Supporting Information

Regioselective C–H Alkenylation and Unsymmetrical Bis-Olefination of Heteroarene Carboxylic Acids with Ruthenium Catalysis in Water

Anup Mandal, Ratnadeep Bera, and Mahiuddin Baidya*

Department of Chemistry, Indian Institute of Technology Madras,

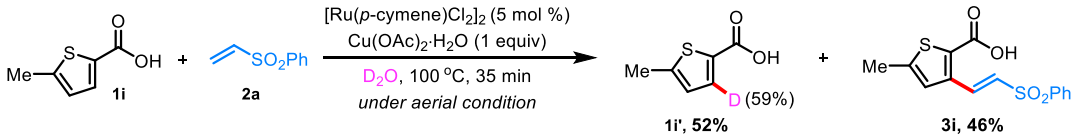
Chennai–600036, Tamil Nadu, India

E-mail: mbaidya@iitm.ac.in

Table of Contents

Mechanistic Studies:

1) H-D Exchange Study



5-Methyl-2-thiophenecarboxylic acid **1i** (0.2 mmol), phenyl vinyl sulfone **2a** (1.1 equiv), [Ru(*p*-cymene)Cl_2]$_2$ (5 mol %), and Cu(OAc)$_2$·H_2O (1 equiv) were added in an oven-dried screw cap reaction tube with a magnetic stir bar under open-air. Then, D_2O (1 mL) was added with a syringe. The reaction tube was capped and the resulting mixture was heated at 100 ºC (in oil-bath) for 35 minutes. After that, it was allowed to cool at room temperature and then quenched with AcOH and diluted with NH_4Cl solution. The mixture was extracted with ethyl acetate (10 mL, two times). The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting deuterium incorporated 5-methyl-2-thiophenecarboxylic acid and the olefinated product were purified by silica gel column chromatography. The H/D exchange result was determined by ^{1}H NMR spectroscopy.

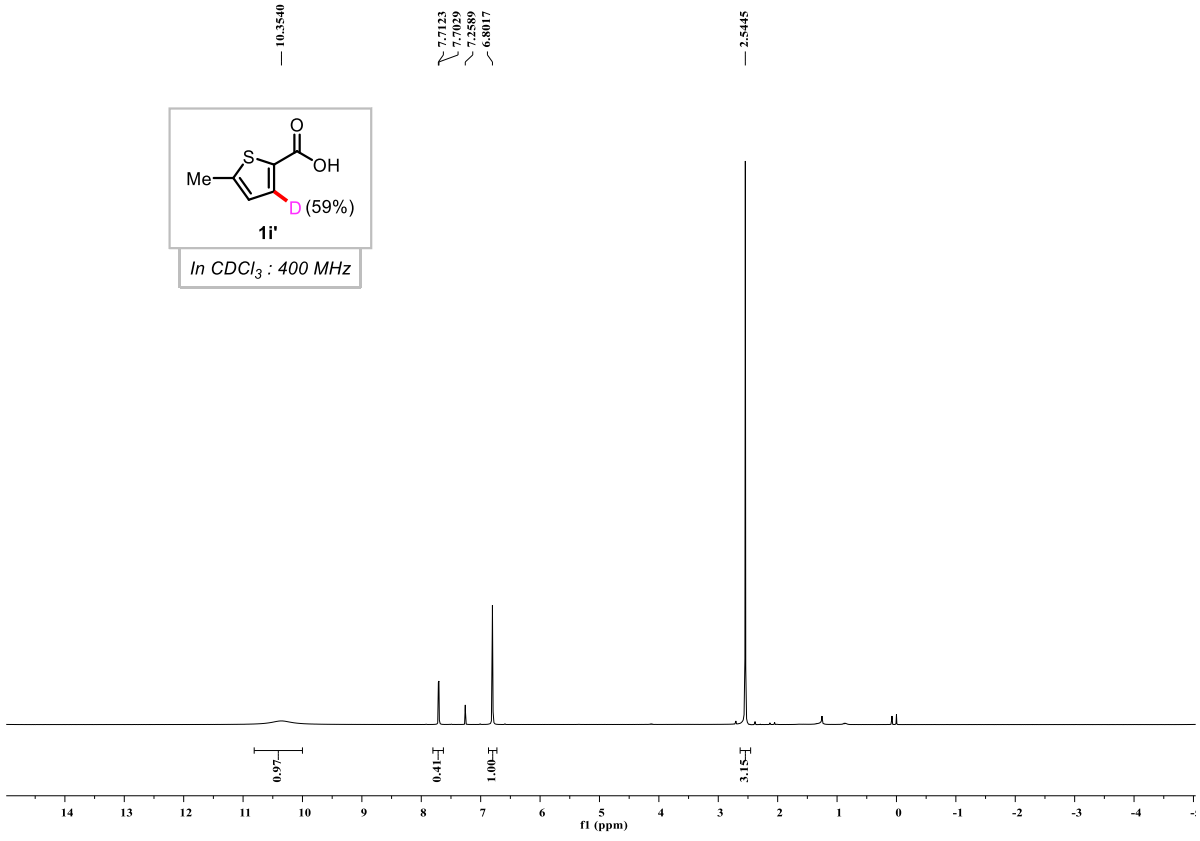
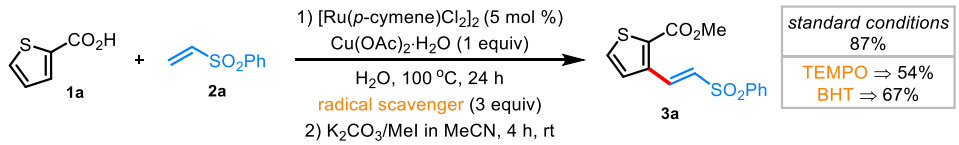


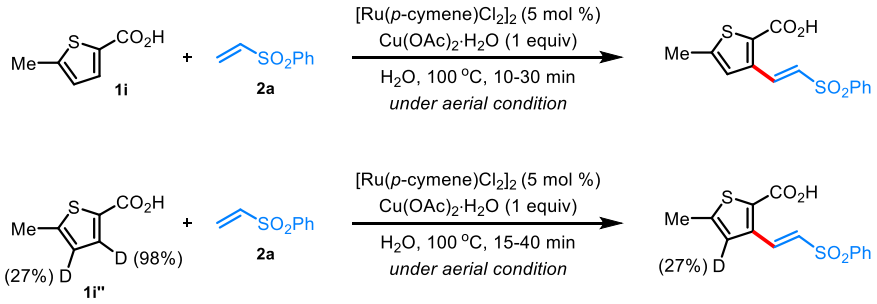
Figure S1. ^{1}H NMR spectrum of deuterium-incorporated 5-methyl-2-thiophenecarboxylic acid **1i′**.

2) Radical Scavenger Experiment



2-Thiophenecarboxylic acid **1a** (0.2 mmol) and phenyl vinyl sulfone **2a** (1.1 equiv), [Ru(*p*-cymene)Cl_2]$_2$ (5 mol %), Cu(OAc)$_2$·H_2O (1 equiv), and corresponding radical scavenger (3 equiv) were added in an oven-dried screw cap reaction tube with a magnetic stir bar under open-air. Then, H_2O (1 mL) was added with a syringe. The reaction tube was capped and the resulting mixture was heated at 100 ºC (in oil-bath) for 24 h. After that, it was allowed to cool at room temperature and then quenched with AcOH and diluted with NH_4Cl solution. The mixture was extracted with ethyl acetate (10 mL, two times). The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. The crude product was treated with K_2CO_3 (2 equiv), MeI (3 equiv) in MeCN (1 mL) at room temperature for 4 h and then concentrated under reduced pressure. The resulting residue was purified by silica gel column chromatography.

3) Kinetic Isotope Effect Study via Parallel Experiment



***KIE* = k_H/k_D = 1.68**

5-Methyl-2-thiophenecarboxylic acid **1i** and deuterated-5-methyl-2-thiophenecarboxylic acid **1i″** were independently reacted with **2a** for five different time intervals (10-40 minutes) under the standard reaction conditions [**1i** (0.2 mmol) or **1i″** (0.2 mmol), phenyl vinyl sulfone **2a** (1.1 equiv), [Ru(*p*-cymene)Cl_2]$_2$ (5 mol %), and Cu(OAc)$_2$·H_2O (1 equiv), H_2O (1 mL)]. The product distributions were analyzed from the worked-up crude reaction mixture by ^{1}H NMR spectroscopy using dibromomethane as an internal standard.

time (min)	10 min	15 min	20 min	25 min	30 min	35 min	40 min
1i → Product (%)	27.1	33.2	36.3	41.2	43.2	-	-
1i″ → Product (%)	-	17.4	20.9	-	24.2	26.7	30.3

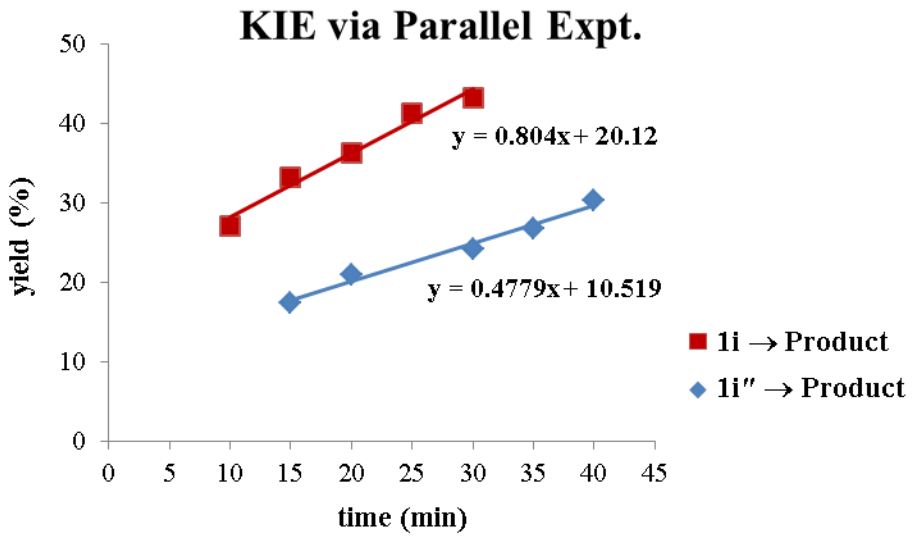


Figure S2. Time vs yield plot for KIE determination.

Crystallographic Experimental Section:

Single Crystal X-ray Crystallography: X-ray data of the crystals were collected and integrated using a Bruker Axs (Kappa Apex 2) CCD diffractometer equipped with graphite monochromatic Mo (Kα) radiation. Crystals were mounted over fine nylon loop which was attached to the copper mounting pin held on by a magnetic base. The APEX 3 and APEX 3-SAINT/Bruker SAINT programs were used for the data collection and unit-cell determination, respectively. The crystal structures were solved by direct methods using SHELXL-2014/4 or SHELXS-97 and refined by full-matrix least-squares on F^2 method using program SHELXL-2014/7 or SHELXL-2018/3.

Method of Crystallization: All the single crystals were grown in a small glass vial by slow evaporation technique from DCM/Hexane solvent system at room temperature over a period of 1-2 weeks.

Crystal Structure of Compound 3c: ORTEP representation (40% ellipsoids probability) of **3c** (CCDC 1975675).

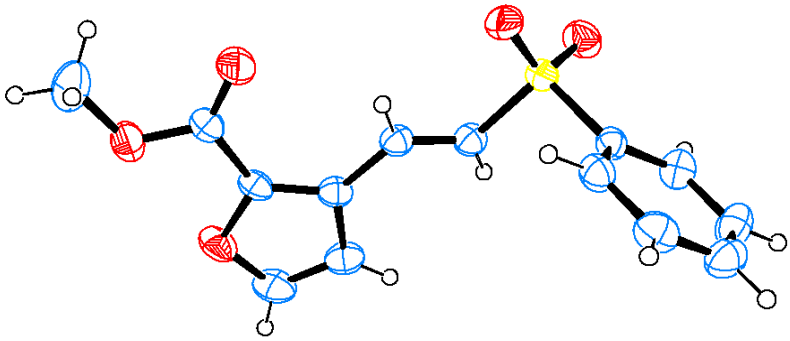


Table S1. Crystal data and structure refinement for **3c** (**CCDC** 1975675).

Identification code	532
Empirical formula	C14 H12 O5 S
Formula weight	292.30
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, Pbca
Unit cell dimensions	a = 7.6046(2) Å α = 90° b = 15.5125(5) Å β = 90° c = 23.2195(7) Å γ = 90°
Volume	2739.12(14) A^3
Z, Calculated density	8, 1.418 Mg/m^3
Absorption coefficient	0.252 mm^-1
F(000)	1216
Crystal size	0.250 x 0.220 x 0.100 mm
Theta range for data collection	2.769 to 24.999 deg.
Limiting indices	-7<=h<=9, -18<=k<=15, -27<=l<=27
Reflections collected / unique	15544 / 2406 [R(int) = 0.0244]
Completeness to theta = 25.000	99.9 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2406 / 0 / 182
Goodness-of-fit on F^2	1.054

Final R indices [I>2sigma(I)]	R1 = 0.0352, wR2 = 0.0837
R indices (all data)	R1 = 0.0445, wR2 = 0.0916
Extinction coefficient	n/a
Largest diff. peak and hole	0.184 and -0.305 e.A^-3

Crystal Structure of Compound 3f: ORTEP representation (40% ellipsoids probability) of **3f** (CCDC 1975672).

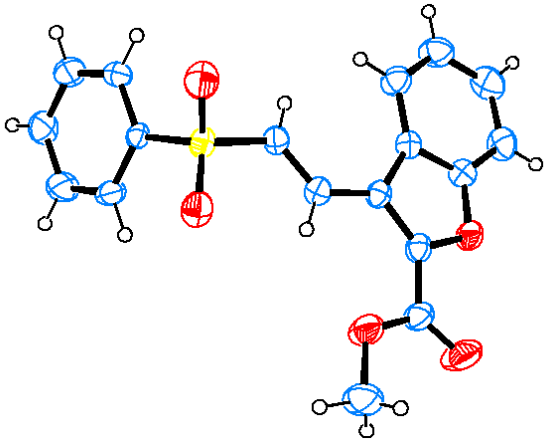


Table S2. Crystal data and structure refinement for **3f** (**CCDC** 1975672).

Identification code	787
Empirical formula	C18 H14 O5 S
Formula weight	342.35
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, Pbca
Unit cell dimensions	a = 17.7265(7) Å α = 90° b = 11.8090(4) Å β = 90° c = 15.2926(4) Å γ = 90°
Volume	3201.23(19) A^3
Z, Calculated density	8, 1.421 Mg/m^3
Absorption coefficient	0.227 mm^-1
F(000)	1424
Crystal size	0.250 x 0.220 x 0.100 mm
Theta range for data collection	2.072 to 24.998 deg.

Limiting indices	-13<=h<=21, -13<=k<=14, -18<=l<=12
Reflections collected / unique	10818 / 2815 [R(int) = 0.0224]
Completeness to theta = 25.000	99.9 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2815 / 0 / 218
Goodness-of-fit on F^2	1.053
Final R indices [I>2sigma(I)]	R1 = 0.0403, wR2 = 0.0959
R indices (all data)	R1 = 0.0531, wR2 = 0.1040
Extinction coefficient	n/a
Largest diff. peak and hole	0.277 and -0.301 e.A^-3

Crystal Structure of Compound 3h: ORTEP representation (40% ellipsoids probability) of **3h** (CCDC 1975673).

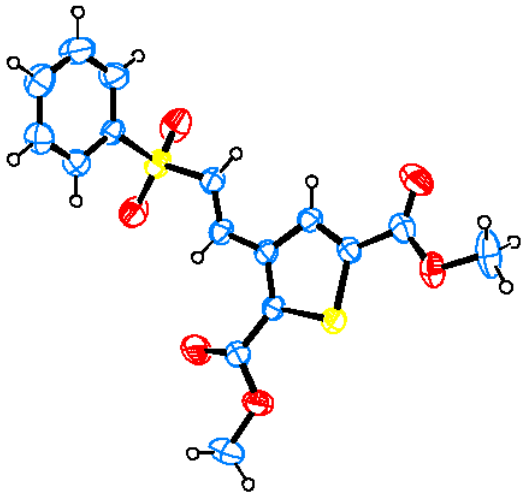


Table S3. Crystal data and structure refinement for **3h** (**CCDC** 1975673).

Identification code	28
Empirical formula	C16 H14 O6 S2
Formula weight	366.39
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, Pbca
Unit cell dimensions	a = 8.0160(2) Å α = 90°

	b = 19.6309(8) Å β = 90° c = 21.6558(8) Å γ = 90°
Volume	3407.8(2) A^3
Z, Calculated density	8, 1.428 Mg/m^3
Absorption coefficient	0.341 mm^-1
F(000)	1520
Crystal size	0.250 x 0.220 x 0.100 mm
Theta range for data collection	1.881 to 24.996 deg.
Limiting indices	-9<=h<=9, -21<=k<=23, -25<=l<=19
Reflections collected / unique	7967 / 2995 [R(int) = 0.0402]
Completeness to theta = 25.000	99.9 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2995 / 0 / 219
Goodness-of-fit on F^2	1.009
Final R indices [I>2sigma(I)]	R1 = 0.0486, wR2 = 0.0978
R indices (all data)	R1 = 0.0957, wR2 = 0.1207
Extinction coefficient	n/a
Largest diff. peak and hole	0.243 and -0.300 e.A^-3

Crystal Structure of Compound 4g: ORTEP representation (40% ellipsoids probability) of **4g** (CCDC 1975676).

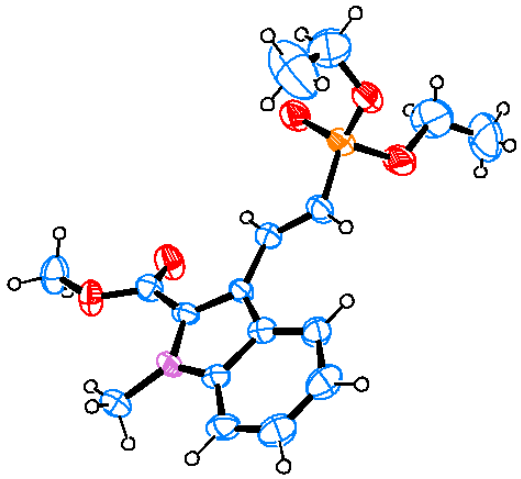


Table S4. Crystal data and structure refinement for **4g** (**CCDC** 1975676).

Identification code	891
Empirical formula	C17 H22 N O5 P
Formula weight	351.32
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 7.4902(15) Å α = 88.249(6)° b = 10.012(2) Å β = 82.343(6)° c = 25.476(5) Å γ = 71.214(6)°
Volume	1792.4(6) Å^3
Z, Calculated density	4, 1.302 Mg/m^3
Absorption coefficient	0.179 mm^-1
F(000)	744
Crystal size	0.200 x 0.150 x 0.150 mm^3
Theta range for data collection	0.807 to 24.997 deg.
Limiting indices	-8<=h<=8, -11<=k<=11, -30<=l<=30
Reflections collected / unique	32648 / 32648 [R(int) = ?]
Completeness to theta = 25.000	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.5243
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	32648 / 39 / 454
Goodness-of-fit on F^2	1.107
Final R indices [I>2sigma(I)]	R1 = 0.1044, wR2 = 0.2823
R indices (all data)	R1 = 0.1436, wR2 = 0.3062
Extinction coefficient	0.017(3)
Largest diff. peak and hole	0.470 and -0.495 e.Å^-3

Crystal Structure of Compound 5g: ORTEP representation (40% ellipsoids probability) of **5g** (CCDC 1975674).

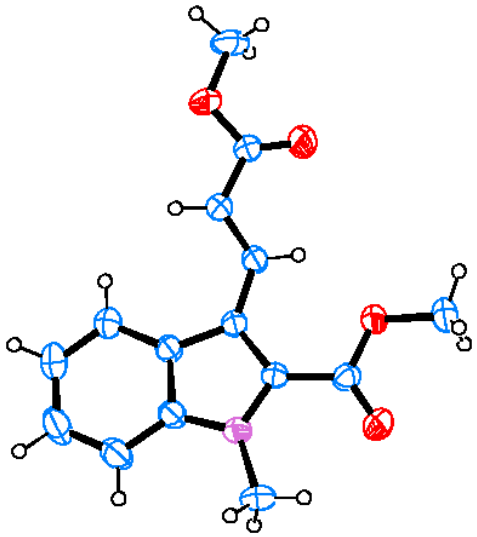


Table S5. Crystal data and structure refinement for **5g** (**CCDC** 1975674).

Identification code	21
Empirical formula	C15 H15 N O4
Formula weight	546.56
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 10.9033(4) Å α = 100.1998(15)° b = 11.1118(4) Å β = 92.2428(17)° c = 13.1220(4) Å γ = 118.6290(14)°
Volume	1359.16(8) A^3
Z, Calculated density	2, 1.336 Mg/m^3
Absorption coefficient	0.098 mm^-1
F(000)	576
Crystal size	0.250 x 0.220 x 0.100 mm
Theta range for data collection	1.593 to 24.999 deg.
Limiting indices	-12<=h<=12, -13<=k<=12, -15<=l<=15
Reflections collected / unique	20423 / 4779 [R(int) = 0.0272]

Completeness to theta = 25.000	99.6 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4779 / 0 / 368
Goodness-of-fit on F^2	1.026
Final R indices [I>2sigma(I)]	R1 = 0.0453, wR2 = 0.1172
R indices (all data)	R1 = 0.0625, wR2 = 0.1333
Extinction coefficient	0.014(2)
Largest diff. peak and hole	0.305 and -0.264 e.A^-3

Crystal Structure of Compound 6b: ORTEP representation (40% ellipsoids probability) of **6b** (CCDC 2017084).

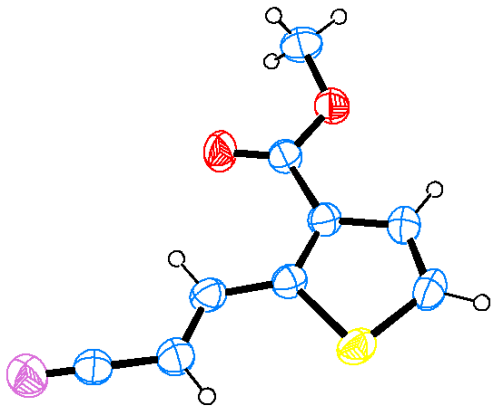


Table S6. Crystal data and structure refinement for **6b** (**CCDC** 2017084).

Identification code	999	
Empirical formula	C9 H7 N O2 S	
Formula weight	193.22	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	F d d 2	
Unit cell dimensions	a = 21.3018(14) Å	α= 90°.
	b = 43.351(3) Å	β= 90°.
	c = 3.9547(3) Å	γ = 90°.

Volume	3652.0(4) $Å^3$
Z	16
Density (calculated)	1.406 Mg/m^3
Absorption coefficient	0.317 mm^{-1}
F(000)	1600
Crystal size	0.300 x 0.250 x 0.200 mm^3
Theta range for data collection	3.407 to 28.310°.
Index ranges	-28<=h<=28, -56<=k<=56, -5<=l<=5
Reflections collected	11871
Independent reflections	2267 [R(int) = 0.0603]
Completeness to theta = 25.242°	99.4 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7457 and 0.5387
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2267 / 1 / 119
Goodness-of-fit on F^2	1.119
Final R indices [I>2sigma(I)]	R1 = 0.0433, wR2 = 0.0983
R indices (all data)	R1 = 0.0560, wR2 = 0.1076
Absolute structure parameter	0.04(4)
Extinction coefficient	n/a
Largest diff. peak and hole	0.219 and -0.277 $e.Å^{-3}$

Crystal Structure of Compound 8p: ORTEP representation (40% ellipsoids probability) of **8p** (CCDC 2017085).

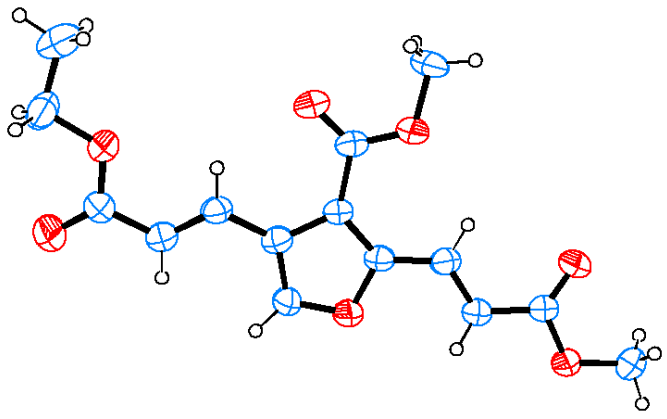


Table S7. Crystal data and structure refinement for **8p** (**CCDC** 2017085).

Identification code	1013	
Empirical formula	C15 H16 O7	
Formula weight	308.28	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 4.03230(10) Å	α= 94.501(2)°.
	b = 12.1406(2) Å	β= 91.3350(10)°.
	c = 15.7776(2) Å	γ = 93.6930(10)°.
Volume	768.09(2) $Å^3$	
Z	2	
Density (calculated)	1.333 Mg/m^3	
Absorption coefficient	0.107 mm^{-1}	
F(000)	324	
Crystal size	0.300 x 0.250 x 0.200 mm^3	
Theta range for data collection	2.976 to 24.997°.	
Index ranges	-4<=h<=4, -14<=k<=14, -18<=l<=18	
Reflections collected	23407	
Independent reflections	2695 [R(int) = 0.0939]	
Completeness to theta = 24.997°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7454 and 0.4080	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2695 / 0 / 202	
Goodness-of-fit on F^2	1.047	
Final R indices [I>2sigma(I)]	R1 = 0.0599, wR2 = 0.1440	
R indices (all data)	R1 = 0.0911, wR2 = 0.1702	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.192 and -0.207 $e.Å^{-3}$	

Analytical Data:

Compounds **5a**,[S1] (**5b**,**g**),[S2] **6a**,[S3] (**7b**, and **8a**,**n**)[S4] are known in literature and thus only ^{1}H NMR data of these compounds are provided.

References:

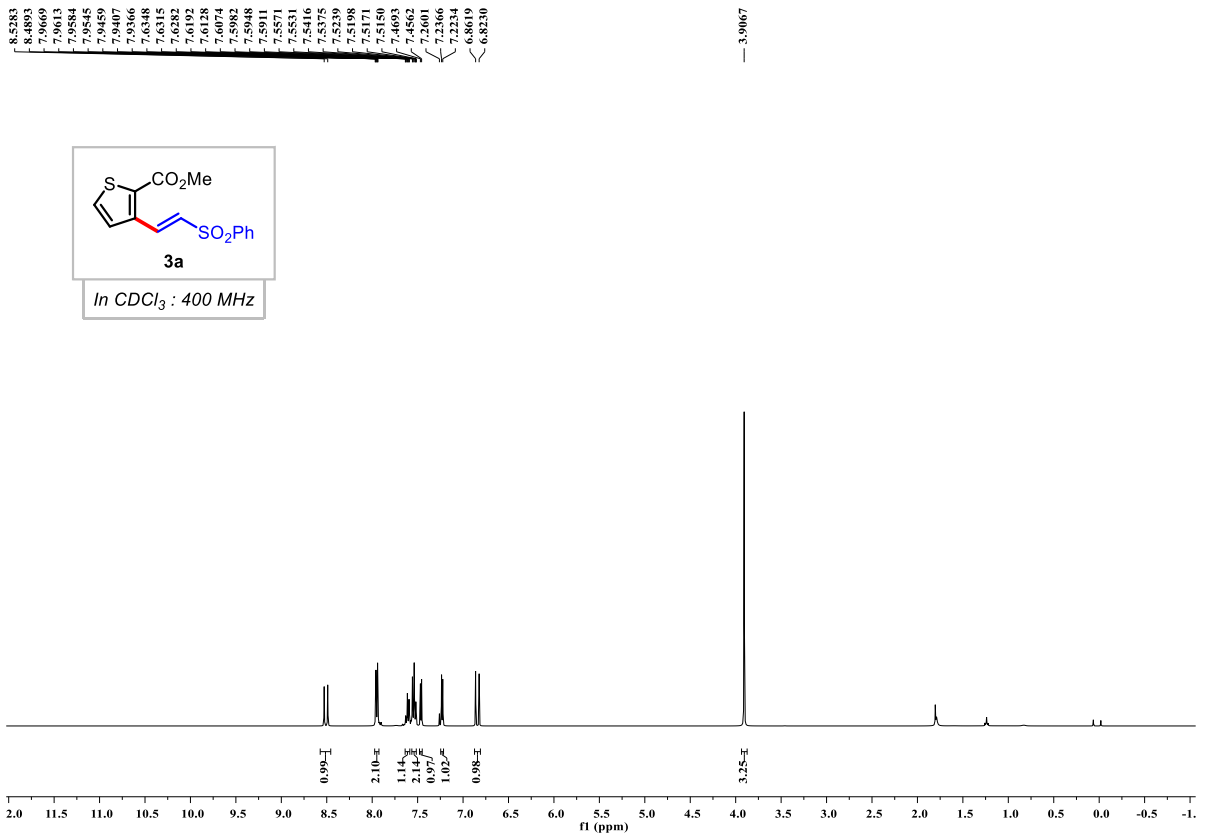
(S1) Oger, N.; Grognec, E. L.; Felpin, F.-X. *J. Org. Chem.* **2014**, *79*, 8255.

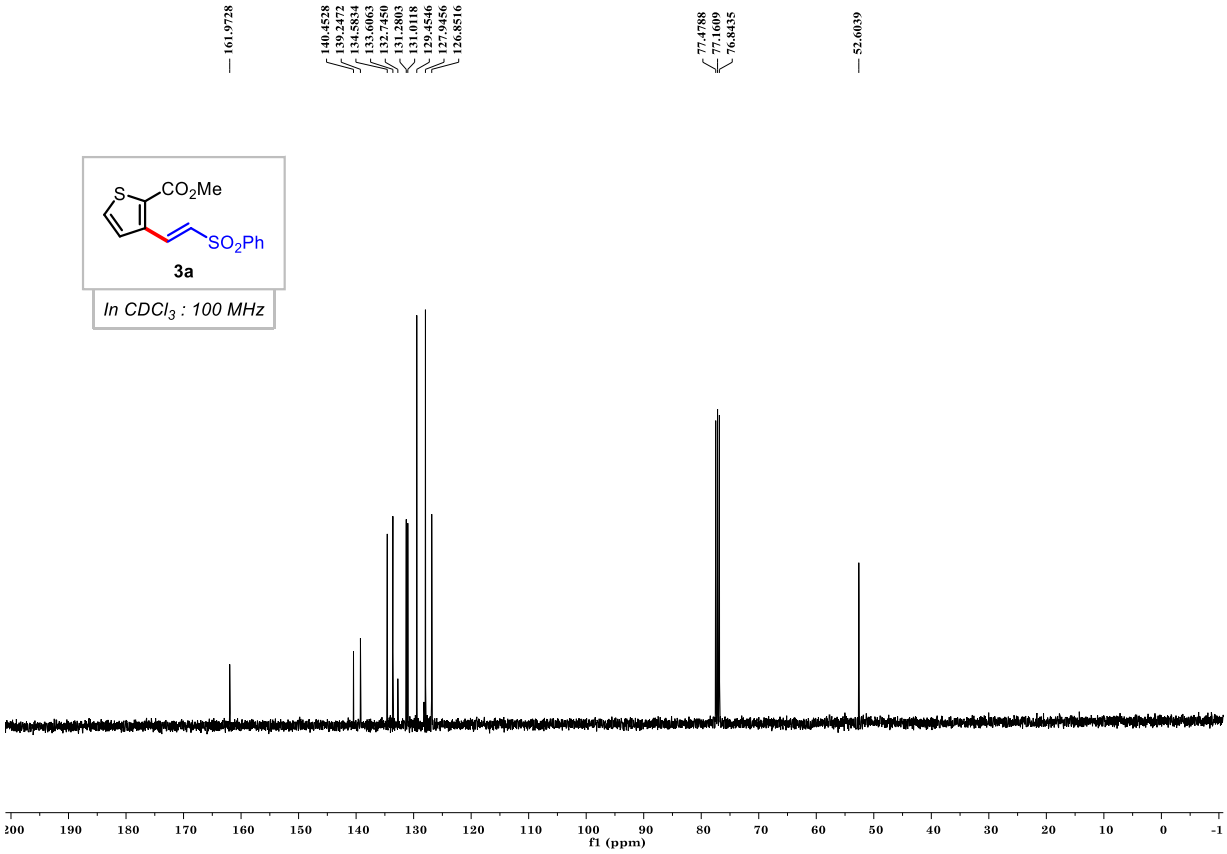
(S2) Padala, K.; Pimparkar, S.; Madasamy, P.; Jeganmohan, M. *Chem. Commun.* **2012**, *48*, 7140.

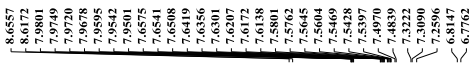
(S3) Ueyama, T.; Mochida, S.; Fukutani, T.; Hirano, K.; Satoh, T.; Miura, M. *Org. Lett.* **2011**, *13*, 706.

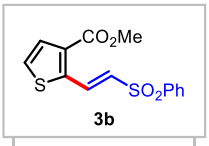
(S4) Mandal, A.; Mehta, G.; Dana, S.; Baidya, M. *Org. Lett.* **2019**, *21*, 5879.

NMR Spectra of Synthesized Compounds


8.5283
8.4893
7.9669
7.9613
7.9584
7.9545
7.9459
7.9407
7.9366
7.6348
7.6315
7.6282
7.6192
7.6128
7.6074
7.5982
7.5948
7.5911
7.5571
7.5531
7.5416
7.5375
7.5239
7.5198
7.5171
7.5150
7.4693
7.4562
7.2601
7.2366
7.2234
6.8619
6.8230
3.9067
CO2Me
S
SO2Ph
3a
In CDCl3 : 400 MHz
0.99
2.10
1.14
2.14
0.97
1.02
0.98
3.25
f1 (ppm)

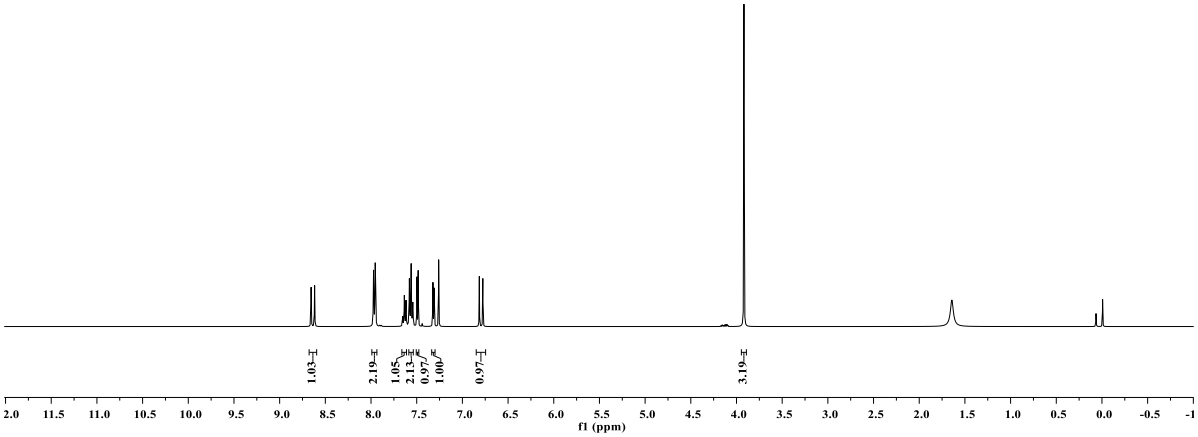

161.9728
140.4528
139.2472
134.5834
133.6063
132.7450
131.2803
131.0118
129.4546
127.9456
126.8516
77.4788
77.1609
76.8435
52.6039
CO2Me
S
SO2Ph
3a
In CDCl3 : 100 MHz
f1 (ppm)

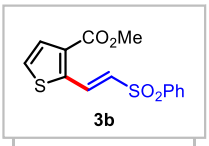




3b

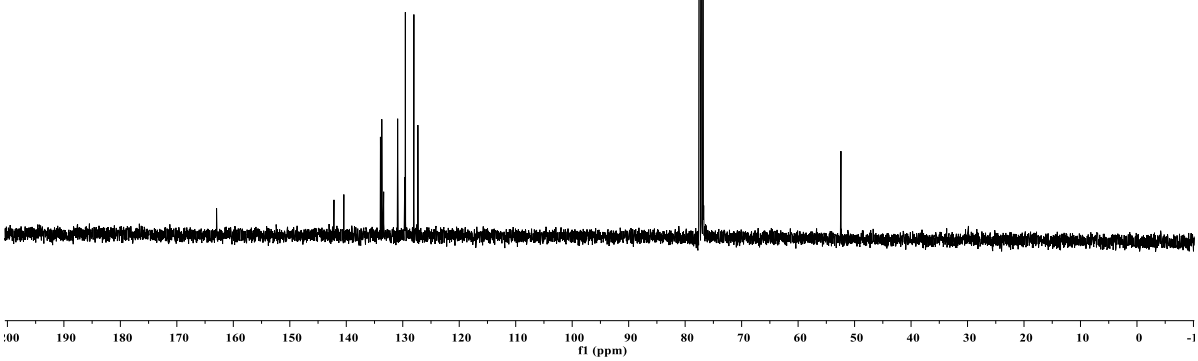
In $CDCl_3$: 400 MHz

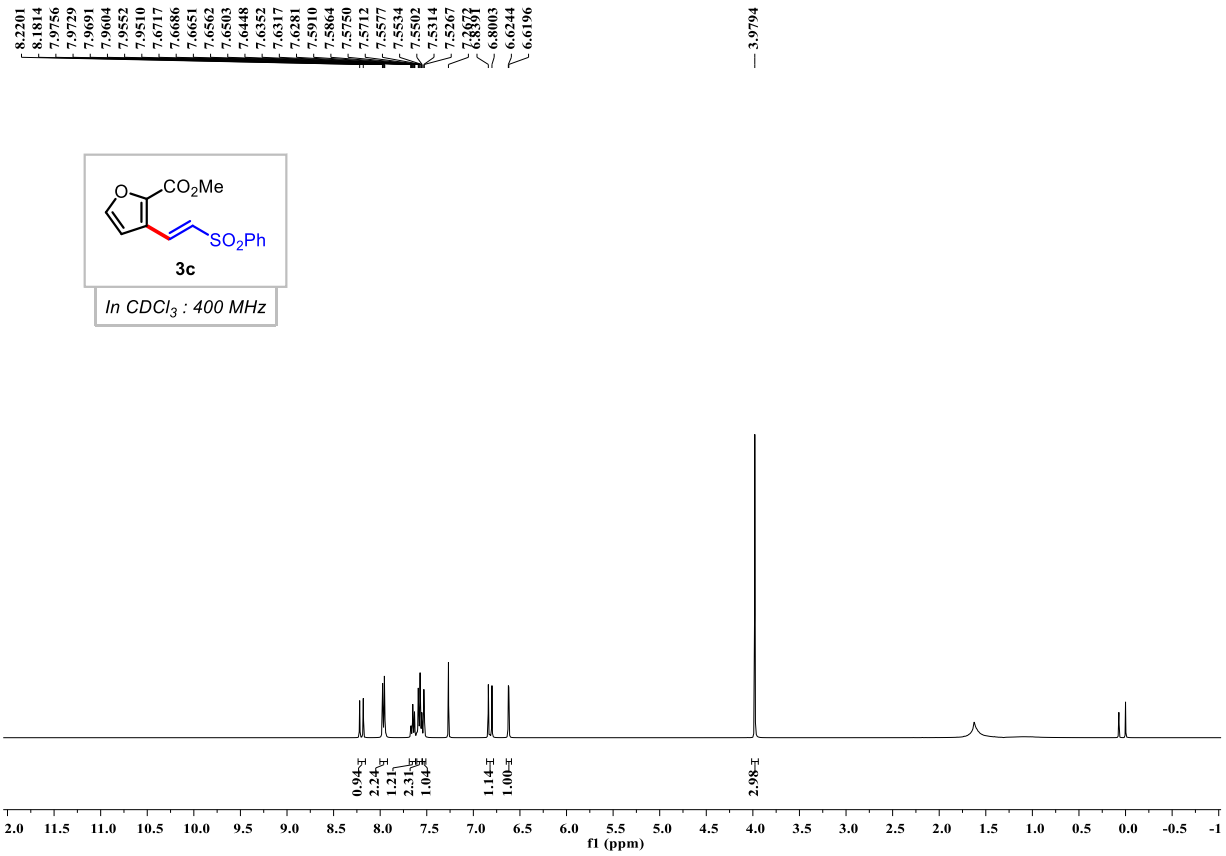


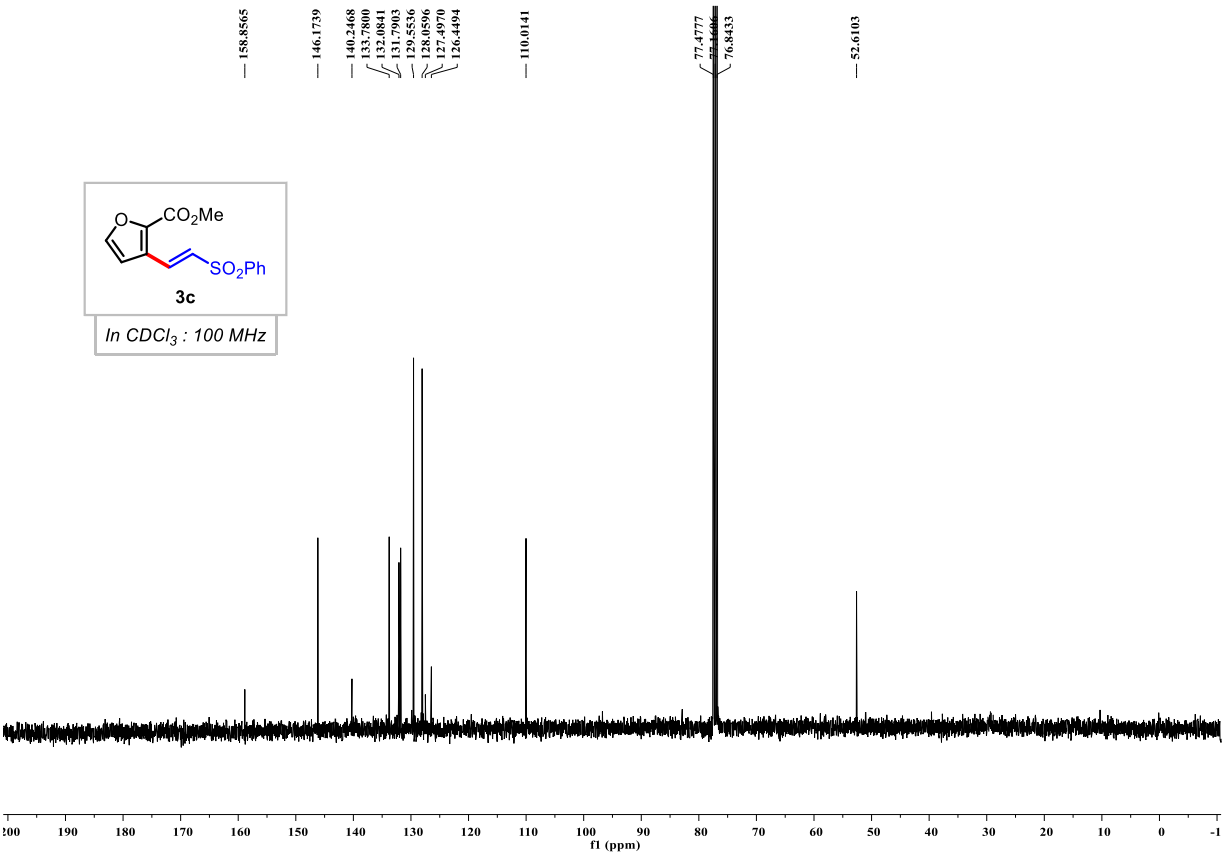


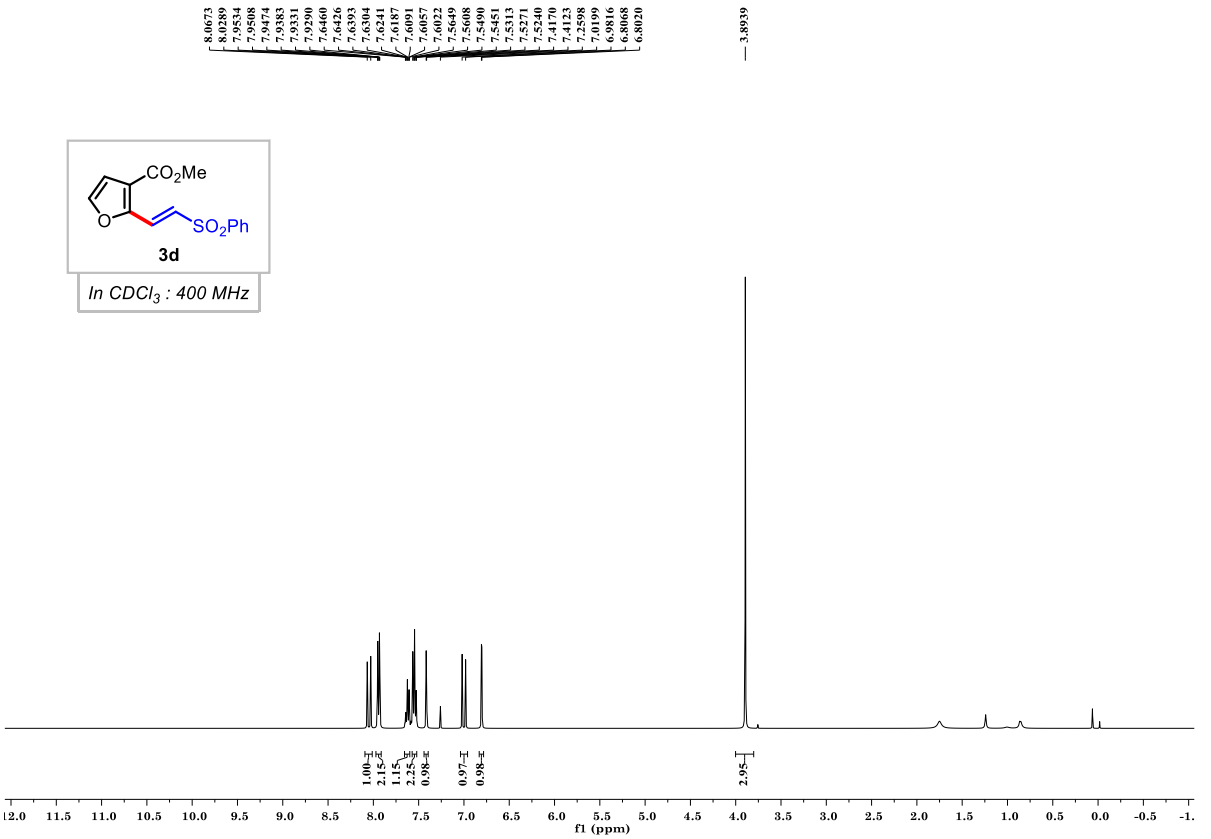
3b

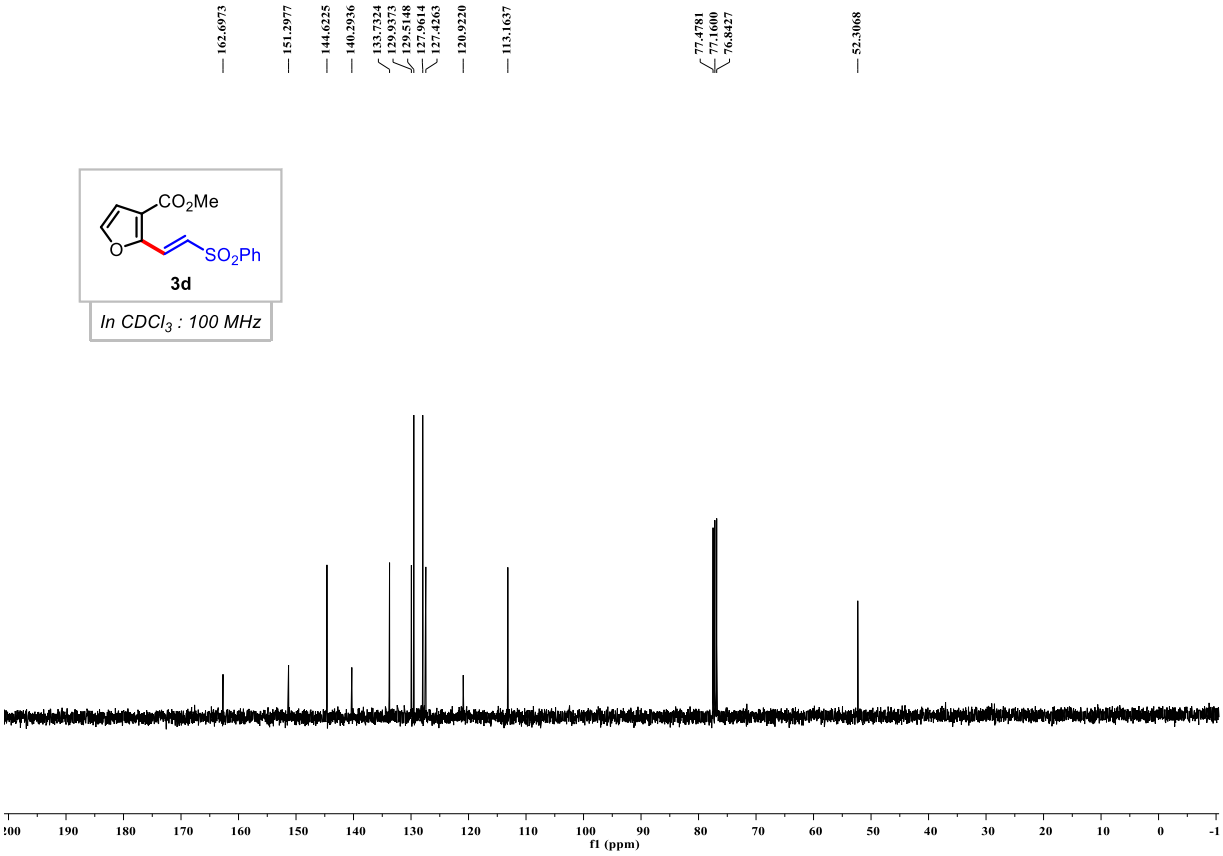
In $CDCl_3$: 100 MHz

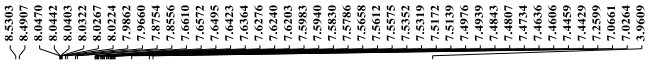


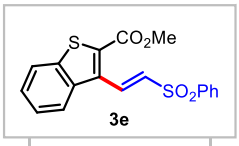

8.2201
8.1814
7.9756
7.9729
7.9691
7.9604
7.9552
7.9510
7.6717
7.6686
7.6651
7.6562
7.6503
7.6448
7.6352
7.6317
7.6281
7.5910
7.5864
7.5750
7.5712
7.5577
7.5534
7.5502
7.5314
7.5267
7.2672
6.8311
6.8003
6.6244
6.6196
3.9794
CO2Me
SO2Ph
3c
In CDCl3 : 400 MHz
0.94
2.24
1.21
2.31
1.04
1.14
1.00
2.98
f1 (ppm)


