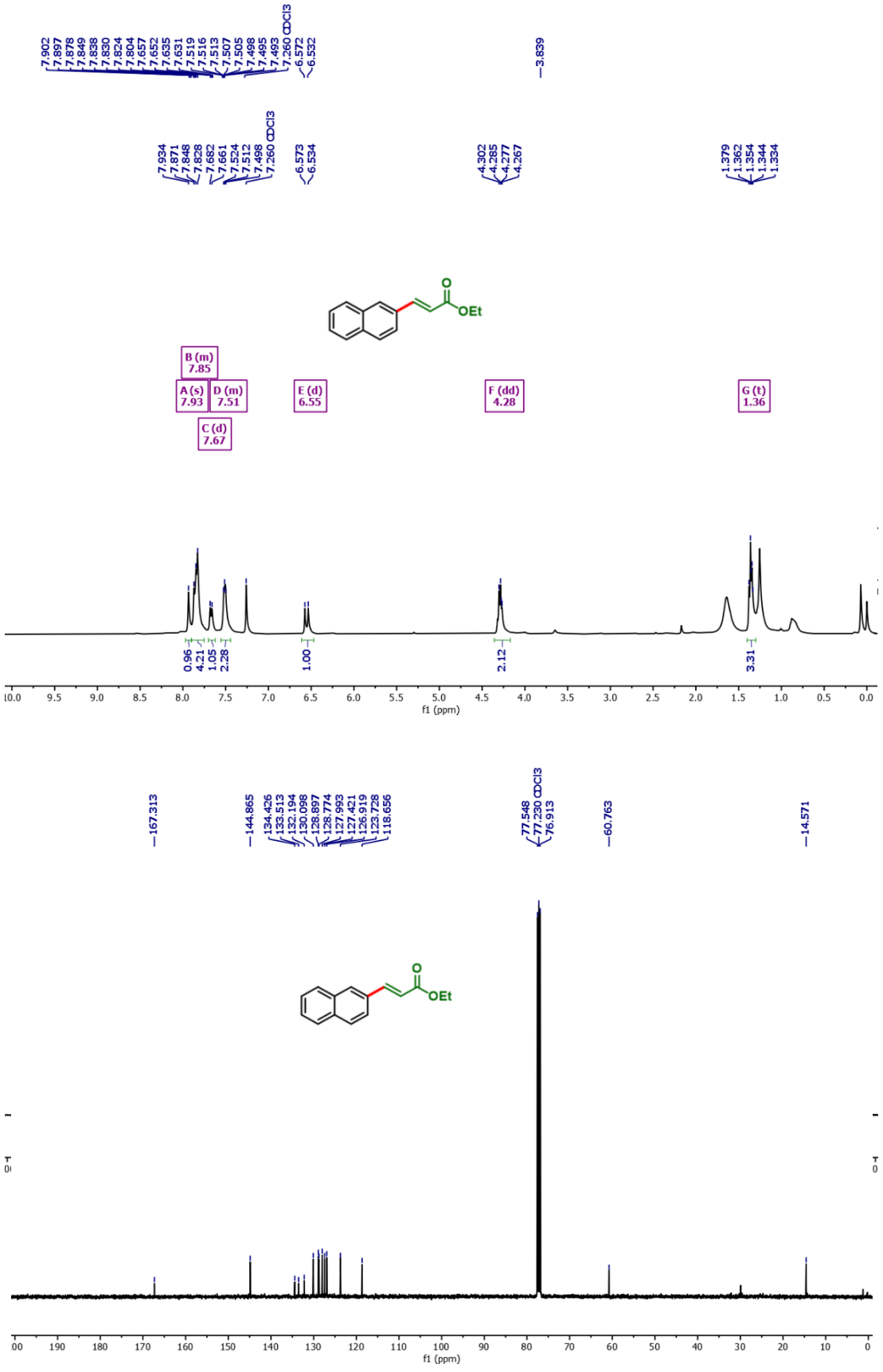

7.801 7.787 7.785 7.746 7.714 7.620 7.608 7.605 7.593 7.590 7.557 7.553 7.541 7.538 7.524 7.521 7.508 7.505 7.490 7.260 CDCl3 7.208 7.201 7.016 6.259 6.228
1.708 1.694 1.680 1.676 1.664 1.651 1.467 1.452 1.437 1.422 1.407 1.393 0.975 0.960 0.945
O2N
S
O
O nBu
D (m) 7.54
B (d) 7.73
G (d) 7.01
A (d) 7.79
F (d) 7.20
H (d) 6.24
C (dd) 7.61
E (td) 7.51
I (t) 4.20
K (h) 1.43
J (m) 1.67
L (t) 0.96
1.00 1.05 1.16 1.04 0.99 0.97 0.93 0.95 1.98 2.21 2.18 3.02
f1 (ppm)

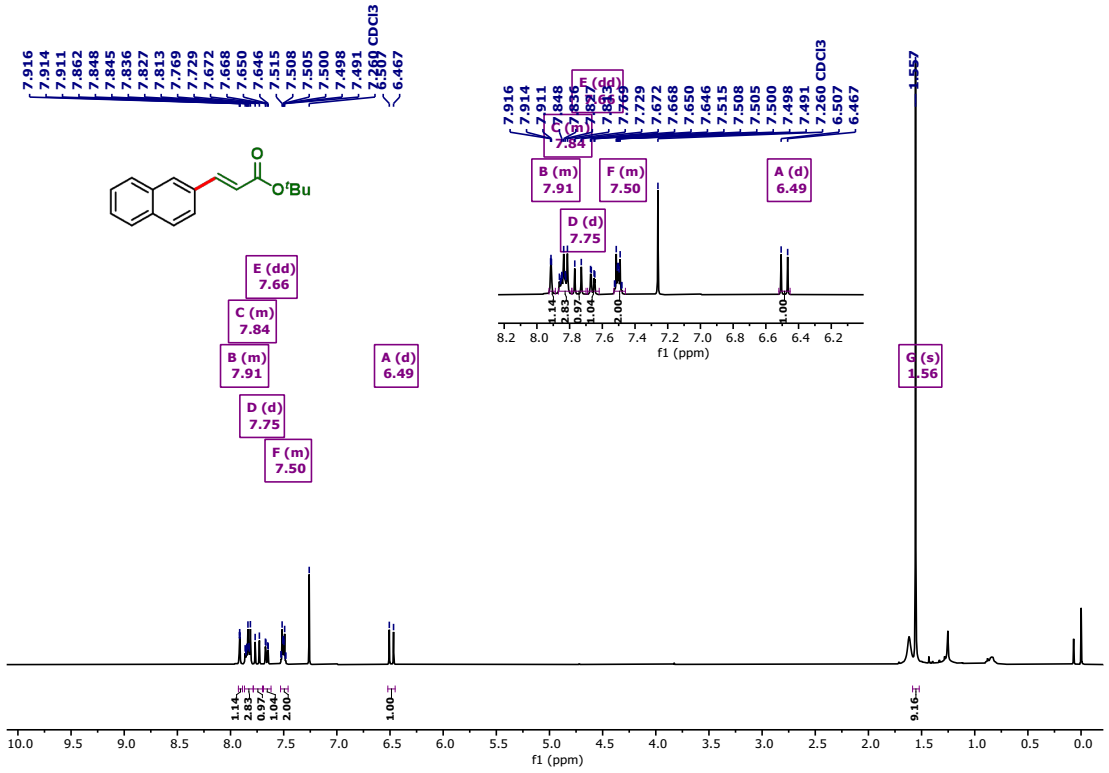

166.931
149.425 141.333 140.036 136.660 132.381 132.285 131.511 129.471 128.287 127.970 124.367 118.077
77.548 77.230 CDCl3 76.913
64.752
30.956
19.387
13.941
O2N
S
O
O nBu
f1 (ppm)

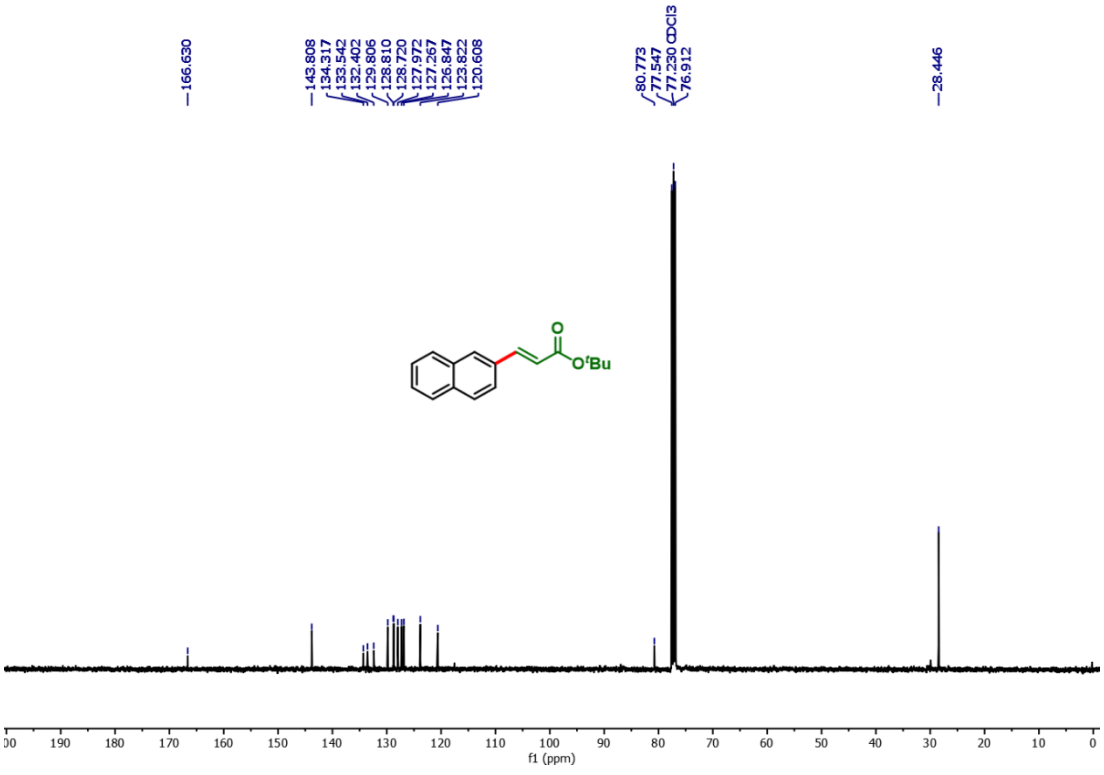

7.815
7.776
7.575
7.571
7.557
7.554
7.551
7.521
7.517
7.470
7.467
7.435
7.432
7.430
7.425
7.412
7.408
7.396
7.392
7.387
7.338
7.320
7.260 CDCl3
6.256
4.224
4.207
4.190
1.724
1.707
1.700
1.692
1.689
1.684
1.670
1.666
1.653
1.647
1.488
1.474
1.469
1.455
1.450
1.447
1.437
1.431
1.418
1.413
1.399
1.394
1.376
0.986
0.967
0.949
Ph
S
CO2nBu
D (d) 7.47
B (m) 7.56
A (d) 7.80
F (m) 7.33
C (d) 7.52
E (m) 7.41
G (d) 6.28
H (t) 4.21
J (m) 1.43
I (m) 1.69
K (t) 0.97
1.00
1.83
0.82
0.91
2.19
0.92
1.01
1.90
2.02
2.26
3.10
f1 (ppm)


167.116
143.440
140.381
137.182
135.203
129.895
129.488
129.146
128.904
127.852
126.487
123.229
117.591
77.548
77.230 CDCl3
76.913
64.717
30.987
19.415
13.965
Ph
S
CO2nBu
f1 (ppm)

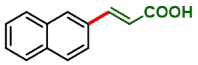

7.71 7.67 7.60 7.59 7.59 7.58 7.26 CDCl3 7.23 7.22
6.39 6.39 6.35 6.35
4.21 4.20 4.19 4.18
2.55 1.71 1.69 1.67 1.67 1.65 1.63 1.44 1.43 1.42 1.41 1.41 1.39 0.97 0.97 0.95 0.95 0.93 0.93
CO2nBu
H3C
O
S
C (d) 7.69
B (d) 7.23
A (dd) 6.37
D (dd) 7.59
G (m) 1.41
E (m) 4.19
I (s) 2.55
F (m) 1.66
H (td) 0.95
f1 (ppm)
190.58 166.38 146.66 145.53 136.14 132.84 130.80 120.66 77.48 77.16 CDCl3 76.84 64.90 30.82 26.88 19.28 13.83