158.8565
146.1739
140.2468
133.7800
132.0841
131.7903
129.5536
128.0596
127.4970
126.4494
110.0141
77.4777
76.8433
52.6103
CO2Me
SO2Ph
3c
In CDCl3 : 100 MHz
f1 (ppm)


8.0673
8.0289
7.9534
7.9508
7.9474
7.9383
7.9331
7.9290
7.6460
7.6426
7.6393
7.6304
7.6244
7.6187
7.6091
7.6057
7.6022
7.5649
7.5608
7.5490
7.5451
7.5313
7.5271
7.5240
7.4170
7.4123
7.2598
7.0199
6.9816
6.8068
6.8020
3.8939
CO2Me
SO2Ph
3d
In CDCl3 : 400 MHz
1.00
2.15
1.15
2.25
0.98
0.97
0.98
2.95
f1 (ppm)

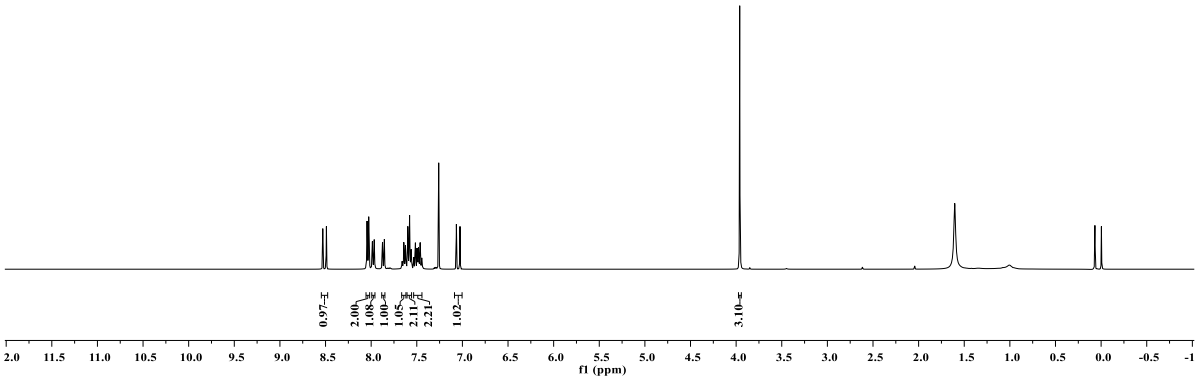

162.6973
151.2977
144.6225
140.2936
133.7324
129.9373
129.5148
127.9614
127.4263
120.9220
113.1637
77.4781
77.1600
76.8427
52.3068
CO2Me
SO2Ph
3d
In CDCl3 : 100 MHz
f1 (ppm)

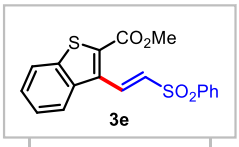




3e

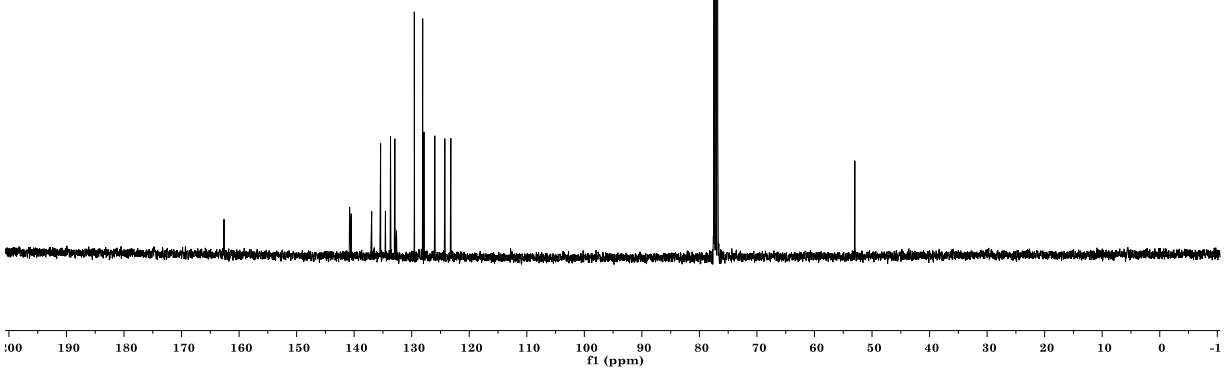
In $CDCl_3$: 400 MHz

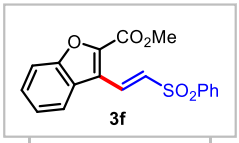




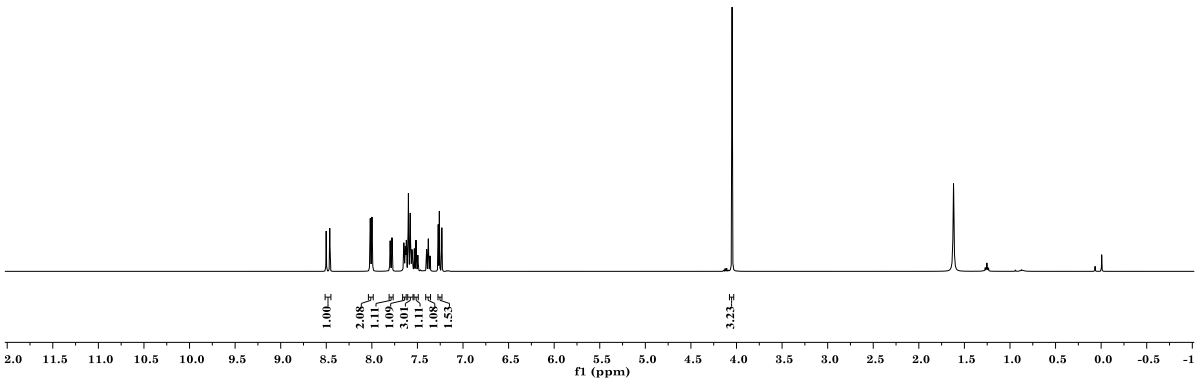
3e

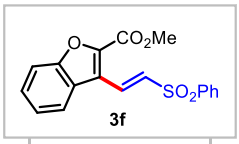
In $CDCl_3$: 100 MHz



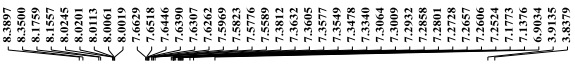


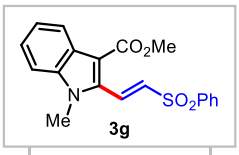
In $CDCl_3$: 400 MHz



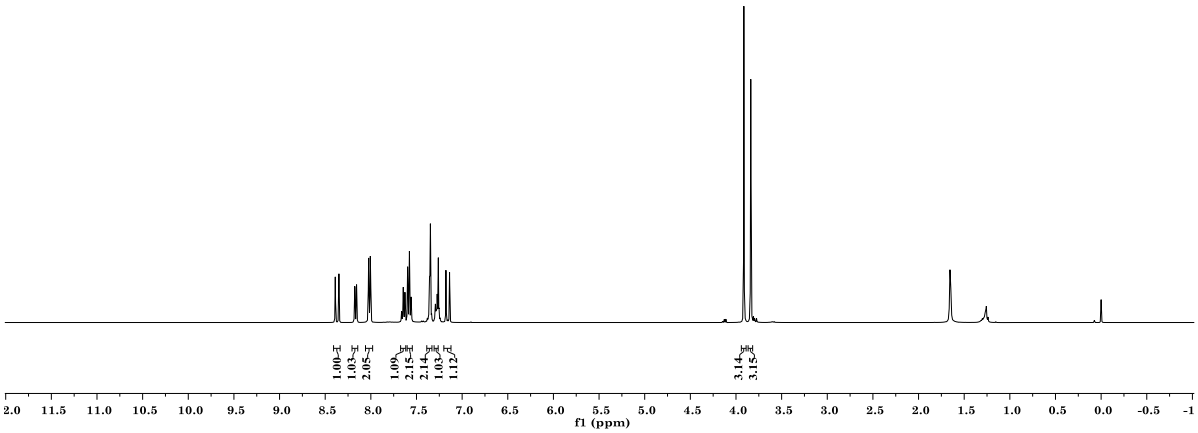


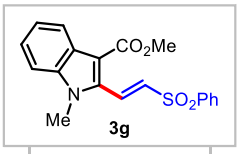
In $CDCl_3$: 100 MHz



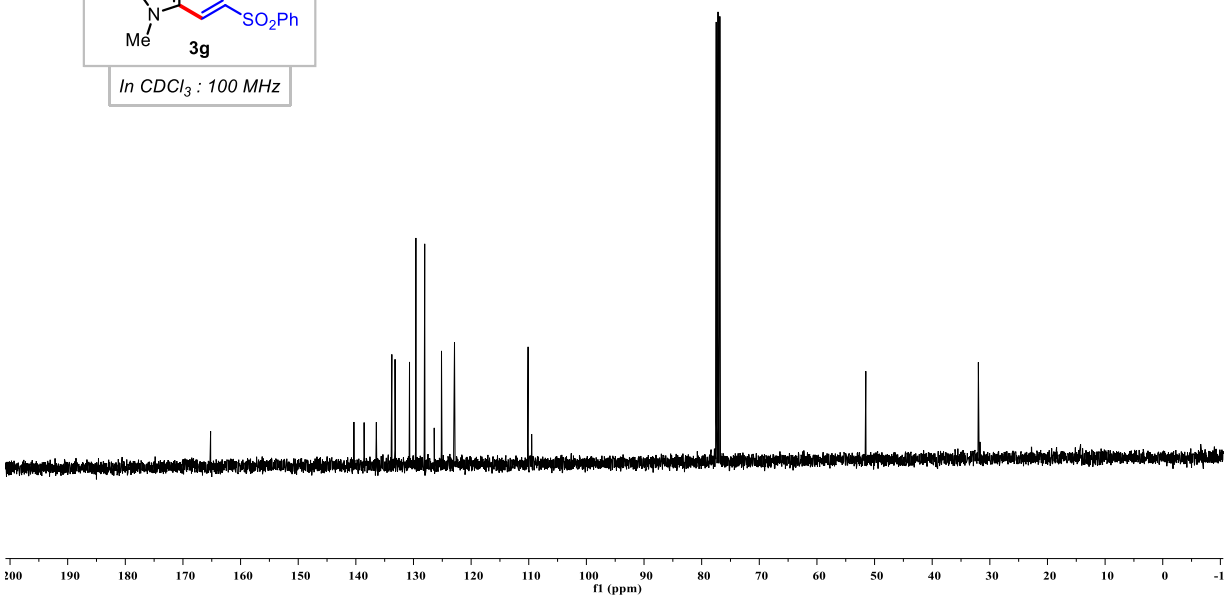


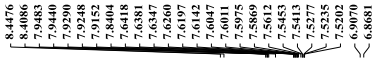
In $CDCl_3$: 400 MHz

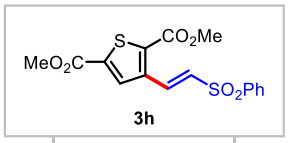




In $CDCl_3$: 100 MHz

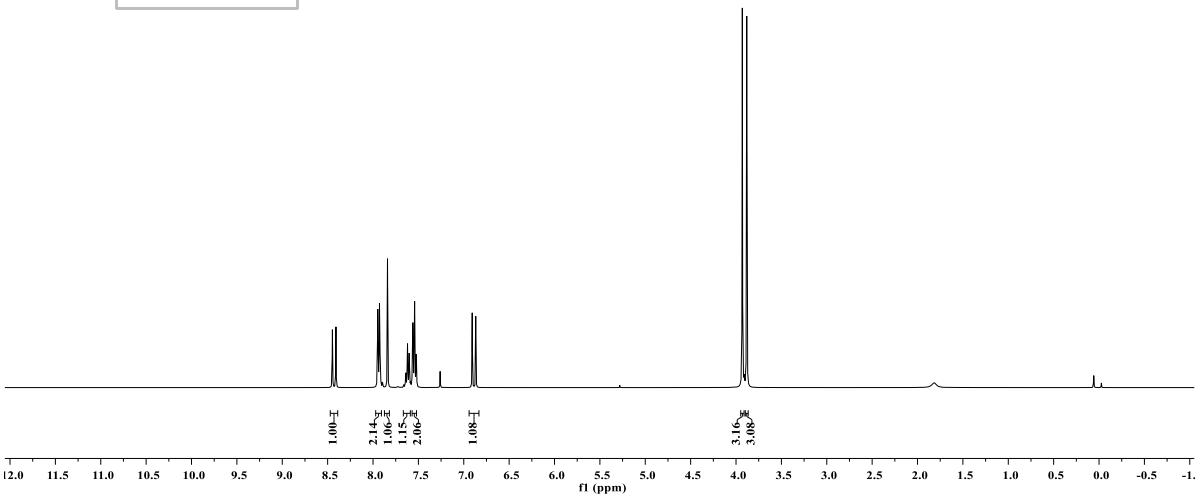


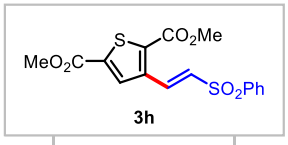




3h

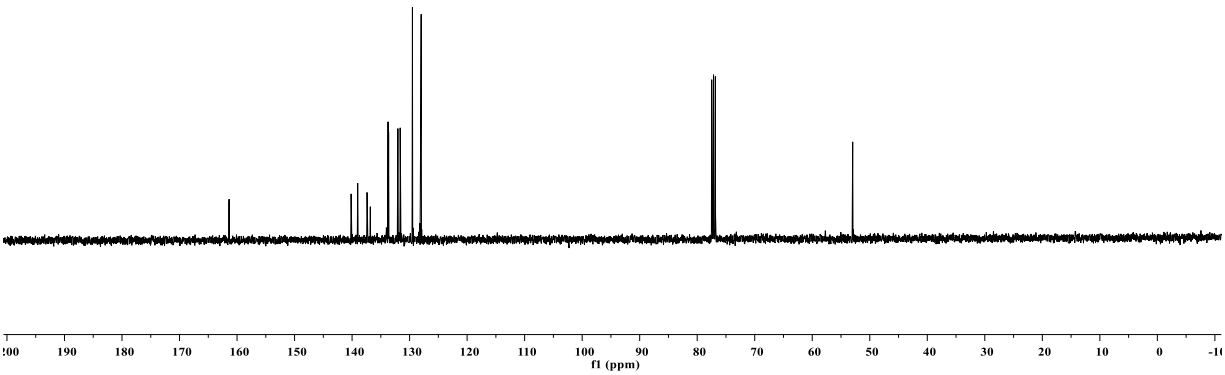
In $CDCl_3$: 400 MHz

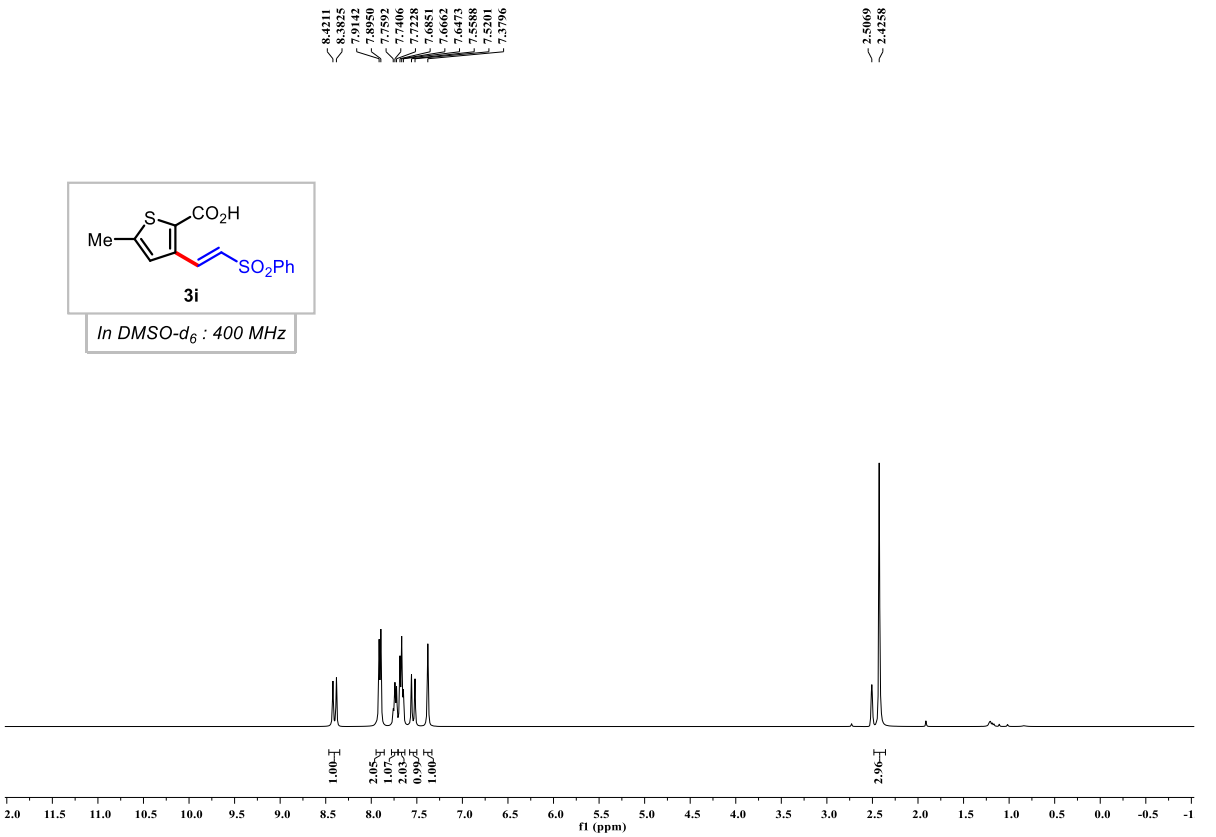


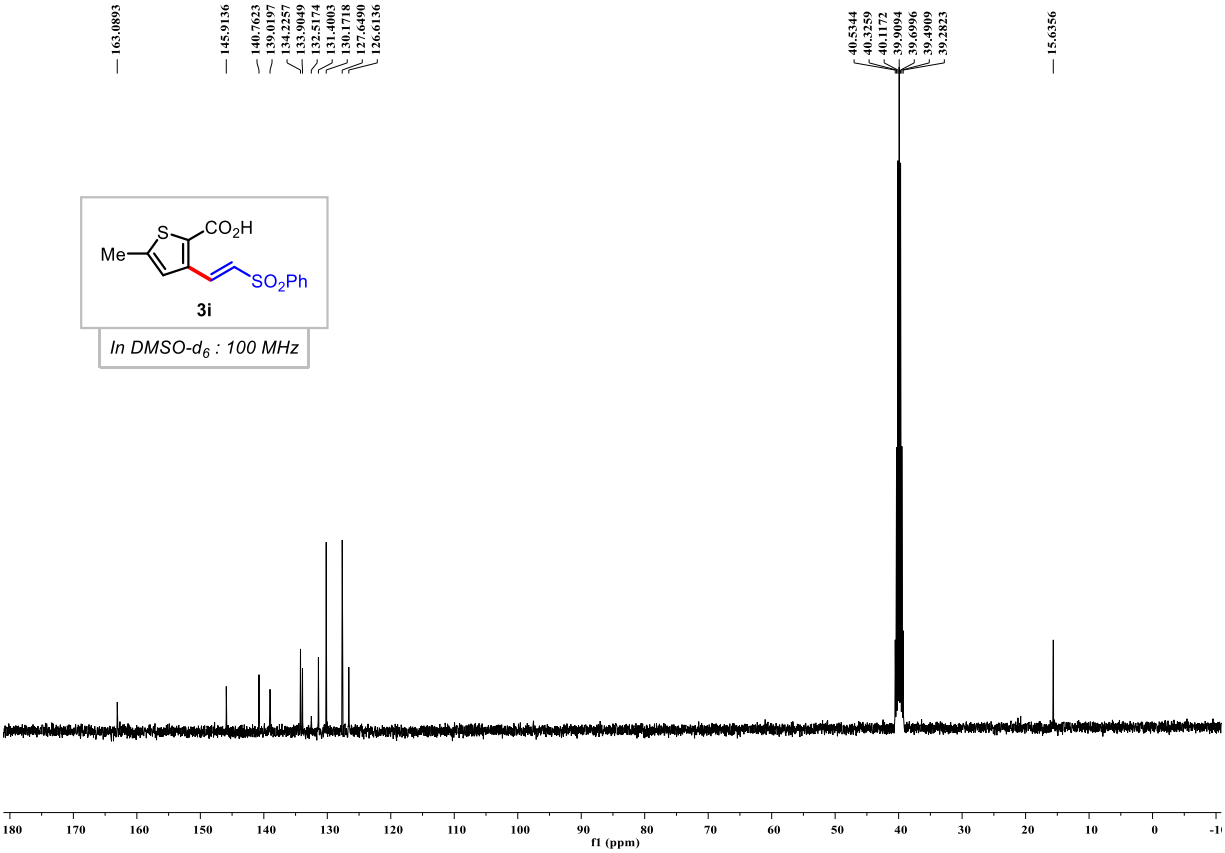


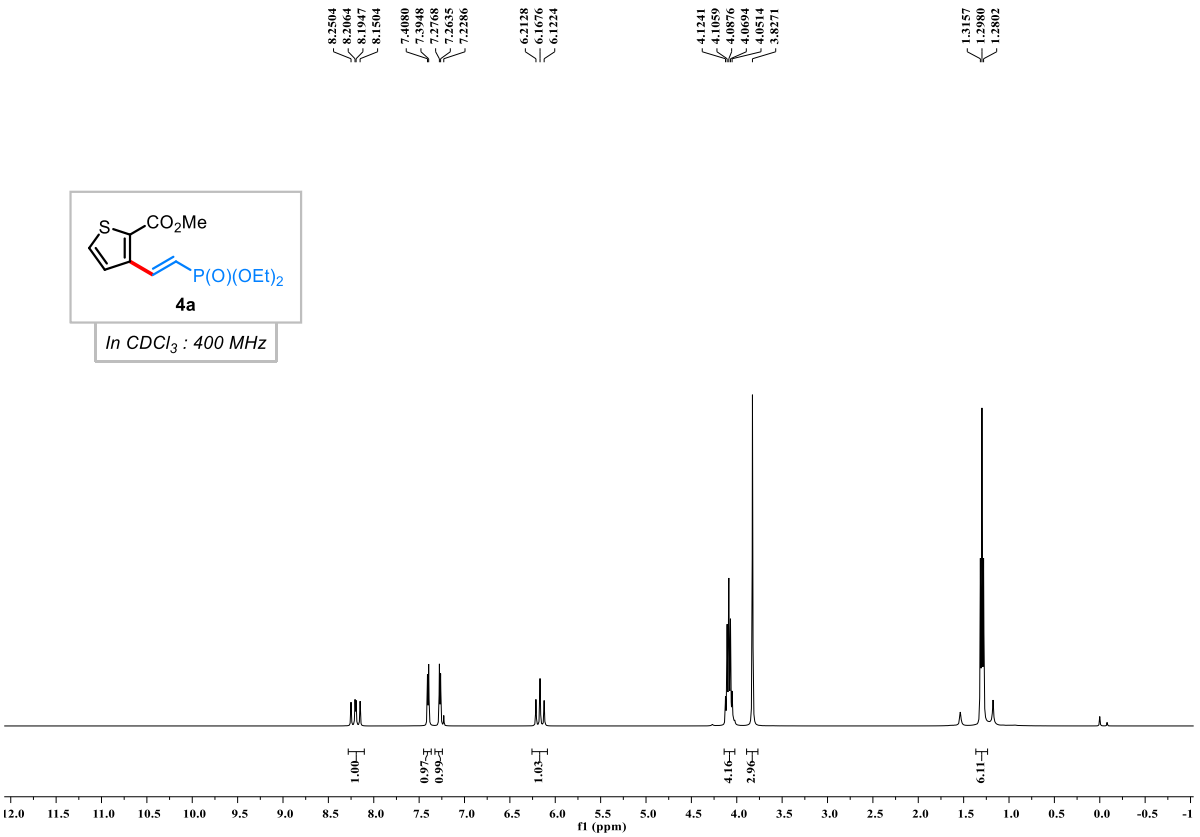
3h

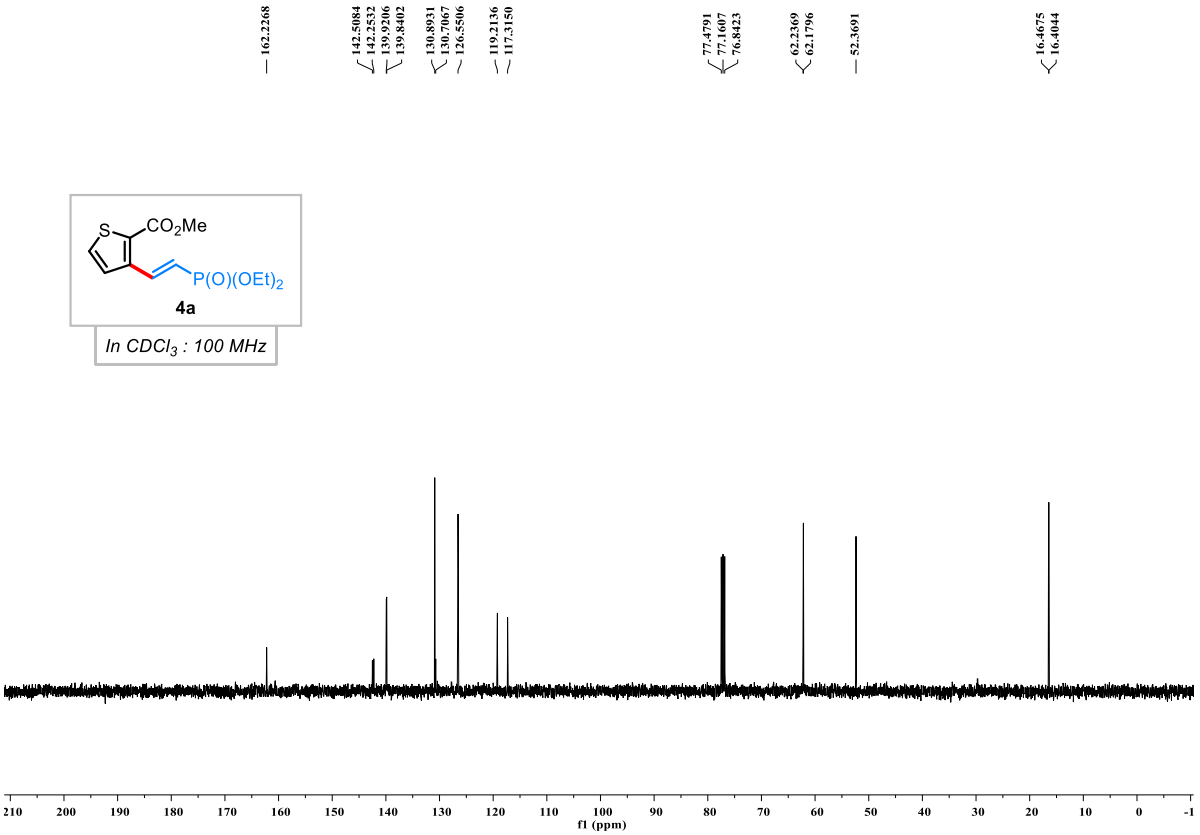
In $CDCl_3$: 100 MHz

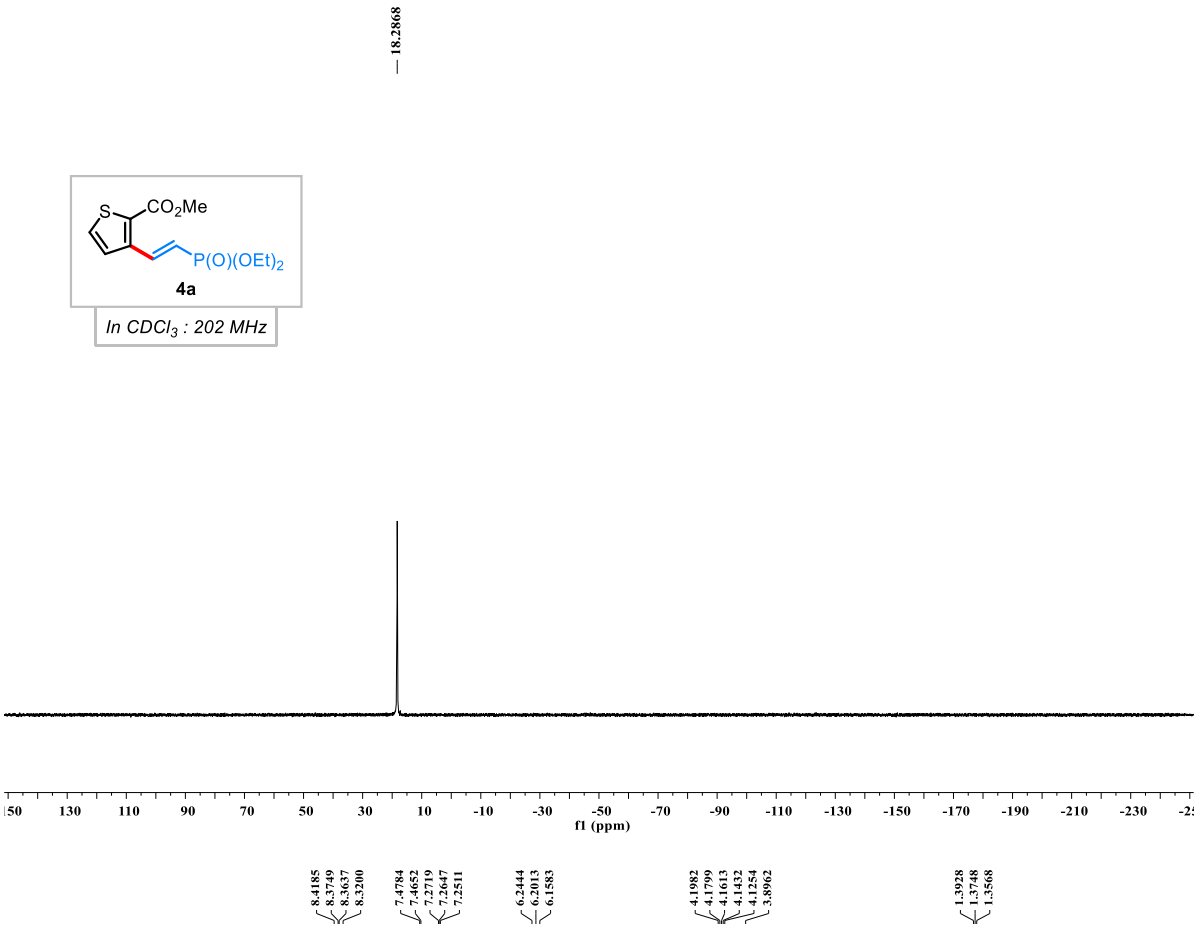


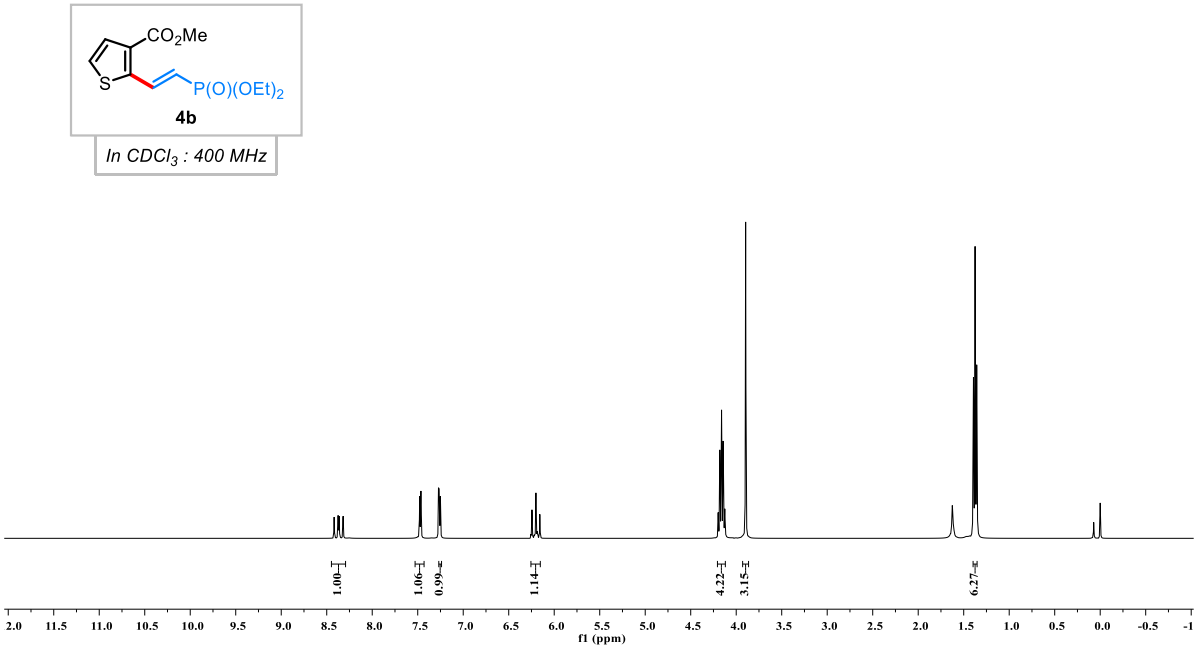

8.4211
8.3825
7.9142
7.8950
7.7592
7.7406
7.7228
7.6851
7.6662
7.6473
7.5588
7.5201
7.3796
2.5069
2.4258
Me
CO2H
SO2Ph
3i
In DMSO-d6 : 400 MHz
1.00
2.05
1.07
2.03
0.99
1.00
2.96
f1 (ppm)

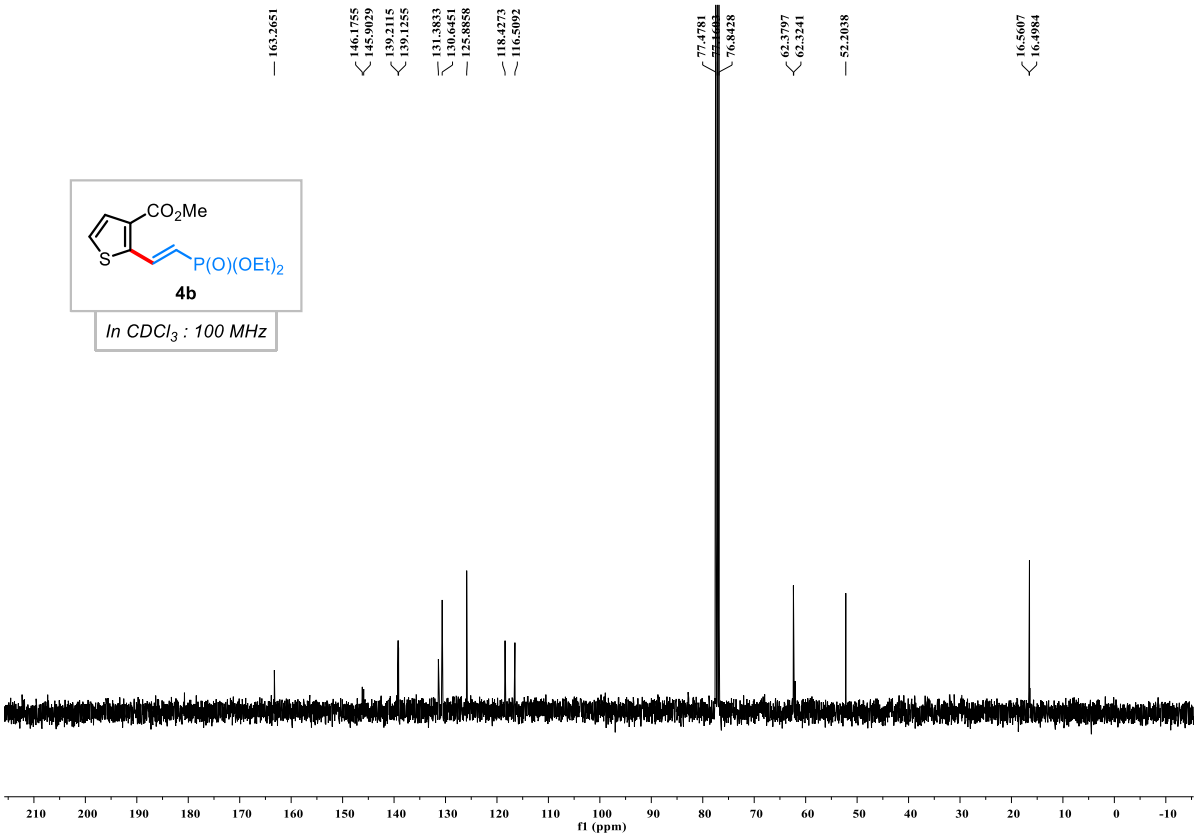

163.0893
145.9136
140.7623
139.0197
134.2257
133.9049
132.5174
131.4003
130.1718
127.6490
126.6136
40.5344
40.3259
40.1172
39.9094
39.6996
39.4909
39.2823
15.6356
Me
CO2H
SO2Ph
3i
In DMSO-d6 : 100 MHz
f1 (ppm)

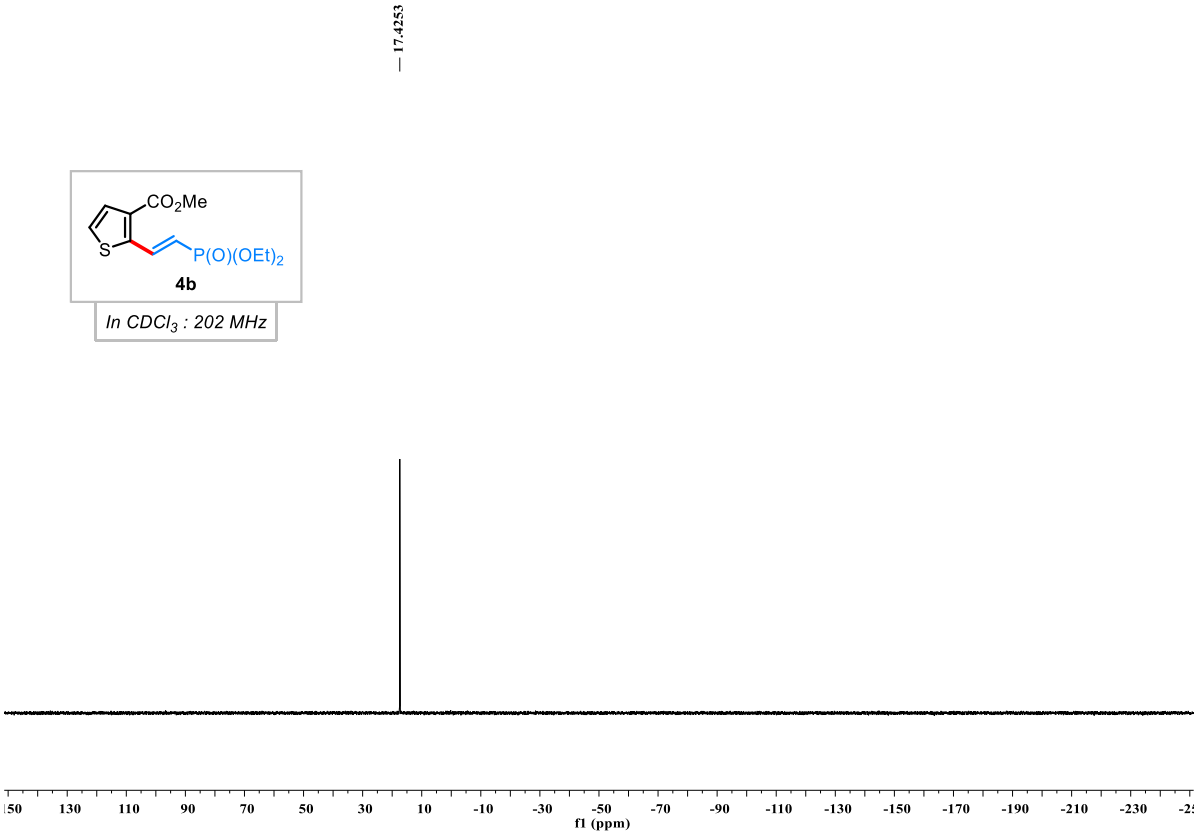

8.2504
8.2064
8.1947
8.1504
7.4080
7.3948
7.2768
7.2635
7.2286
6.2128
6.1676
6.1224
4.1241
4.1059
4.0876
4.0694
4.0514
3.8271
1.3157
1.2980
1.2802
S
CO2Me
P(O)(OEt)2
4a
In CDCl3 : 400 MHz
1.00
0.97
0.99
1.03
4.16
2.96
6.11
f1 (ppm)

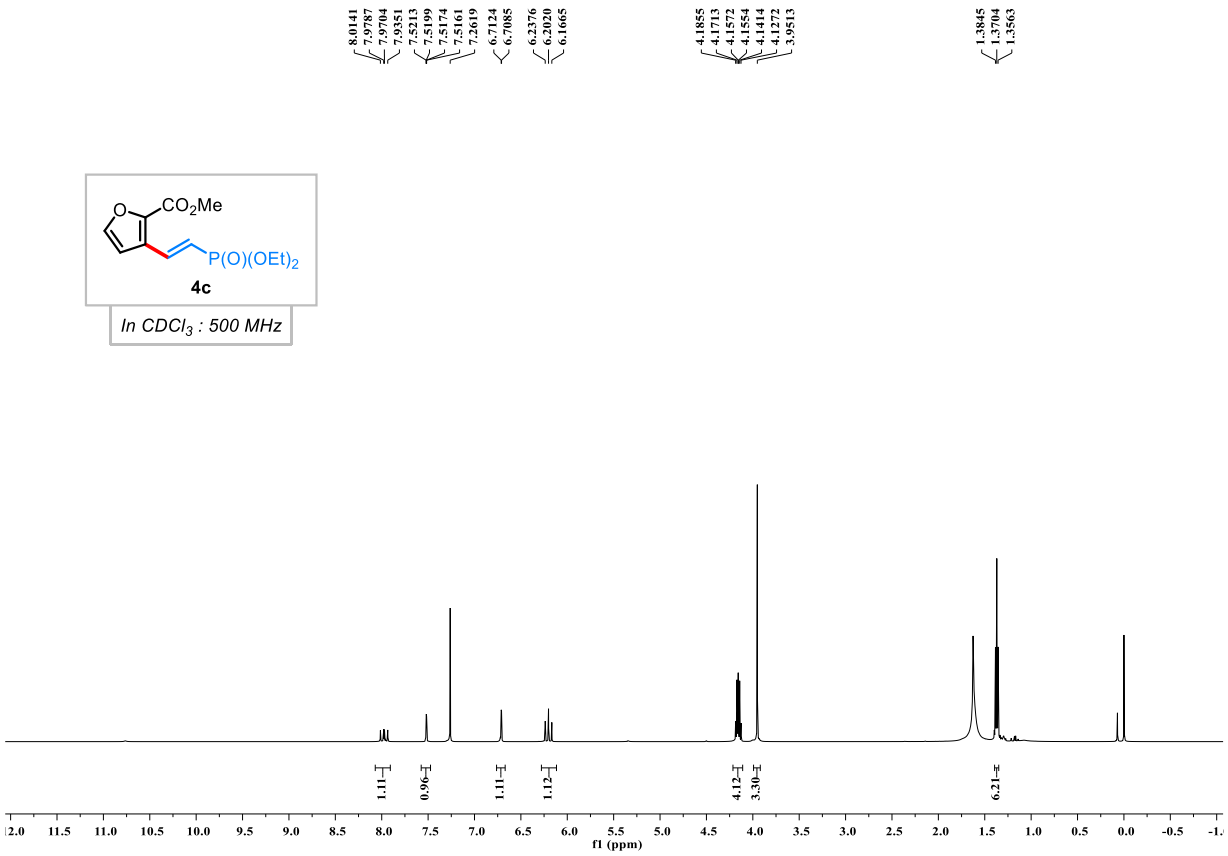

162.2268
142.5084
142.2532
139.9206
139.8402
130.8931
130.7067
126.5506
119.2136
117.3150
77.4791
77.1607
76.8423
62.2369
62.1796
52.3691
16.4675
16.4044
S
CO2Me
P(O)(OEt)2
4a
In CDCl3 : 100 MHz
f1 (ppm)