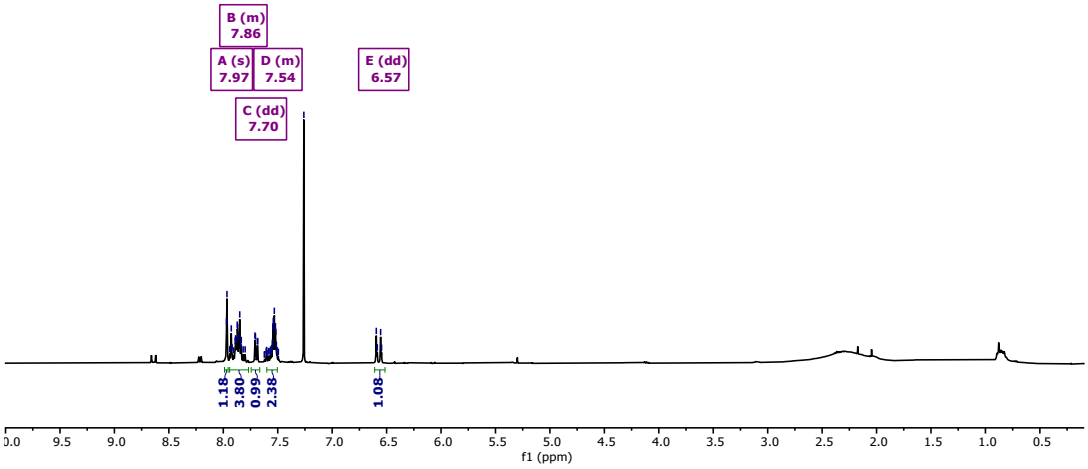

7.902
7.897
7.878
7.849
7.838
7.830
7.824
7.804
7.657
7.652
7.635
7.631
7.519
7.516
7.513
7.507
7.505
7.498
7.495
7.493
7.260 CDCl3
6.572
6.532
3.839
7.934
7.871
7.848
7.828
7.682
7.661
7.524
7.512
7.498
7.260 CDCl3
6.573
6.534
4.302
4.285
4.277
4.267
1.379
1.362
1.354
1.344
1.334
OEt
B (m) 7.85
A (s) 7.93
D (m) 7.51
C (d) 7.67
E (d) 6.55
F (dd) 4.28
G (t) 1.36
0.96
4.21
1.05
2.28
1.00
2.12
3.31
f1 (ppm)
167.313
144.865
134.426
133.513
132.194
130.098
128.897
128.774
127.993
127.421
126.919
123.728
118.656
77.548
77.230 CDCl3
76.913
60.763
14.571
OEt
f1 (ppm)

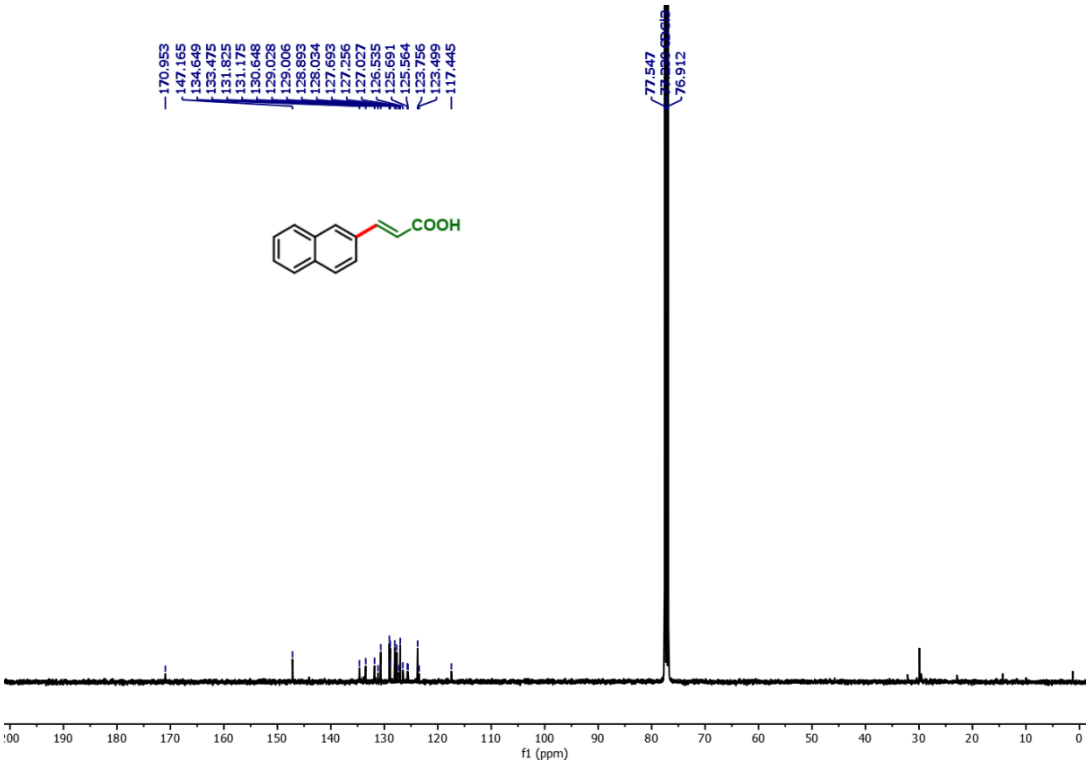

7.916
7.914
7.911
7.862
7.848
7.845
7.836
7.827
7.813
7.769
7.729
7.672
7.668
7.650
7.646
7.515
7.508
7.505
7.500
7.498
7.491
7.260 CDCl3
6.507
6.467
O
O'Bu
E (dd)
7.66
C (m)
7.84
B (m)
7.91
D (d)
7.75
F (m)
7.50
A (d)
6.49
G (s)
1.56
1.14
2.83
0.97
1.04
2.00
1.00
9.16
f1 (ppm)

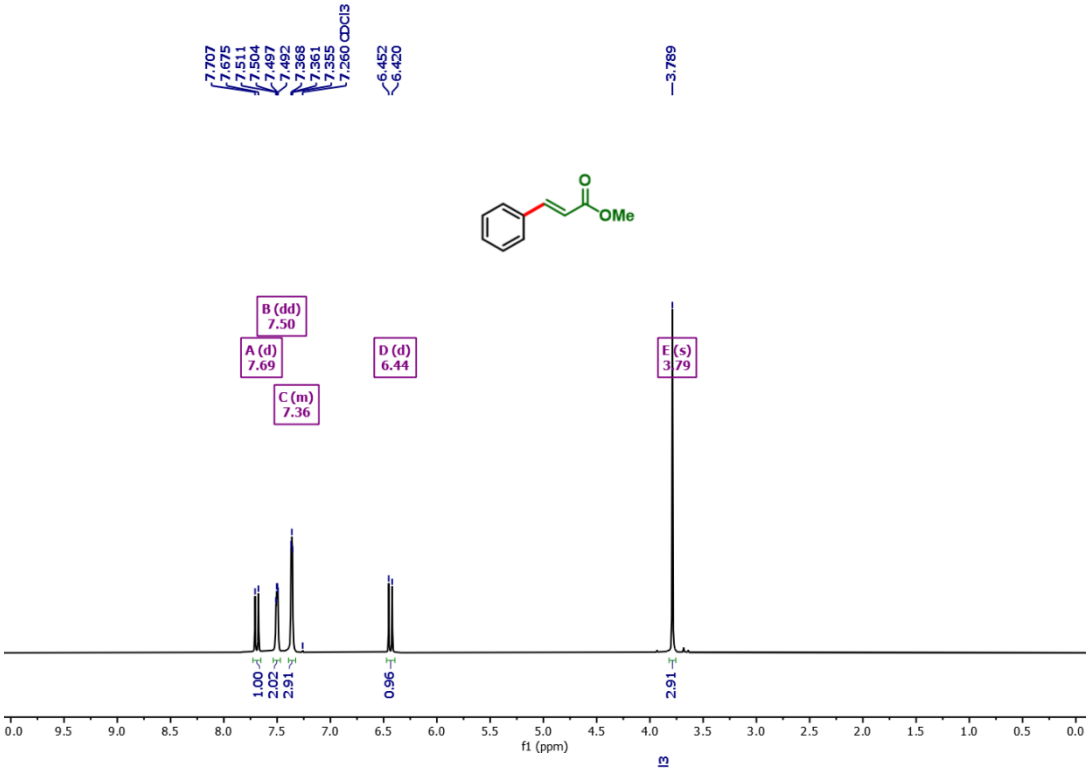

166.630
143.808
134.317
133.542
132.402
129.806
128.810
128.730
127.972
127.267
126.847
123.822
120.608
80.773
77.547
77.230 CDCl3
76.912
28.446
O
O'Bu
f1 (ppm)