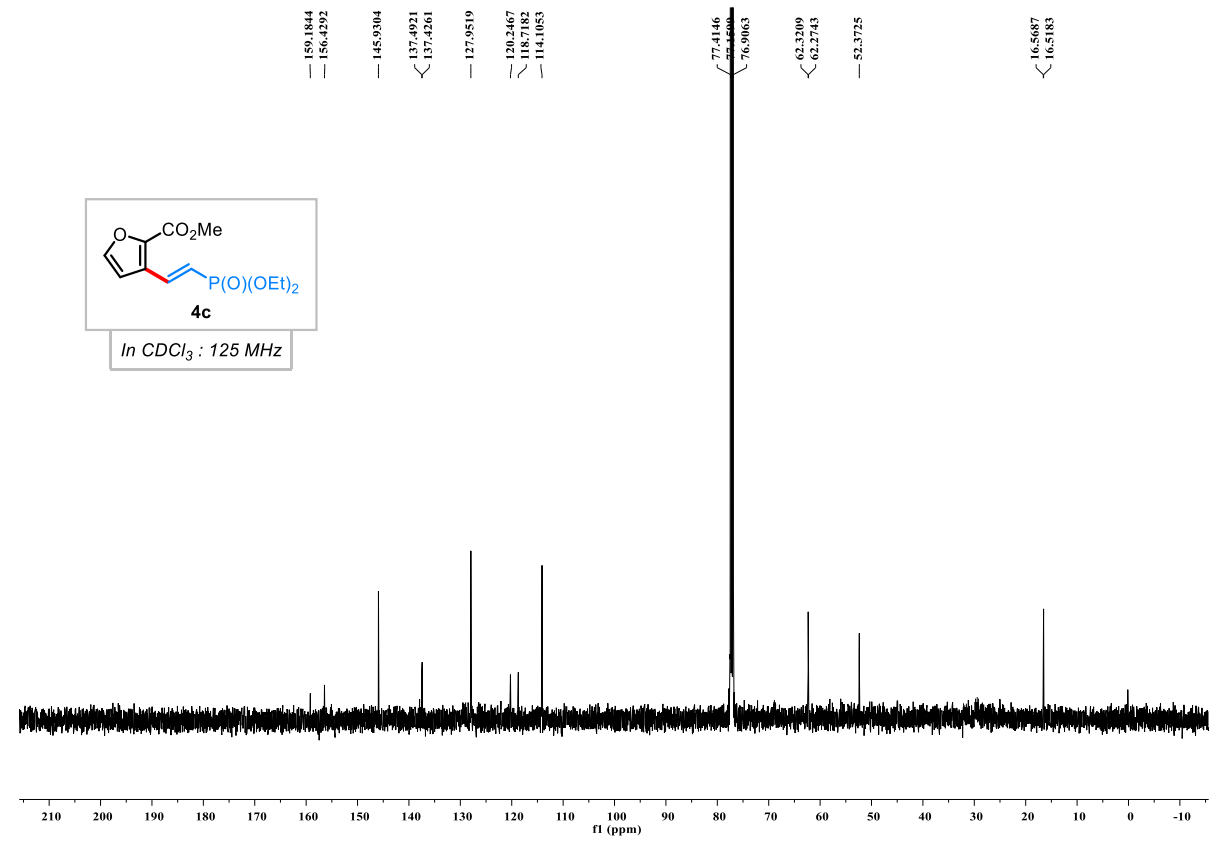

18.2868
CO2Me
S
P(O)(OEt)2
4a
In CDCl3 : 202 MHz
f1 (ppm)
8.4185
8.3749
8.3637
8.3200
7.4784
7.4652
7.2719
7.2647
7.2511
6.2444
6.2013
6.1583
4.1982
4.1799
4.1613
4.1432
4.1254
3.8962
1.3928
1.3748
1.3568

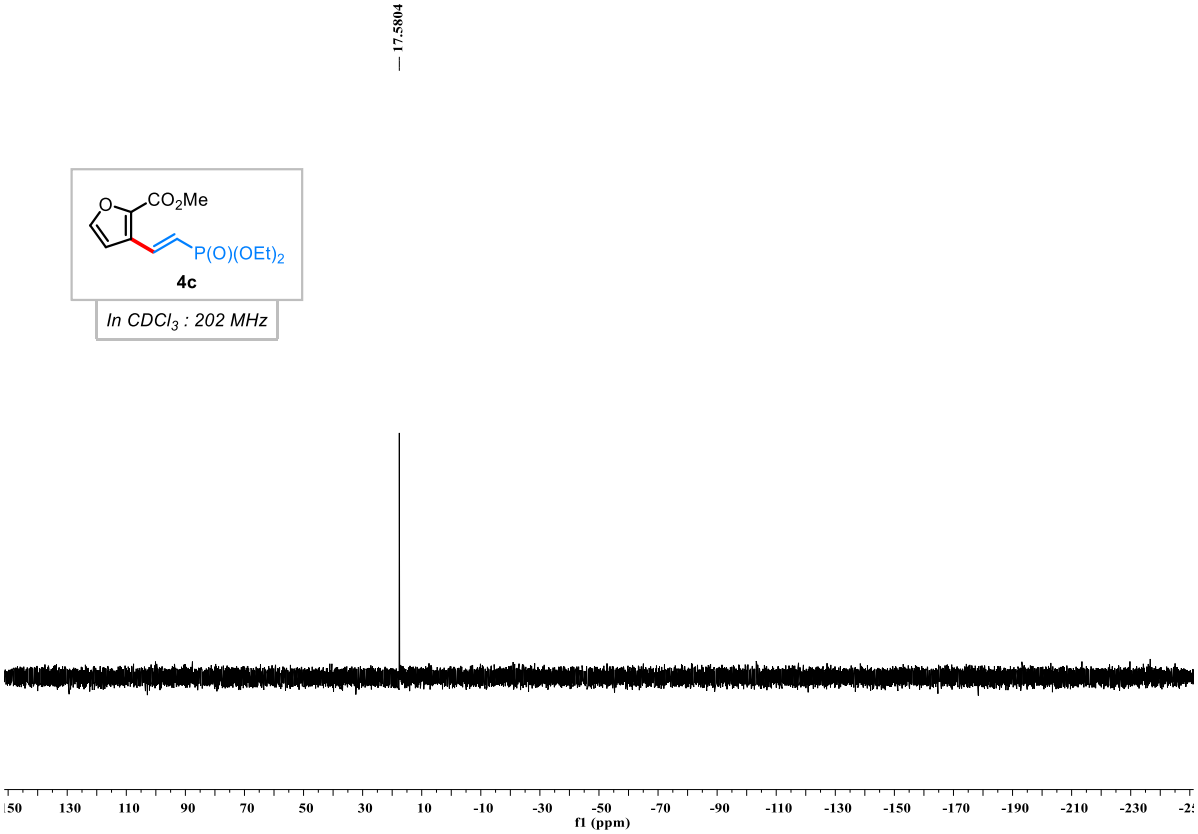

CO2Me
S
P(O)(OEt)2
4b
In CDCl3 : 400 MHz
1.00
1.06
0.99
1.14
4.22
3.15
6.27
f1 (ppm)

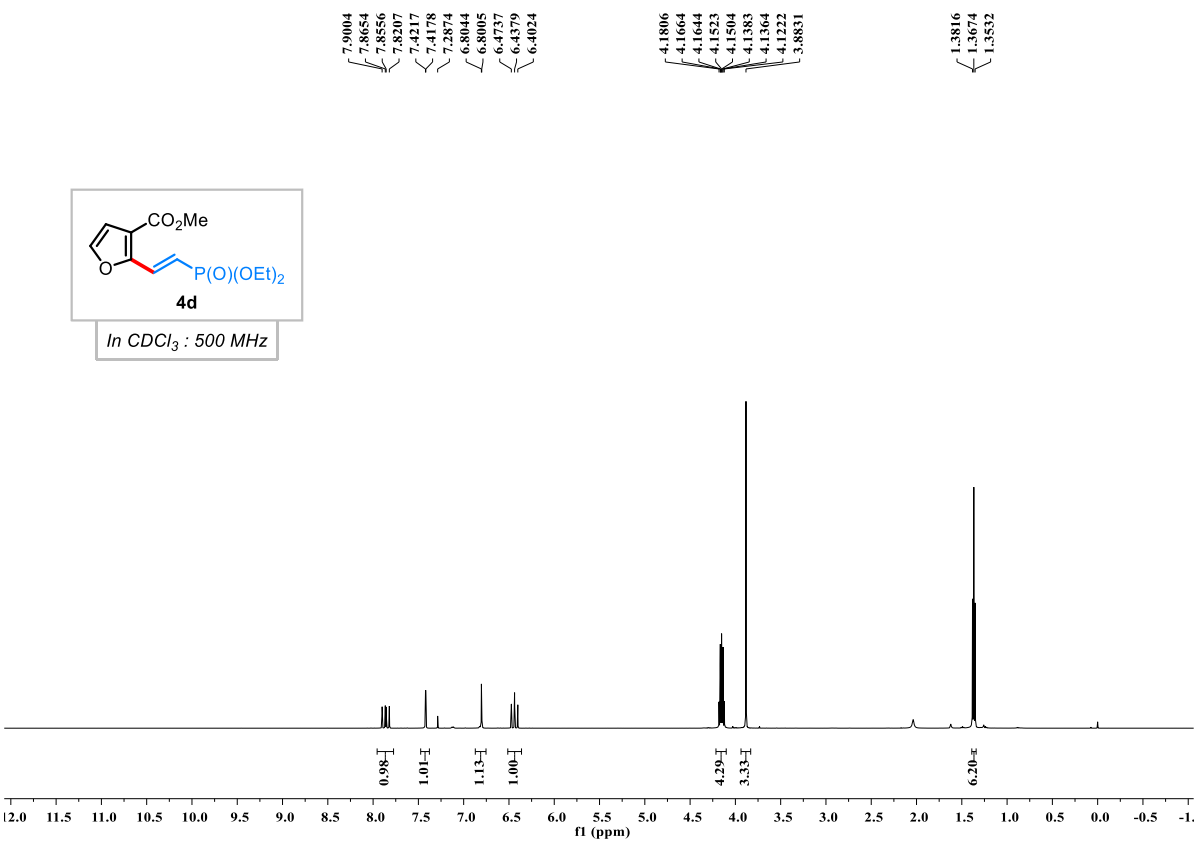

163.2651
146.1755
145.9029
139.2115
139.1255
131.3833
130.6451
125.8858
118.4273
116.5092
77.4781
76.8428
62.3797
62.3241
52.2038
16.5607
16.4984
CO2Me
S
P(O)(OEt)2
4b
In CDCl3 : 100 MHz
f1 (ppm)

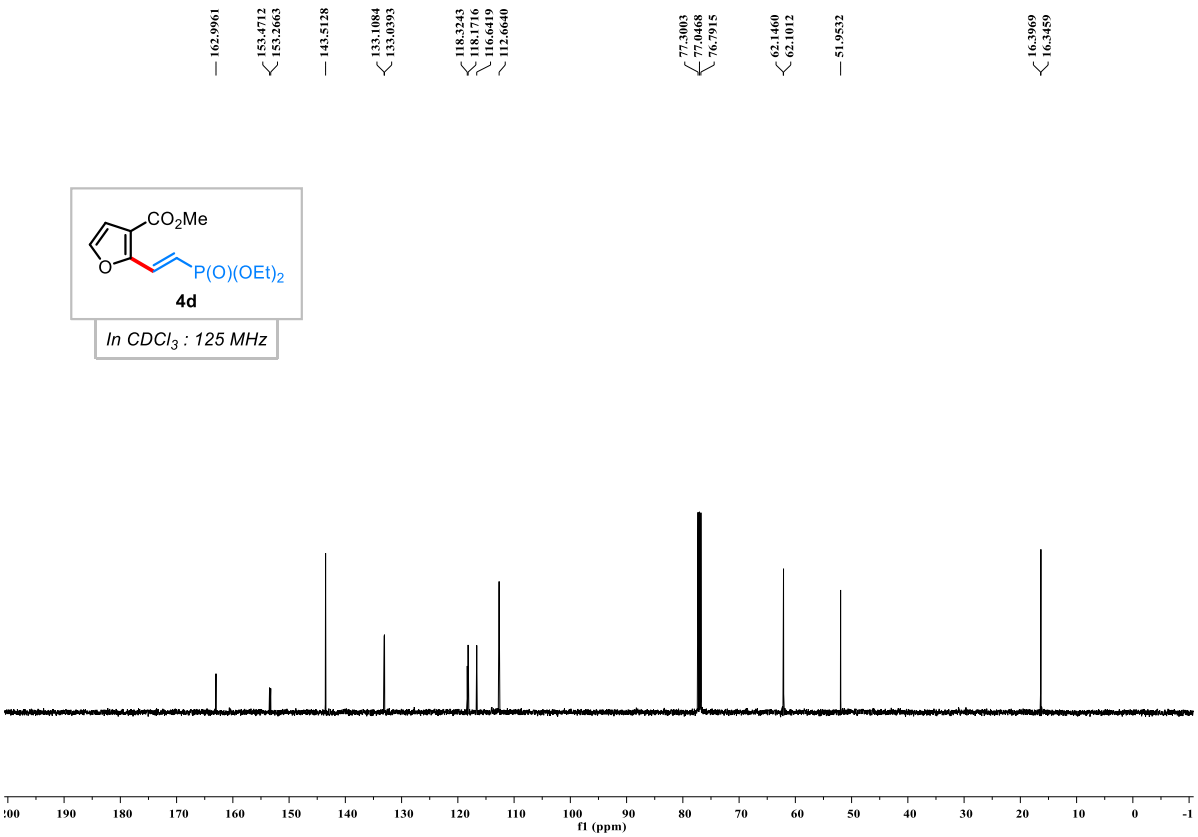

17.4253
CO2Me
S
P(O)(OEt)2
4b
In CDCl3 : 202 MHz
f1 (ppm)

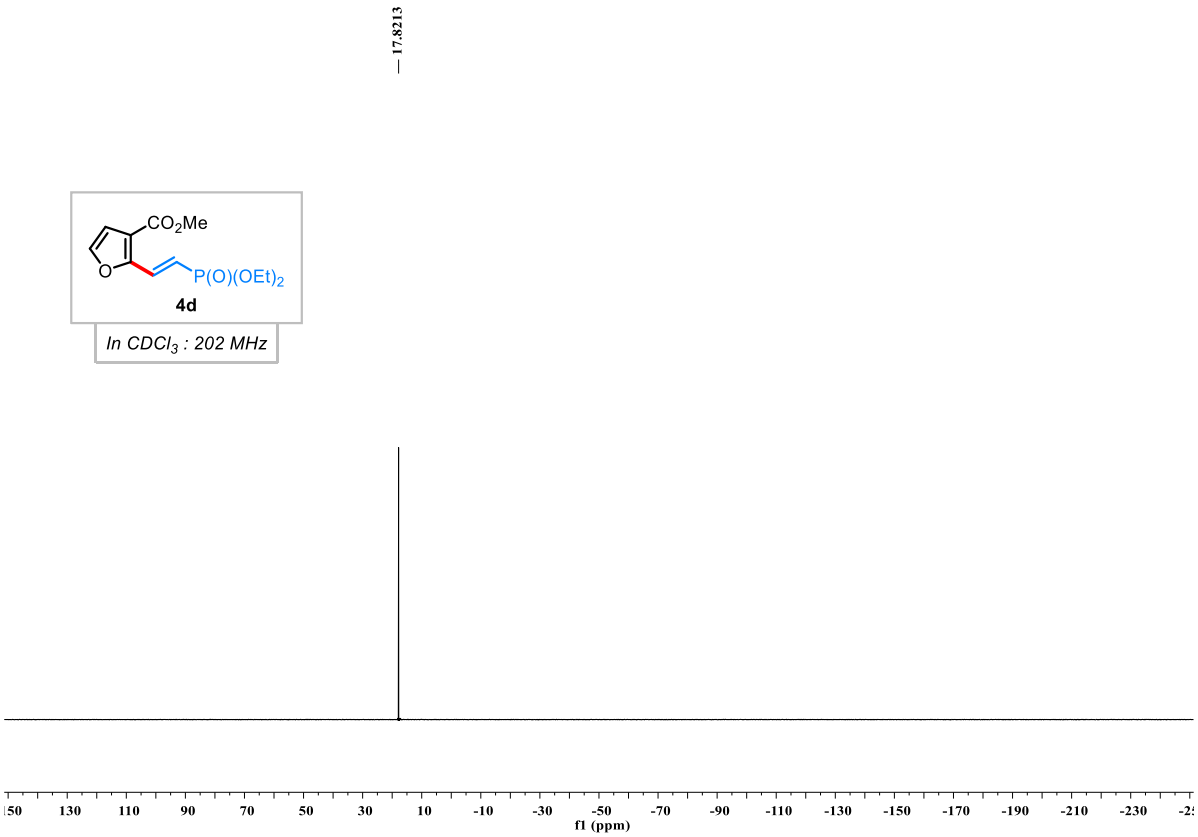

8.0141
7.9787
7.9704
7.9351
7.5213
7.5199
7.5174
7.5161
7.2619
6.7124
6.7085
6.2376
6.2020
6.1665
4.1855
4.1713
4.1572
4.1554
4.1414
4.1272
3.9513
1.3845
1.3704
1.3563
CO2Me
P(O)(OEt)2
4c
In CDCl3 : 500 MHz
1.11
0.96
1.11
1.12
4.12
3.30
6.21
f1 (ppm)

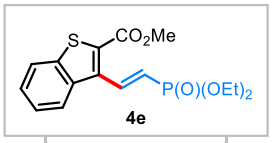

159.1844
156.4292
145.9304
137.4921
137.4261
127.9519
120.2467
118.7182
114.1053
77.4146
77.1500
76.9063
62.3209
62.2743
52.3725
16.5687
16.5183
CO2Me
P(O)(OEt)2
4c
In CDCl3 : 125 MHz
f1 (ppm)


17.5804
CO2Me
P(O)(OEt)2
4c
In CDCl3 : 202 MHz
f1 (ppm)


7.9004
7.8654
7.8556
7.8207
7.4217
7.4178
7.2874
6.8044
6.8005
6.4737
6.4379
6.4024
4.1806
4.1664
4.1644
4.1523
4.1504
4.1383
4.1364
4.1222
3.8831
1.3816
1.3674
1.3532
CO2Me
P(O)(OEt)2
4d
In CDCl3 : 500 MHz
0.98
1.01
1.13
1.00
4.29
3.33
6.20
f1 (ppm)

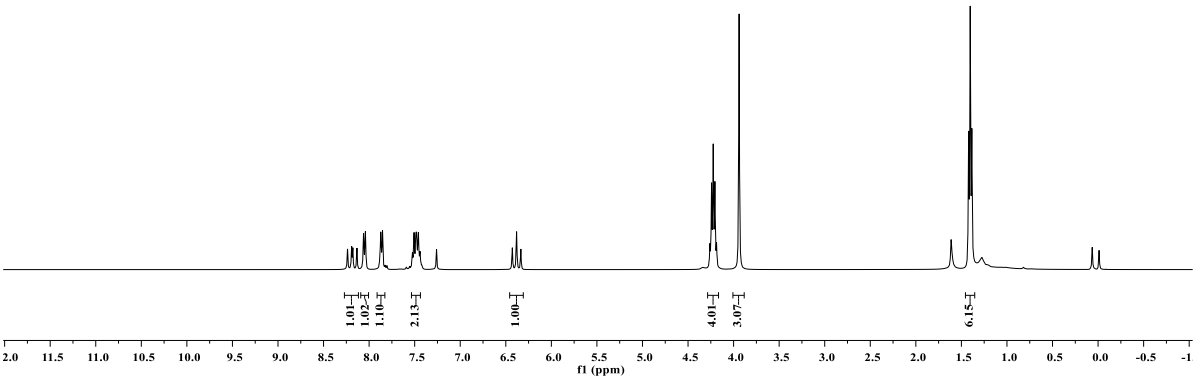

162.9961
153.4712
153.2663
143.5128
133.1084
133.0393
118.3243
118.1716
116.6419
112.6640
77.3003
77.0468
76.7915
62.1460
62.1012
51.9532
16.3969
16.3459
CO2Me
P(O)(OEt)2
4d
In CDCl3 : 125 MHz
f1 (ppm)

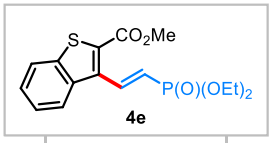

17.8213
CO2Me
P(O)(OEt)2
4d
In CDCl3 : 202 MHz
f1 (ppm)



4e

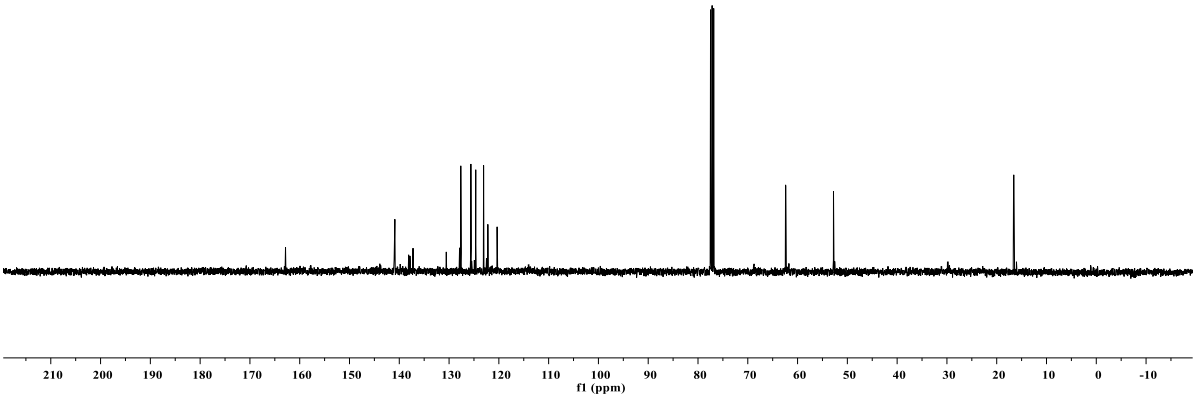
In $CDCl_3$: 400 MHz

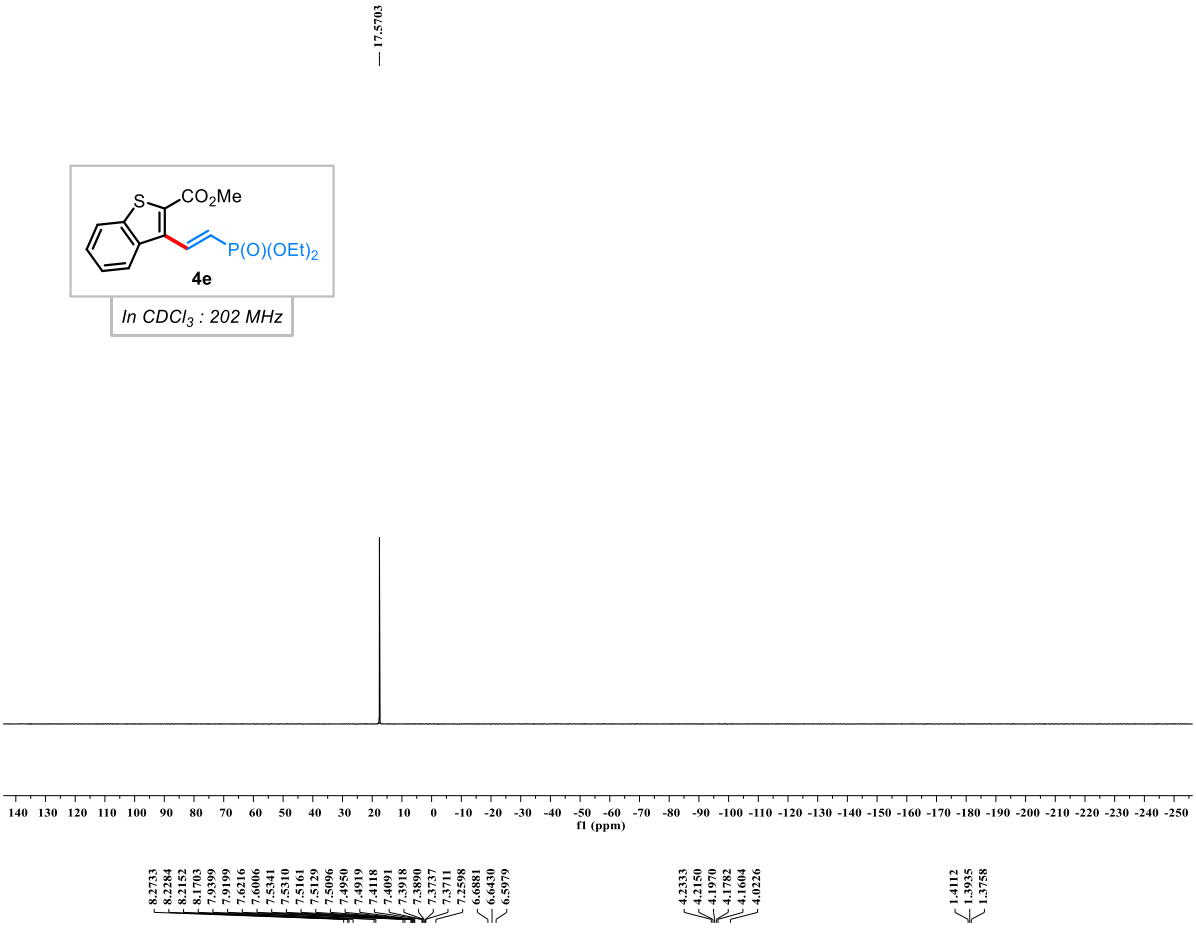


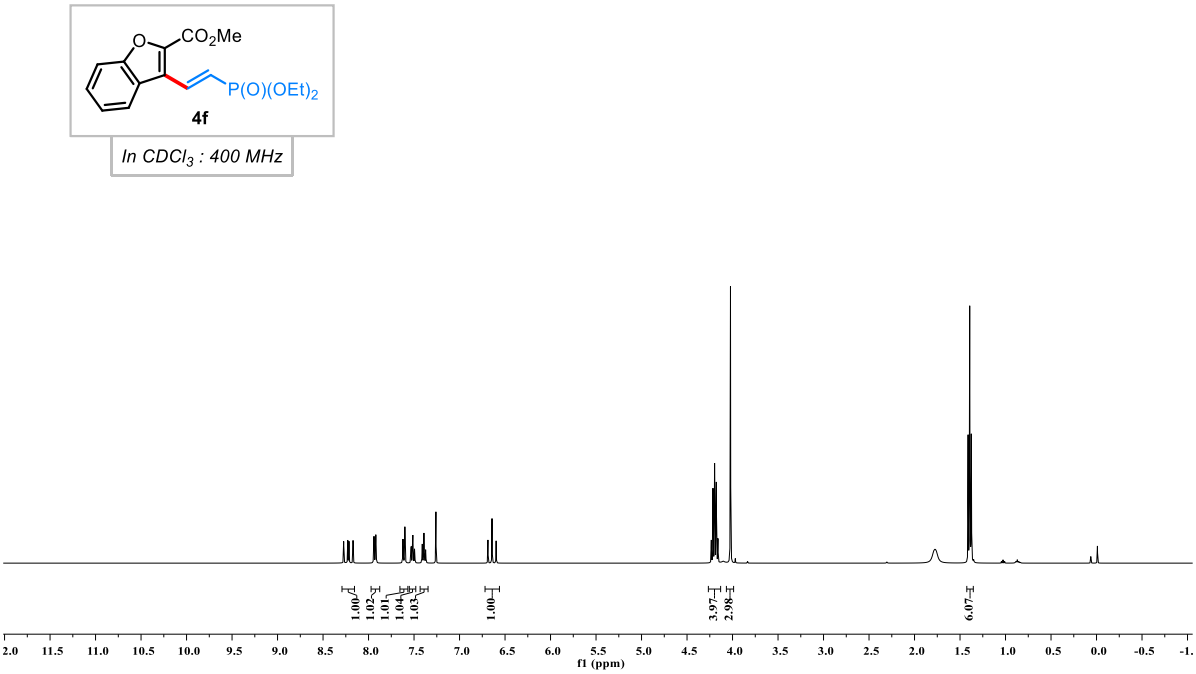


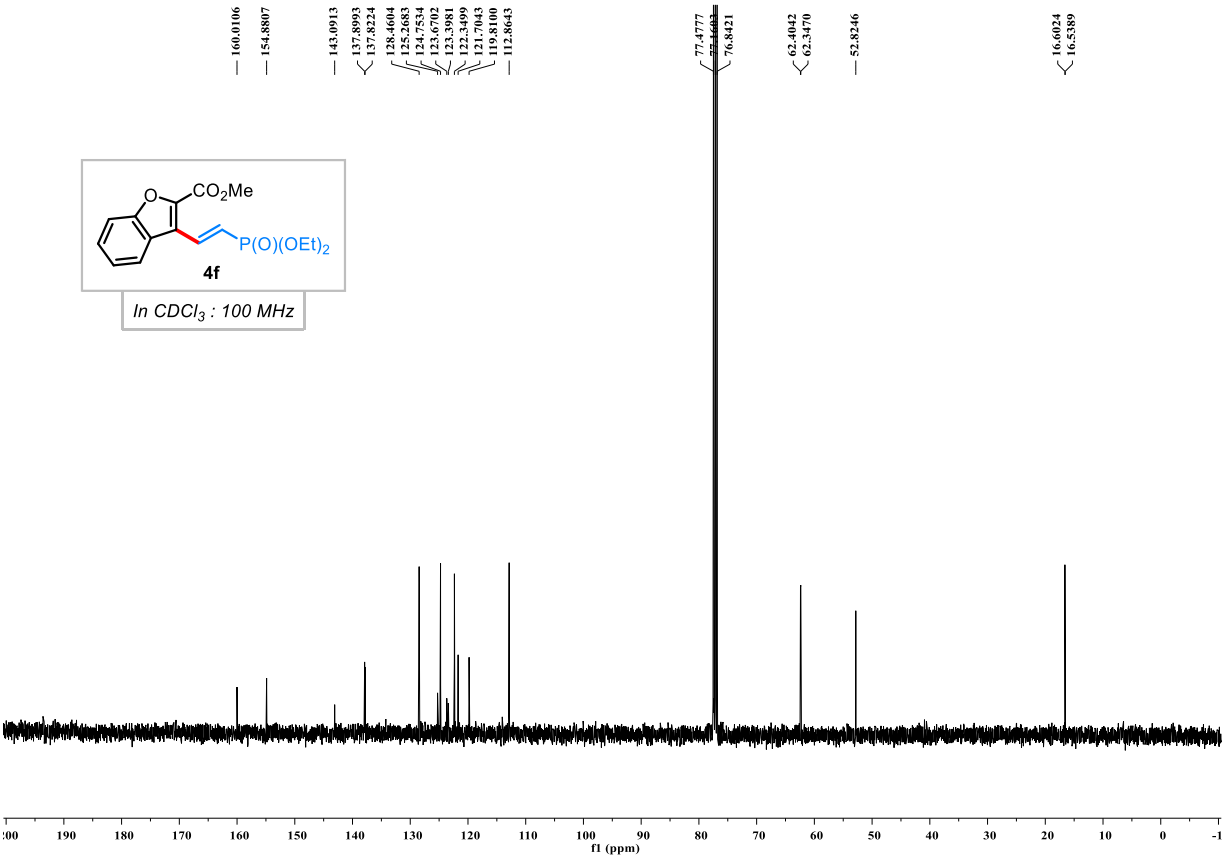
4e

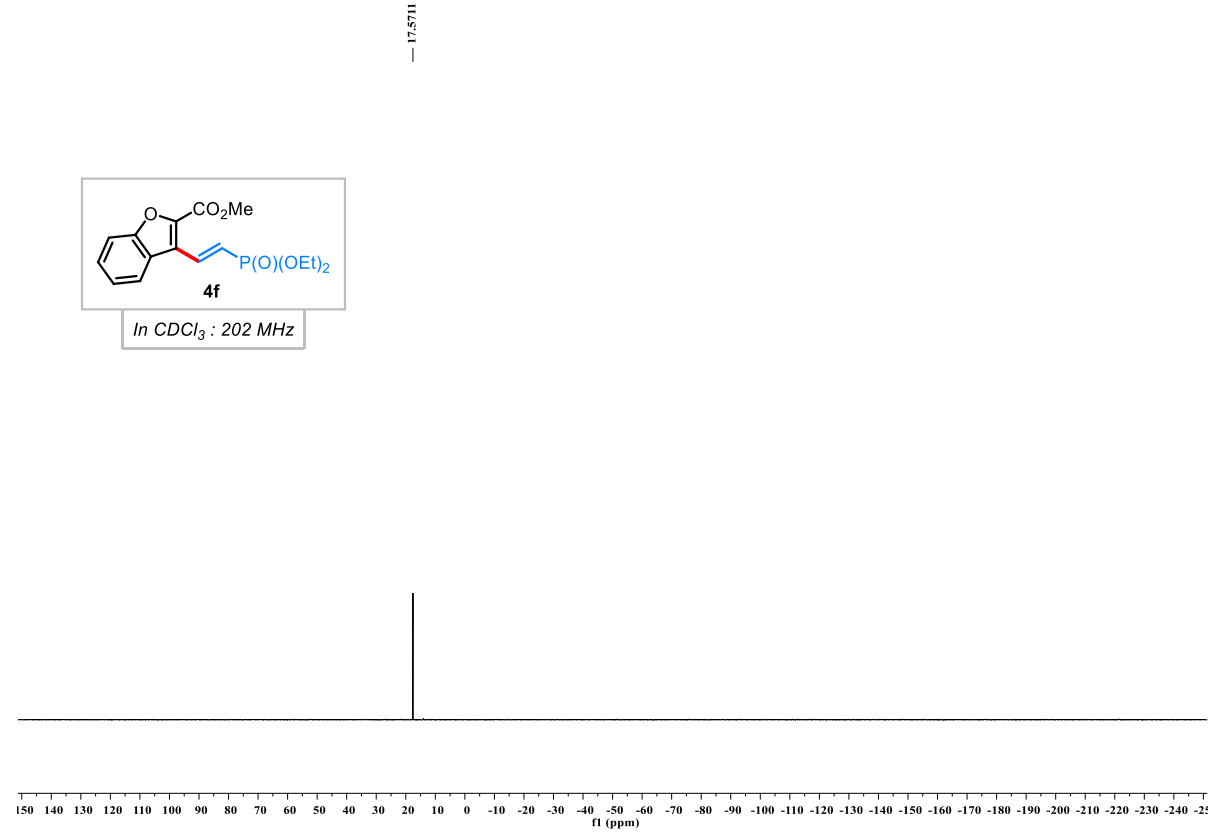
In $CDCl_3$: 100 MHz

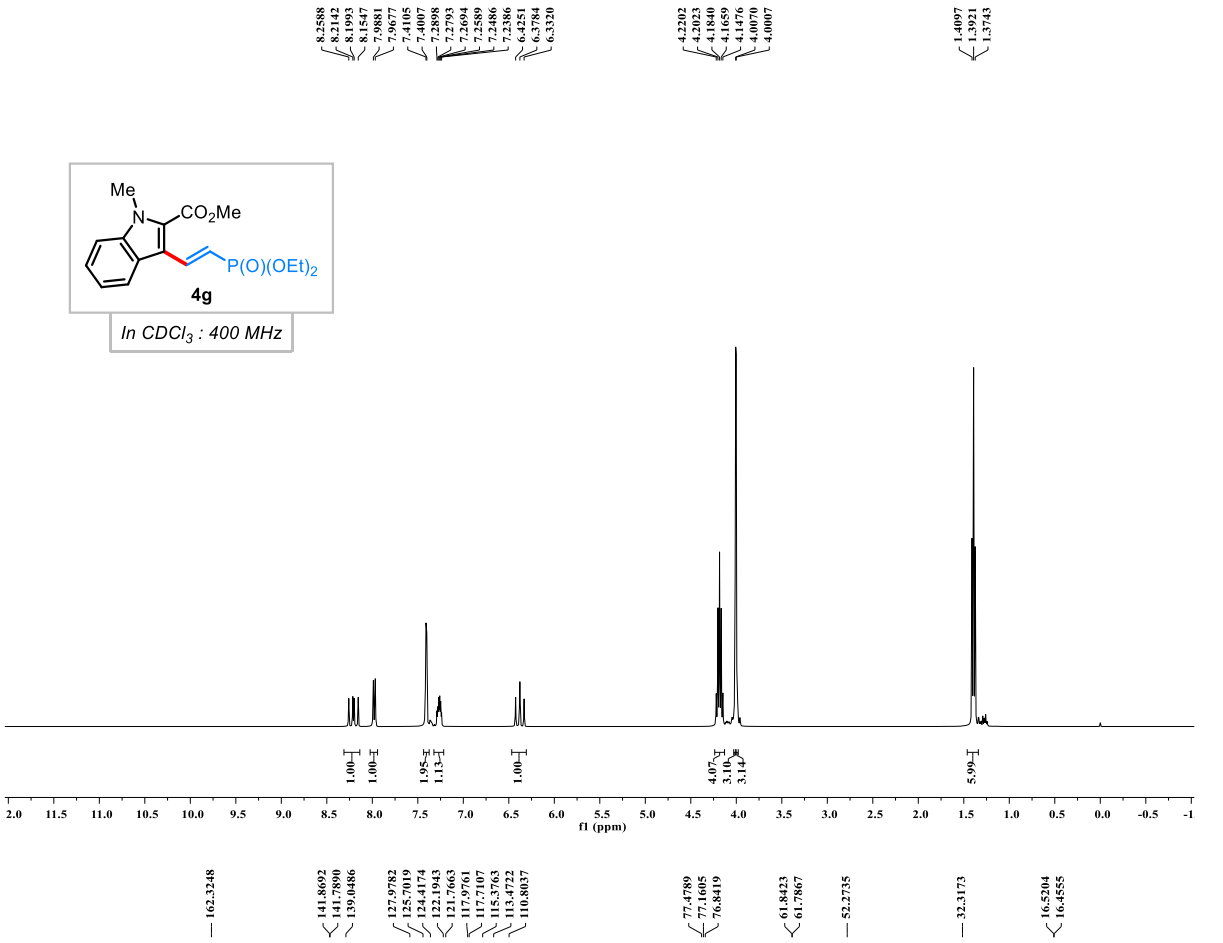


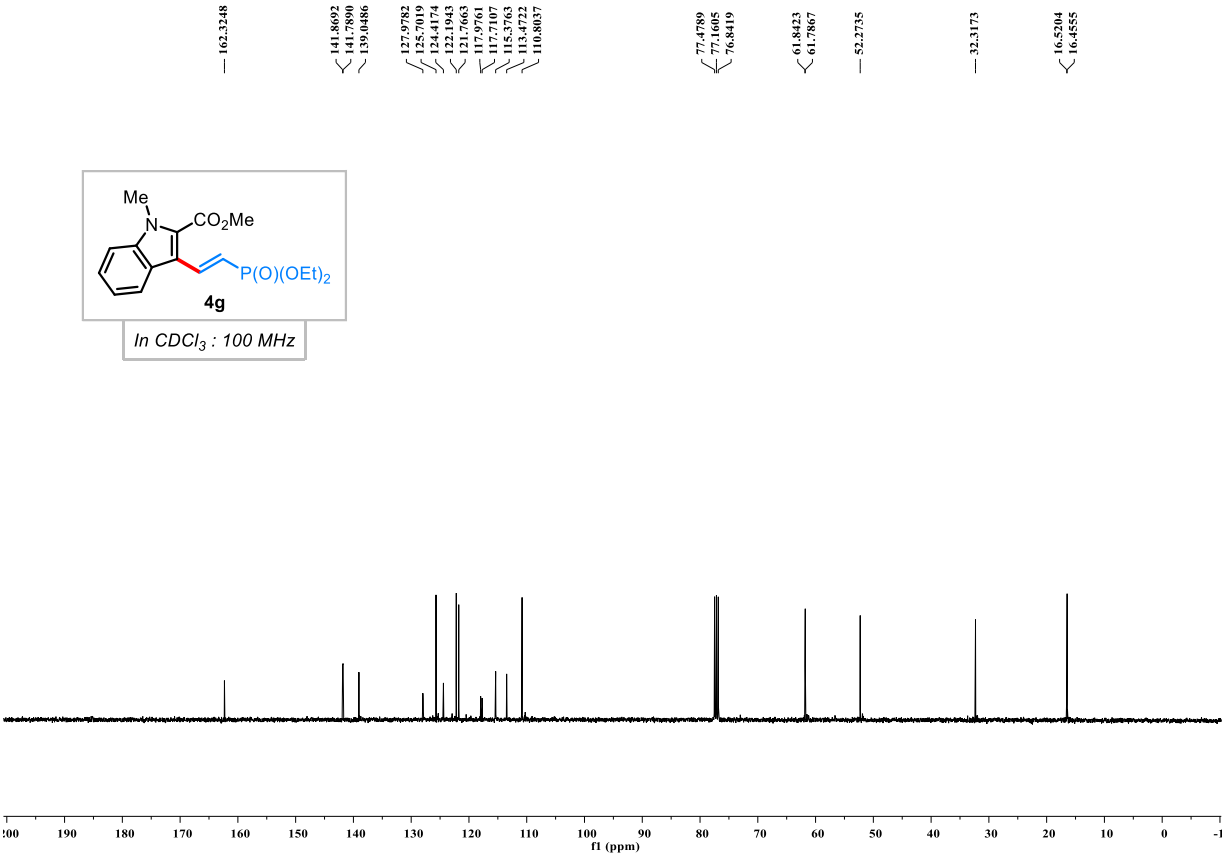

17.5703
CO2Me
S
P(O)(OEt)2
4e
In CDCl3 : 202 MHz
f1 (ppm)
8.2733
8.2284
8.2152
8.1703
7.9399
7.9199
7.6216
7.6006
7.5341
7.5310
7.5161
7.5129
7.5096
7.4950
7.4919
7.4118
7.4091
7.3918
7.3890
7.3737
7.3711
7.2598
6.6881
6.6430
6.5979
4.2333
4.2150
4.1970
4.1782
4.1604
4.0226
1.4112
1.3935
1.3758

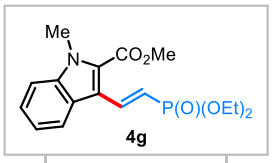

CO2Me
O
P(O)(OEt)2
4f
In CDCl3 : 400 MHz
f1 (ppm)
1.00
1.02
1.01
1.04
1.03
1.00
3.97
2.98
6.07


160.0106
154.8807
143.0913
137.8993
137.8224
128.4604
125.2683
124.7534
123.6702
123.3981
122.3499
121.7043
119.8100
112.8643
77.4777
77.1603
76.8421
62.4042
62.3470
52.8246
16.6024
16.5389
CO2Me
O
P(O)(OEt)2
4f
In CDCl3 : 100 MHz
f1 (ppm)


17.5711
CO2Me
O
P(O)(OEt)2
4f
In CDCl3 : 202 MHz
f1 (ppm)

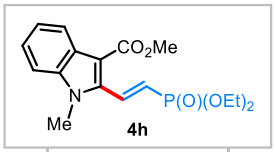

8.2588
8.2142
8.1993
8.1547
7.9881
7.9677
7.4105
7.4007
7.2898
7.2793
7.2694
7.2589
7.2486
7.2386
6.4251
6.3784
6.3320
4.2202
4.2023
4.1840
4.1659
4.1476
4.0070
4.0007
1.4097
1.3921
1.3743
Me
N
CO2Me
P(O)(OEt)2
4g
In CDCl3 : 400 MHz
1.00
1.00
1.95
1.13
1.00
4.07
3.10
3.14
5.99
f1 (ppm)


162.3248
141.8692
141.7890
139.0486
127.9782
125.7019
124.4174
122.1943
121.7663
117.9761
117.7107
115.5763
113.4722
110.8037
77.4789
77.1605
76.8419
61.8423
61.7867
52.2735
32.3173
16.5204
16.4555
Me
N
CO2Me
P(O)(OEt)2
4g
In CDCl3 : 100 MHz
f1 (ppm)



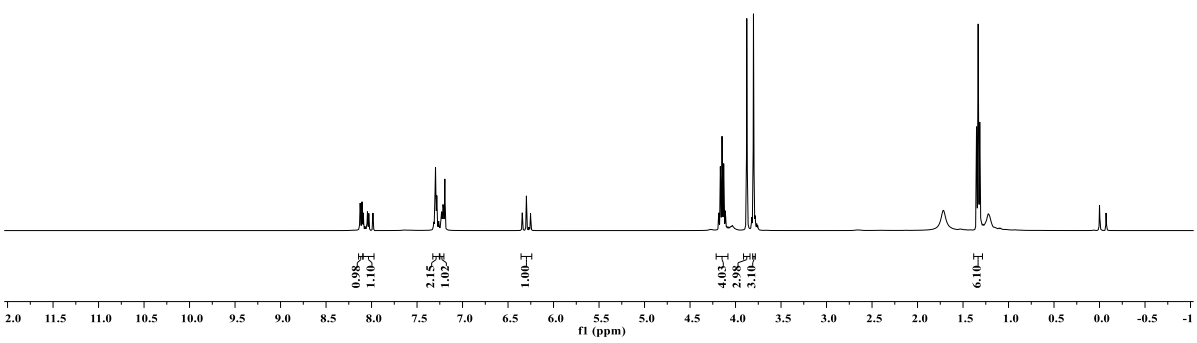
4g

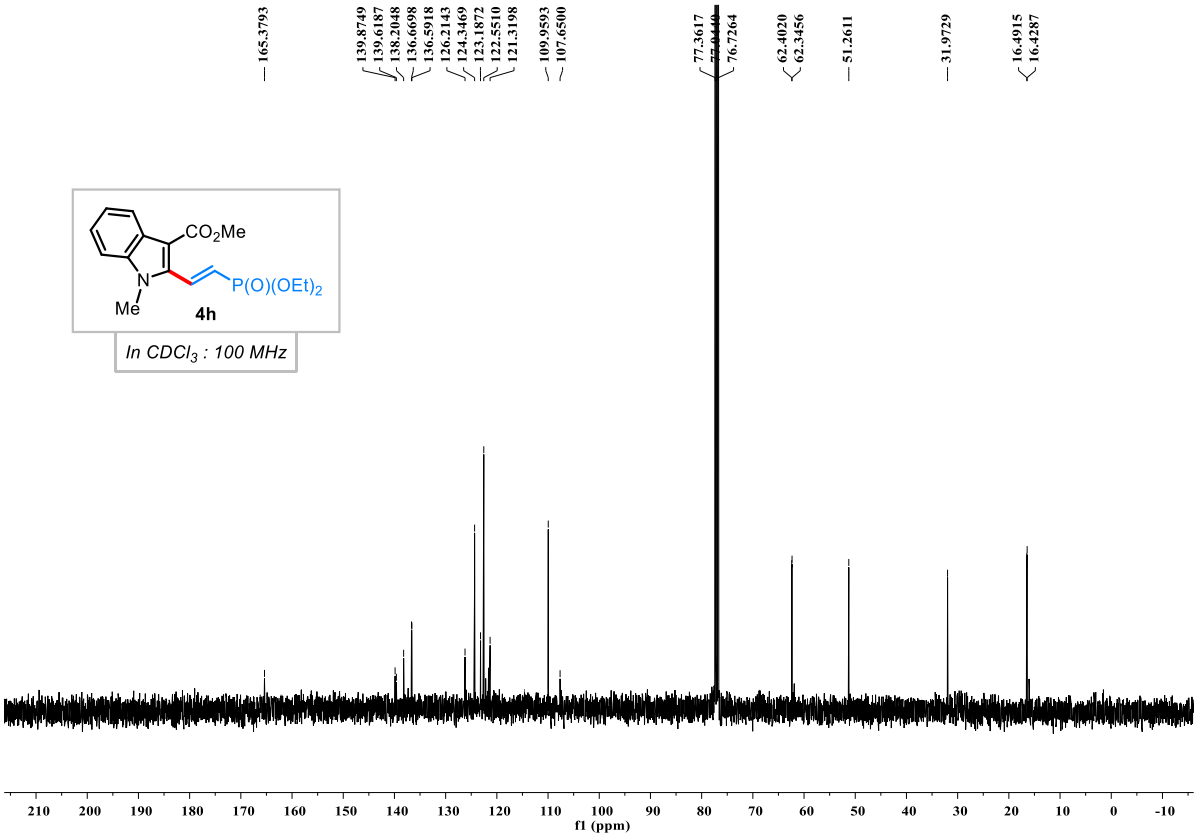
In $CDCl_3$: 202 MHz

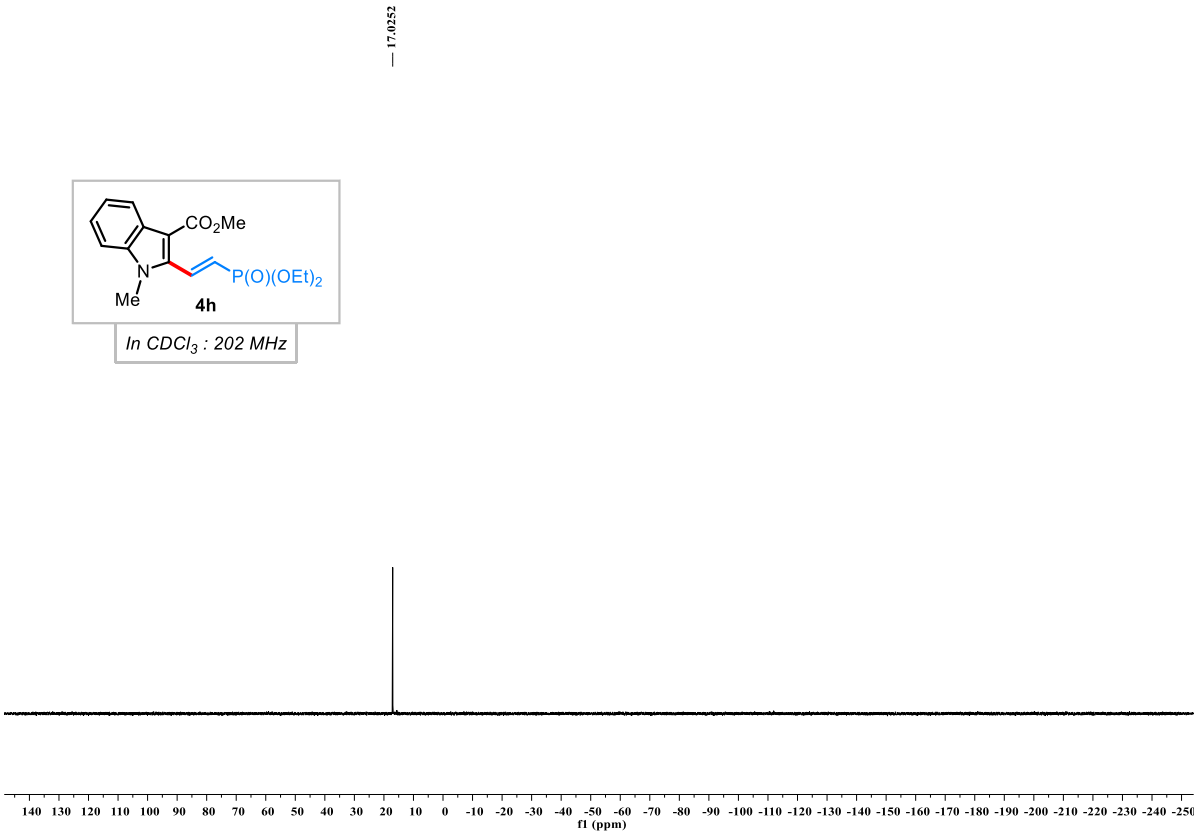


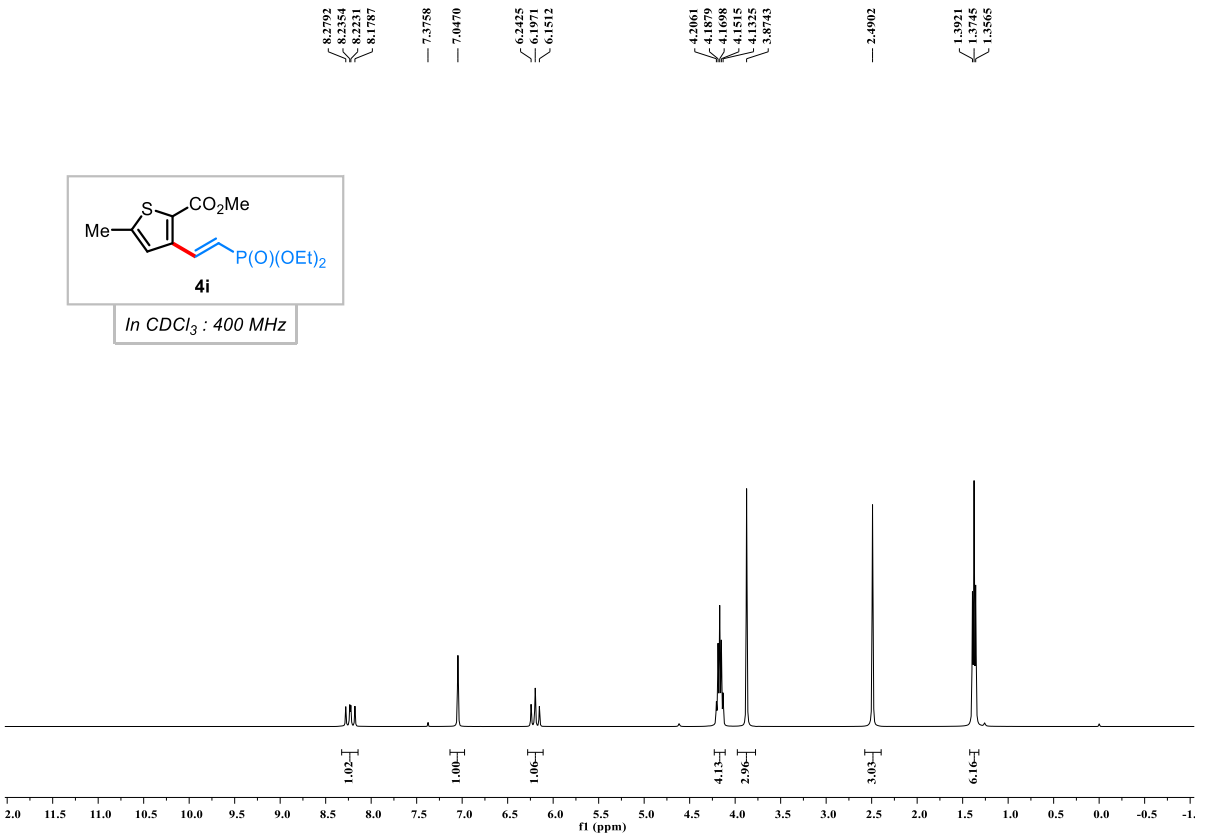
4h

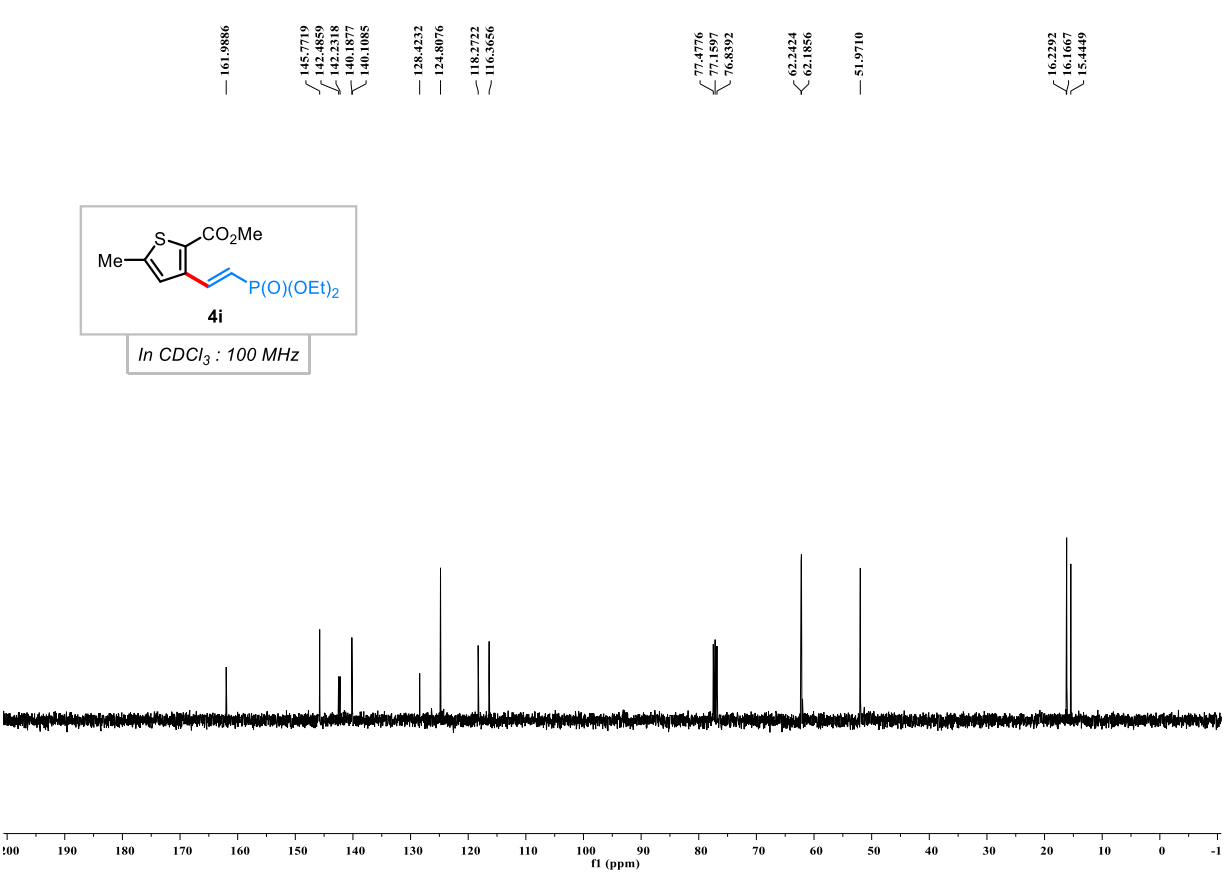
In $CDCl_3$: 400 MHz

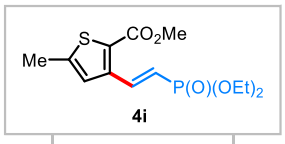



165.3793
139.8749
139.6187
138.2048
136.6698
136.5918
126.2143
124.3469
123.1872
122.5510
121.3198
109.9593
107.6500
77.3617
77.0440
76.7264
62.4020
62.3456
51.2611
31.9729
16.4915
16.4287
CO2Me
N
Me
P(O)(OEt)2
4h
In CDCl3 : 100 MHz
f1 (ppm)


17.0252
CO2Me
N
Me
P(O)(OEt)2
4h
In CDCl3 : 202 MHz
f1 (ppm)

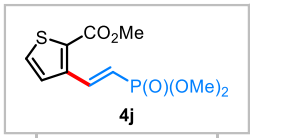

8.2792
8.2354
8.2231
8.1787
7.3758
7.0470
6.2425
6.1971
6.1512
4.2061
4.1879
4.1698
4.1515
4.1325
3.8743
2.4902
1.3921
1.3745
1.3565
Me
S
CO2Me
P(O)(OEt)2
4i
In CDCl3 : 400 MHz
1.02
1.00
1.06
4.13
2.96
3.03
6.16
f1 (ppm)


161.9886
145.7719
142.4859
142.2318
140.1877
140.1085
128.4232
124.8076
118.2722
116.3656
77.4776
77.1597
76.8392
62.2424
62.1856
51.9710
16.2292
16.1667
15.4449
Me
S
CO2Me
P(O)(OEt)2
4i
In CDCl3 : 100 MHz
f1 (ppm)



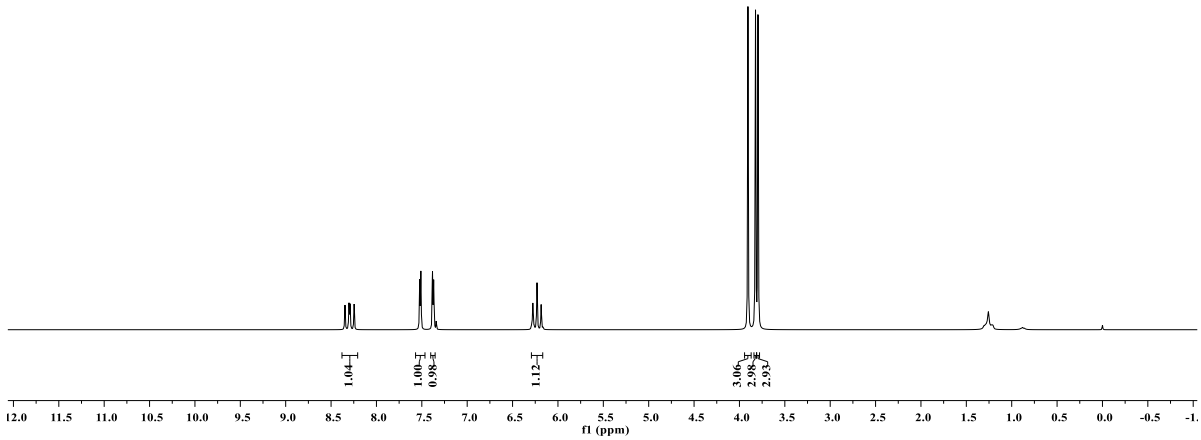
4i

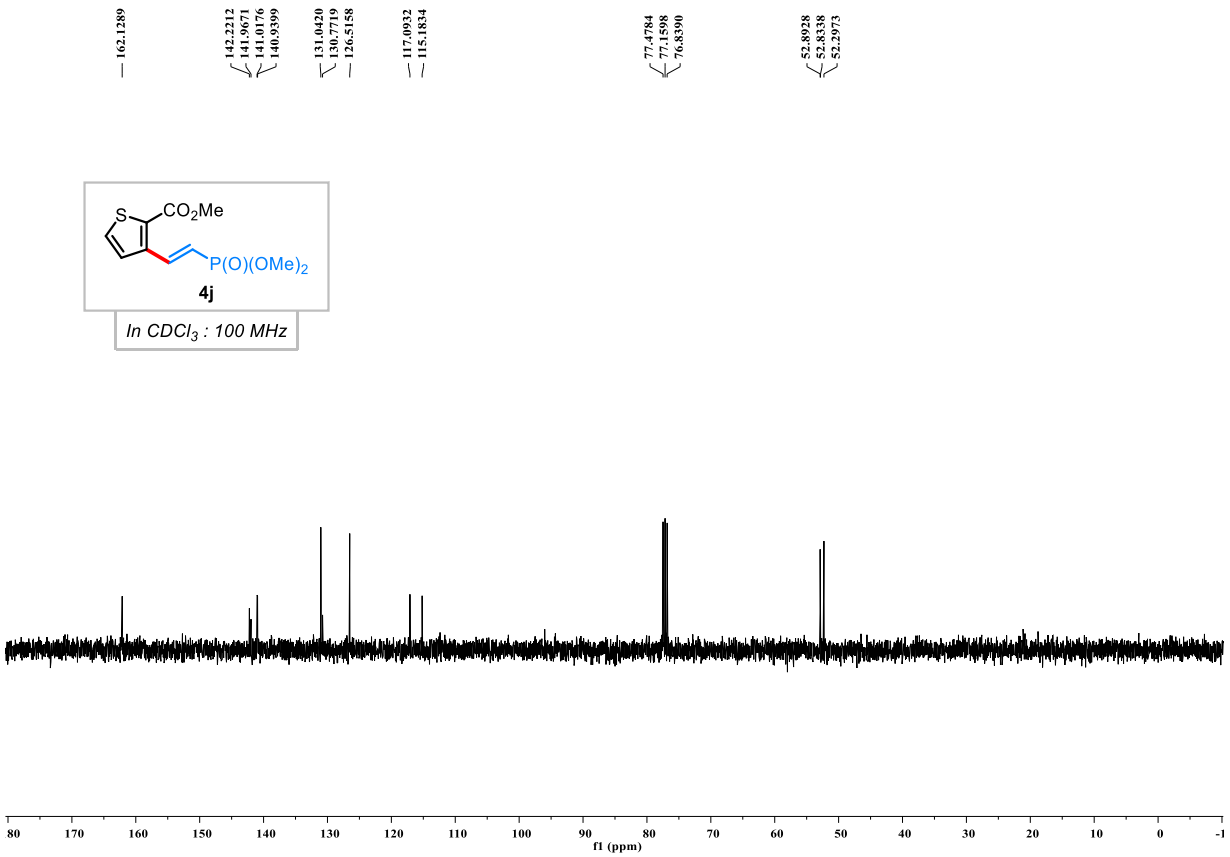
In $CDCl_3$: 202 MHz

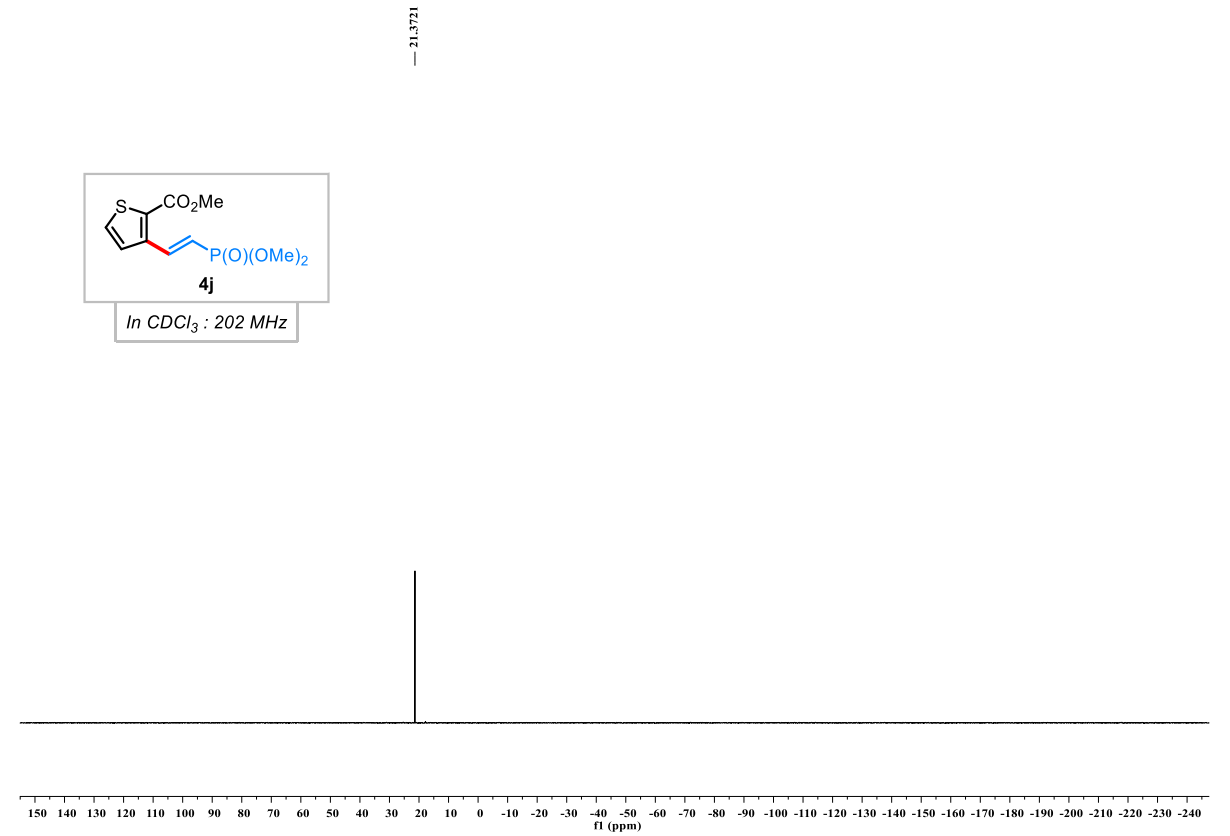


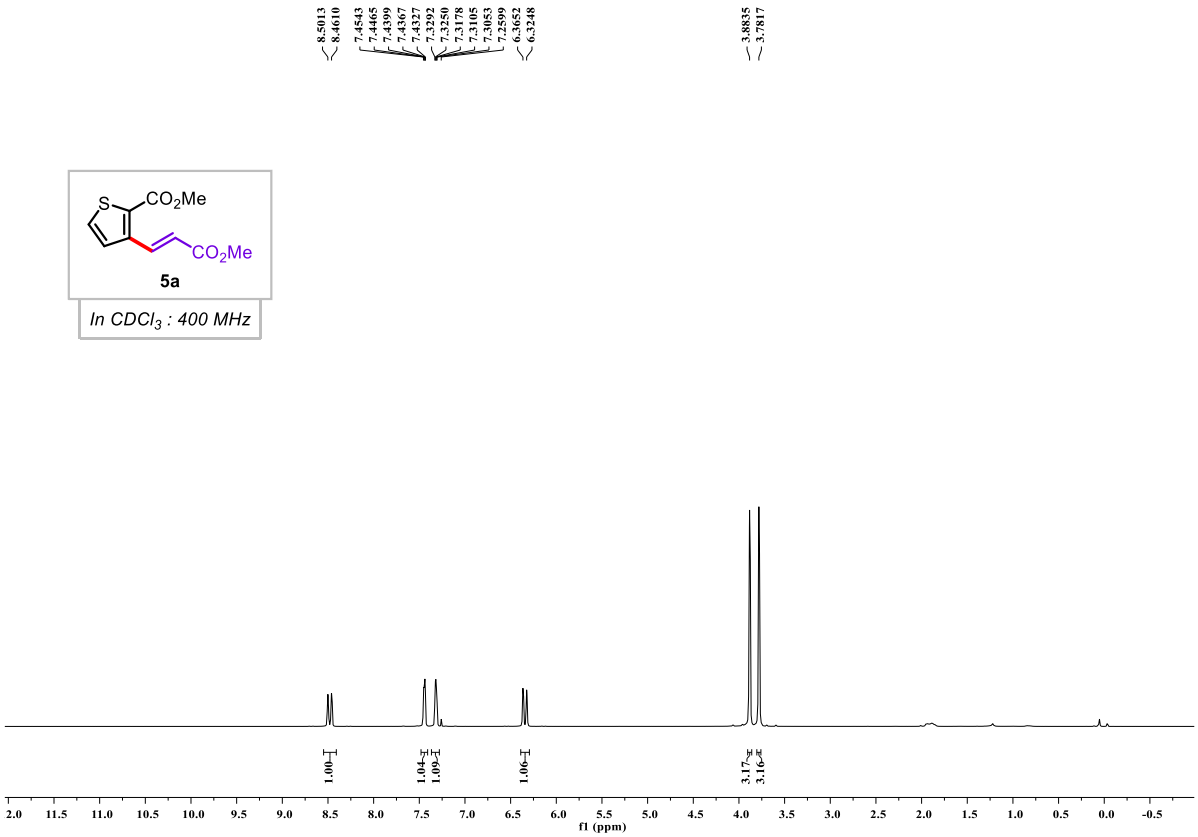
4j

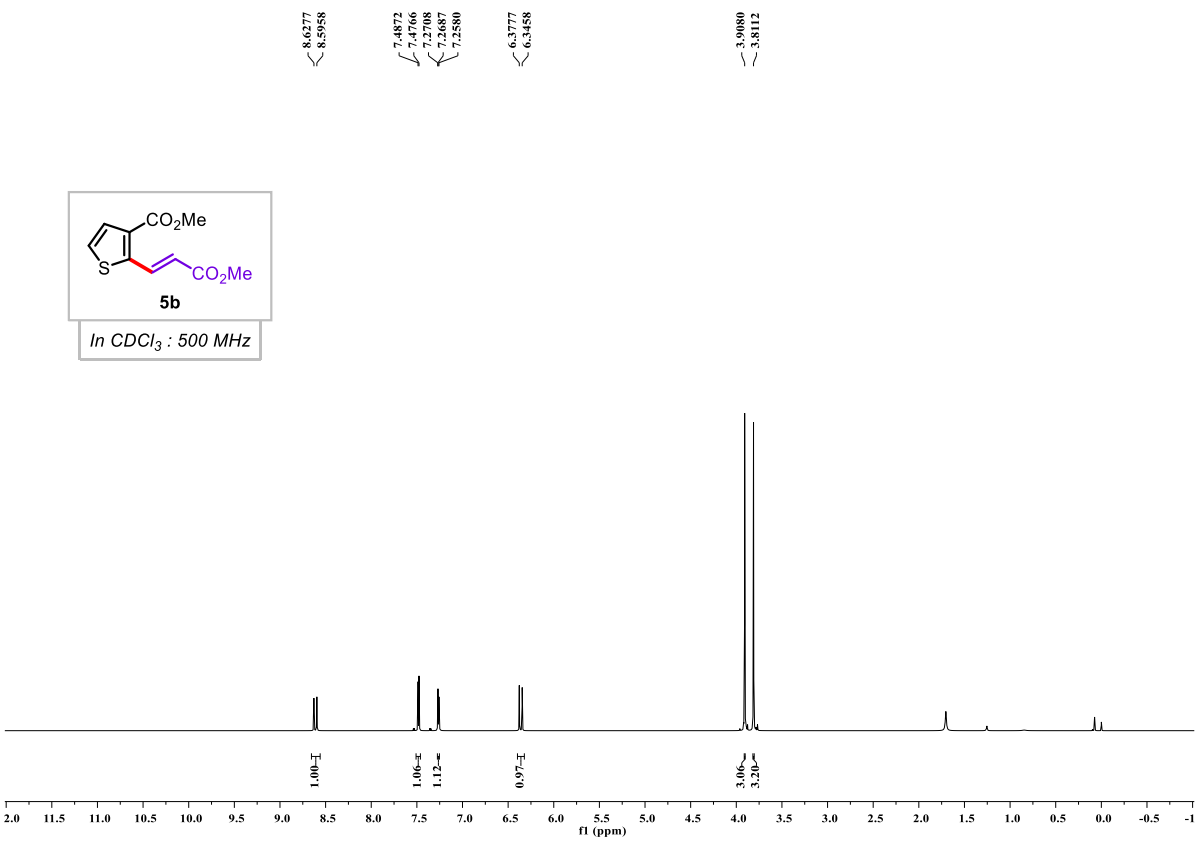
In $CDCl_3$: 400 MHz

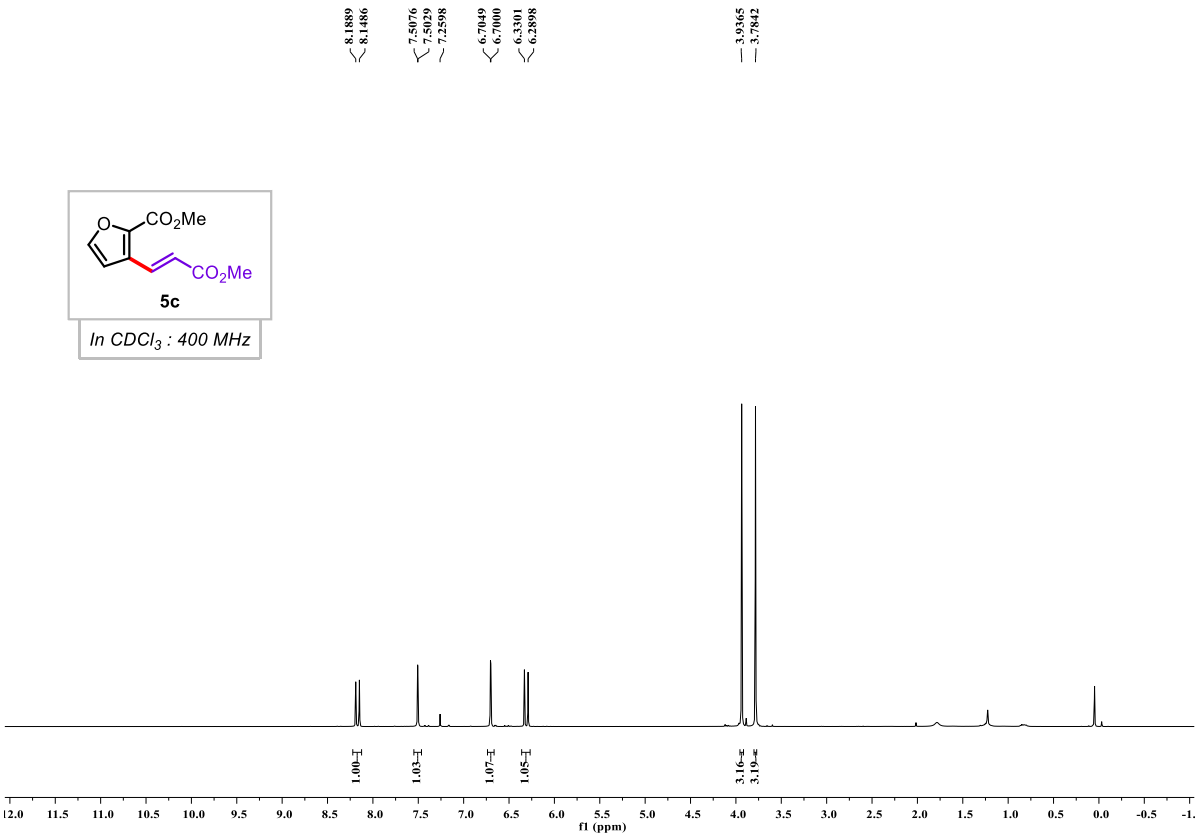


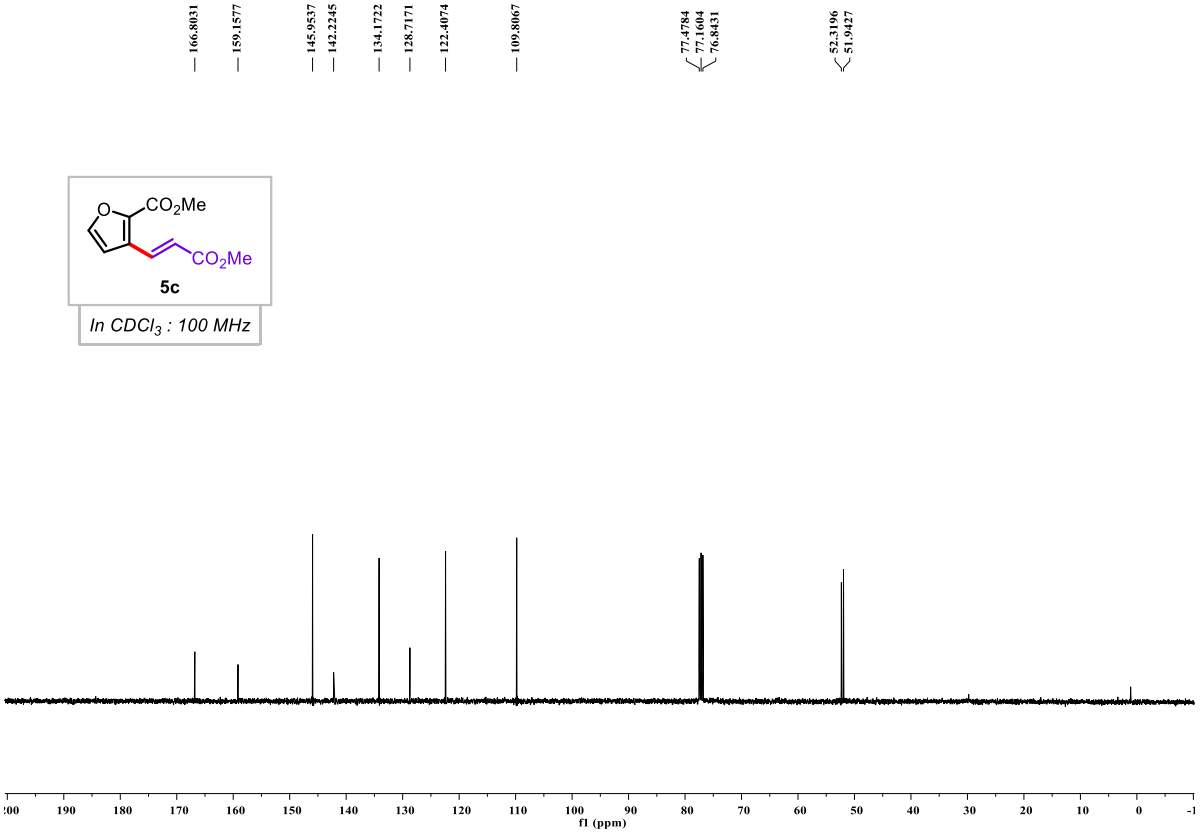

162.1289
142.2212
141.9671
141.0176
140.9399
131.0420
130.7719
126.5158
117.0932
115.1834
77.4784
77.1598
76.8390
52.8928
52.8338
52.2973
CO2Me
S
P(O)(OMe)2
4j
In CDCl3 : 100 MHz
f1 (ppm)

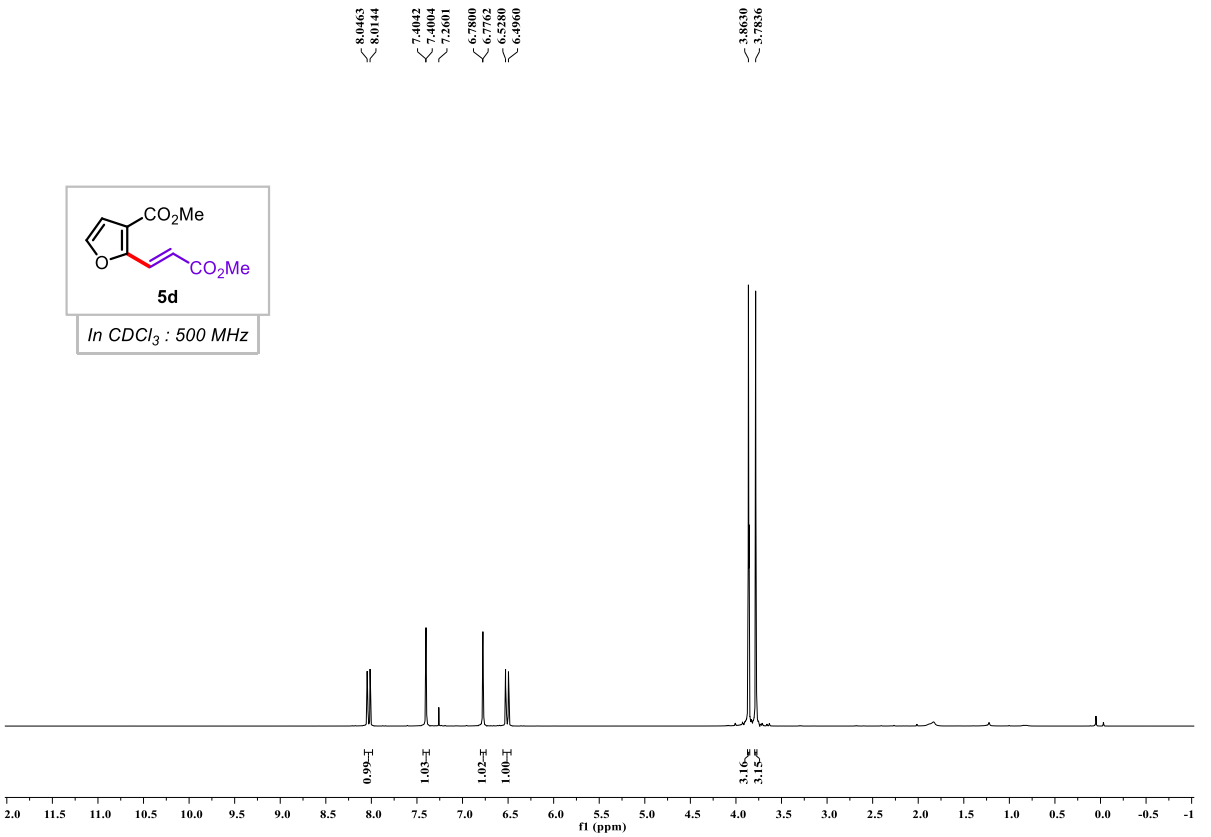

21.3721
CO2Me
S
P(O)(OMe)2
4j
In CDCl3 : 202 MHz
f1 (ppm)

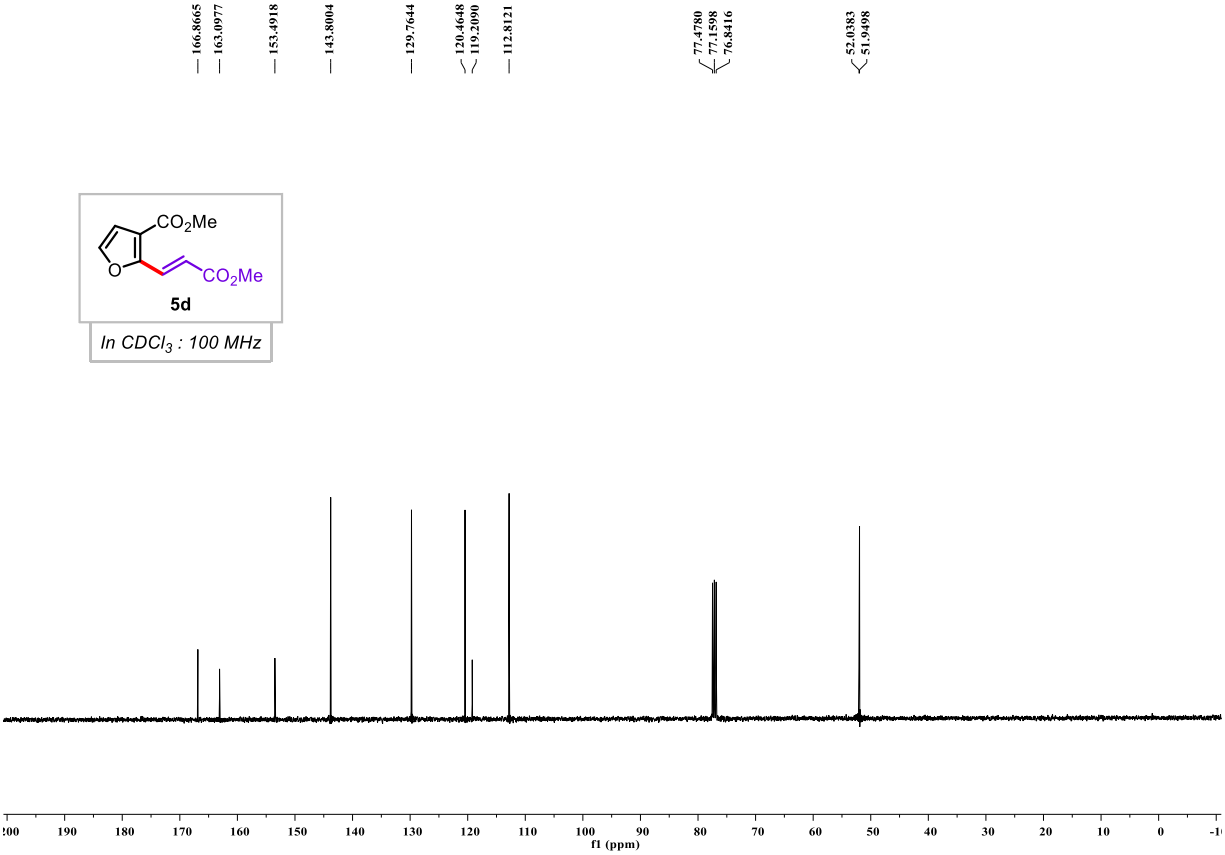

8.5013
8.4610
7.4543
7.4465
7.4399
7.4367
7.4327
7.3292
7.3250
7.3178
7.3105
7.3053
7.2599
6.3652
6.3348
3.8835
3.7817
CO2Me
CO2Me
5a
In CDCl3 : 400 MHz
1.00
1.04
1.09
1.06
3.17
3.16
f1 (ppm)

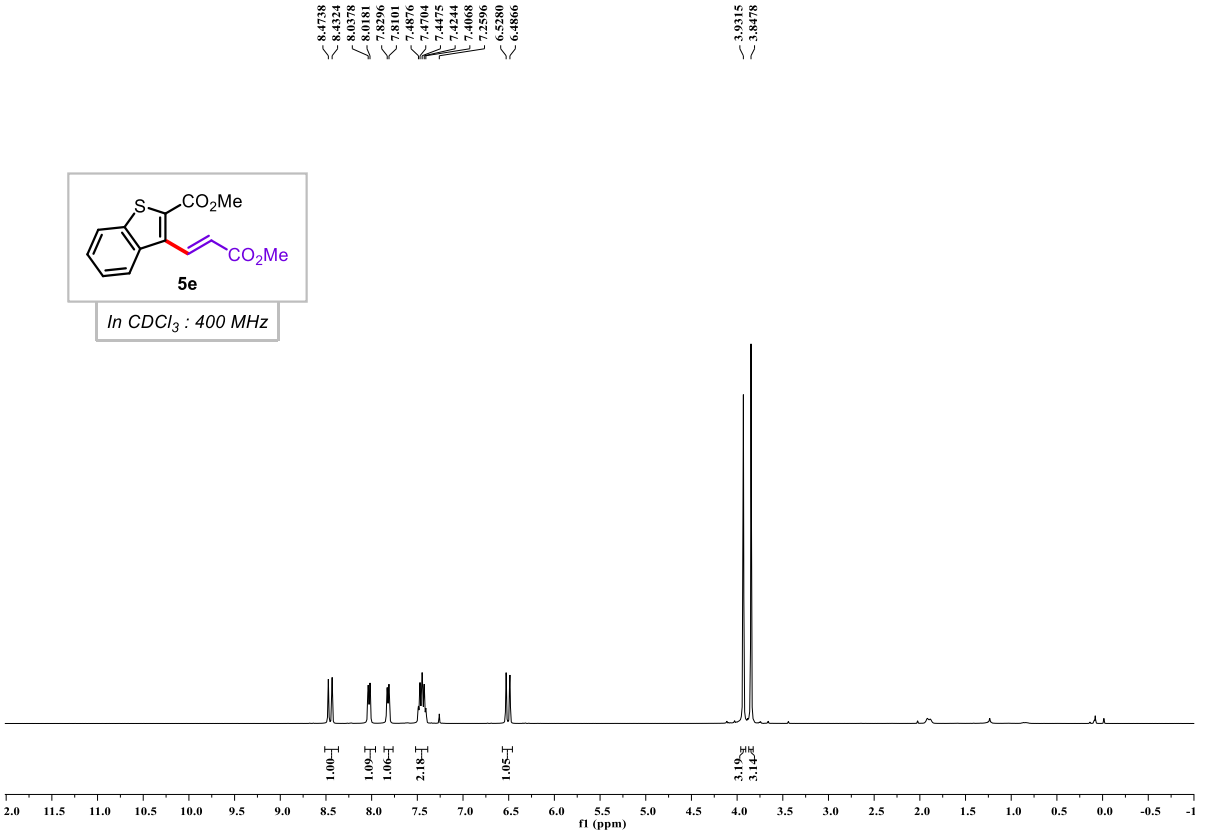

8.6277
8.5958
7.4872
7.4766
7.2708
7.2687
7.2580
6.3777
6.3458
3.9080
3.8112
CO2Me
CO2Me
5b
In CDCl3 : 500 MHz
1.00
1.06
1.12
0.97
3.06
3.20
f1 (ppm)

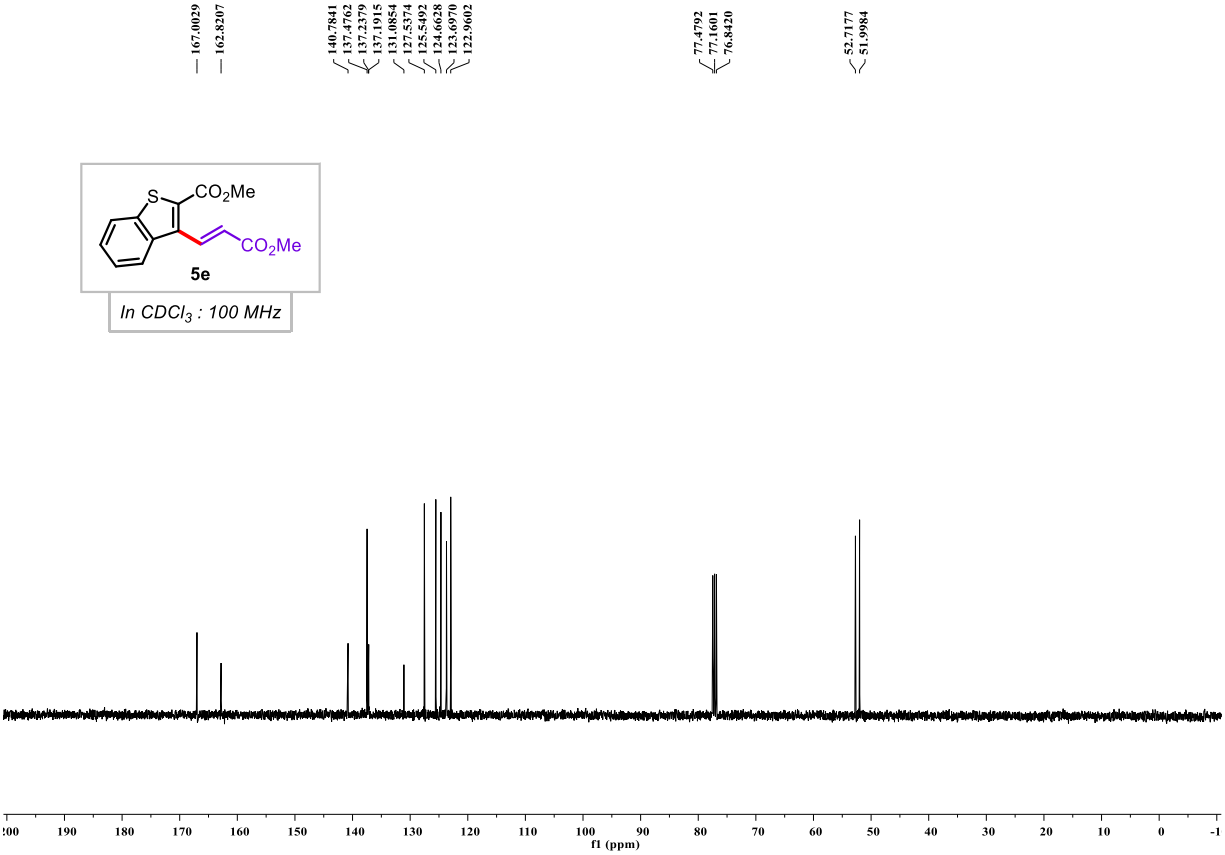

8.1889
8.1486
7.5076
7.5029
7.2598
6.7049
6.7000
6.3301
6.2898
3.9365
3.7842
CO2Me
CO2Me
5c
In CDCl3 : 400 MHz
1.00
1.03
1.07
1.05
3.16
3.19
f1 (ppm)