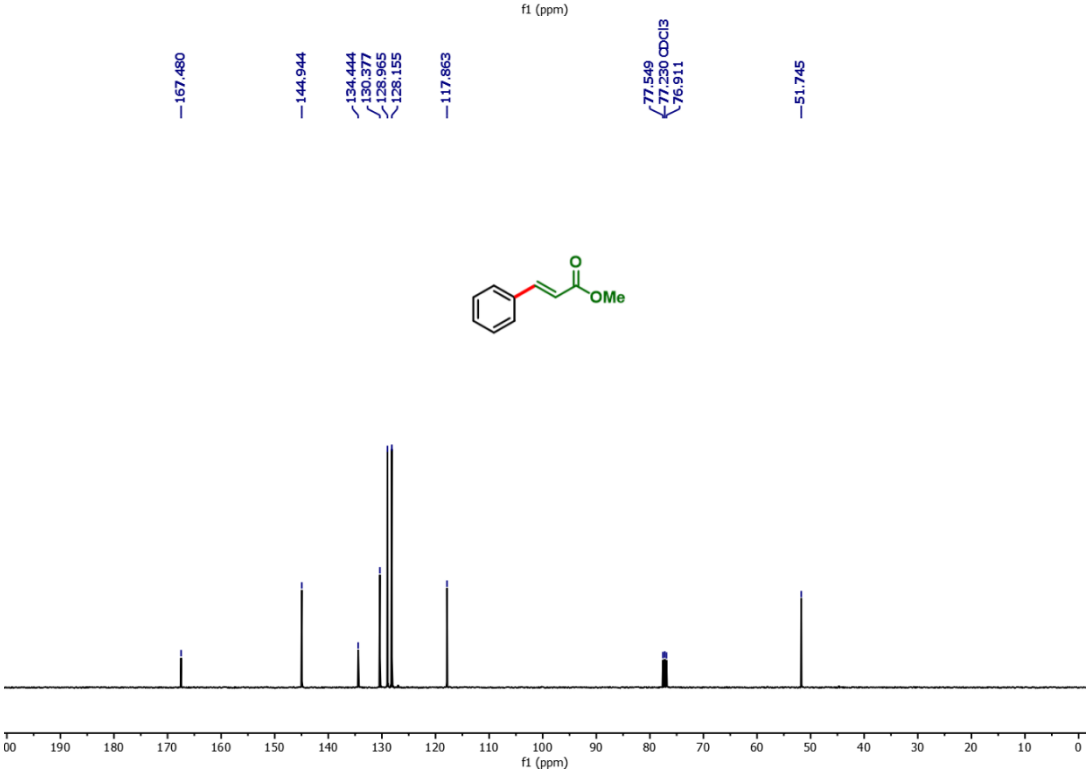

7.970
7.966
7.940
7.927
7.919
7.905
7.891
7.887
7.877
7.872
7.868
7.858
7.849
7.840
7.835
7.817
7.799
7.709
7.705
7.688
7.683
7.623
7.619
7.606
7.602
7.598
7.585
7.581
7.569
7.565
7.562
7.557
7.552
7.548
7.545
7.541
7.531
7.521
7.517
7.512
7.505
7.500
7.494
7.260 CDCl3
6.595
6.586
6.555
6.546

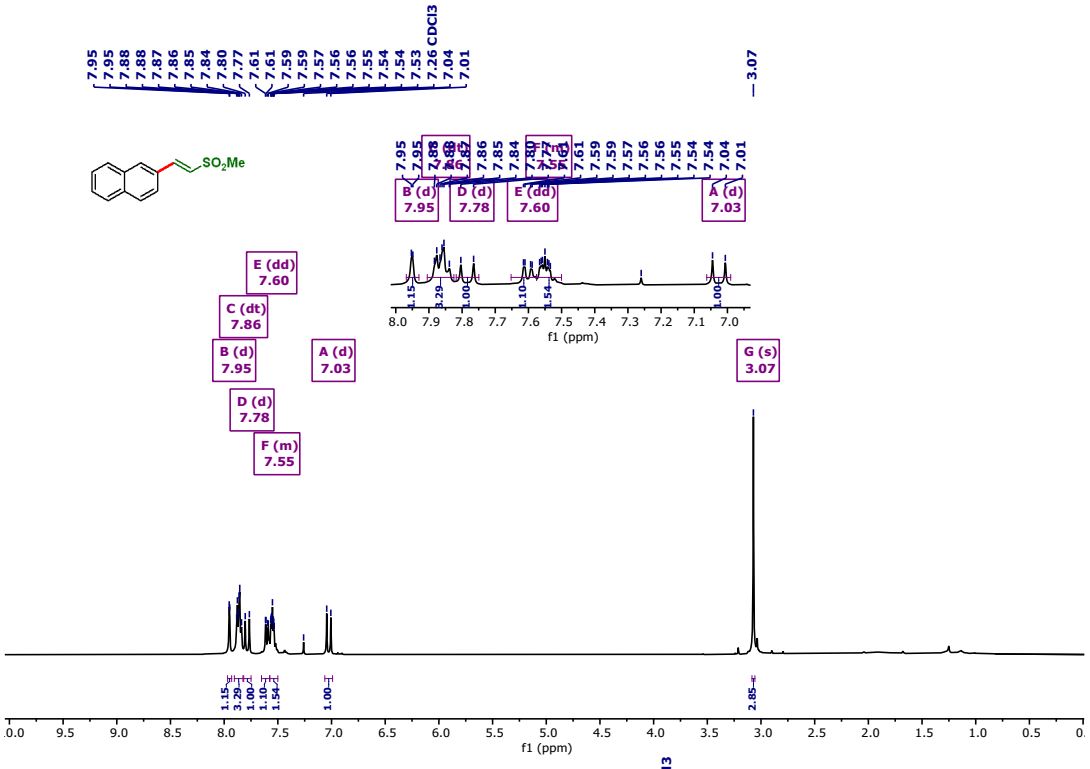

COOH

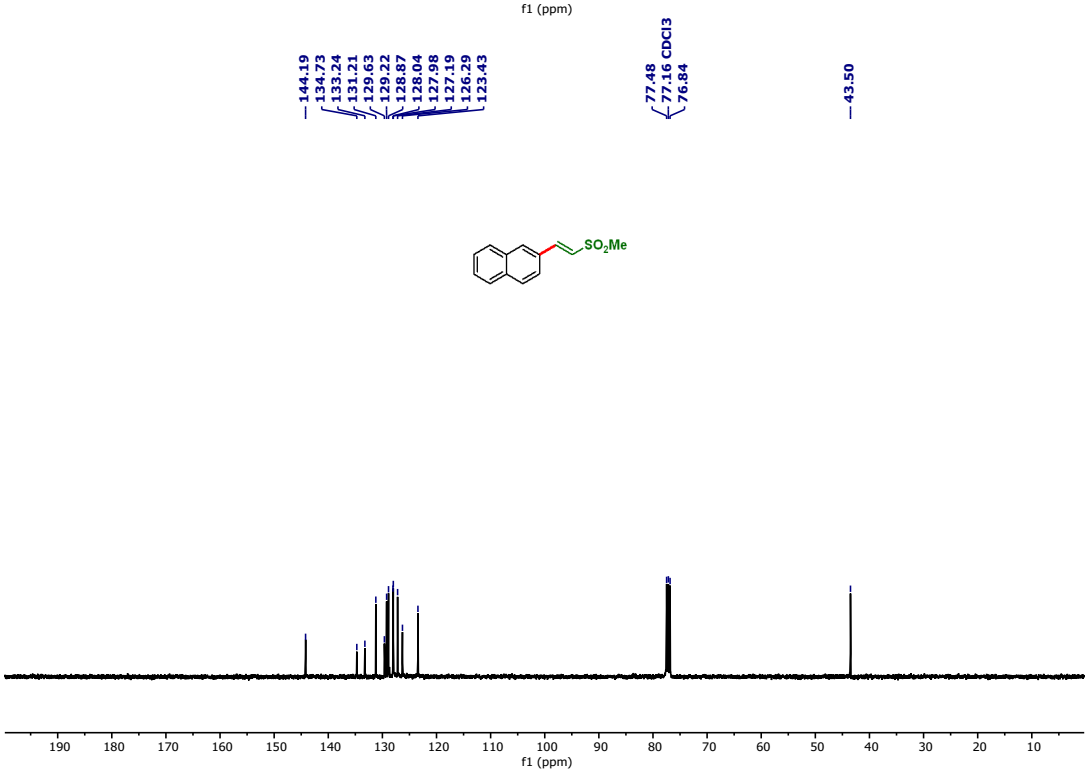

B (m)
7.86
A (s)
7.97
D (m)
7.54
E (dd)
6.57
C (dd)
7.70
1.18
3.80
0.99
2.38
1.08
f1 (ppm)

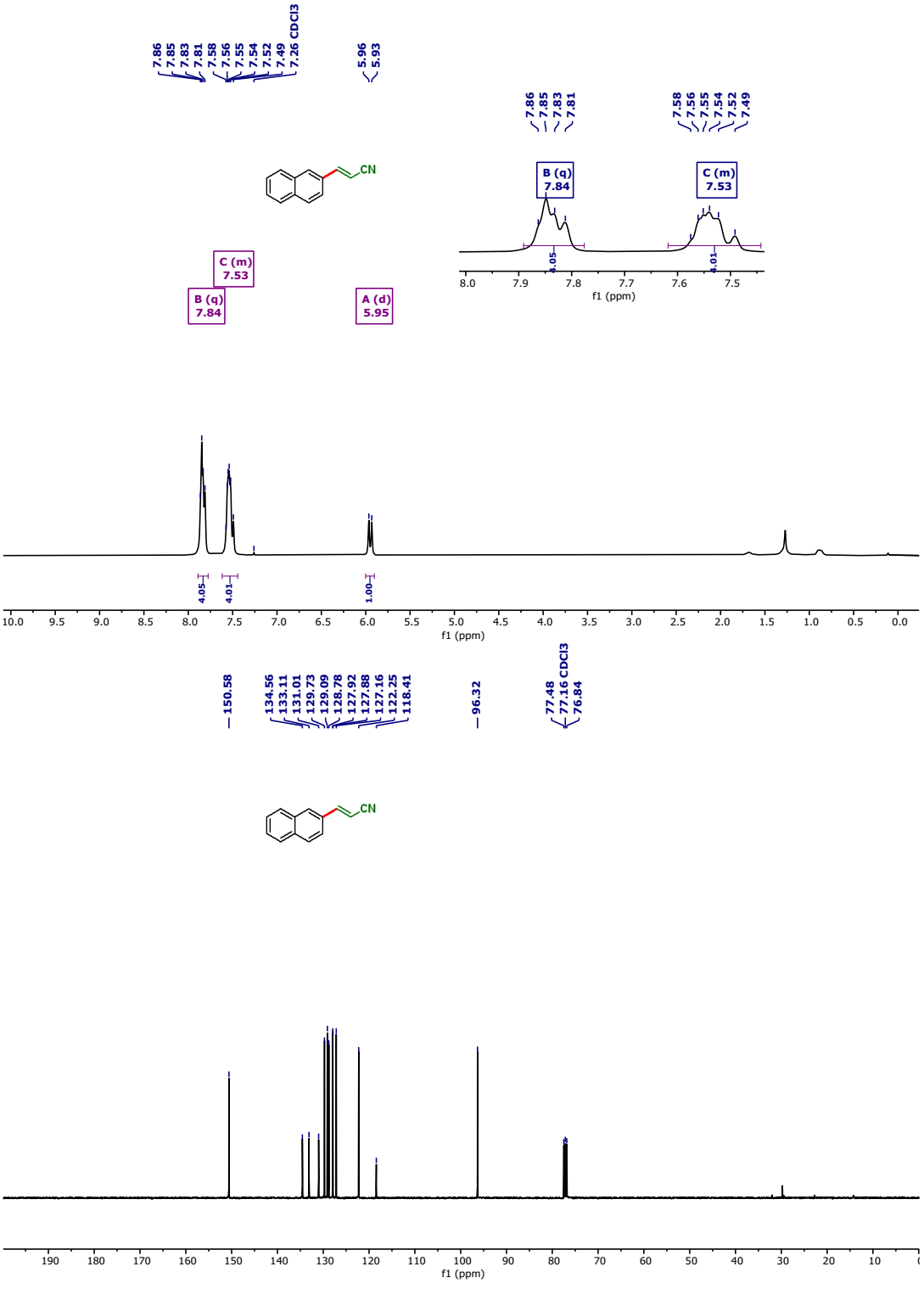

170.953
147.165
134.649
133.475
131.825
131.175
130.648
129.028
129.006
128.893
128.034
127.693
127.256
127.027
126.535
125.691
125.564
123.756
123.499
117.445
77.547
76.912
COOH
f1 (ppm)