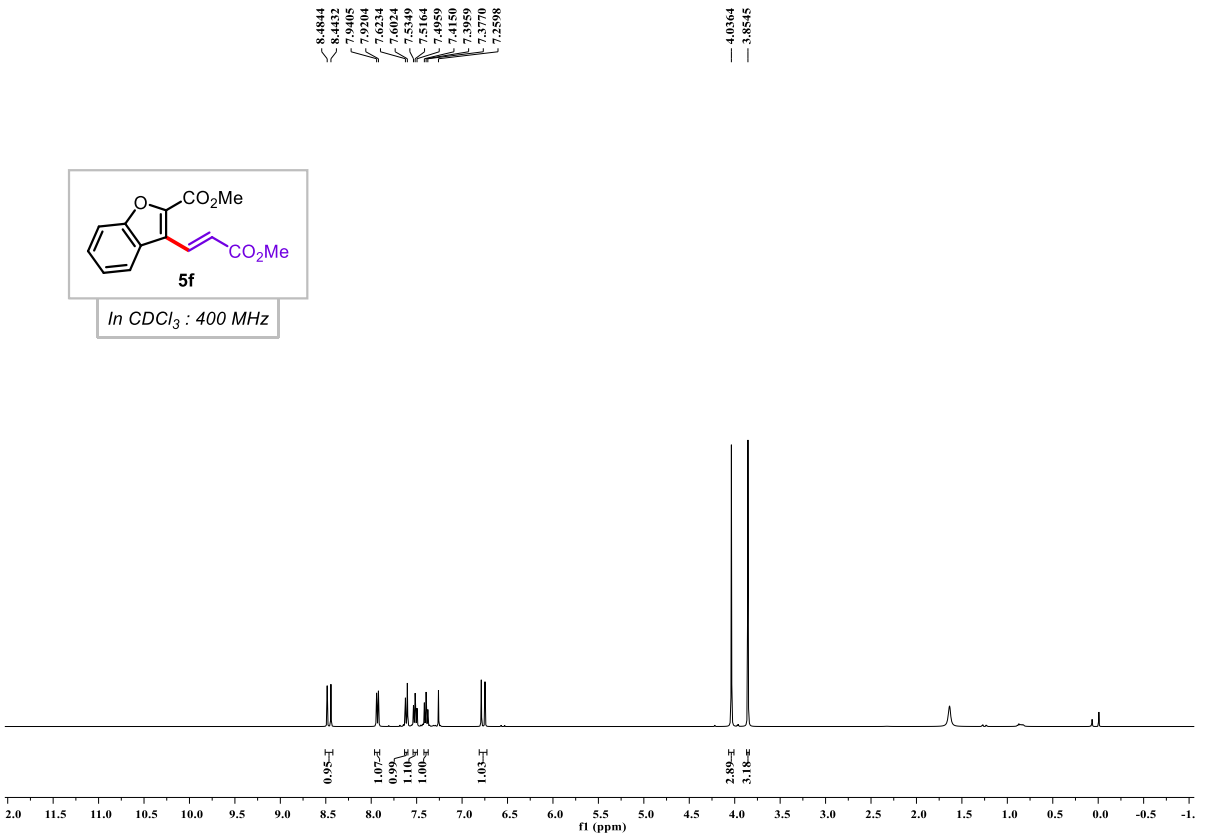

166.8031
159.1577
145.9537
142.2245
134.1722
128.7171
122.4074
109.8067
77.4784
77.1604
76.8431
52.3196
51.9427
CO2Me
CO2Me
5c
In CDCl3 : 100 MHz
f1 (ppm)

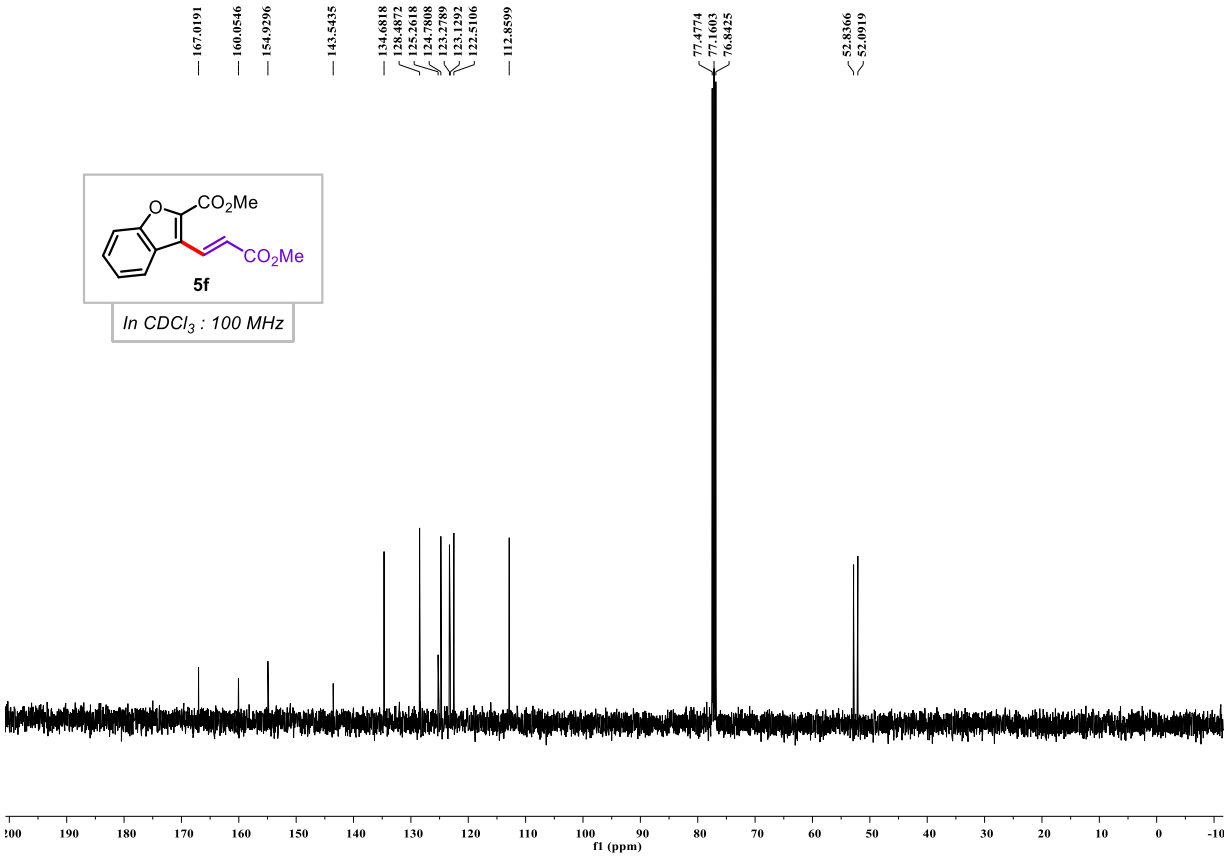

8.0463
8.0144
7.4042
7.4004
7.2601
6.7800
6.7762
6.5280
6.4960
3.8630
3.7836
CO2Me
CO2Me
5d
In CDCl3 : 500 MHz
0.99
1.03
1.02
1.00
3.16
3.15
f1 (ppm)

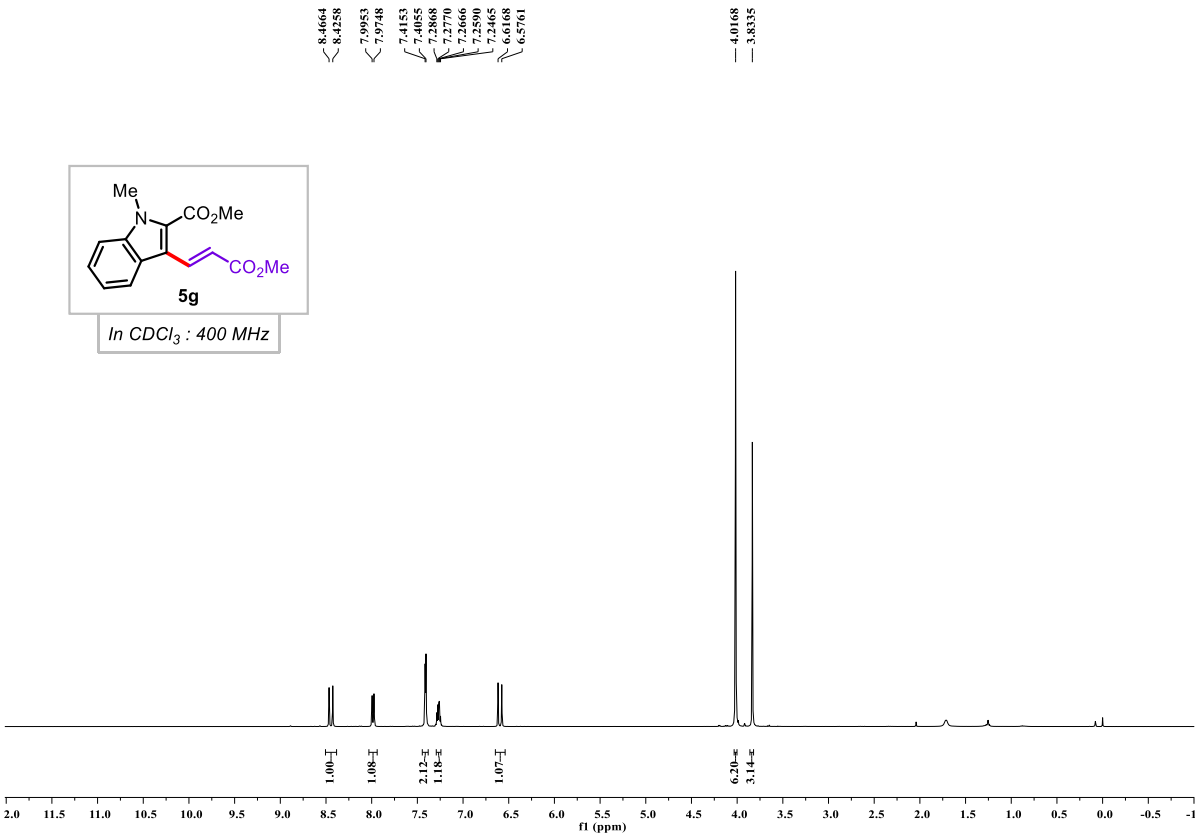

166.8665
163.0977
153.4918
143.8004
129.7644
120.4648
119.2090
112.8121
77.4780
77.1598
76.8416
52.0383
51.9498
CO2Me
CO2Me
5d
In CDCl3 : 100 MHz
f1 (ppm)

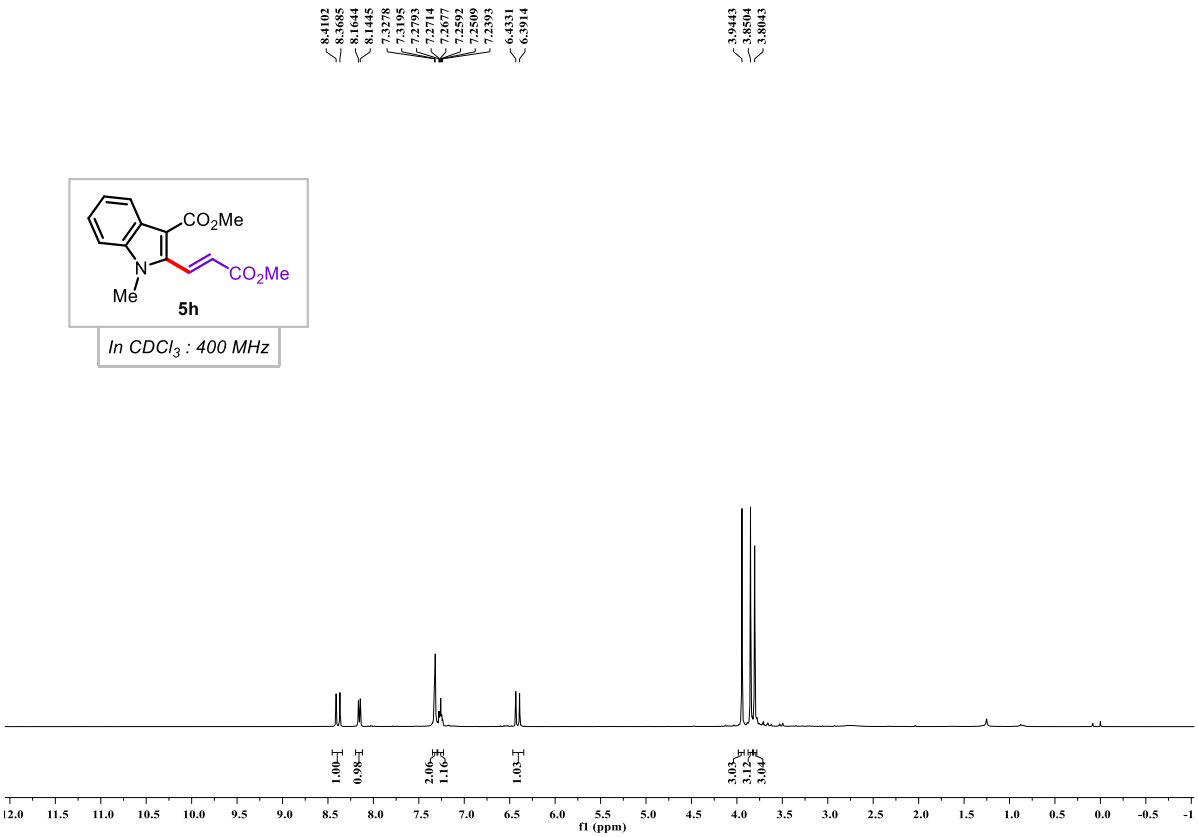

8.4738
8.4324
8.0378
8.0181
7.8296
7.8101
7.4876
7.4704
7.4475
7.4244
7.4068
7.2596
6.5280
6.4866
3.9315
3.8478
CO2Me
S
CO2Me
5e
In CDCl3 : 400 MHz
1.00
1.09
1.06
2.18
1.05
3.19
3.14
f1 (ppm)

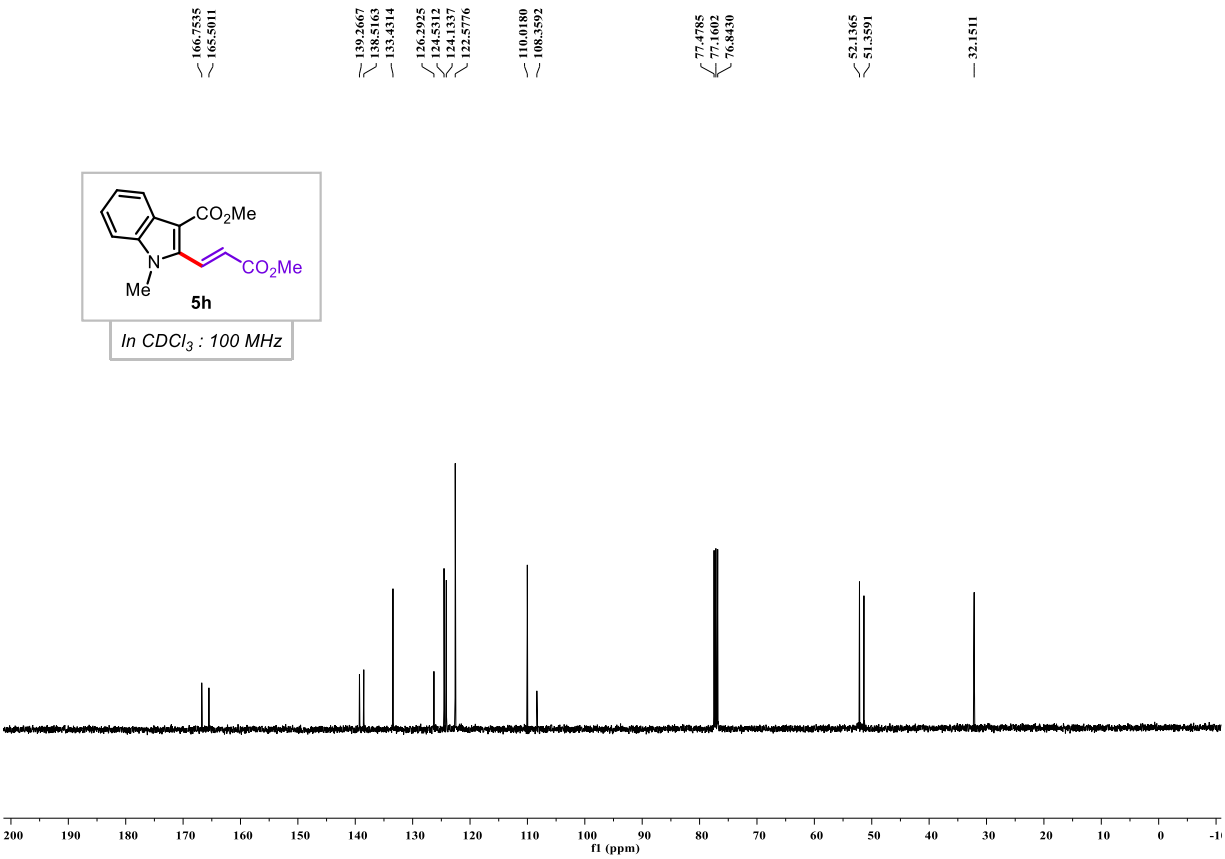

167.0029
162.8207
140.7841
137.4762
137.2379
137.1915
131.0854
127.5374
125.5492
124.6628
123.6970
122.9602
77.4792
77.1601
76.8420
52.7177
51.9984
CO2Me
S
CO2Me
5e
In CDCl3 : 100 MHz
f1 (ppm)

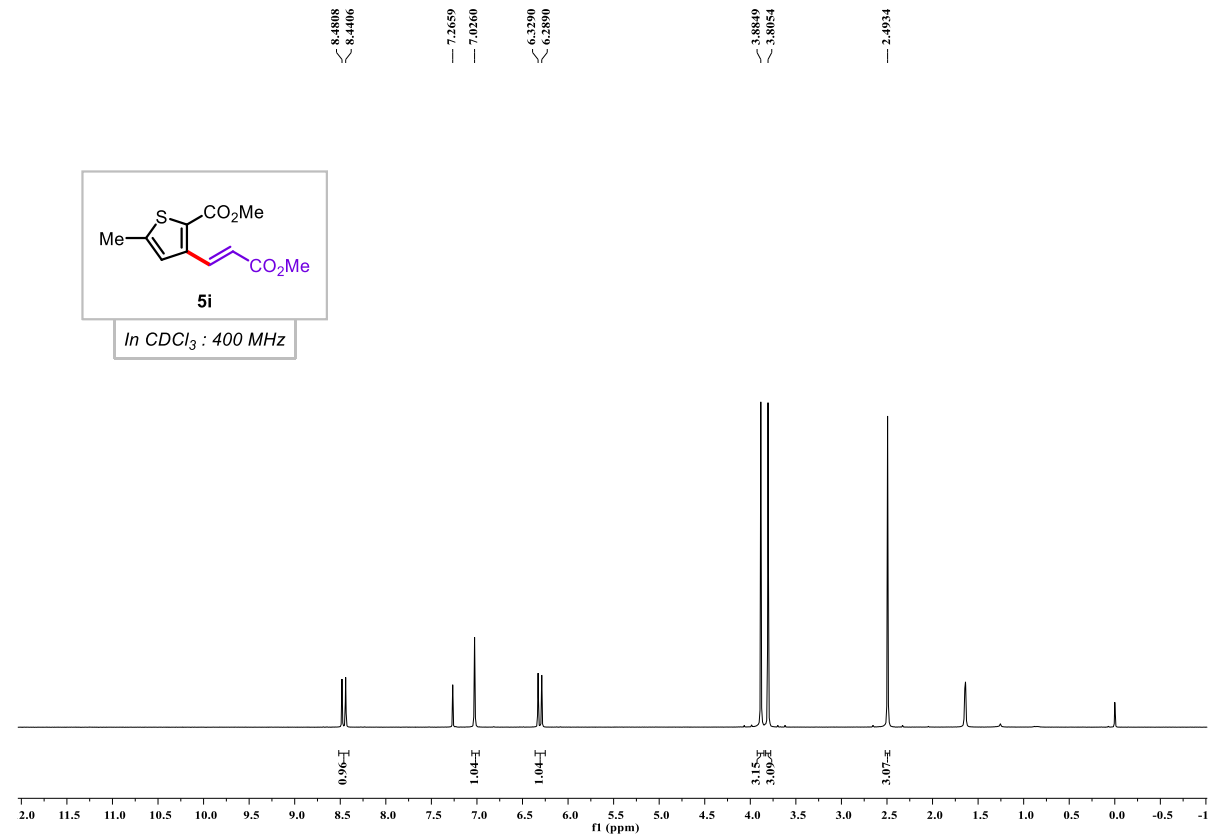

8.4844
8.4432
7.9405
7.9204
7.6234
7.6024
7.5349
7.5164
7.4959
7.4150
7.3959
7.3770
7.2598
4.0364
3.8545
CO2Me
CO2Me
5f
In CDCl3 : 400 MHz
0.95
1.07
0.99
1.10
1.00
1.03
2.89
3.18
f1 (ppm)

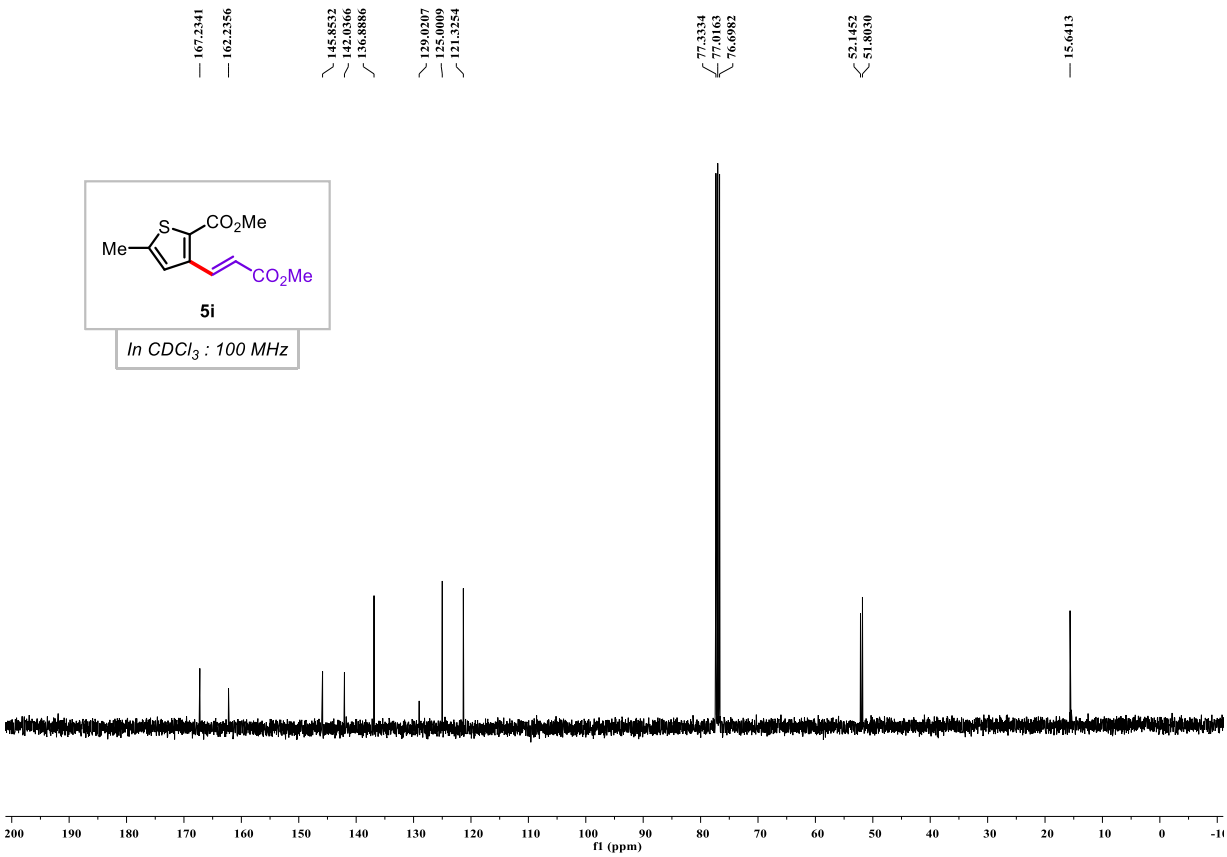

167.0191
160.0546
154.9296
143.5435
134.6818
128.4872
125.2618
124.7808
123.2789
123.1292
122.5106
112.8599
77.4774
77.1603
76.8425
52.8366
52.0919
CO2Me
CO2Me
5f
In CDCl3 : 100 MHz
f1 (ppm)

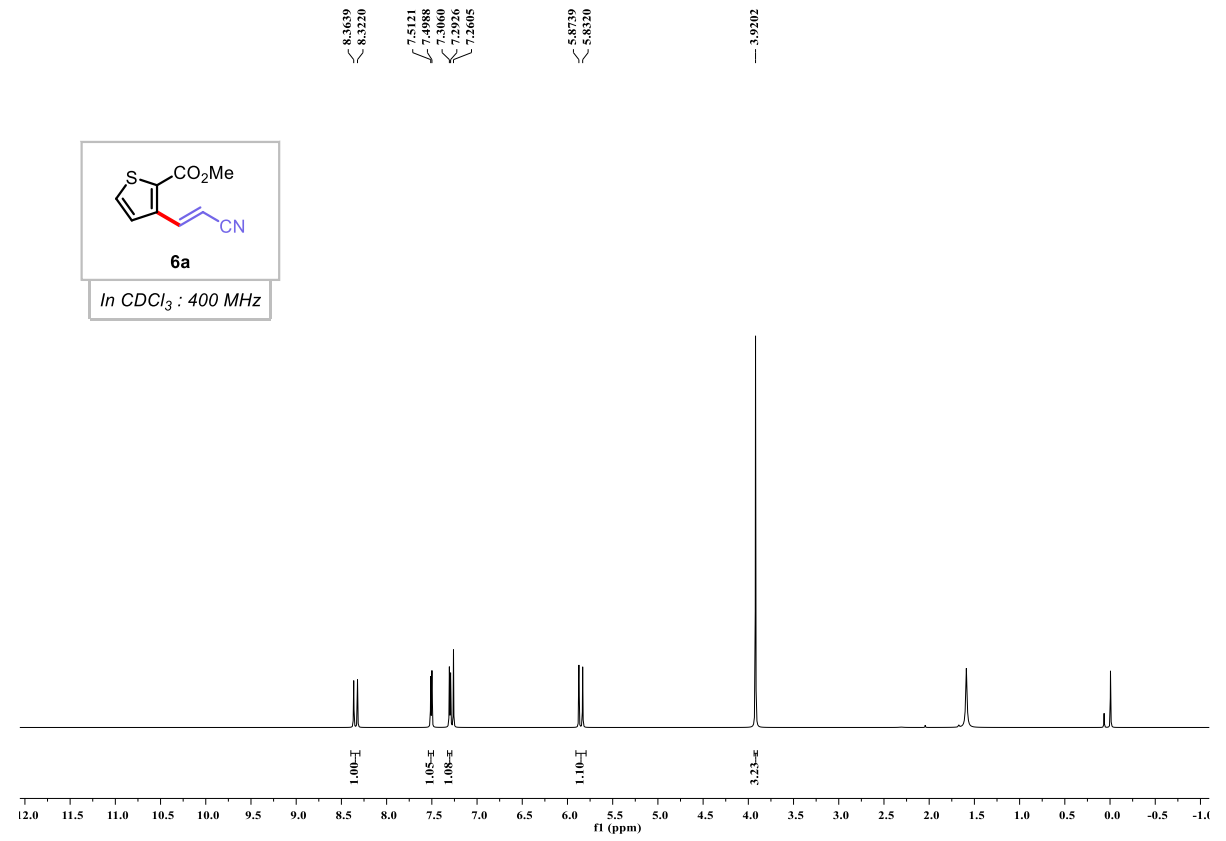

8.4664
8.4258
7.9953
7.9748
7.4153
7.4055
7.2868
7.2770
7.2666
7.2590
7.2465
6.6168
6.5761
4.0168
3.8335
Me
N
CO2Me
CO2Me
5g
In CDCl3 : 400 MHz
1.00
1.08
2.12
1.18
1.07
6.20
3.14
f1 (ppm)

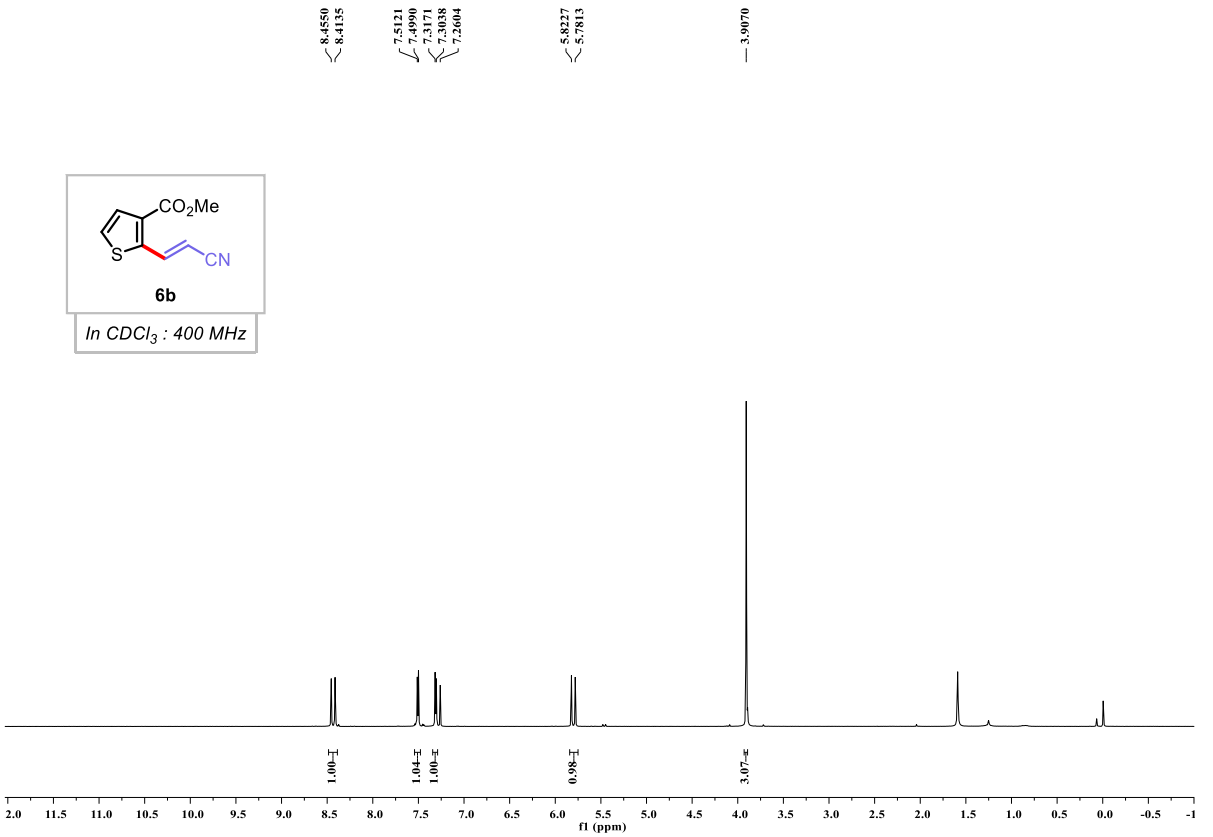

8.4102
8.3685
8.1644
8.1445
7.3278
7.3195
7.2793
7.2714
7.2677
7.2592
7.2509
7.2393
6.4331
6.3914
3.9443
3.8504
3.8043
CO2Me
N
Me
CO2Me
5h
In CDCl3 : 400 MHz
1.00
0.98
2.06
1.16
1.03
3.03
3.12
3.04
f1 (ppm)

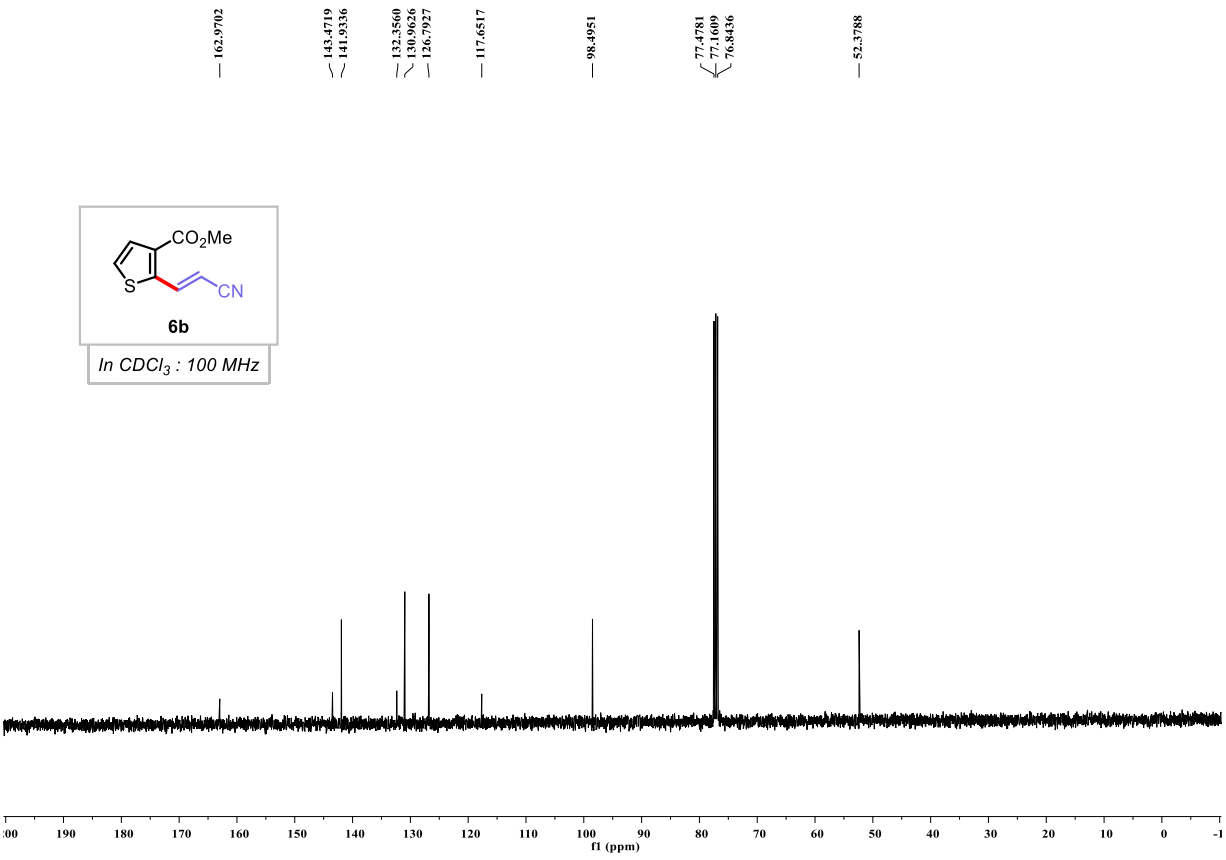

166.7535
165.5011
139.2667
138.5163
133.4314
126.2925
124.5312
124.1337
122.5776
110.0180
108.3592
77.4785
77.1602
76.8430
52.1365
51.3591
32.1511
CO2Me
CO2Me
N
Me
5h
In CDCl3 : 100 MHz
f1 (ppm)

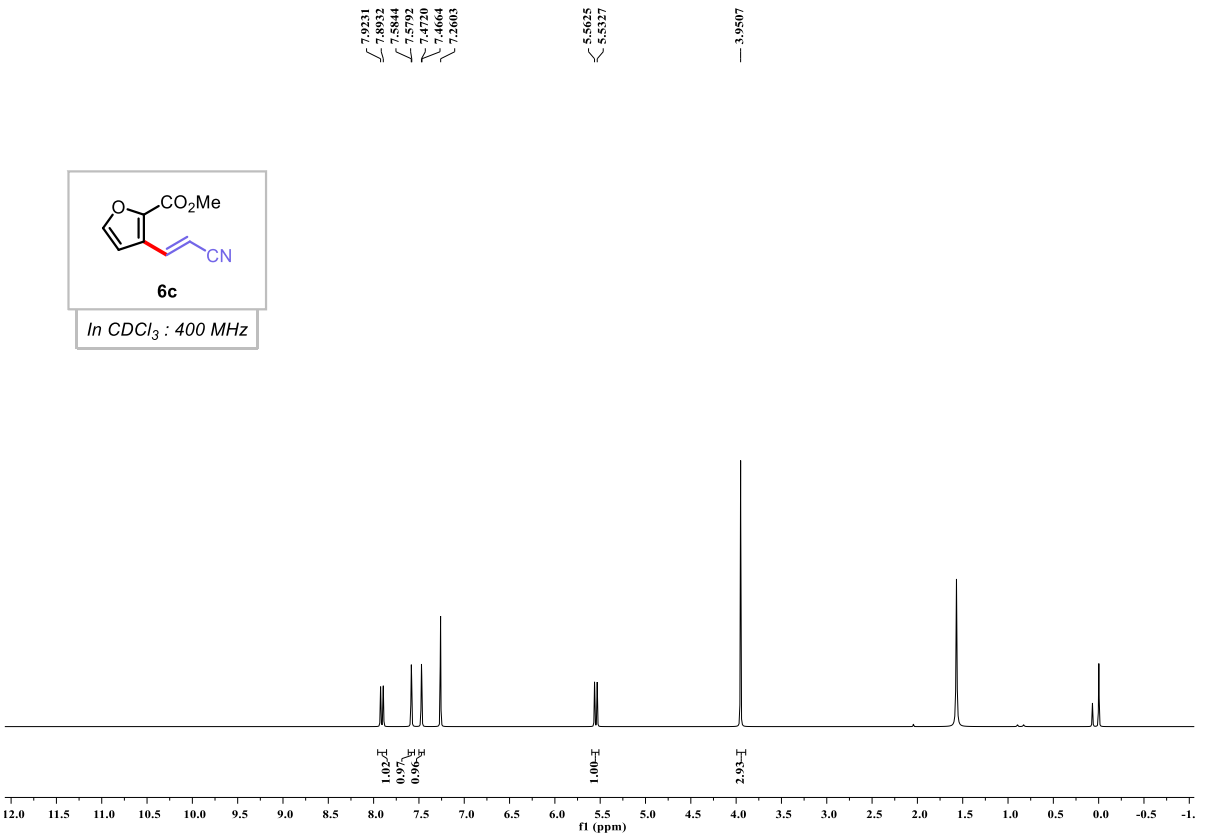

8.4808
8.4406
7.2659
7.0260
6.3290
6.2890
3.8849
3.8054
2.4934
S
CO2Me
Me
CO2Me
5i
In CDCl3 : 400 MHz
0.96
1.04
1.04
3.15
3.09
3.07
f1 (ppm)

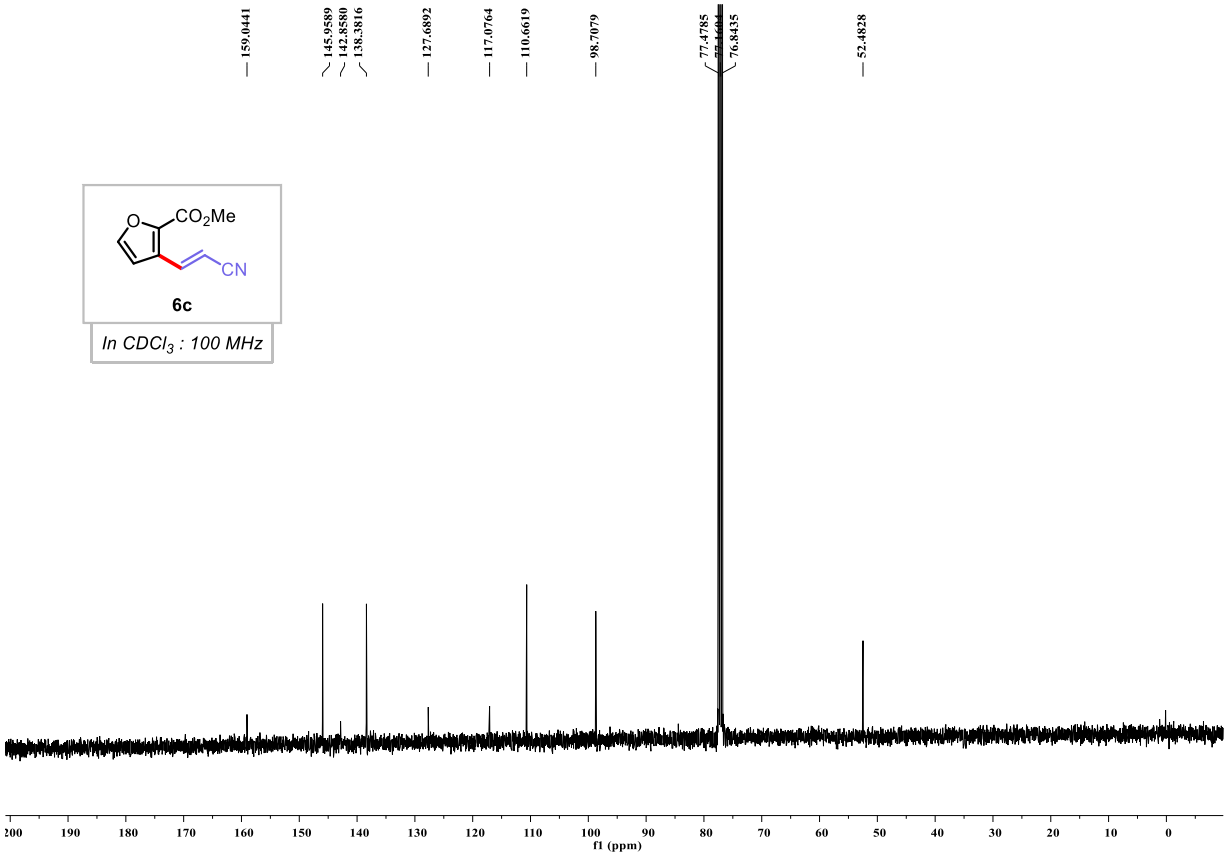

167.2341
162.2356
145.8532
142.0366
136.8886
129.0207
125.0009
121.3254
77.3334
77.0163
76.6982
52.1452
51.8030
15.6413
S
CO2Me
Me
CO2Me
5i
In CDCl3 : 100 MHz
f1 (ppm)

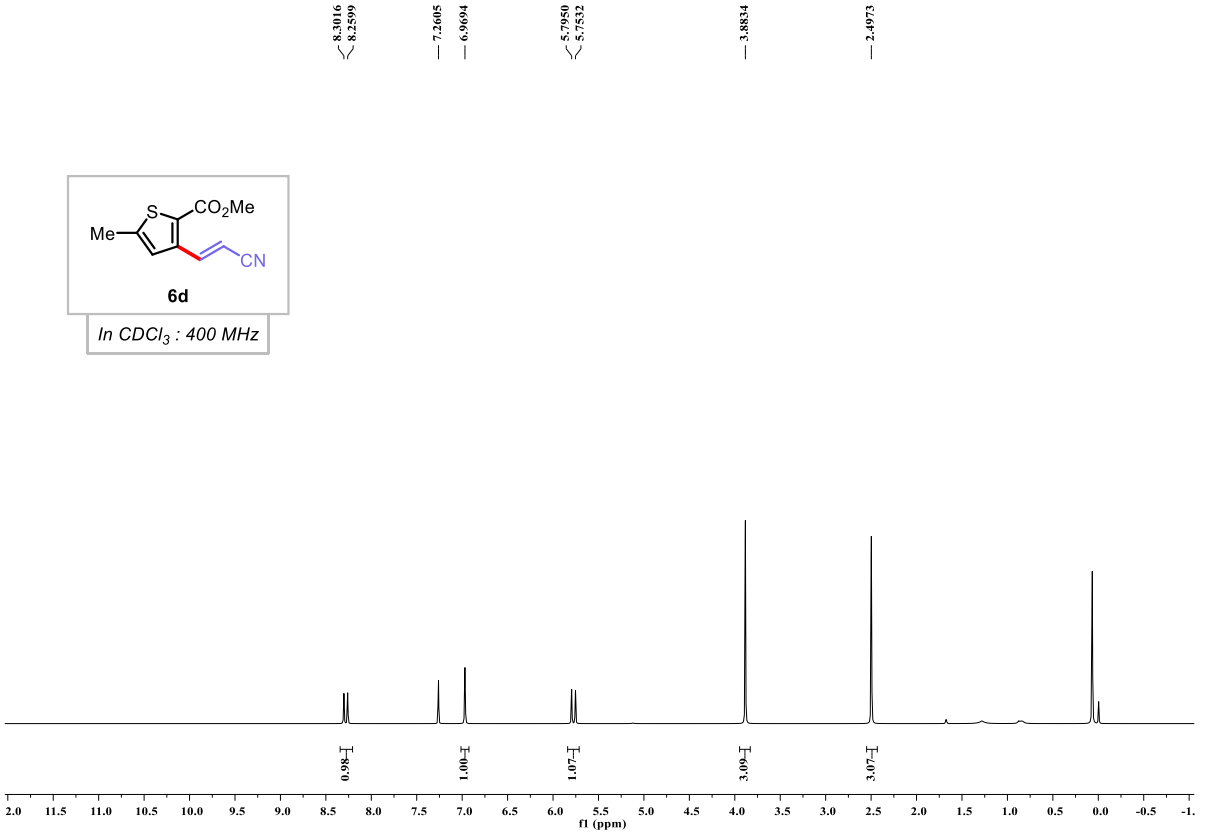

8.3639
8.3220
7.5121
7.4988
7.3060
7.2926
7.2605
5.8739
5.8320
3.9202
S
CO2Me
CN
6a
In CDCl3 : 400 MHz
1.00
1.05
1.08
1.10
3.23
f1 (ppm)

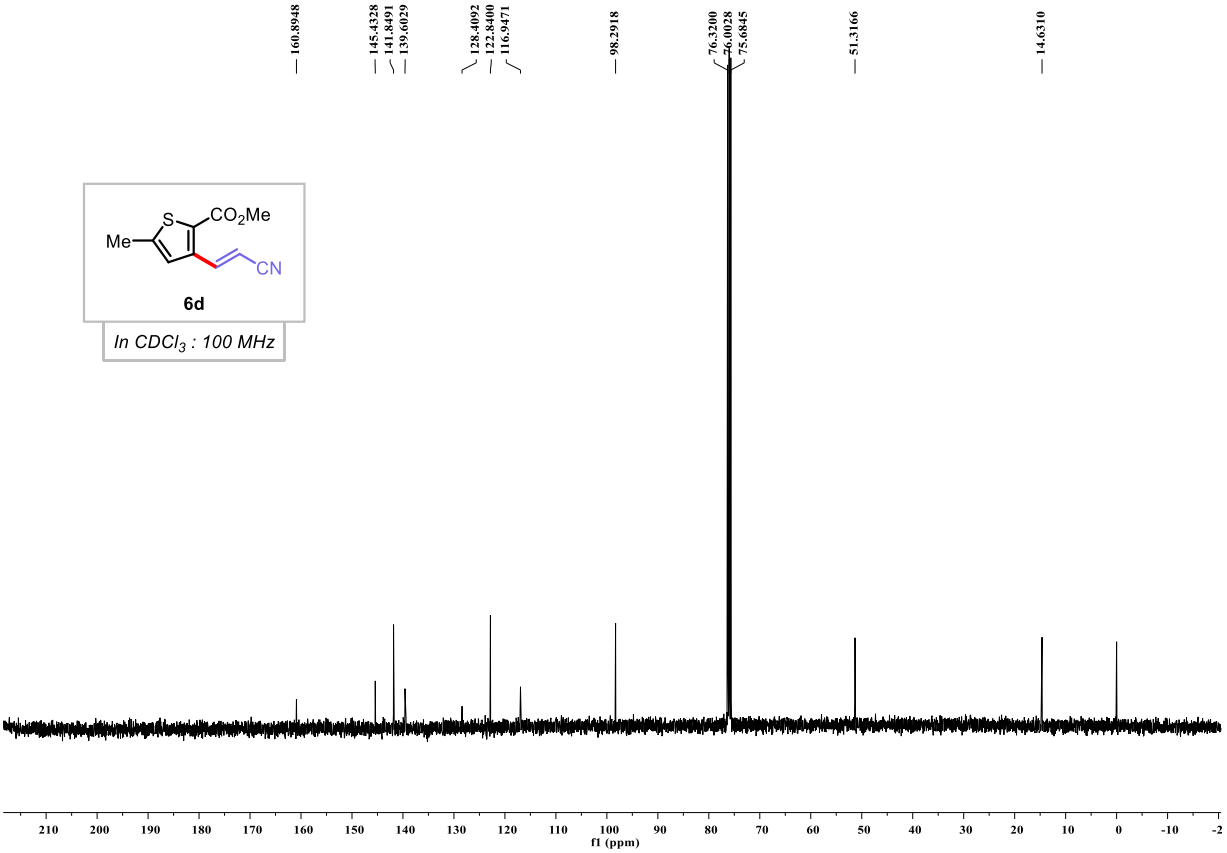

8.4550
8.4135
7.5121
7.4990
7.3171
7.3038
7.2604
5.8227
5.7813
3.9070
CO2Me
S
CN
6b
In CDCl3 : 400 MHz
1.00
1.04
1.00
0.98
3.07
f1 (ppm)

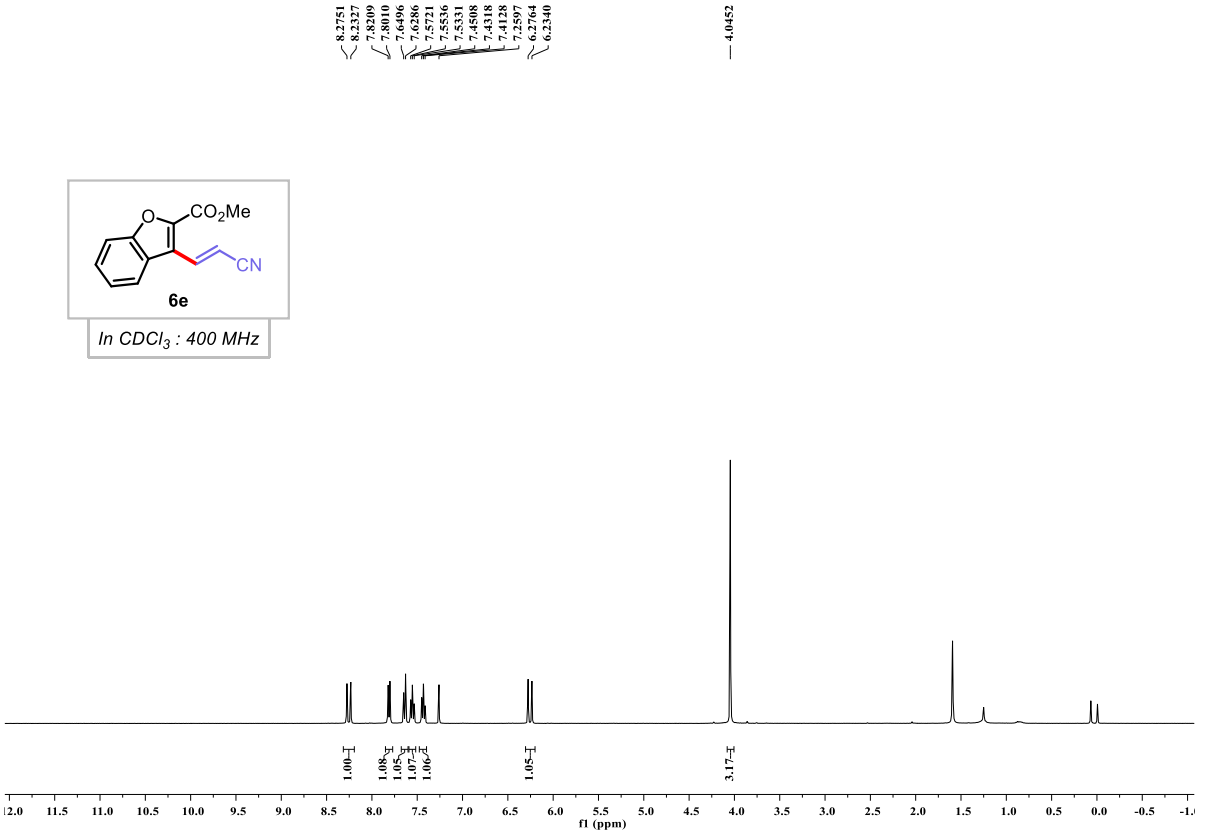

162.9702
143.4719
141.9336
132.3560
130.9626
126.7927
117.6517
98.4951
77.4781
77.1609
76.8436
52.3788
CO2Me
S
CN
6b
In CDCl3 : 100 MHz
f1 (ppm)

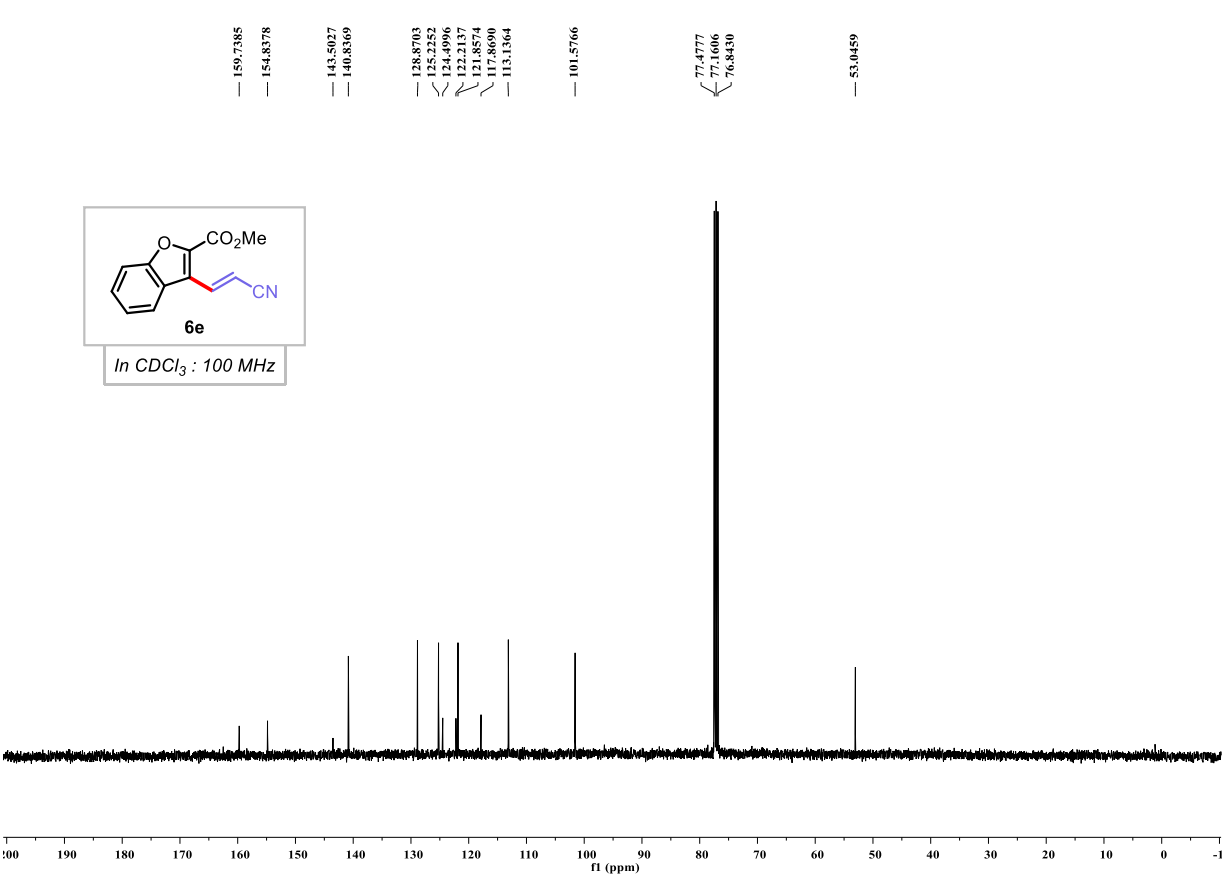

7.9231
7.8932
7.5844
7.5792
7.4720
7.4664
7.2603
5.5625
5.5327
3.9507
CO2Me
CN
6c
In CDCl3 : 400 MHz
1.02
0.97
0.96
1.00
2.93
f1 (ppm)

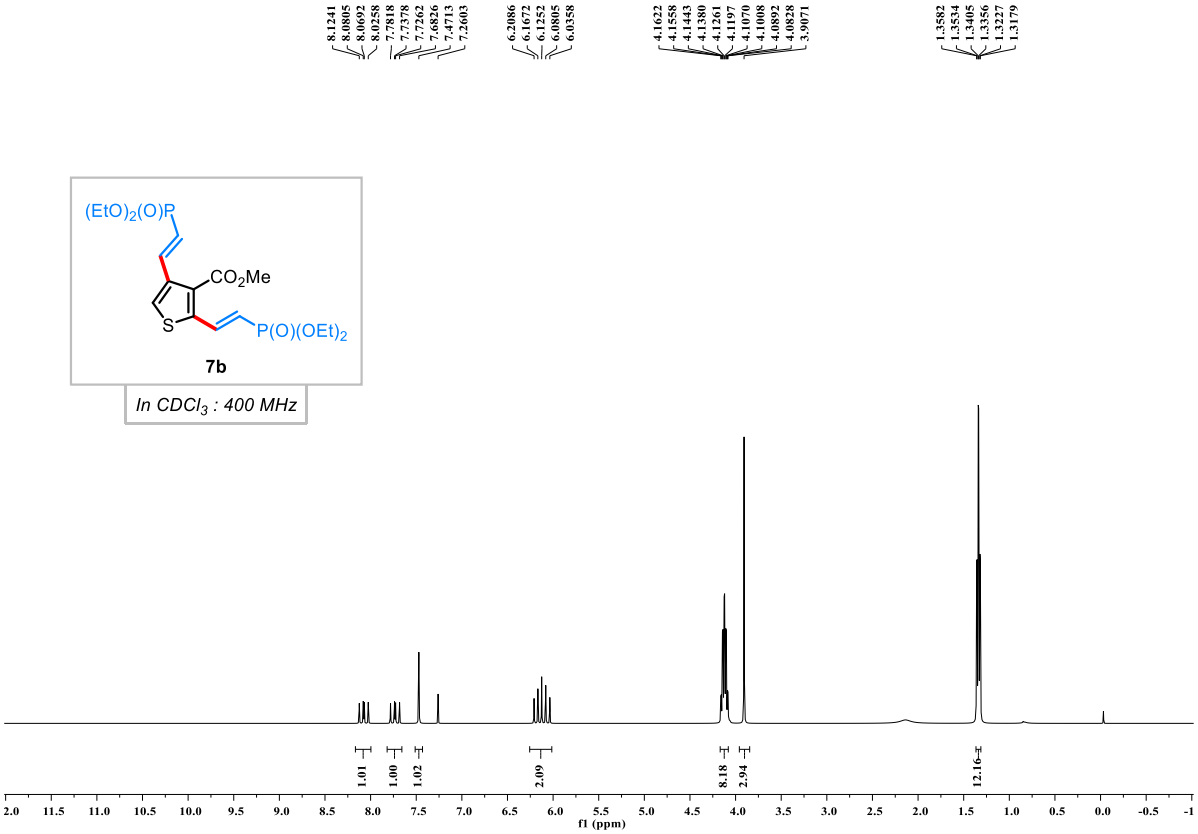

159.0441
145.9589
142.8580
138.3816
127.6892
117.0764
110.6619
98.7079
77.4785
76.8435
52.4828
CO2Me
CN
6c
In CDCl3 : 100 MHz
f1 (ppm)

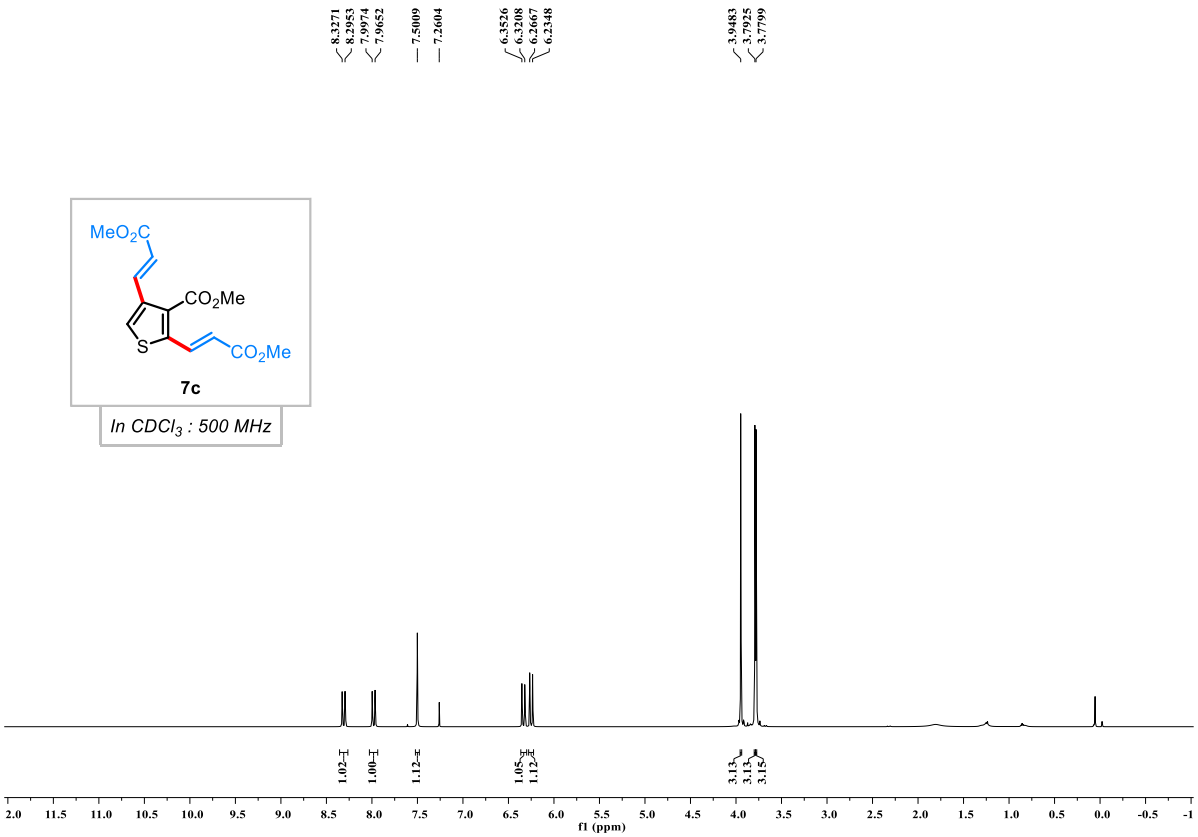

8.3016
8.2599
7.2605
6.9694
5.7950
5.7532
3.8834
2.4973
S
CO2Me
Me
CN
6d
In CDCl3 : 400 MHz
0.98
1.00
1.07
3.09
3.07
f1 (ppm)

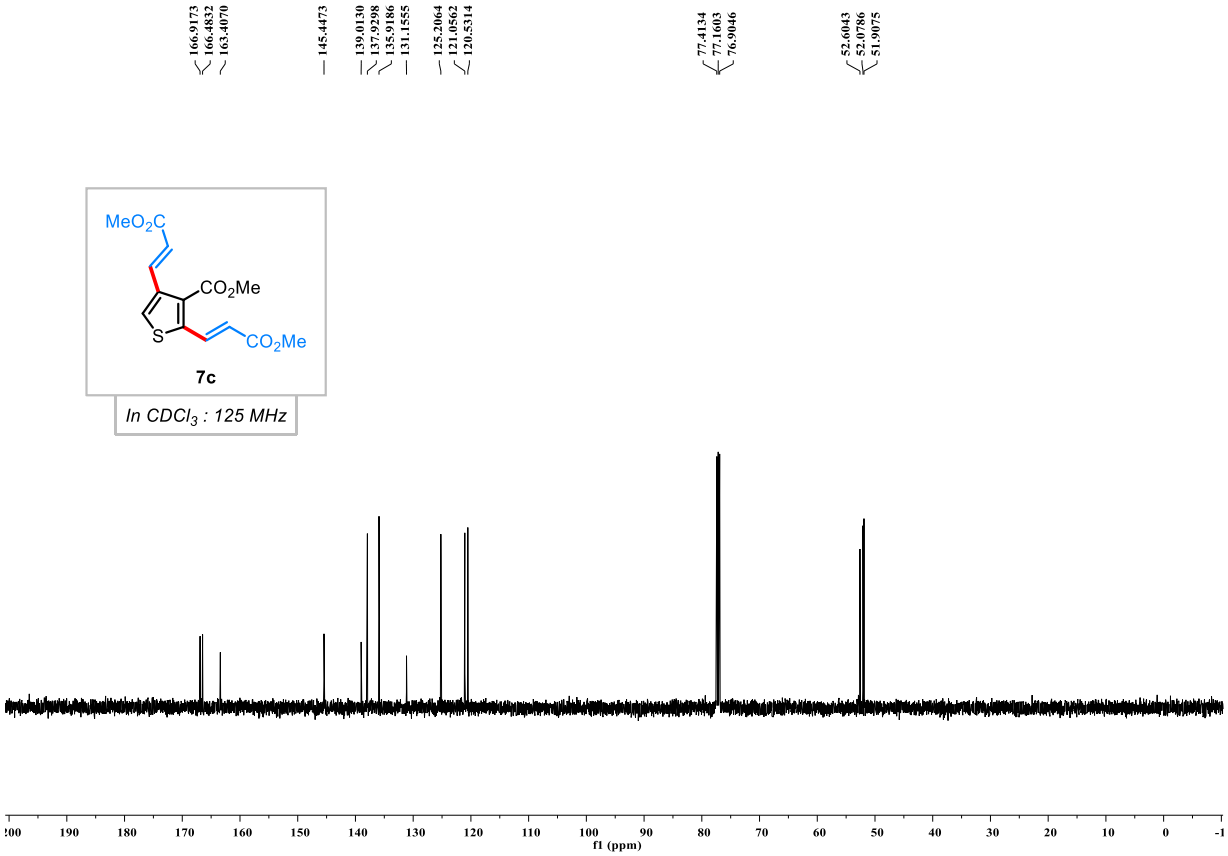

160.8948
145.4328
141.8491
139.6029
128.4092
122.8400
116.9471
98.2918
76.3200
76.0028
75.6845
51.3166
14.6310
S
CO2Me
Me
CN
6d
In CDCl3 : 100 MHz
f1 (ppm)

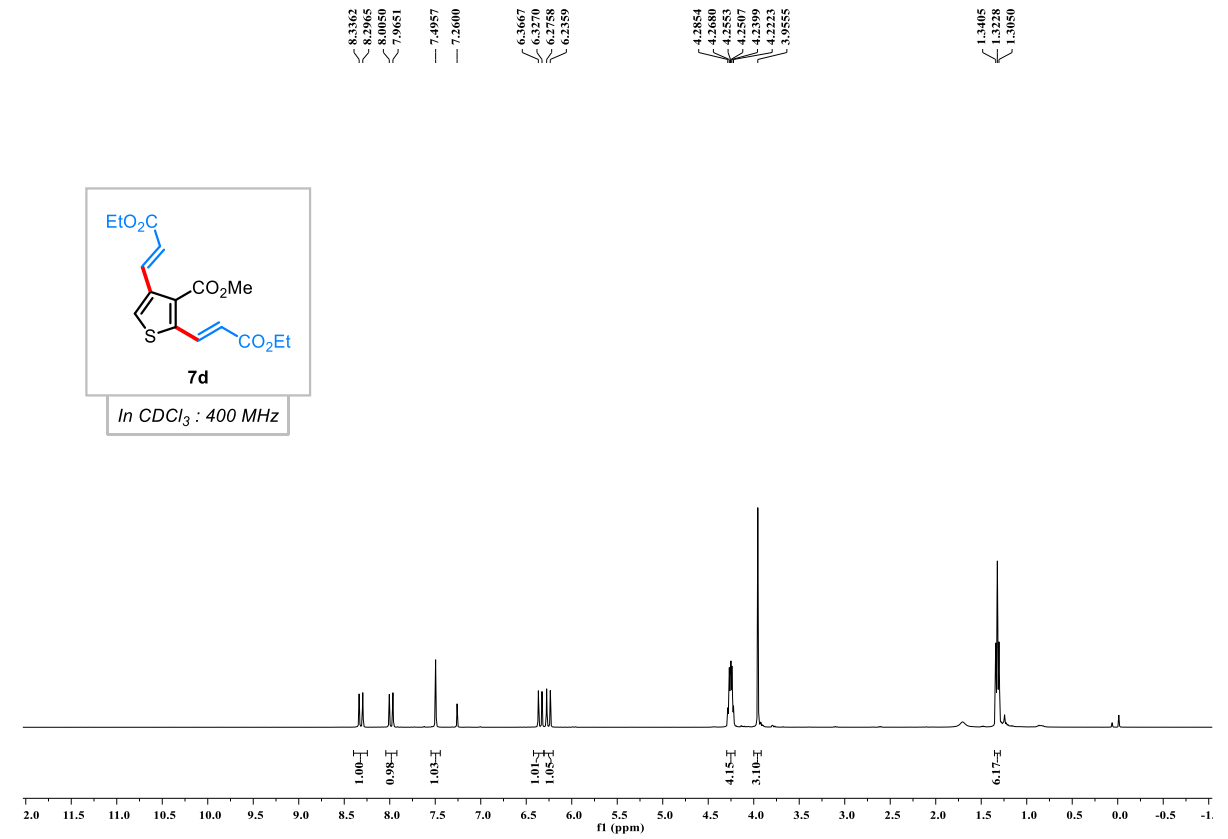

8.2751
8.2327
7.8209
7.8010
7.6496
7.6286
7.5721
7.5536
7.5331
7.4508
7.4318
7.4128
7.2597
6.2764
6.2340
4.0452
CO2Me
CN
6e
In CDCl3 : 400 MHz
1.00
1.08
1.05
1.07
1.06
1.05
3.17
f1 (ppm)

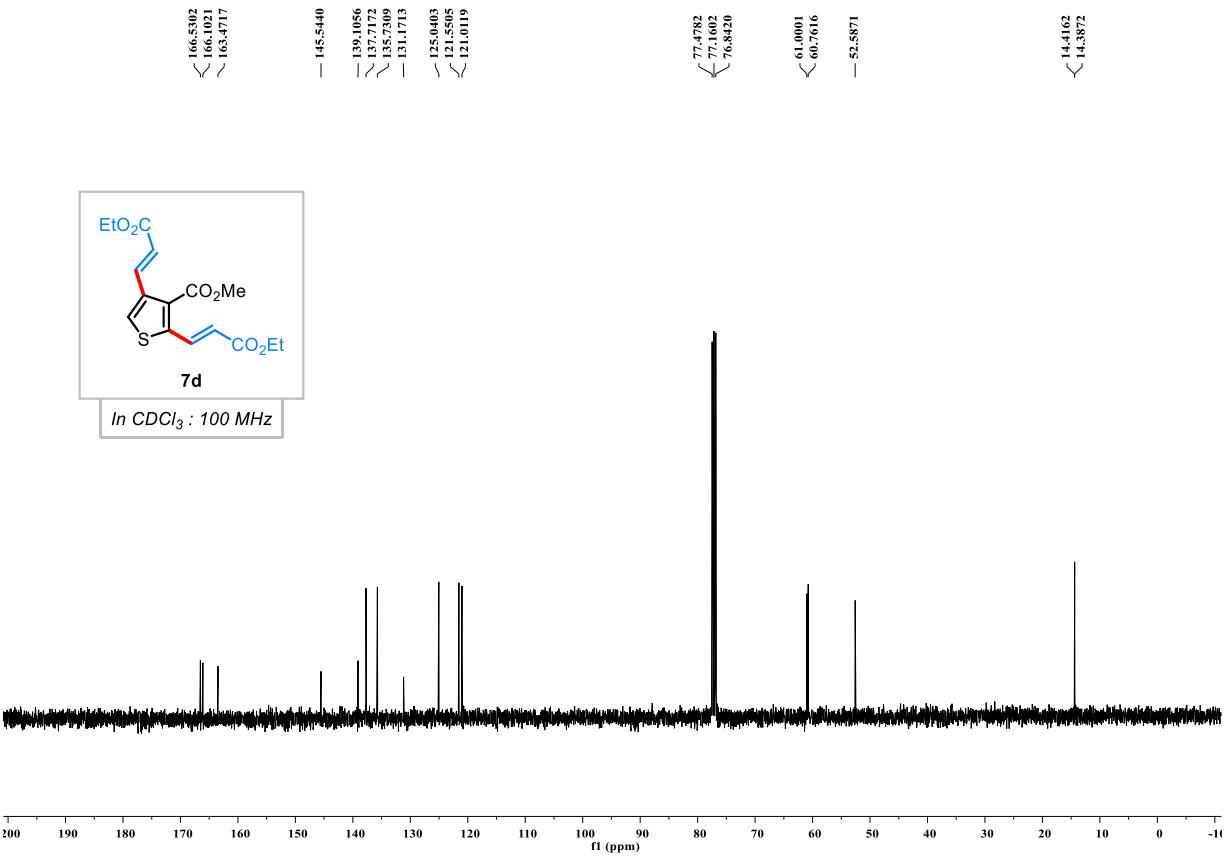

159.7385
154.8378
143.5027
140.8369
128.8703
125.2252
124.4996
122.2137
121.8574
117.8690
113.1364
101.5766
77.4777
77.1606
76.8430
53.0459
CO2Me
CN
6e
In CDCl3 : 100 MHz
f1 (ppm)

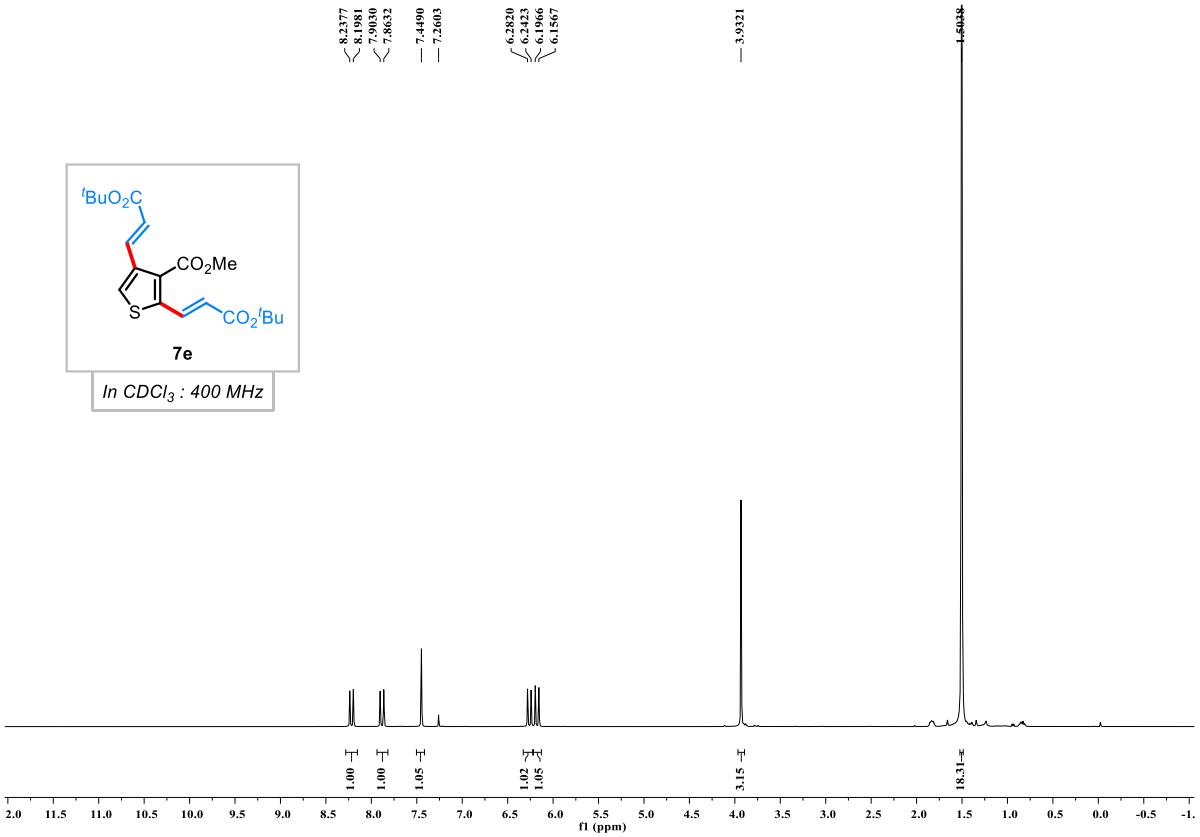

8.1241
8.0805
8.0692
8.0258
7.7818
7.7378
7.7262
7.6826
7.4713
7.2603
6.2086
6.1672
6.1252
6.0805
6.0358
4.1622
4.1558
4.1443
4.1380
4.1261
4.1197
4.1070
4.1008
4.0892
4.0828
3.9071
1.3582
1.3534
1.3405
1.3356
1.3227
1.3179
(EtO)2(O)P
CO2Me
S
P(O)(OEt)2
7b
In CDCl3 : 400 MHz
1.01
1.00
1.02
2.09
8.18
2.94
12.16
f1 (ppm)

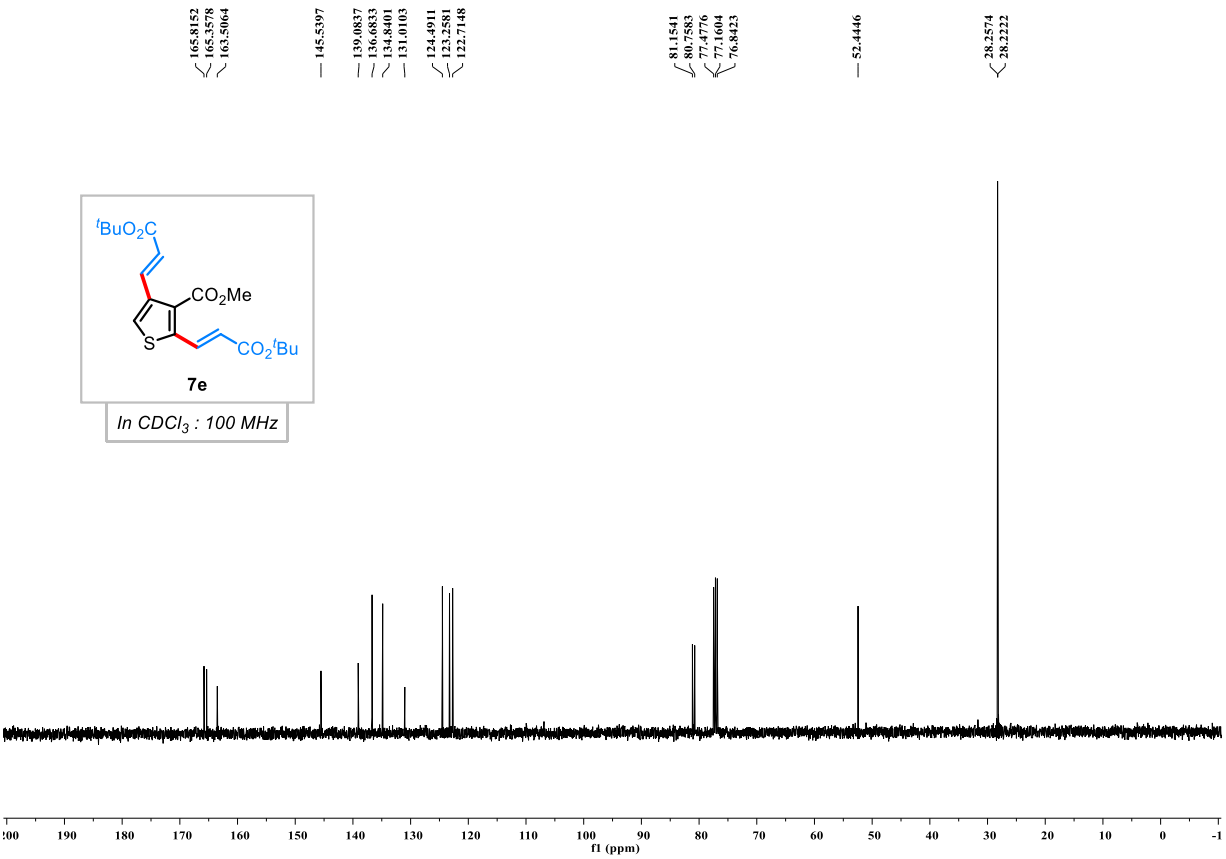

8.3271
8.2953
7.9974
7.9652
7.5009
7.2604
6.3526
6.3208
6.2667
6.2348
3.9483
3.7925
3.7799
MeO2C
CO2Me
S
CO2Me
7c
In CDCl3 : 500 MHz
1.02
1.00
1.12
1.05
1.12
3.13
3.13
3.15
f1 (ppm)

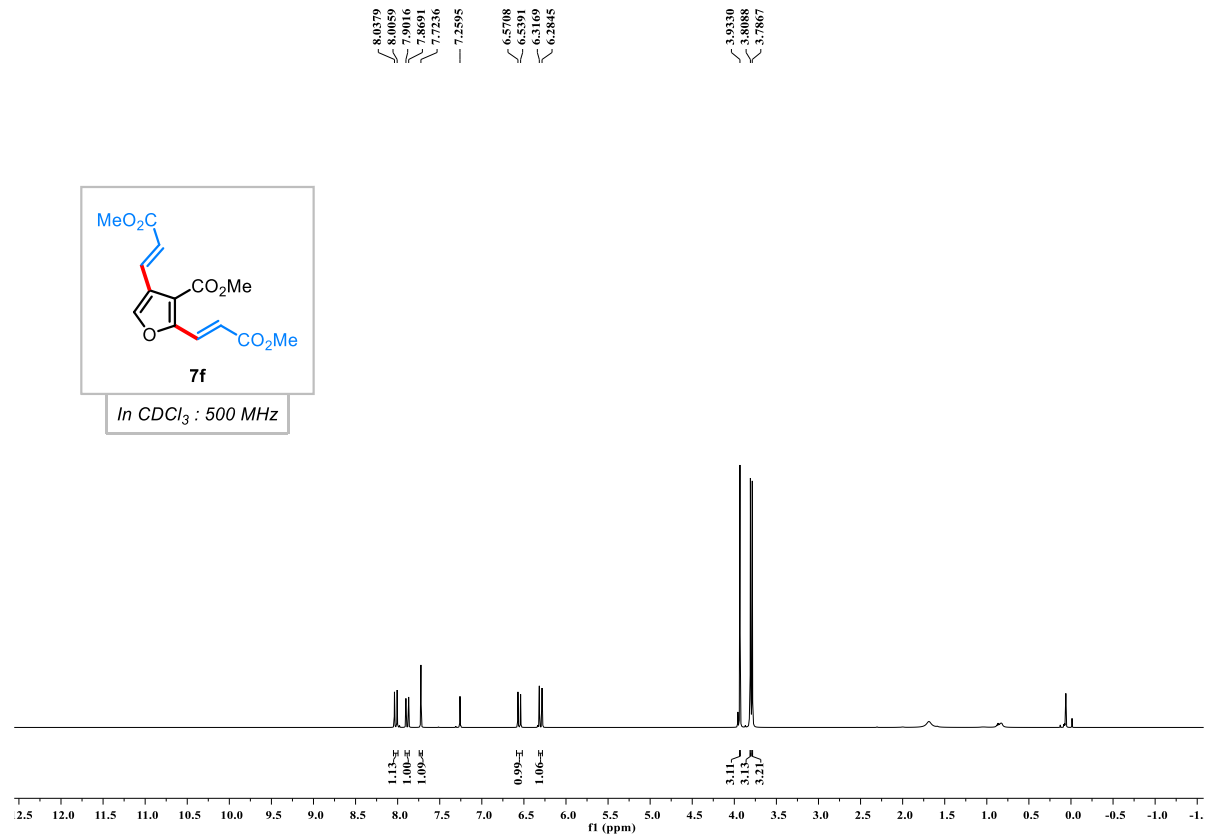

166.9173
166.4832
163.4070
145.4473
139.0130
137.9298
135.9186
131.1555
125.2064
121.0562
120.5314
77.4134
77.1603
76.9046
52.6043
52.0786
51.9075
MeO2C
CO2Me
S
CO2Me
7c
In CDCl3 : 125 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

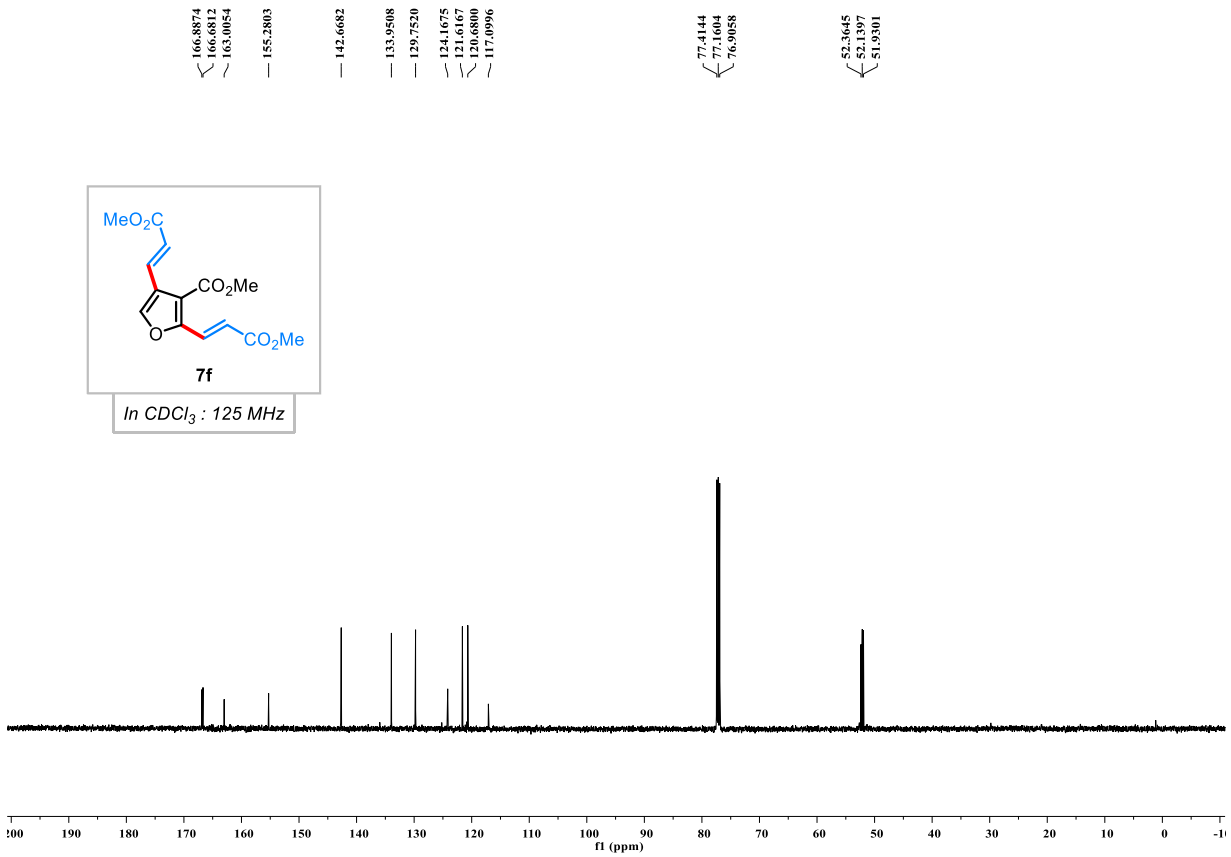

8.3362
8.2965
8.0050
7.9651
7.4957
7.2600
6.3667
6.3270
6.2758
6.2359
4.2854
4.2680
4.2553
4.2507
4.2399
4.2223
3.9555
1.3405
1.3228
1.3050
EtO2C
CO2Me
S
CO2Et
7d
In CDCl3 : 400 MHz
1.00
0.98
1.03
1.01
1.05
4.15
3.10
6.17
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

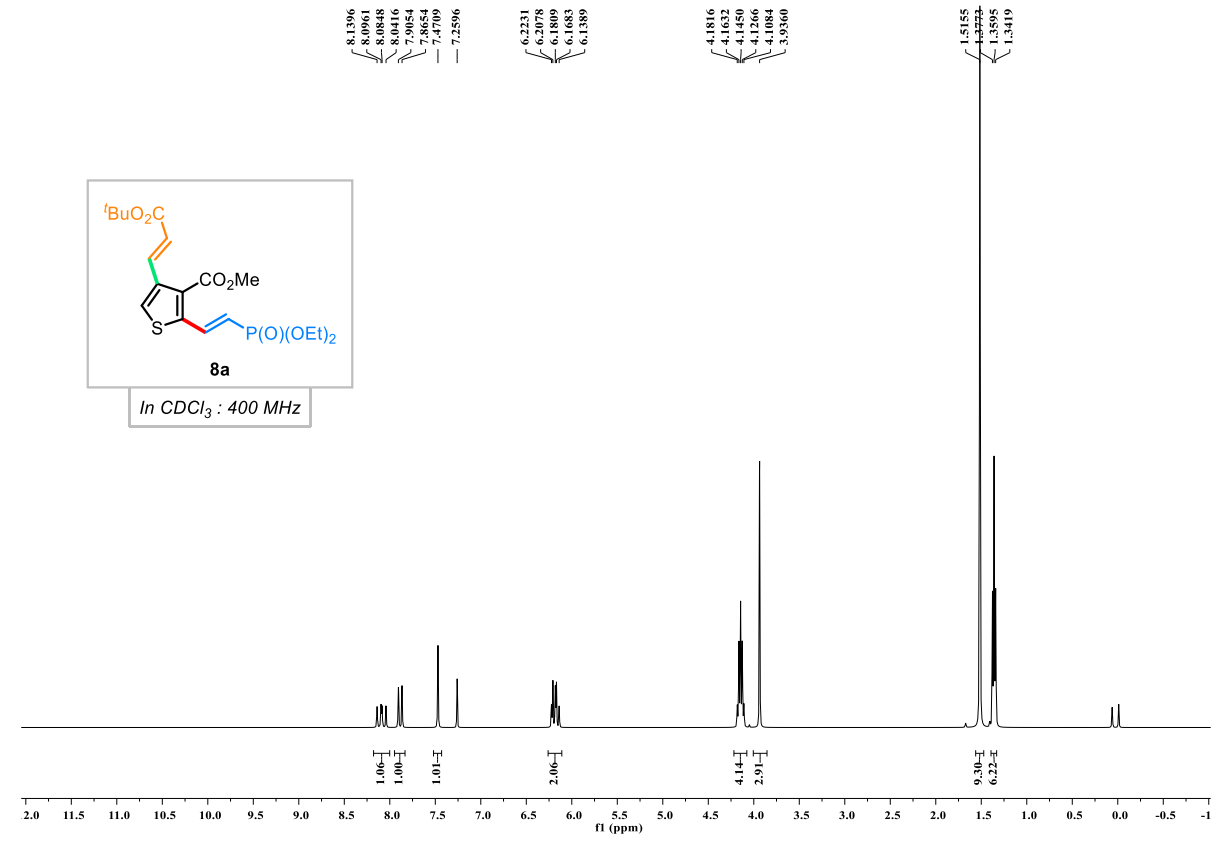

166.5302
166.1021
163.4717
145.5440
139.1056
137.7172
135.7309
131.1713
125.0403
121.5505
121.0119
77.4782
77.1602
76.8420
61.0001
60.7616
52.5871
14.4162
14.3872
EtO2C
CO2Me
CO2Et
S
7d
In CDCl3 : 100 MHz
f1 (ppm)

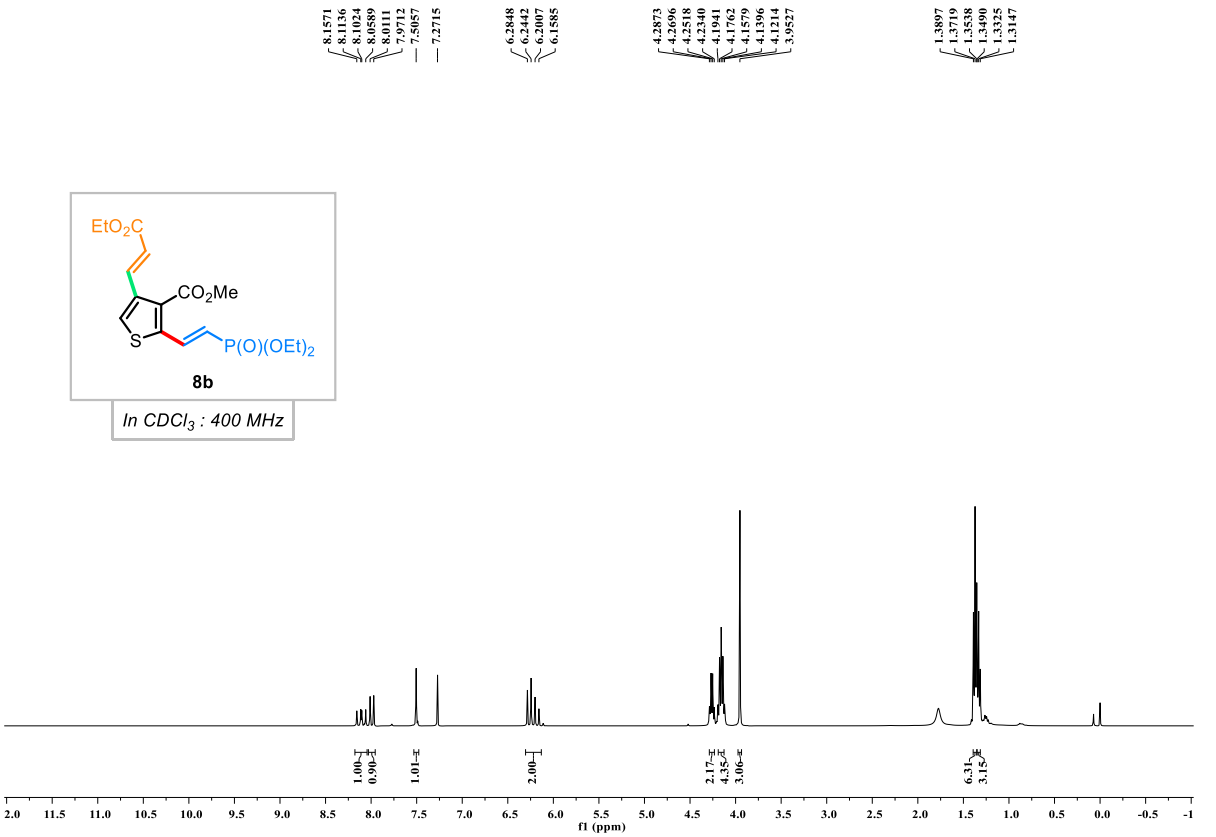

8.2377
8.1981
7.9030
7.8632
7.4490
7.2603
6.2820
6.2423
6.1966
6.1567
3.9321
1.5033
tBuO2C
CO2Me
CO2tBu
S
7e
In CDCl3 : 400 MHz
1.00
1.00
1.05
1.02
1.05
3.15
18.31
f1 (ppm)