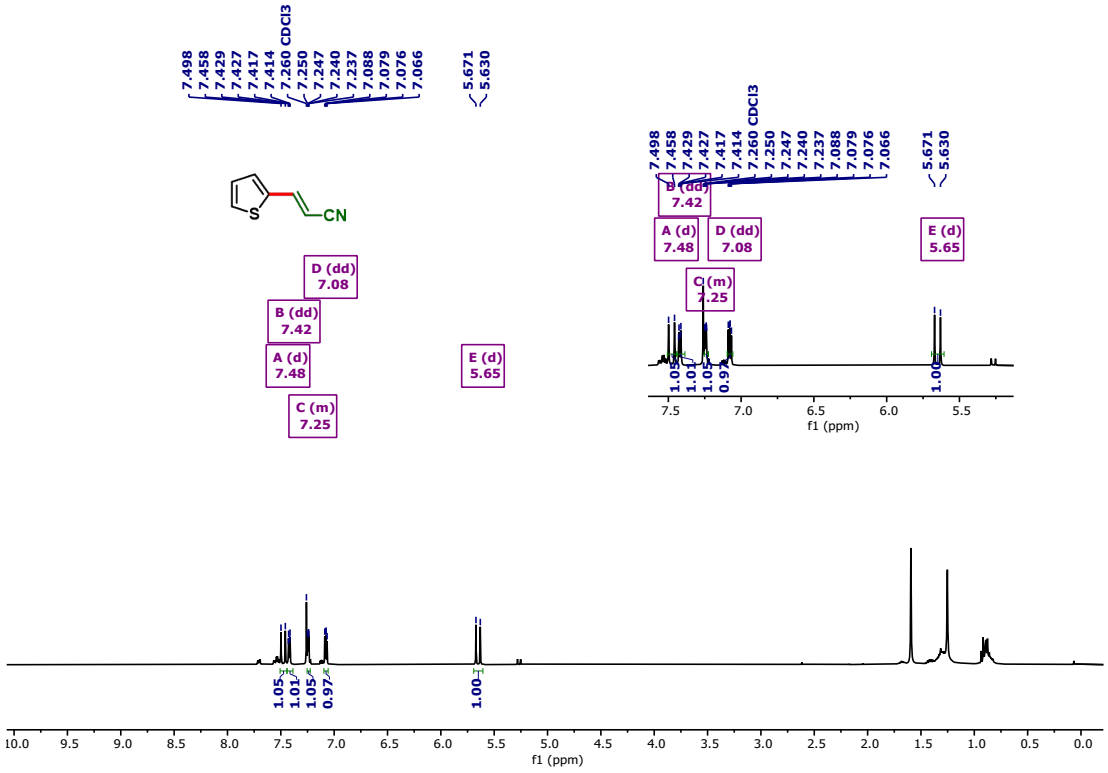

7.707
7.675
7.511
7.504
7.497
7.492
7.368
7.361
7.355
7.260 CDCl3
6.452
6.420
3.789
OMe
B (dd)
7.50
A (d)
7.69
C (m)
7.36
D (d)
6.44
E (s)
3.79
1.00
2.02
2.91
0.96
2.91
f1 (ppm)

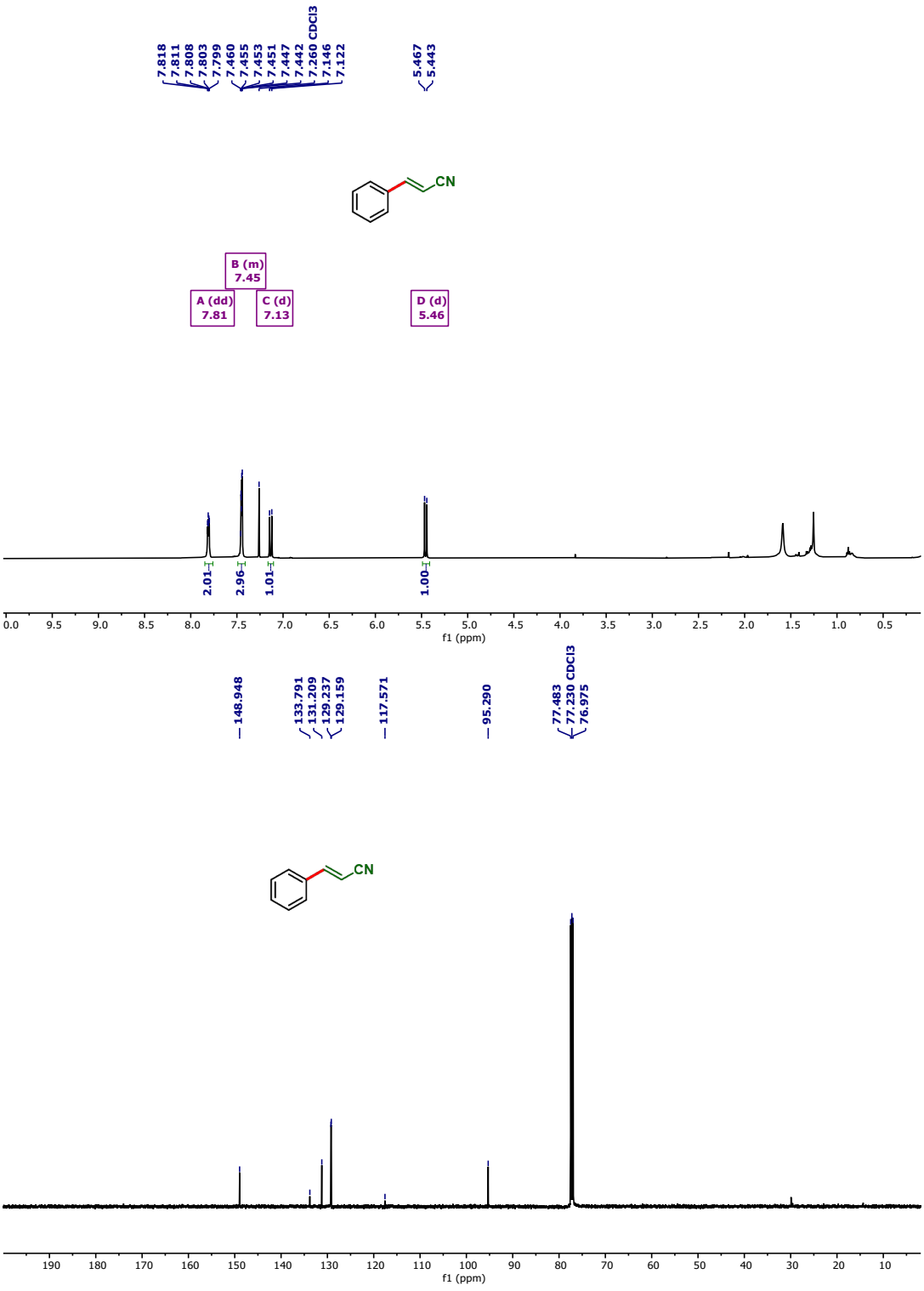

167.480
144.944
134.444
130.377
128.965
128.155
117.863
77.549
77.230 CDCl3
76.911
51.745
OMe
f1 (ppm)