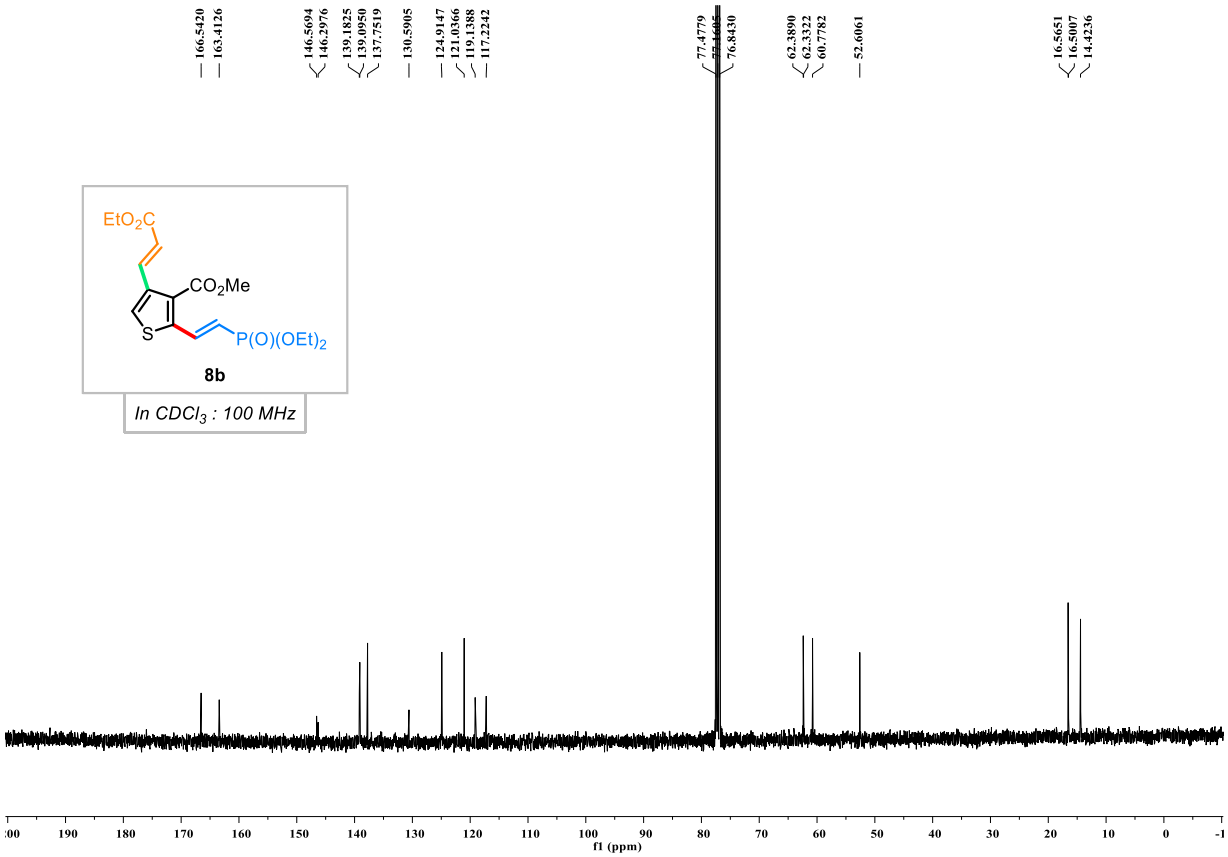

165.8152
165.3578
163.5064
145.5397
139.0837
136.6833
134.8401
131.0103
124.4911
123.2581
122.7148
81.1541
80.7583
77.4776
77.1604
76.8423
52.4446
28.2574
28.2222
tBuO2C
CO2Me
CO2tBu
S
7e
In CDCl3 : 100 MHz
f1 (ppm)

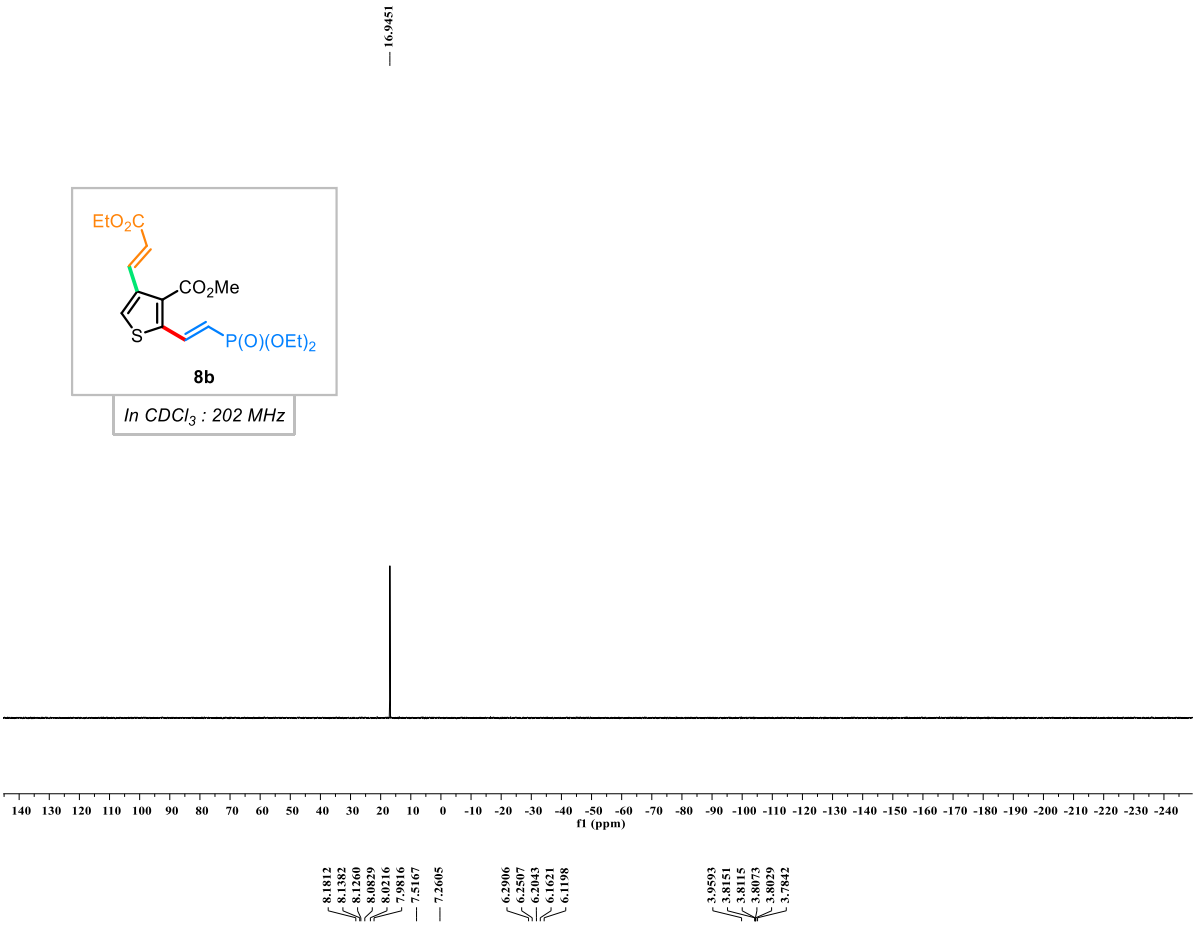

8.0379
8.0059
7.9016
7.8691
7.7236
7.2595
6.5708
6.5391
6.3169
6.2845
3.9330
3.9088
3.7867
MeO2C
CO2Me
CO2Me
O
7f
In CDCl3 : 500 MHz
1.13
1.00
1.09
0.99
1.06
3.11
3.13
3.21
f1 (ppm)

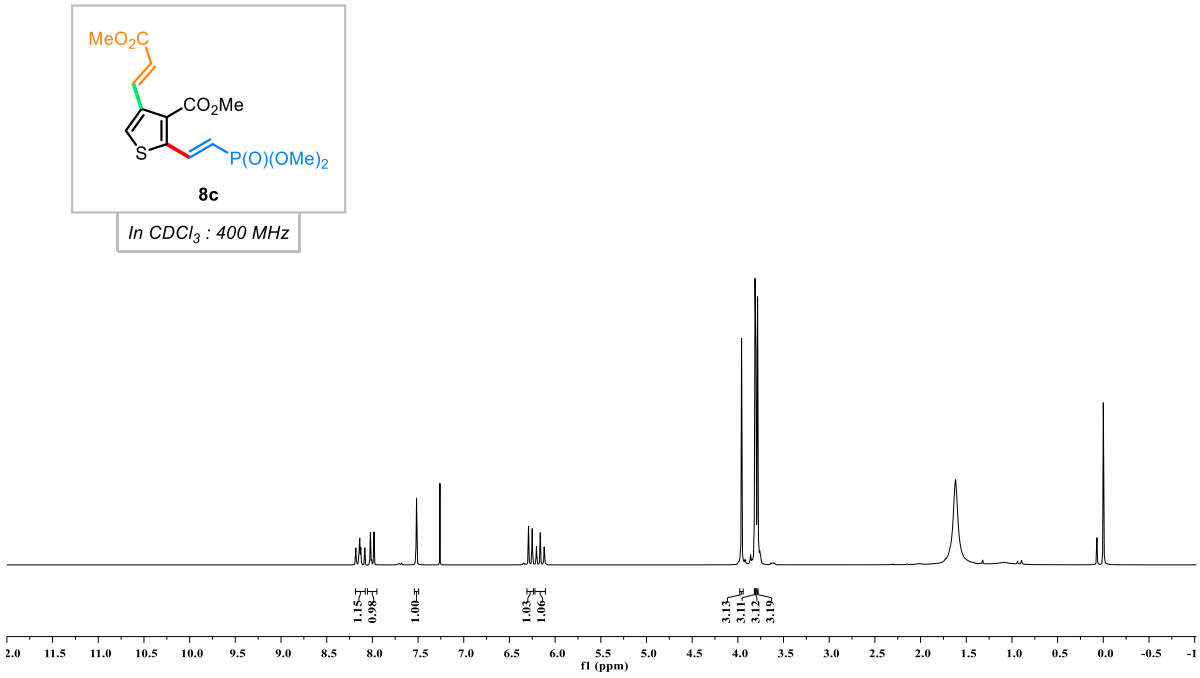

166.8874
166.6812
163.0054
155.2803
142.6682
133.9508
129.7520
124.1675
121.6167
120.6800
117.0996
77.4144
77.1604
76.9058
52.3645
52.1397
51.9301
MeO2C
CO2Me
CO2Me
O
7f
In CDCl3 : 125 MHz
f1 (ppm)

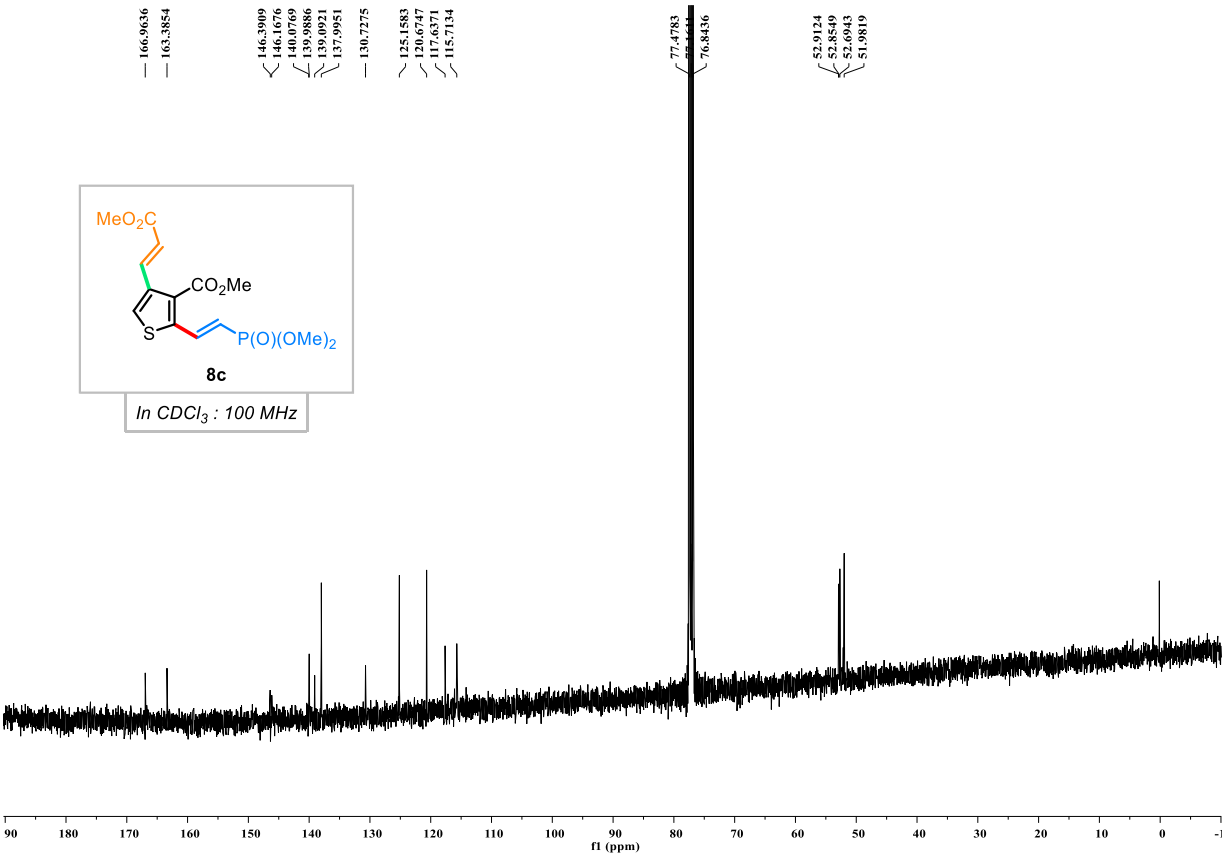

8.1396
8.0961
8.0848
8.0416
7.9054
7.8654
7.4709
7.2596
6.2231
6.2078
6.1809
6.1683
6.1389
4.1816
4.1632
4.1450
4.1266
4.1084
3.9360
1.5155
1.3723
1.3595
1.3419
tBuO2C
CO2Me
S
P(O)(OEt)2
8a
In CDCl3 : 400 MHz
1.06
1.00
1.01
2.06
4.14
2.91
9.30
6.22
f1 (ppm)

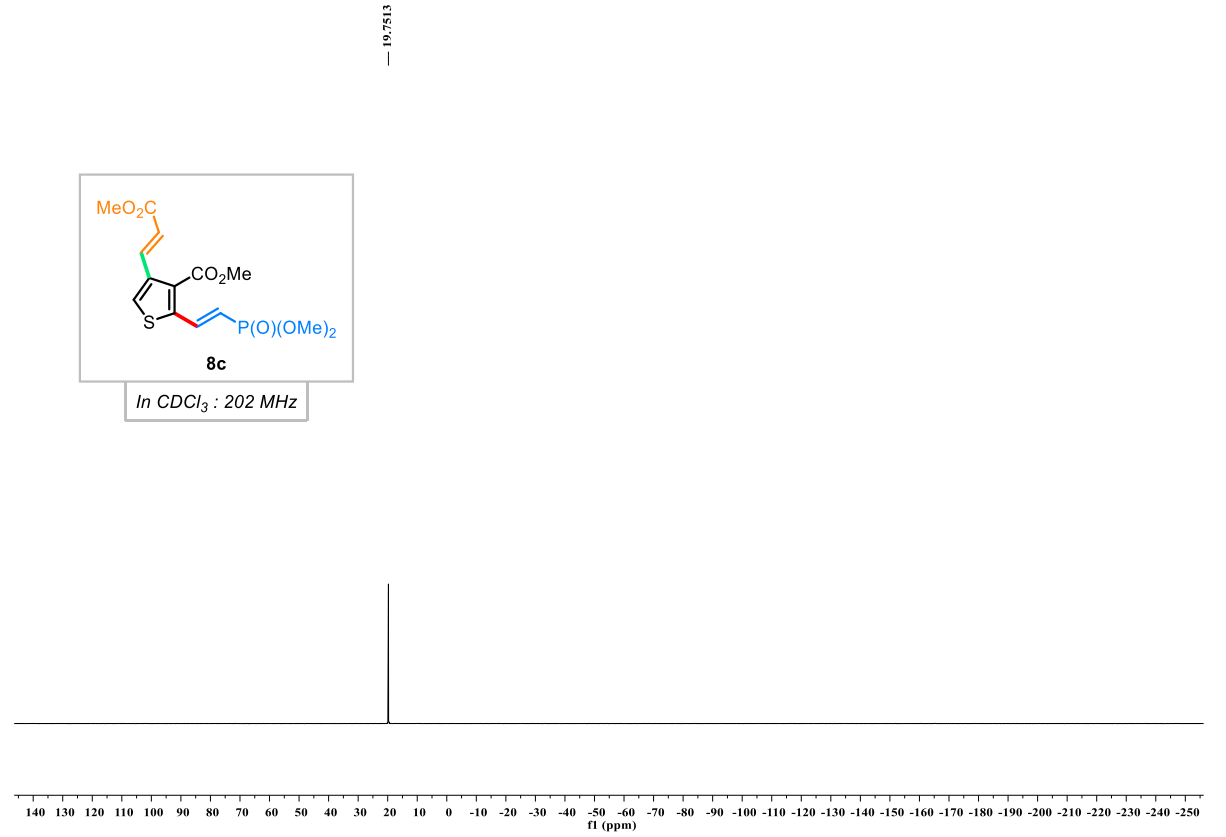

8.1571
8.1136
8.1024
8.0589
8.0111
7.9712
7.5057
7.2715
6.2848
6.2442
6.2007
6.1585
4.2873
4.2696
4.2518
4.2340
4.1941
4.1762
4.1579
4.1396
4.1214
3.9527
1.3897
1.3719
1.3538
1.3490
1.3325
1.3147
EtO2C
CO2Me
S
P(O)(OEt)2
8b
In CDCl3 : 400 MHz
1.00
0.90
1.01
2.00
2.17
4.35
3.06
6.31
3.15
f1 (ppm)

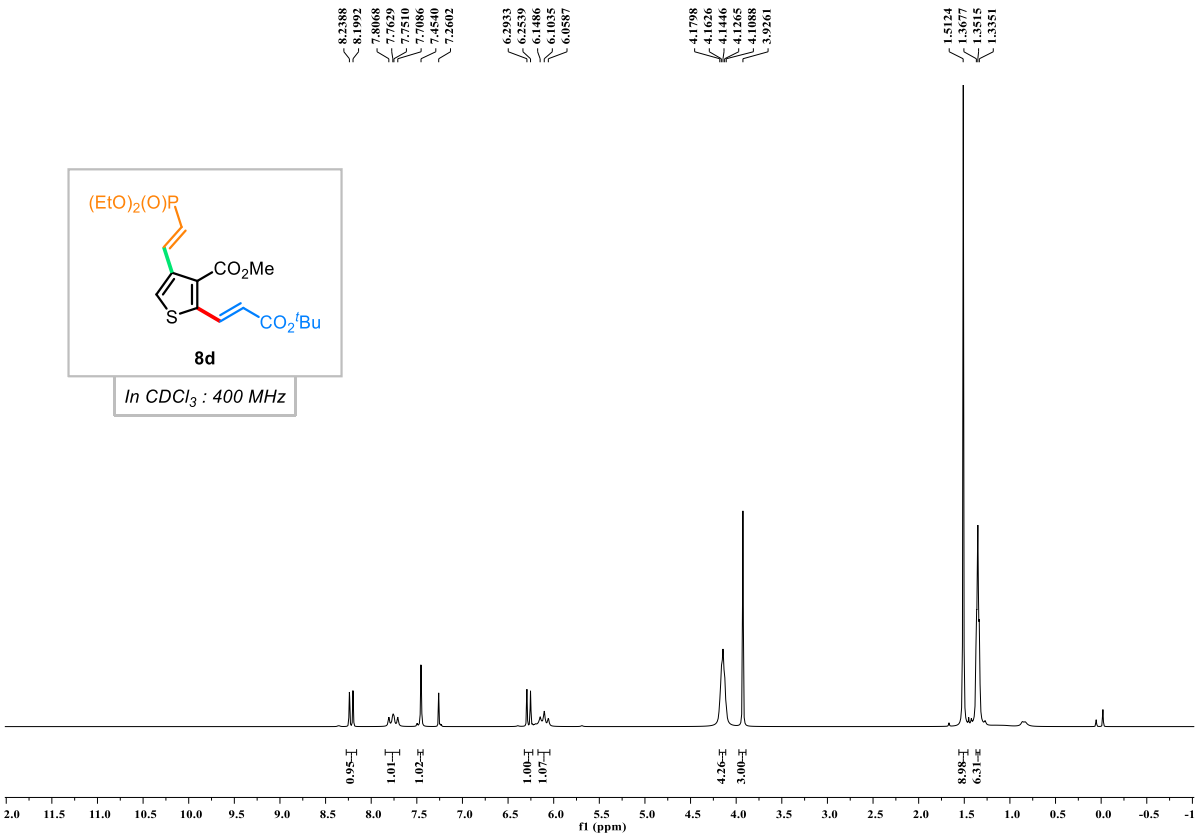

166.5420
163.4126
146.5694
146.2976
139.1825
139.0950
137.7519
130.5905
124.9147
121.0366
119.1388
117.2242
77.4779
76.8430
62.3890
62.3322
60.7782
52.6061
16.5651
16.5007
14.4236
EtO2C
CO2Me
S
P(O)(OEt)2
8b
In CDCl3 : 100 MHz
f1 (ppm)

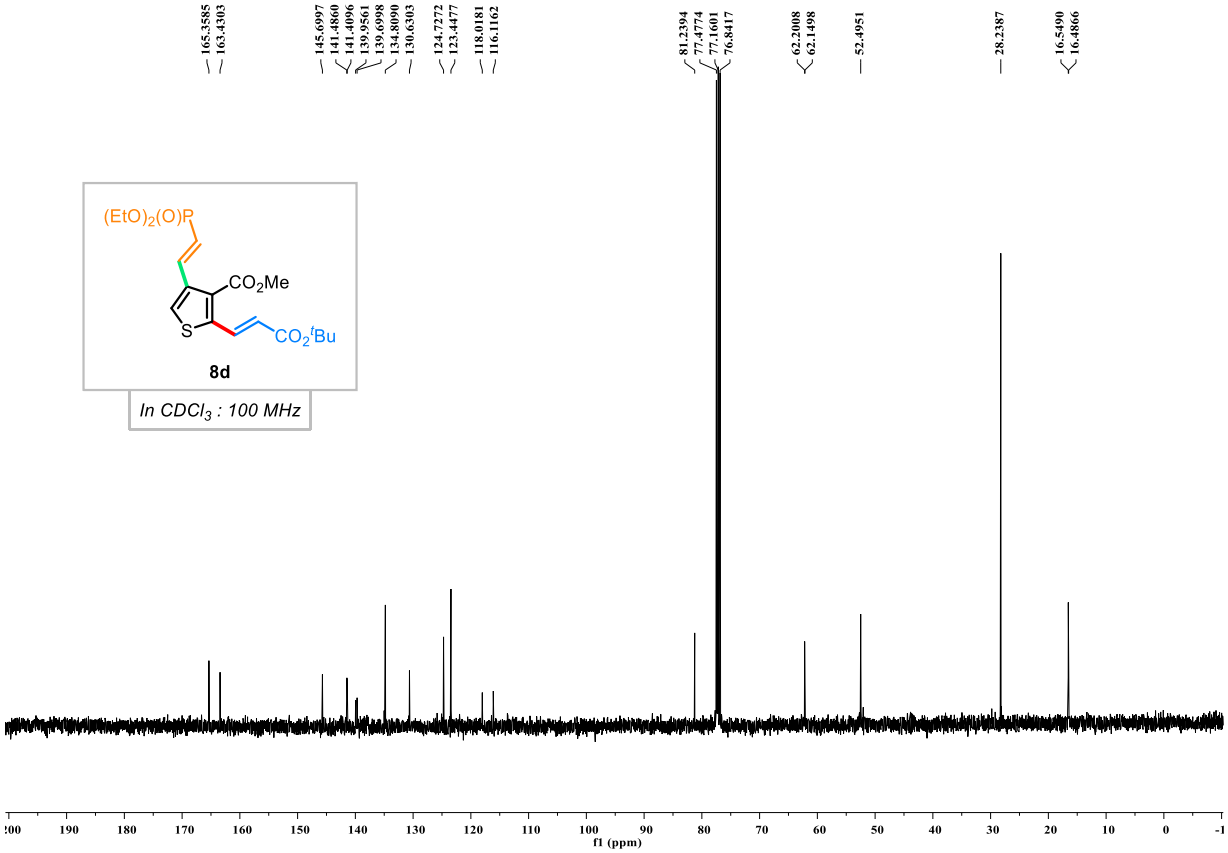

16.9451
EtO2C
CO2Me
S
P(O)(OEt)2
8b
In CDCl3 : 202 MHz
fl (ppm)
8.1812
8.1382
8.1260
8.0829
8.0216
7.9816
7.5167
7.2605
6.2906
6.2507
6.2043
6.1621
6.1198
3.9593
3.8151
3.8115
3.8073
3.8029
3.7842

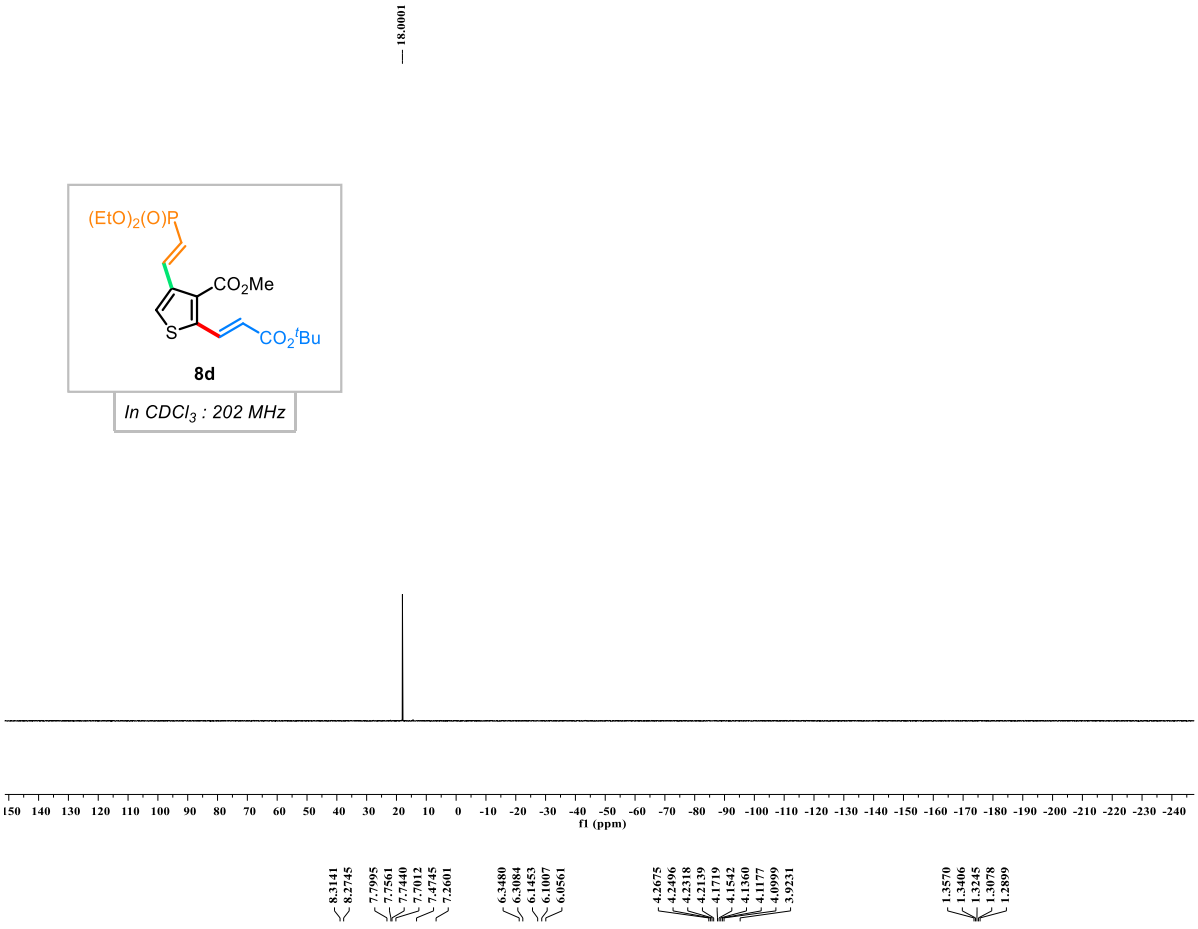

MeO2C
CO2Me
S
P(O)(OMe)2
8c
In CDCl3 : 400 MHz
fl (ppm)
1.15
0.98
1.00
1.03
1.06
3.13
3.11
3.12
3.19

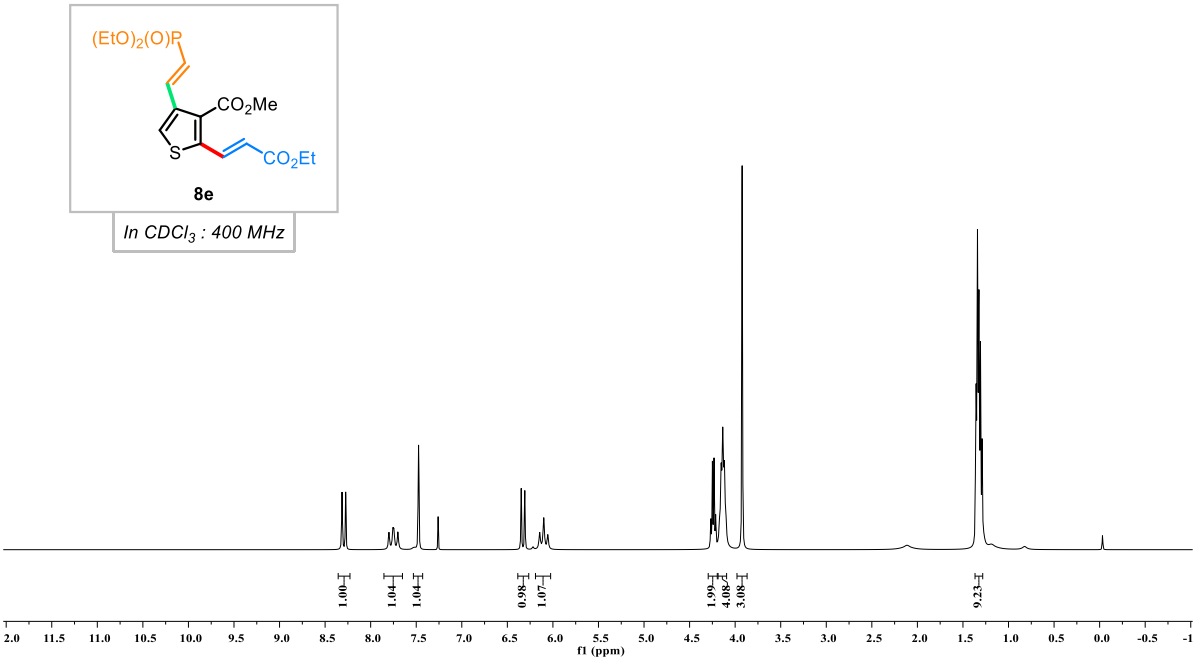

166.9636
163.3854
146.3909
146.1676
140.0769
139.9886
139.0921
137.9951
130.7275
125.1583
120.6747
117.6371
115.7134
77.4783
77.1644
76.8436
52.9124
52.8549
52.6943
51.9819
MeO2C
CO2Me
S
P(O)(OMe)2
8c
In CDCl3 : 100 MHz
f1 (ppm)

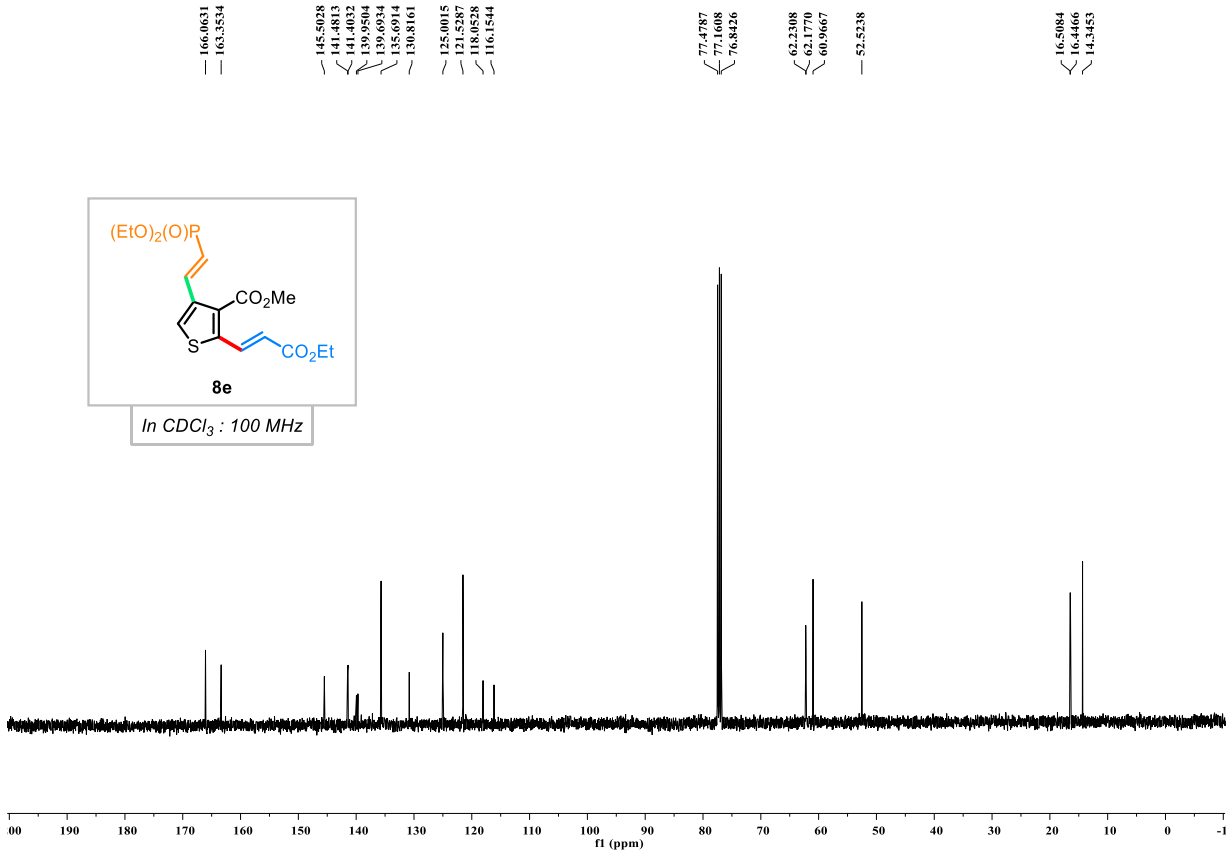

19.7513
MeO2C
CO2Me
S
P(O)(OMe)2
8c
In CDCl3 : 202 MHz
f1 (ppm)

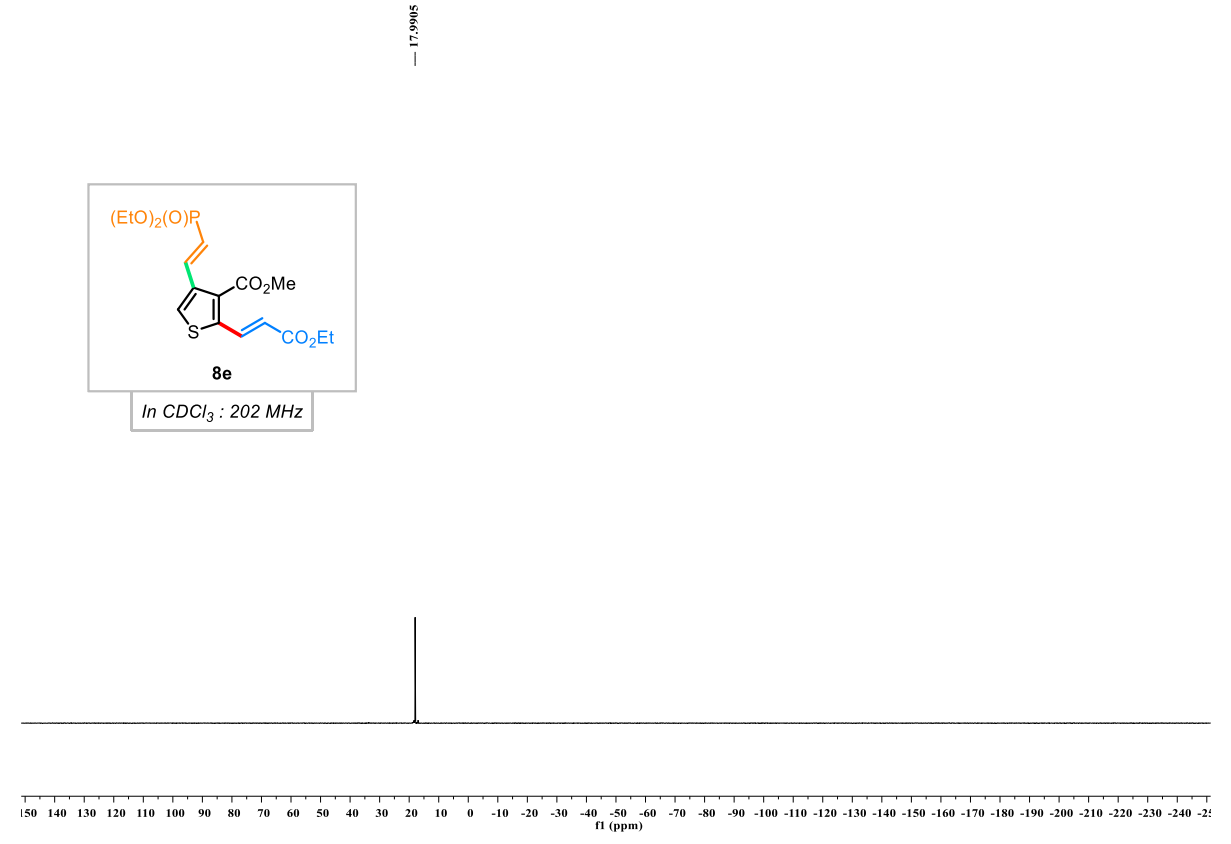

8.2388
8.1992
7.8068
7.7629
7.7510
7.7086
7.4540
7.2602
6.2933
6.2539
6.1486
6.1035
6.0587
4.1798
4.1626
4.1446
4.1265
4.1088
3.9261
1.5124
1.3677
1.3515
1.3351
(EtO)2(O)P
CO2Me
S
CO2tBu
8d
In CDCl3 : 400 MHz
0.95
1.01
1.02
1.00
1.07
4.26
3.00
8.98
6.31
f1 (ppm)

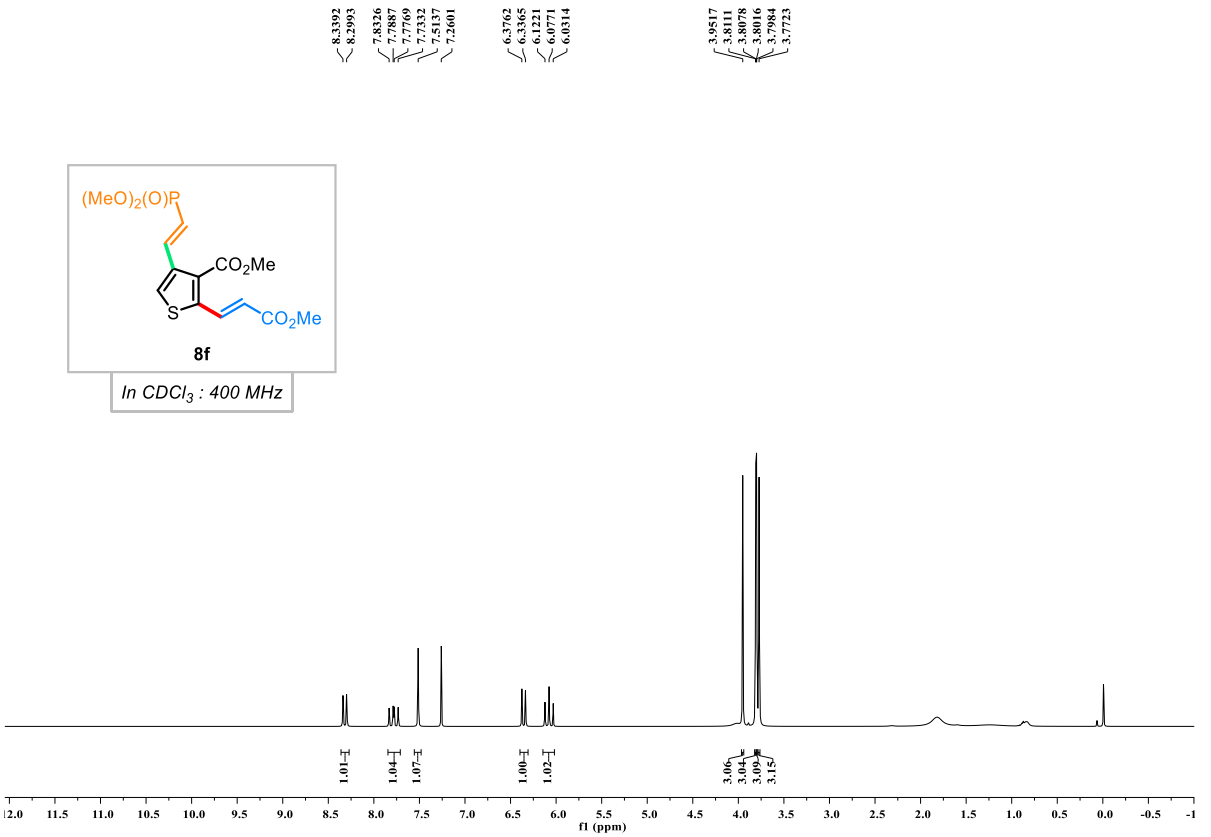

165.3585
163.4303
145.6997
141.4860
141.4096
139.9561
139.6998
134.8090
130.6303
124.7272
123.4477
118.0181
116.1162
81.2394
77.4774
77.1601
76.8417
62.2008
62.1498
52.4951
28.2387
16.5490
16.4866
(EtO)2(O)P
CO2Me
S
CO2tBu
8d
In CDCl3 : 100 MHz
f1 (ppm)

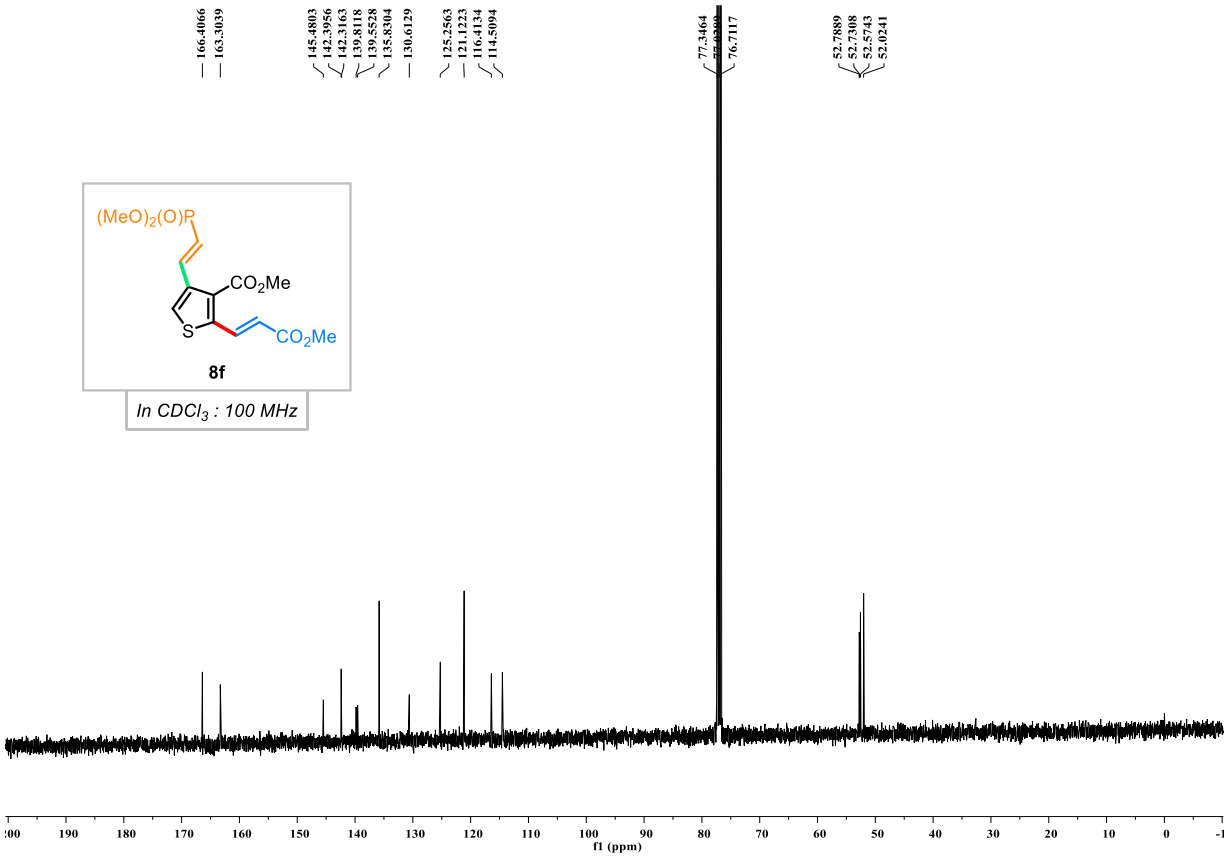

18.0001
(EtO)2(O)P
CO2Me
S
CO2tBu
8d
In CDCl3 : 202 MHz
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230 -240
f1 (ppm)
8.3141
8.2745
7.7995
7.7561
7.7440
7.7012
7.4745
7.2601
6.3480
6.3084
6.1453
6.1007
6.0561
4.2675
4.2496
4.2318
4.2139
4.1719
4.1542
4.1360
4.1177
4.0999
3.9231
1.3570
1.3406
1.3245
1.3078
1.2899

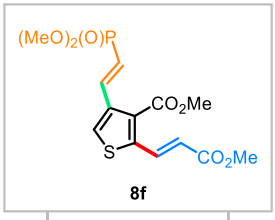

(EtO)2(O)P
CO2Me
S
CO2Et
8e
In CDCl3 : 400 MHz
1.00
1.04
1.04
0.98
1.07
1.99
4.08
3.08
9.23
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)


166.0631
163.3534
145.5028
141.4813
141.4032
139.9504
139.6934
135.6914
130.8161
125.0015
121.5287
118.0528
116.1544
77.4787
77.1608
76.8426
62.2308
62.1770
60.9667
52.5238
16.5084
16.4466
14.3453
(EtO)2(O)P
CO2Me
CO2Et
S
8e
In CDCl3 : 100 MHz
f1 (ppm)


17.9905
(EtO)2(O)P
CO2Me
CO2Et
S
8e
In CDCl3 : 202 MHz
f1 (ppm)

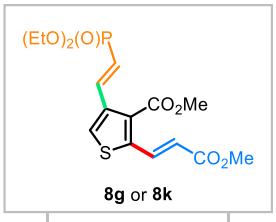

8.3392
8.2993
7.8326
7.7887
7.7769
7.7332
7.5137
7.2601
6.3762
6.3365
6.1221
6.0771
6.0314
3.9517
3.8111
3.8078
3.8016
3.7984
3.7723
(MeO)2(O)P
CO2Me
S
CO2Me
8f
In CDCl3 : 400 MHz
1.01
1.04
1.07
1.00
1.02
3.06
3.04
3.09
3.15
f1 (ppm)


166.4066
163.3039
145.4803
142.3956
142.3163
139.8118
139.5528
135.8304
130.6129
125.2563
121.1223
116.4134
114.5094
77.3464
76.7117
52.7889
52.7308
52.5743
52.0241
(MeO)2(O)P
CO2Me
S
CO2Me
8f
In CDCl3 : 100 MHz
f1 (ppm)



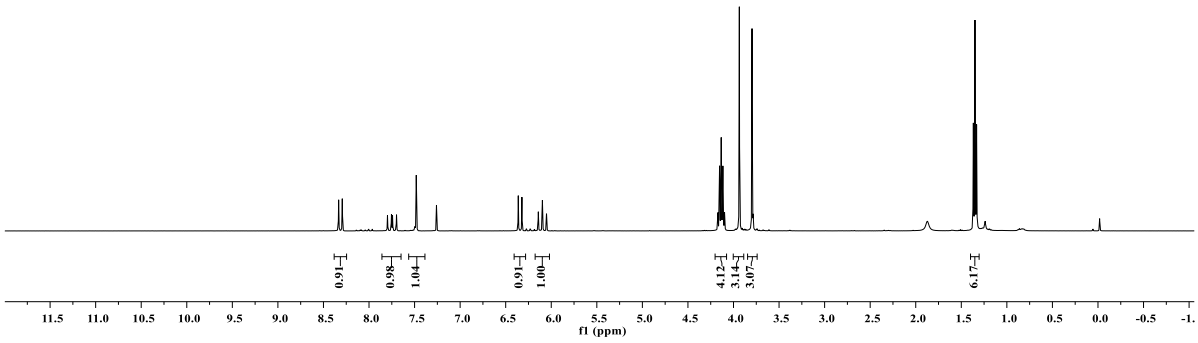
8f

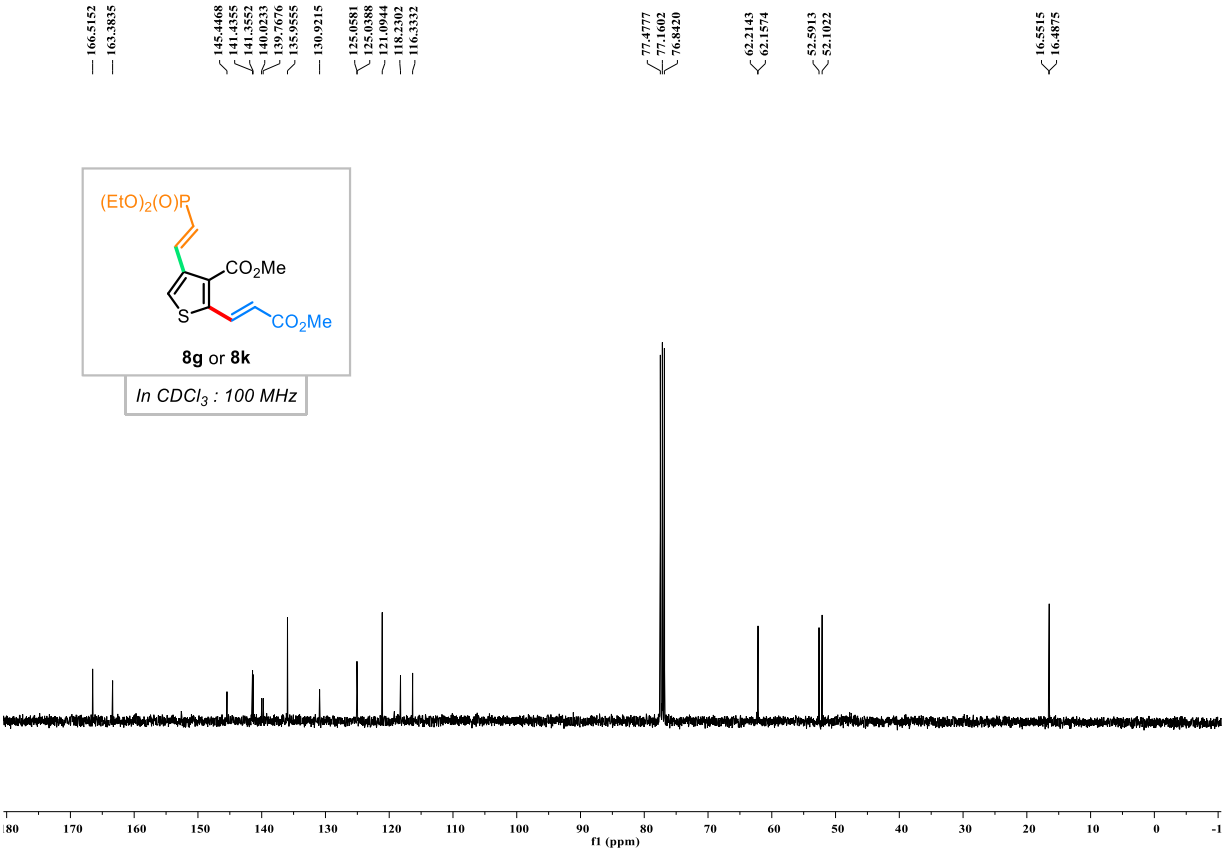
In $CDCl_3$: 202 MHz

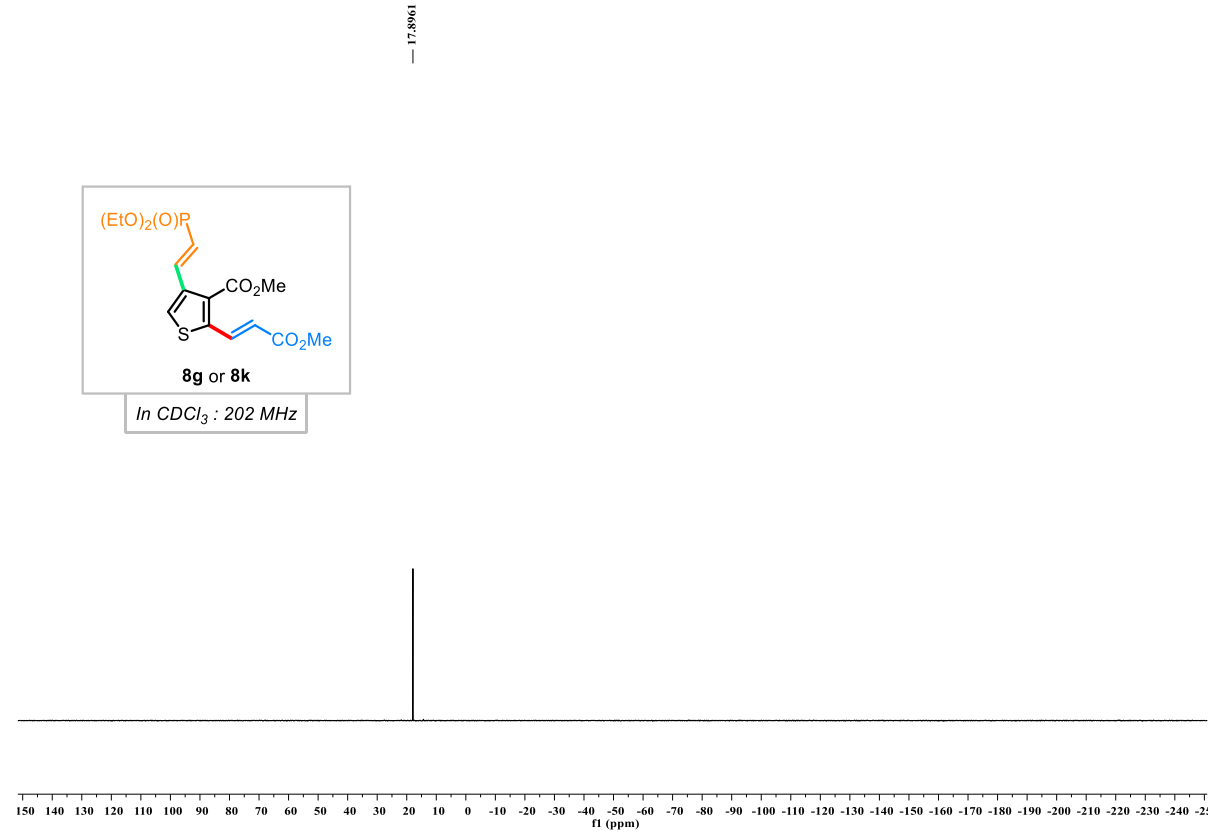


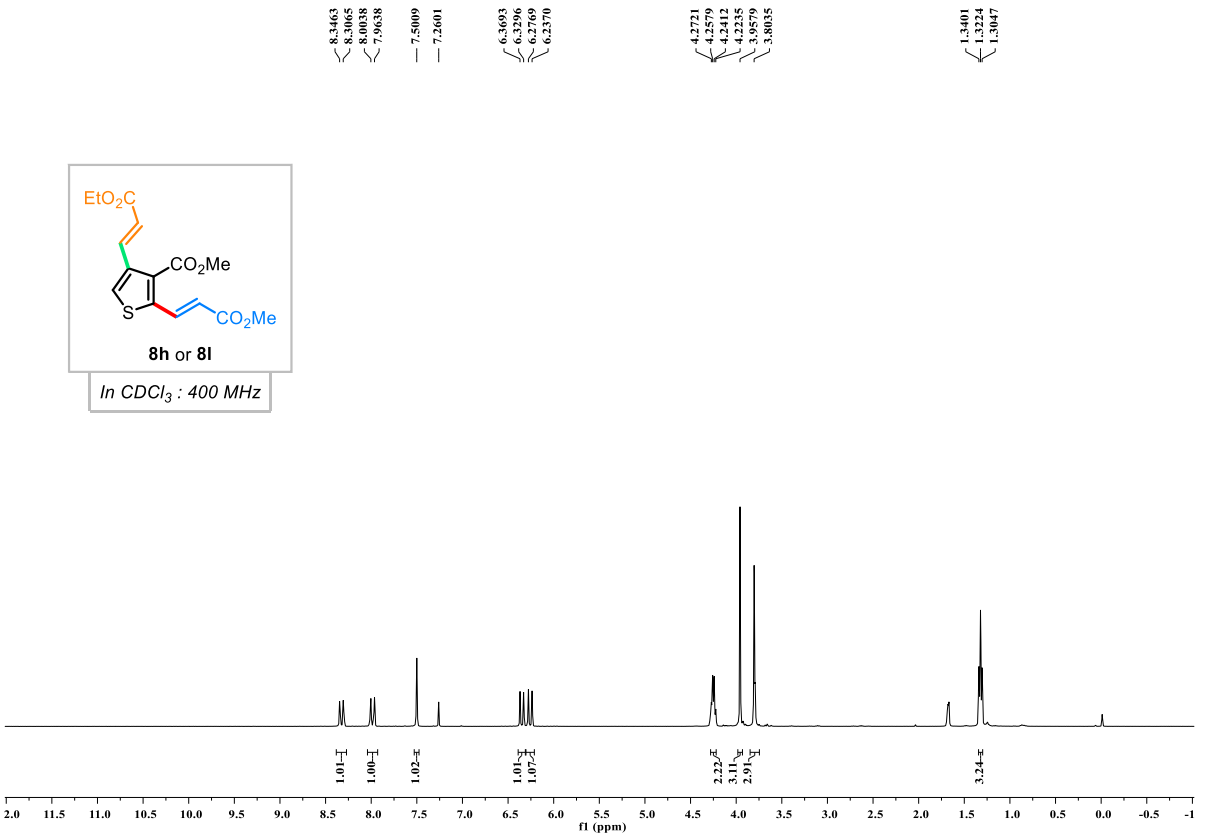
8g or **8k**

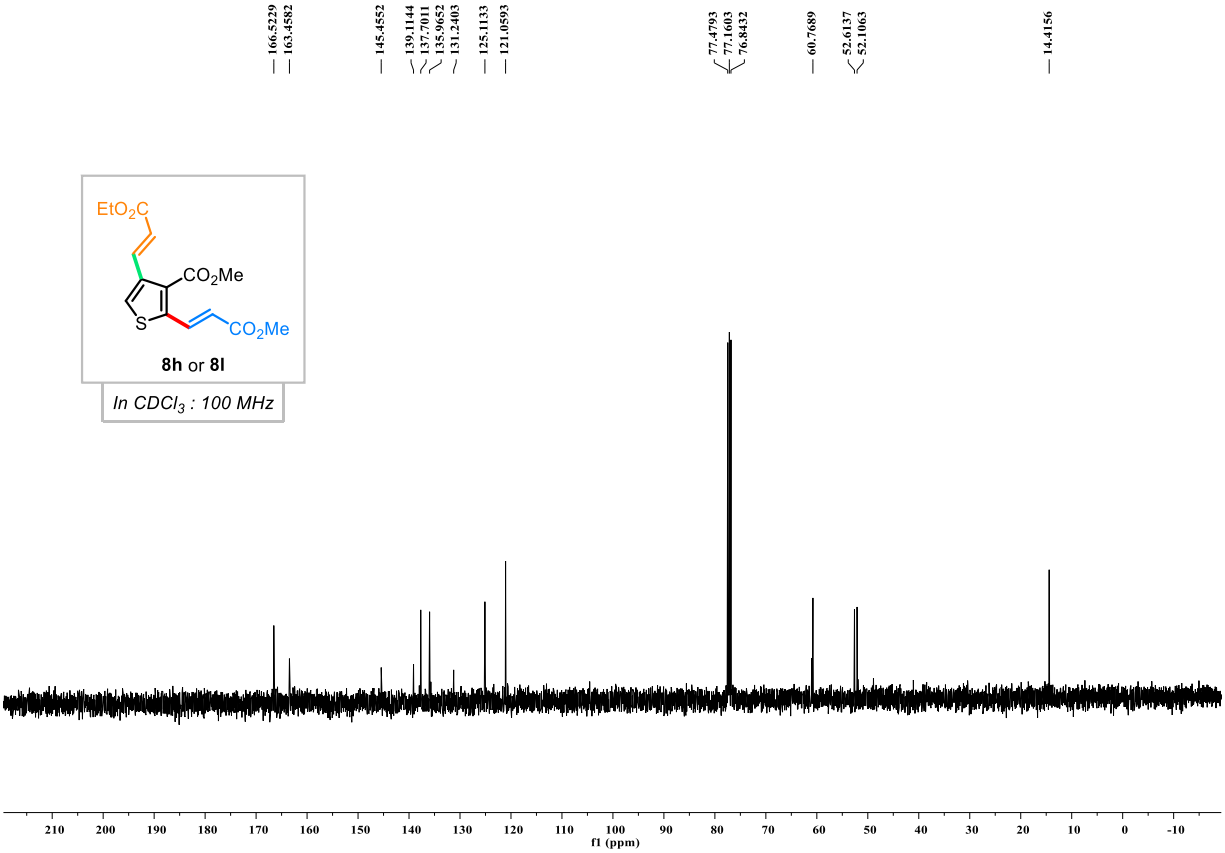
In $CDCl_3$: 400 MHz

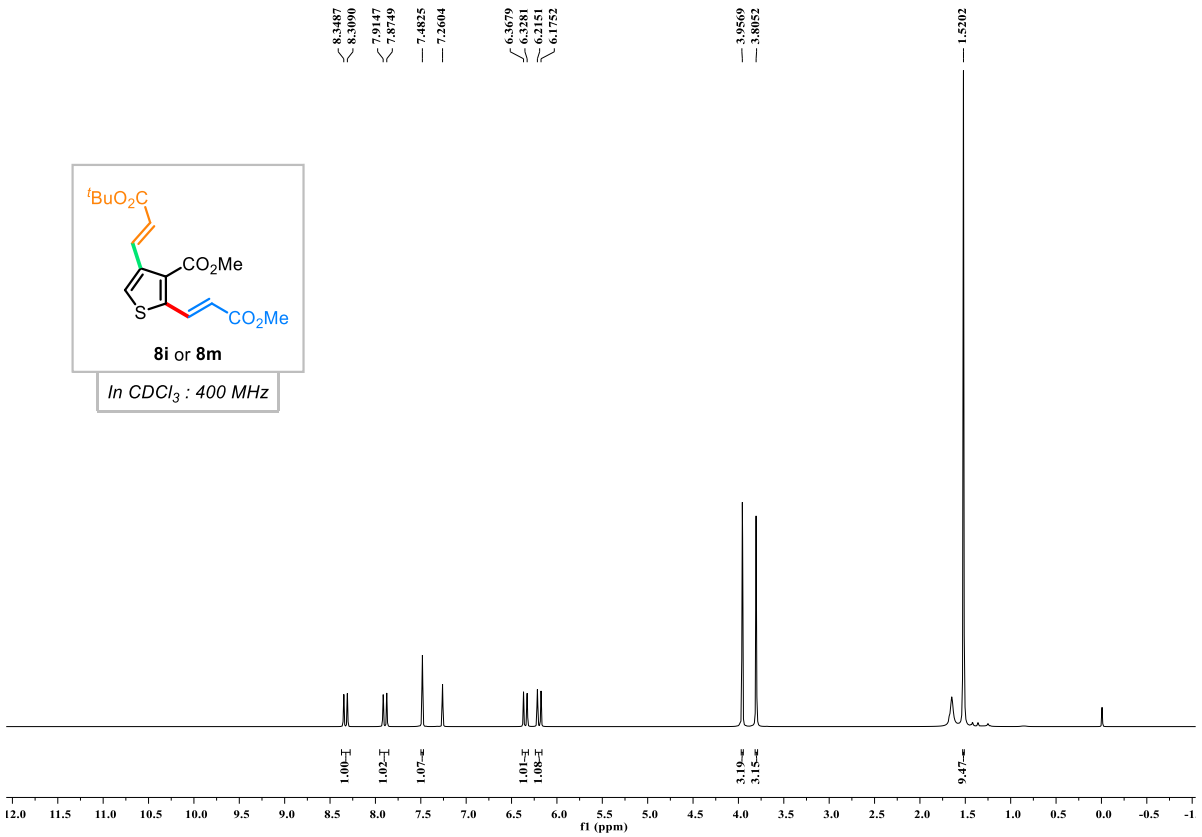


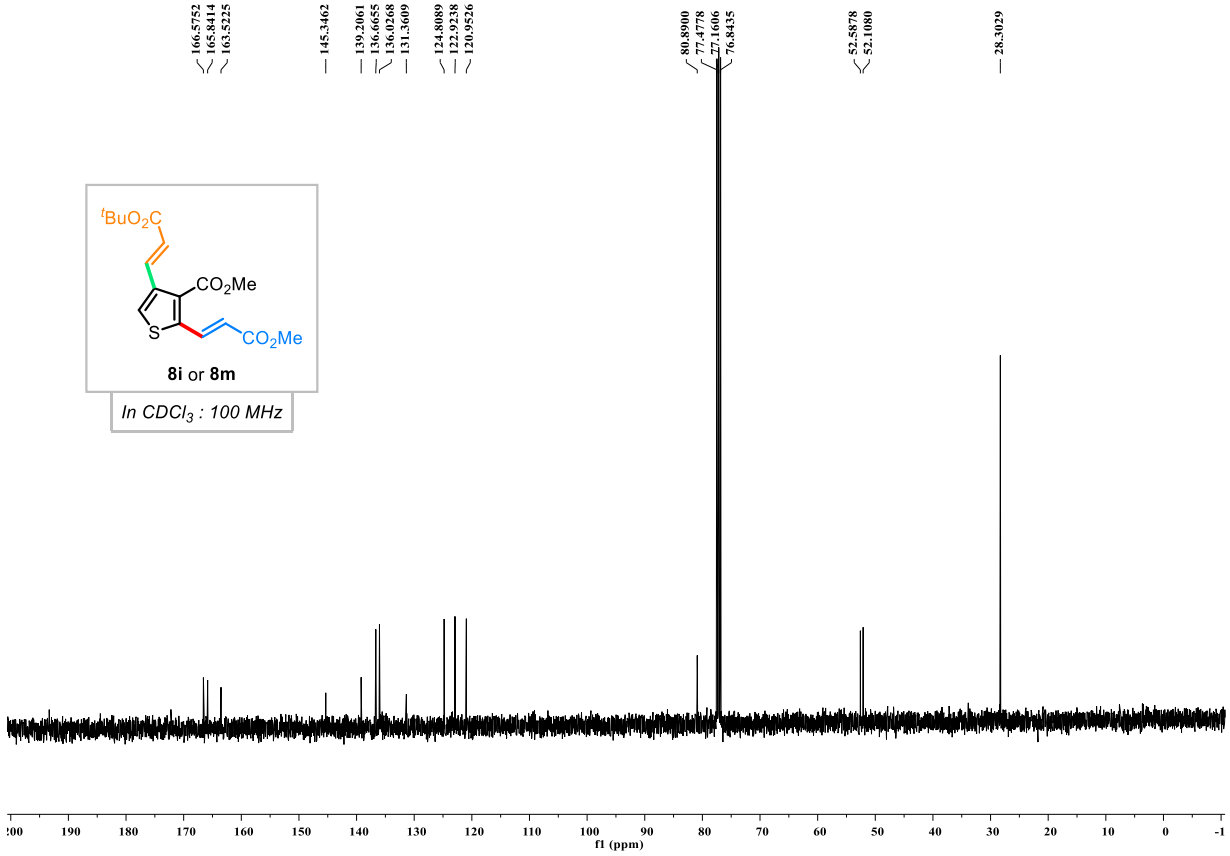

166.5152
163.3835
145.4468
141.4355
141.3552
140.0233
139.7676
135.9555
130.9215
125.0581
125.0388
121.0944
118.2302
116.3332
77.4777
77.1602
76.8420
62.2143
62.1574
52.5913
52.1022
16.5515
16.4875
(EtO)2(O)P
CO2Me
CO2Me
S
8g or 8k
In CDCl3 : 100 MHz
f1 (ppm)

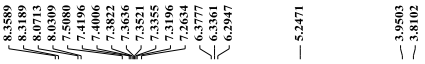

17.8961
(EtO)2(O)P
CO2Me
CO2Me
S
8g or 8k
In CDCl3 : 202 MHz
f1 (ppm)

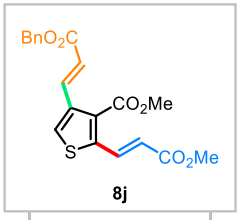

8.3463
8.3065
8.0038
7.9638
7.5009
7.2601
6.3693
6.3296
6.2769
6.2370
4.2721
4.2579
4.2412
4.2235
3.9579
3.8035
1.3401
1.3224
1.3047
EtO2C
CO2Me
CO2Me
S
8h or 8l
In CDCl3 : 400 MHz
1.01
1.00
1.02
1.01
1.07
2.22
3.11
2.91
3.24
f1 (ppm)


166.5229
163.4582
145.4552
139.1144
137.7011
135.9652
131.2403
125.1133
121.0593
77.4793
77.1603
76.8432
60.7689
52.6137
52.1063
14.4156
EtO2C
CO2Me
CO2Me
S
8h or 8l
In CDCl3 : 100 MHz
f1 (ppm)


8.3487
8.3090
7.9147
7.8749
7.4825
7.2604
6.3679
6.3281
6.2151
6.1752
3.9569
3.8052
1.5202
tBuO2C
CO2Me
CO2Me
S
8i or 8m
In CDCl3 : 400 MHz
1.00
1.02
1.07
1.01
1.08
3.19
3.15
9.47
f1 (ppm)

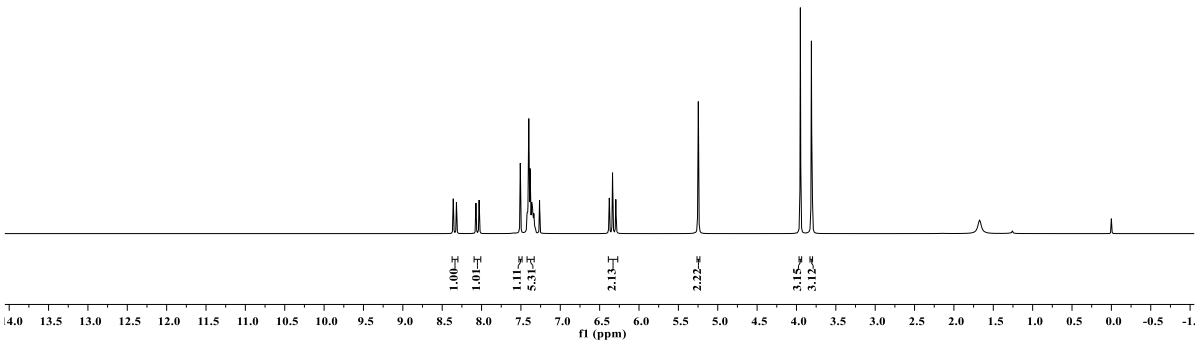

166.5752
165.8414
163.5225
145.3462
139.2061
136.6655
136.0268
131.3609
124.8089
122.9238
120.9526
80.8900
77.4778
27.1606
76.8435
52.5878
52.1080
28.3029
tBuO2C
CO2Me
CO2Me
S
8i or 8m
In CDCl3 : 100 MHz
f1 (ppm)

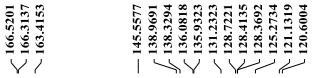


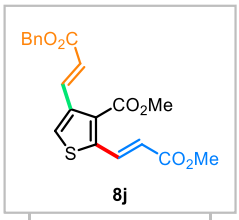


8j

In $CDCl_3$: 400 MHz

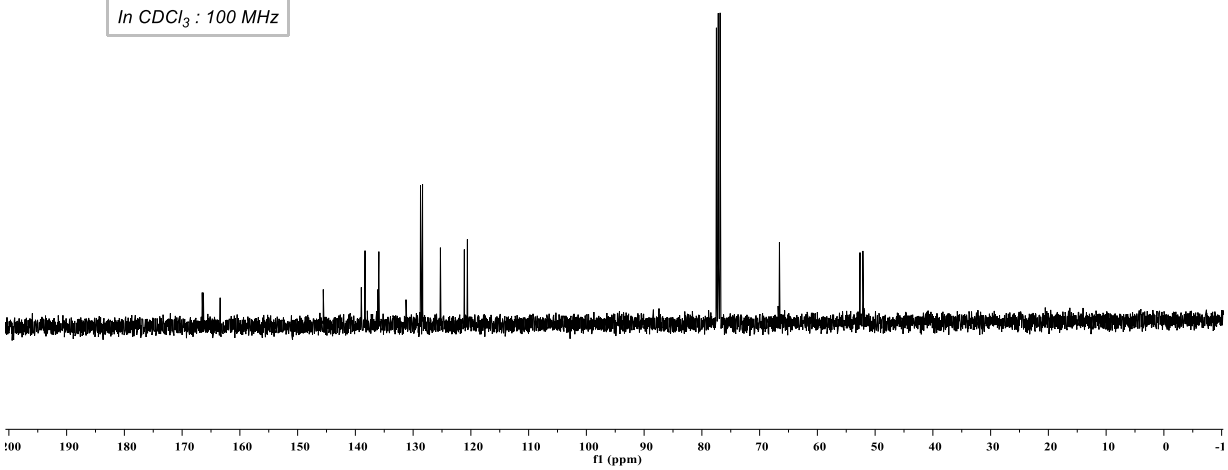


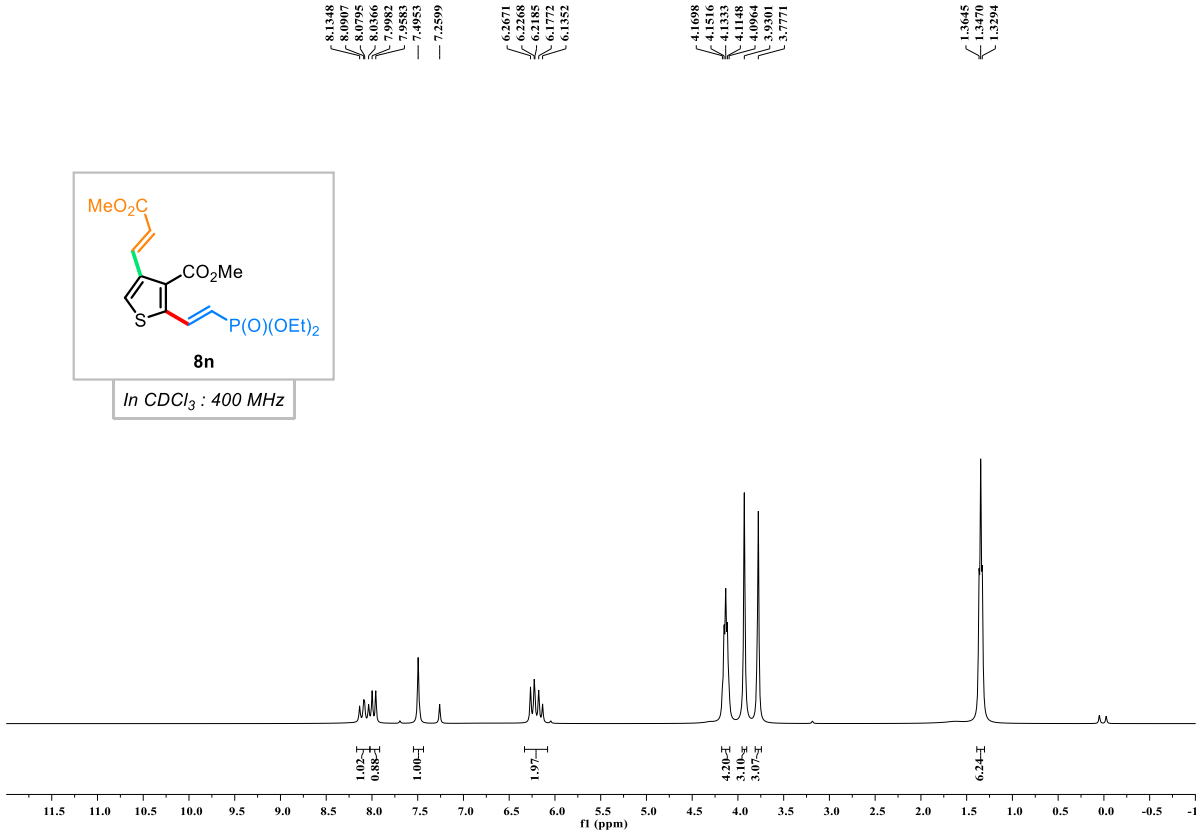


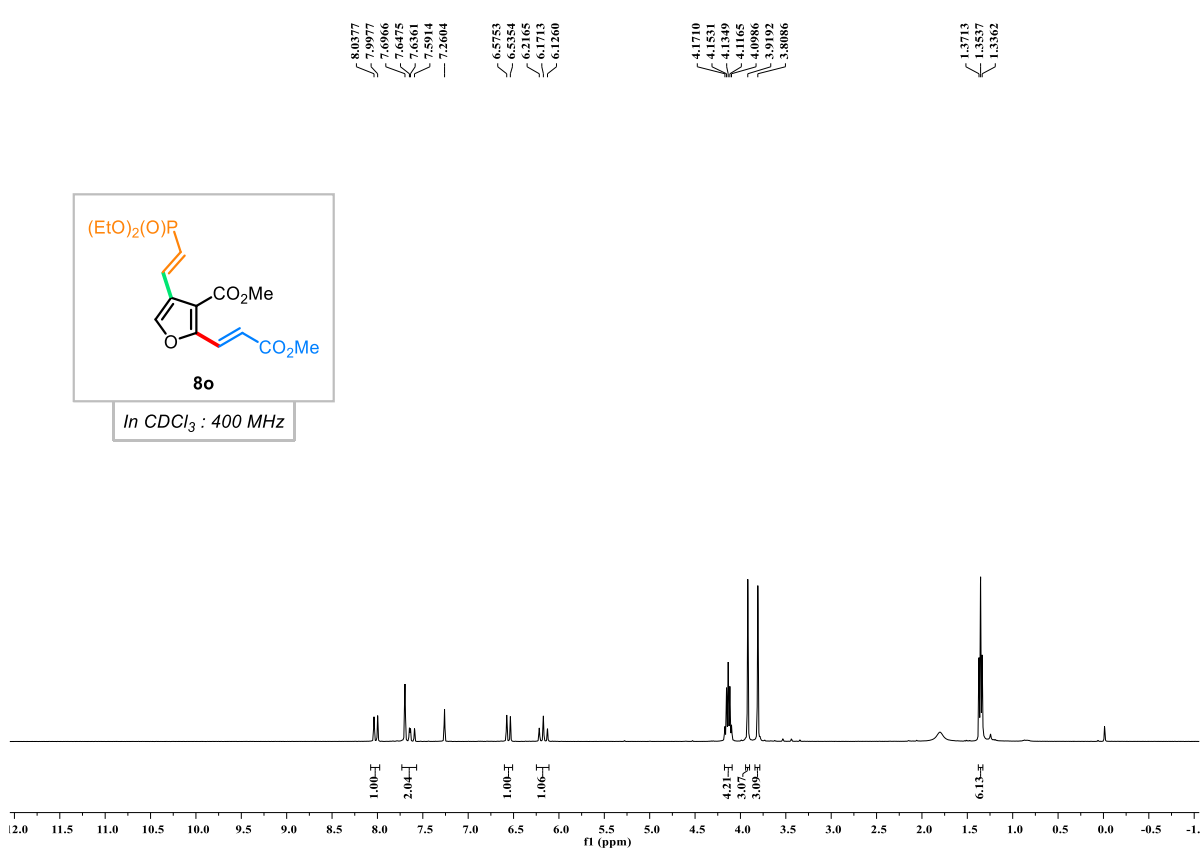


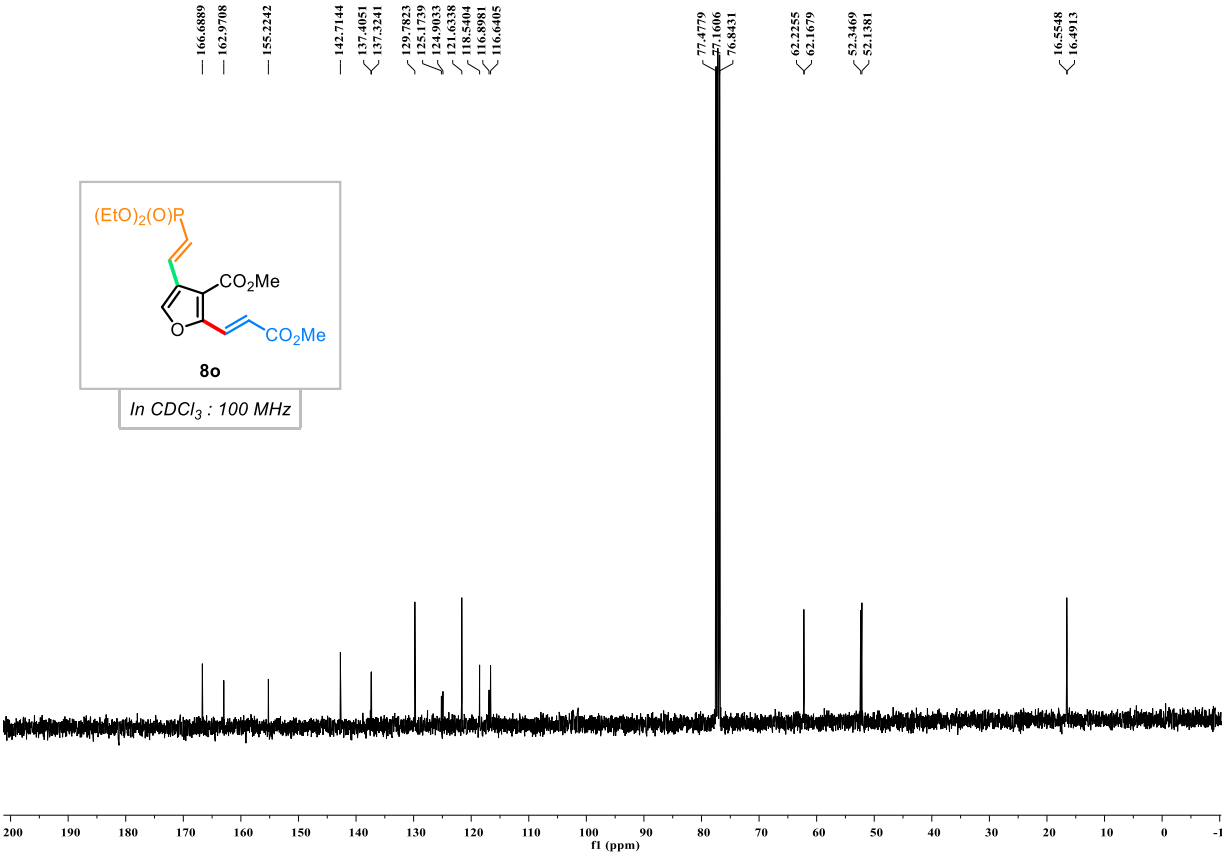
8j

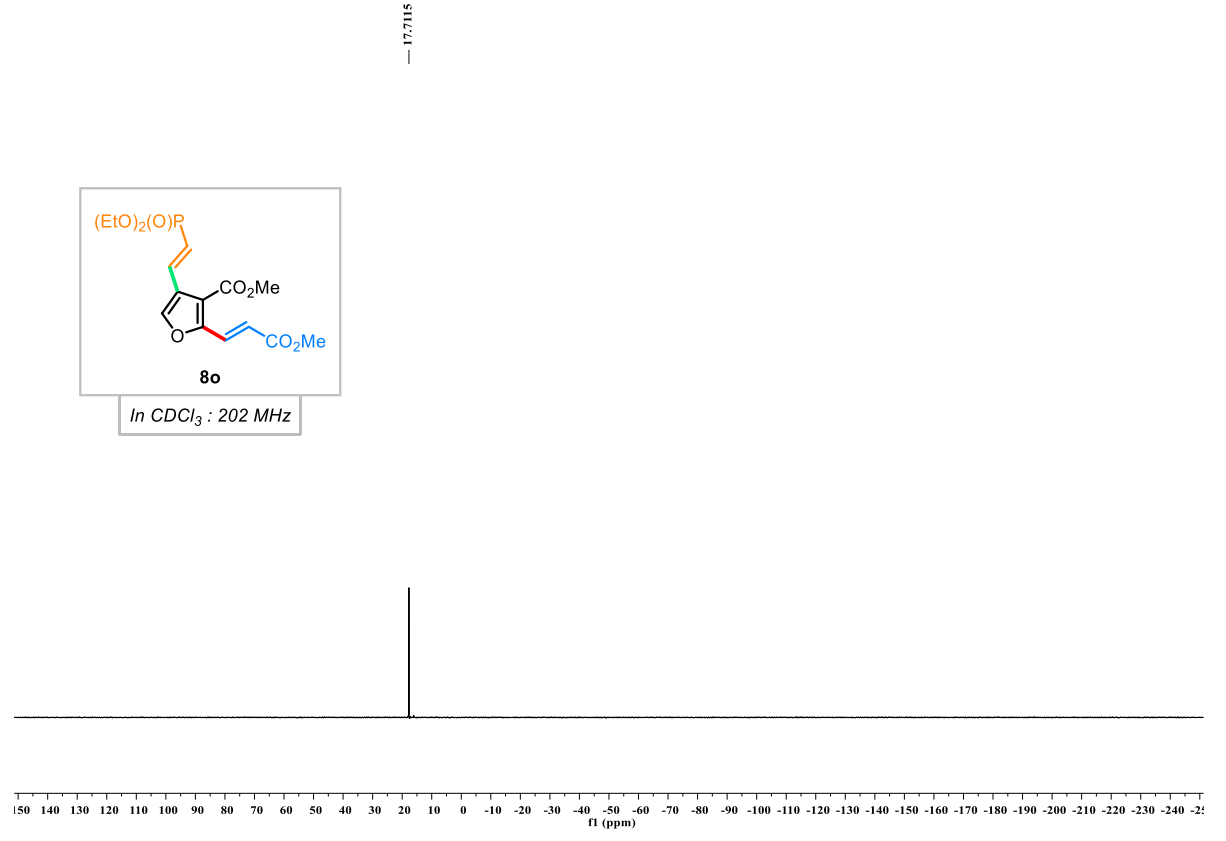
In $CDCl_3$: 100 MHz

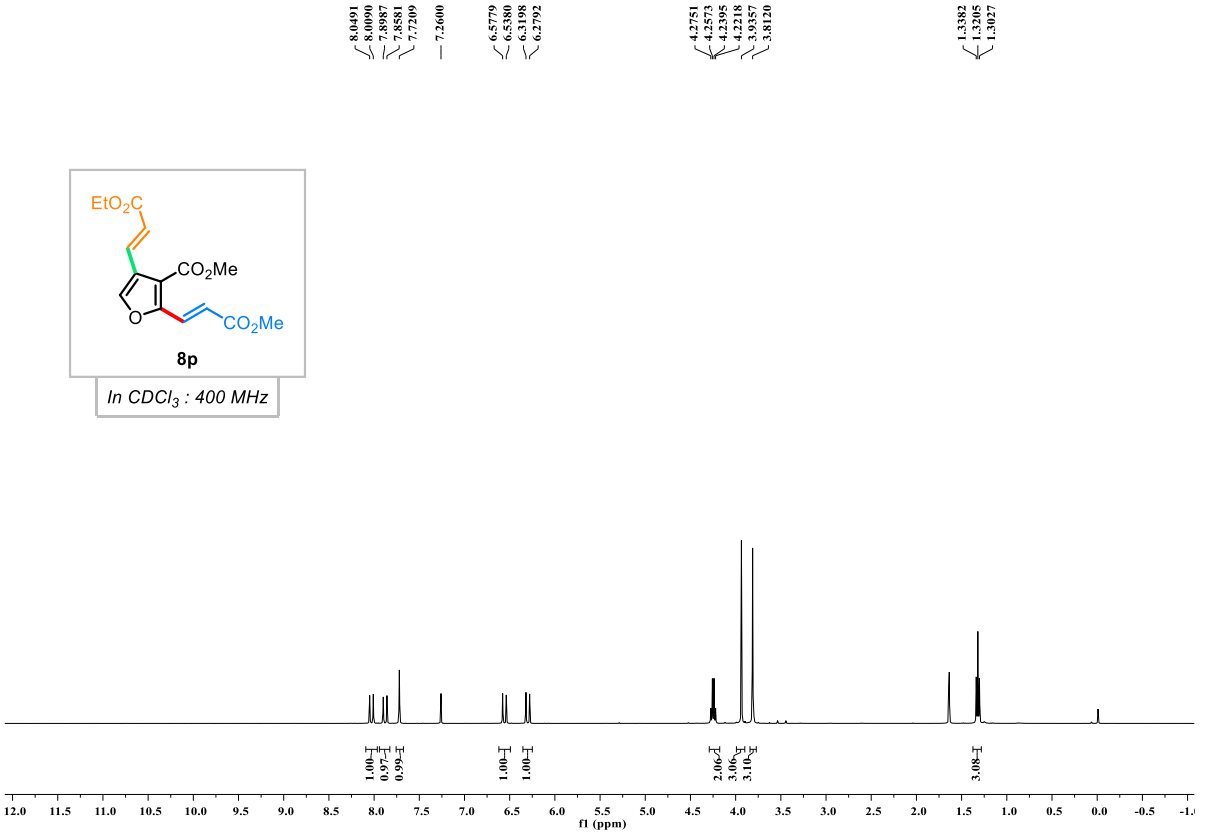


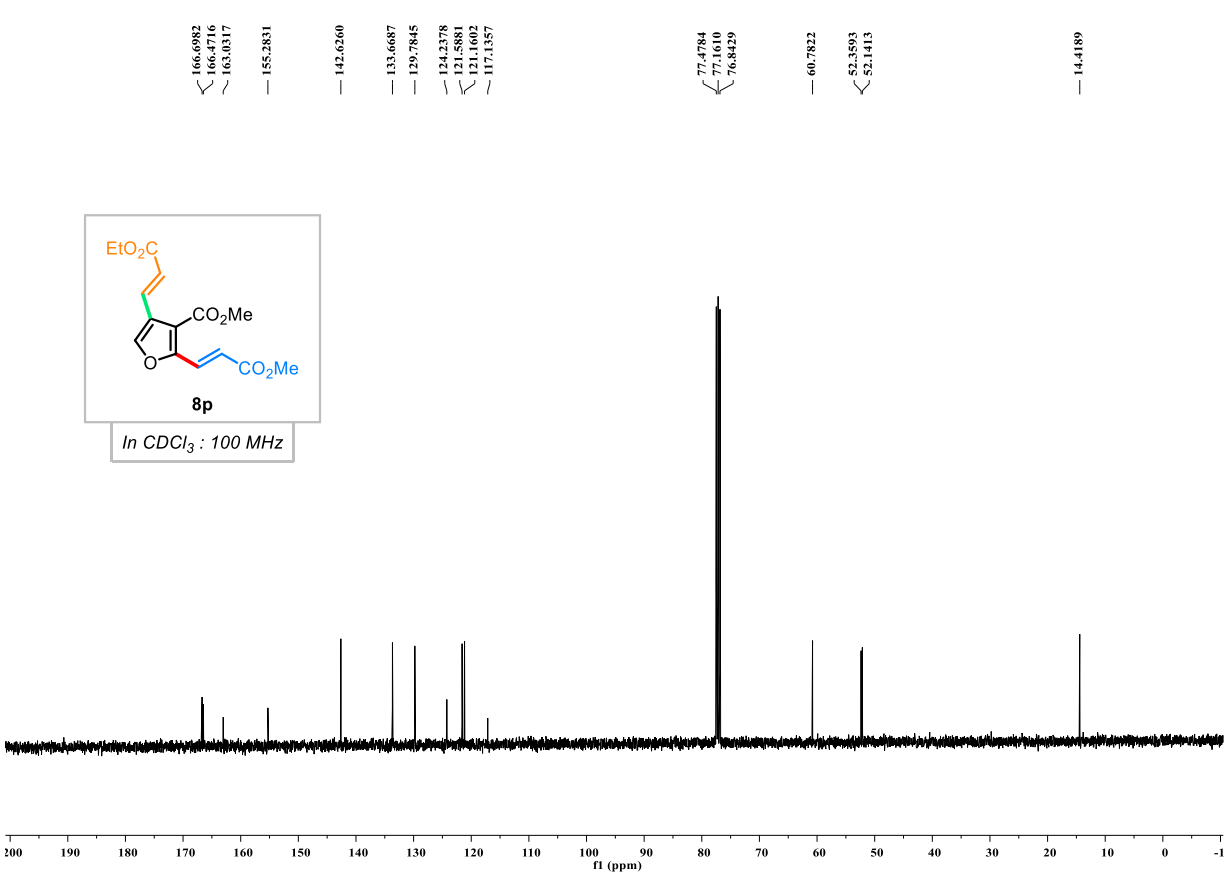

8.1348
8.0997
8.0795
8.0366
7.9982
7.9583
7.4953
7.2599
6.2671
6.2268
6.2185
6.1772
6.1352
4.1698
4.1516
4.1333
4.1148
4.0964
3.9301
3.7771
1.3645
1.3470
1.3294
MeO2C
CO2Me
S
P(O)(OEt)2
8n
In CDCl3 : 400 MHz
1.02
0.88
1.00
1.97
4.20
3.10
3.07
6.24
f1 (ppm)


8.0377
7.9977
7.6966
7.6475
7.6361
7.5914
7.2604
6.5753
6.5354
6.2165
6.1713
6.1260
4.1710
4.1531
4.1349
4.1165
4.0986
3.9192
3.8086
1.3713
1.3537
1.3362
(EtO)2(O)P
CO2Me
O
CO2Me
8o
In CDCl3 : 400 MHz
1.00
2.04
1.00
1.06
4.21
3.07
3.09
6.13
f1 (ppm)


166.6889
162.9708
155.2242
142.7144
137.4051
137.3241
129.7823
125.1739
124.9033
121.6338
118.5404
116.8981
116.6405
77.4779
77.1606
76.8431
62.2255
62.1679
52.3469
52.1381
16.5548
16.4913
(EtO)2(O)P
CO2Me
CO2Me
O
8o
In CDCl3 : 100 MHz
f1 (ppm)


17.7115
(EtO)2(O)P
CO2Me
CO2Me
O
8o
In CDCl3 : 202 MHz
f1 (ppm)


8.0491
8.0090
7.8987
7.8581
7.7209
7.2600
6.5779
6.5380
6.3198
6.2792
4.2751
4.2573
4.2395
4.2218
3.9357
3.8120
1.3382
1.3205
1.3027
EtO2C
CO2Me
CO2Me
8p
In CDCl3 : 400 MHz
1.00
0.97
0.99
1.00
1.00
2.06
3.06
3.16
3.08
f1 (ppm)


166.6982
166.4716
163.0317
155.2831
142.6260
133.6687
129.7845
124.2378
121.5881
121.1602
117.1357
77.4784
77.1610
76.8429
60.7822
52.3593
52.1413
14.4189
EtO2C
CO2Me
CO2Me
8p
In CDCl3 : 100 MHz
f1 (ppm)