7.95
7.95
7.88
7.88
7.87
7.86
7.85
7.84
7.80
7.77
7.61
7.61
7.59
7.59
7.57
7.56
7.56
7.55
7.54
7.54
7.53
7.26 CDCl3
7.04
7.01
3.07
SO2Me
B (d) 7.95
D (d) 7.78
E (dd) 7.60
A (d) 7.03
E (dd) 7.60
C (dt) 7.86
B (d) 7.95
A (d) 7.03
G (s) 3.07
D (d) 7.78
F (m) 7.55
1.15
3.29
1.00
1.10
1.54
1.00
2.85
f1 (ppm)


144.19
134.73
133.24
131.21
129.63
129.22
128.87
128.04
127.98
127.19
126.29
123.43
77.48
77.16 CDCl3
76.84
43.50
SO2Me
f1 (ppm)


7.86
7.85
7.83
7.81
7.58
7.56
7.55
7.54
7.52
7.49
7.26 CDCl3
5.96
5.93
CN
C (m)
7.53
B (q)
7.84
A (d)
5.95
4.05
4.01
1.00
f1 (ppm)
150.58
134.56
133.11
131.01
129.73
129.09
128.78
127.92
127.88
127.16
122.25
118.41
96.32
77.48
77.16 CDCl3
76.84


7.498
7.458
7.429
7.427
7.417
7.414
7.260 CDCl3
7.250
7.247
7.240
7.237
7.088
7.079
7.076
7.066
5.671
5.630
S
CN
D (dd)
7.08
B (dd)
7.42
A (d)
7.48
C (m)
7.25
E (d)
5.65
1.05
1.01
1.05
0.97
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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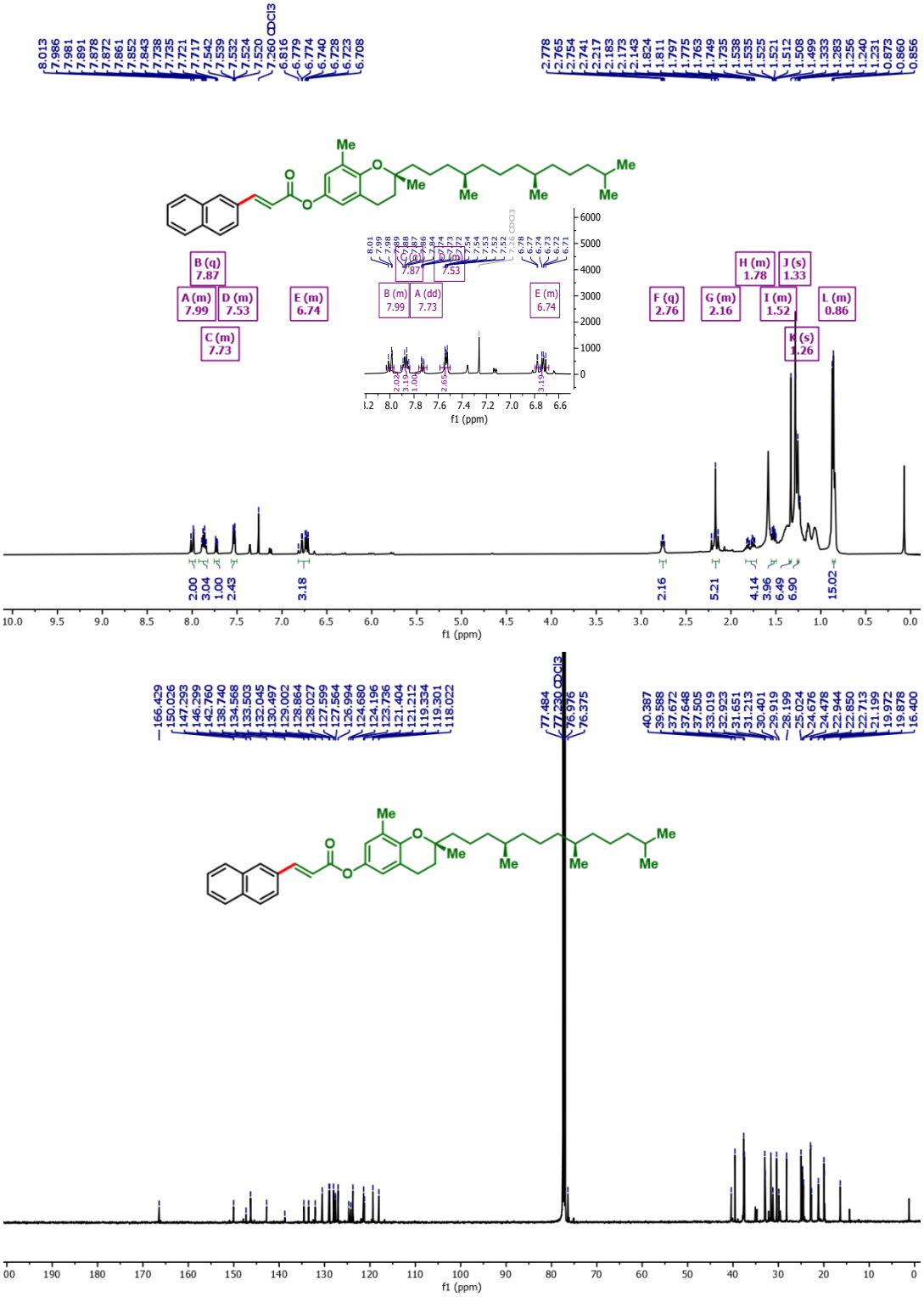
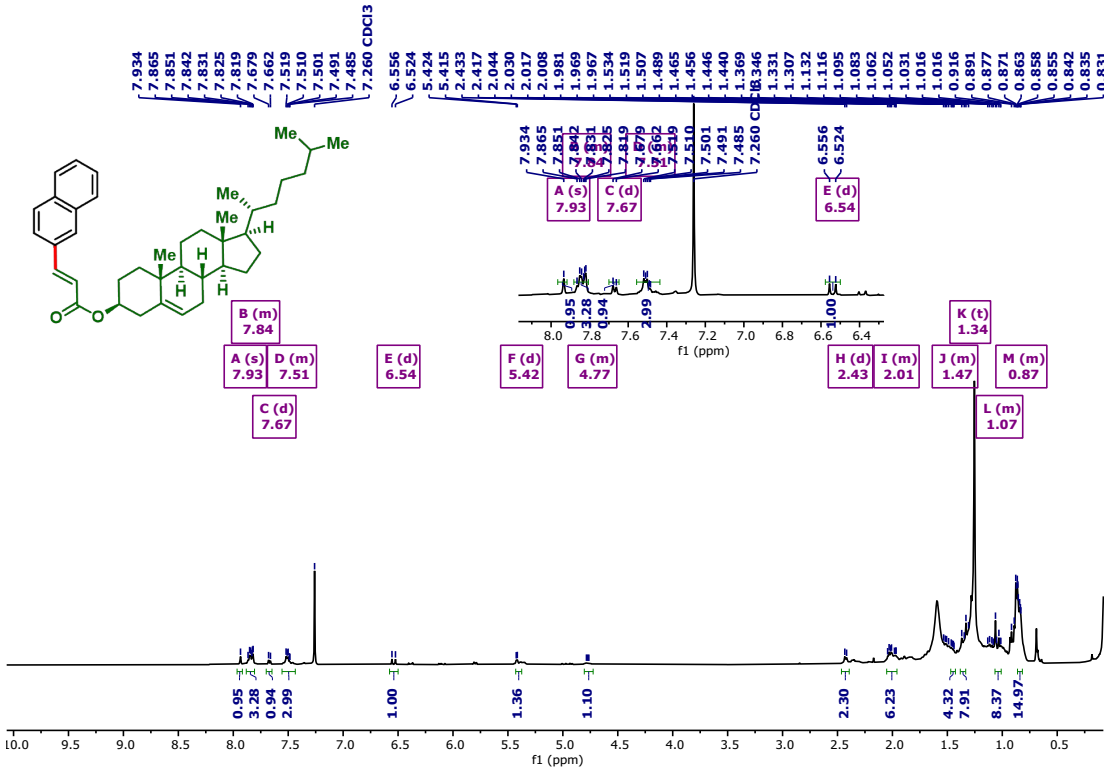
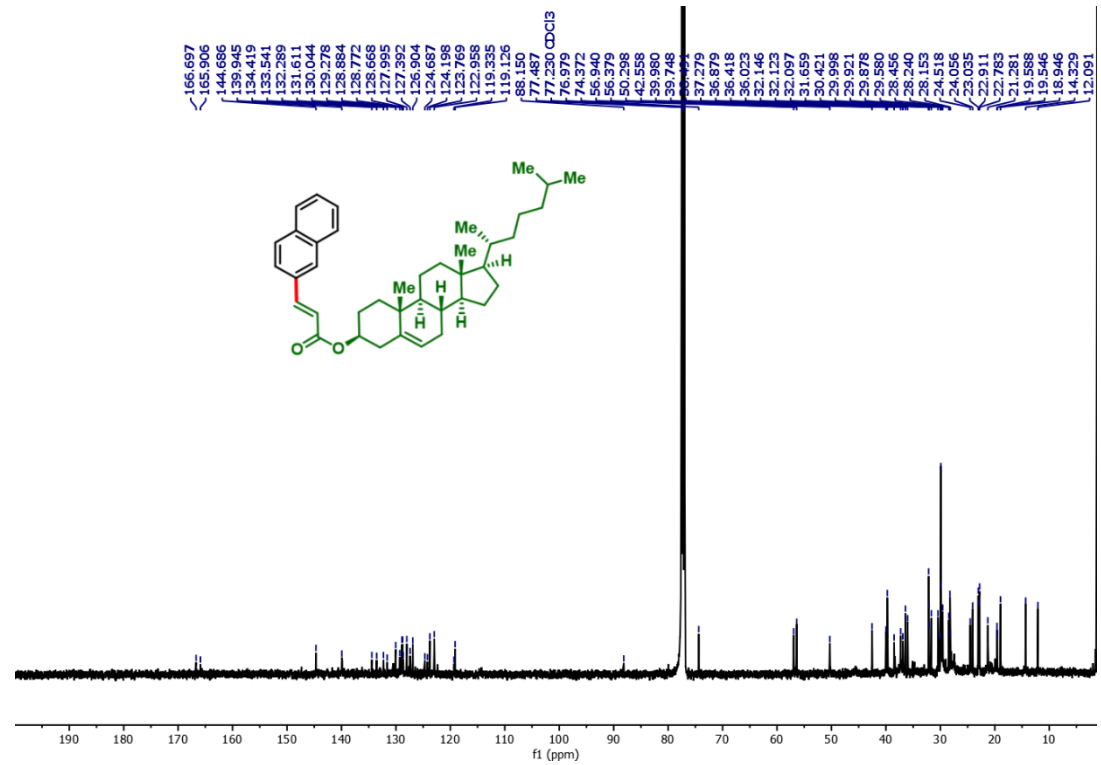
7.811
7.808
7.803
7.799
7.460
7.455
7.453
7.451
7.447
7.442
7.260 CDCl3
7.146
7.122
5.467
5.443
CN
B (m)
7.45
A (dd)
7.81
C (d)
7.13
D (d)
5.46
2.01
2.96
1.01
1.00
f1 (ppm)
148.948
133.791
131.209
129.237
129.159
117.571
95.290
77.483
77.230 CDCl3
76.975
CN
f1 (ppm)


8.013
7.986
7.981
7.891
7.878
7.872
7.861
7.852
7.843
7.738
7.735
7.721
7.717
7.542
7.539
7.532
7.524
7.520
7.260 CDCl3
6.816
6.779
6.774
6.740
6.728
6.723
6.708
2.778
2.765
2.754
2.741
2.217
2.183
2.173
2.143
1.824
1.811
1.797
1.775
1.763
1.749
1.735
1.538
1.535
1.525
1.521
1.512
1.508
1.499
1.333
1.283
1.256
1.240
1.231
0.873
0.860
0.856
Me
O
Me
Me
Me
Me
O
B (q) 7.87
A (m) 7.99
D (m) 7.53
C (m) 7.73
E (m) 6.74
F (q) 2.76
G (m) 2.16
H (m) 1.78
I (m) 1.52
J (s) 1.33
K (s) 1.26
L (m) 0.86
2.00
3.04
1.00
2.43
3.18
2.16
5.21
4.14
3.96
6.49
6.90
15.02
f1 (ppm)
166.429
150.026
147.293
146.299
142.760
138.740
134.568
133.503
132.045
130.497
129.002
128.864
128.027
127.599
127.564
126.994
124.680
124.196
123.736
121.404
121.212
119.334
119.301
118.022
77.484
77.230 CDCl3
76.976
76.375
40.387
39.588
37.672
37.648
37.505
33.019
32.923
31.651
31.213
30.401
29.919
28.199
25.024
24.676
24.478
22.944
22.850
22.713
21.199
19.972
19.878
16.400
f1 (ppm)


A (s) 7.93
B (m) 7.84
C (d) 7.67
D (m) 7.51
E (d) 6.54
F (d) 5.42
G (m) 4.77
H (d) 2.43
I (m) 2.01
J (m) 1.47
K (t) 1.34
L (m) 1.07
M (m) 0.87
f1 (ppm)


f1 (ppm)