Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Highly Regioselective Palladium-Catalysed Oxidative Allylic C-H Carbonylation of Alkenes*

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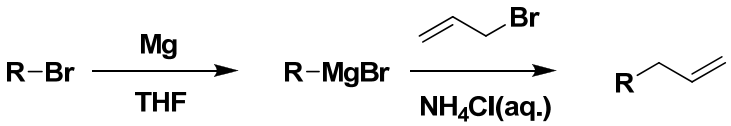
List of Contents

A. General method

^{1}H and ^{13}C NMR spectra were recorded on BRUKER DRX-400 spectrometer using $CDCl_3$ as solvent and TMS as an internal standard. Gas chromatograph mass spectra were obtained with a SHIMADZU model GCMS–QP 5000 spectrometer. HRMS was carried out on a MAT 95XP (Thermo).

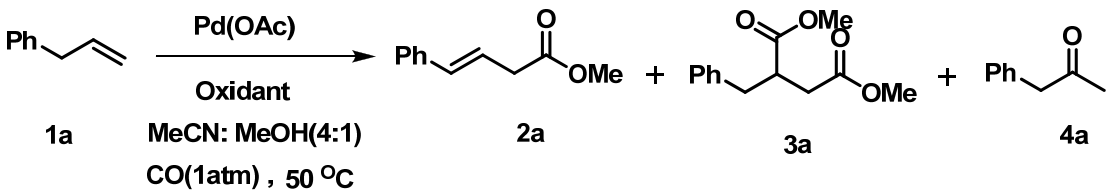
1. Typical reaction procedure for the synthesis of alkenes 1.[1]



R = Ar

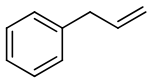
2. The deuterium-labeled substrates *d,d*-1c and *d*-1c were synthesized from $AlLiD_4$ according to a previously reported literature.[1]

3. Optimization of Reaction Conditions



Entry	Ratio of the Oxidant	Yield (%)[b]		
		2a	**3a**	**4a**
1	**BQ(2.0 eq.)/ DDQ(0.5 eq.)**	**82**	**3**	**5**
2	BQ(1.5 eq.)/ DDQ(0.5 eq.)	13	28	11
3	BQ(1.0 eq.)/ DDQ(0.5 eq.)	8	77	3
4	BQ(1.0 eq.)/ DDQ(1.0 eq.)	22	3	38
5	BQ(1.0 eq.)/ DDQ(1.5 eq.)	14	0	53

4. Substrates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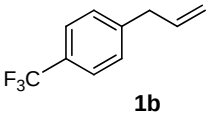

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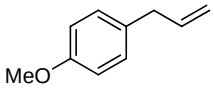

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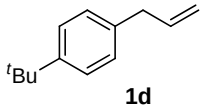

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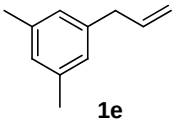
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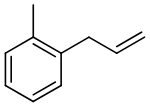
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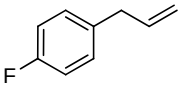

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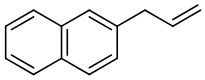
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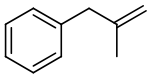
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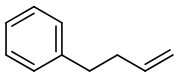
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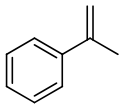
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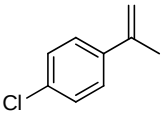

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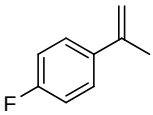

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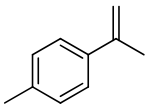
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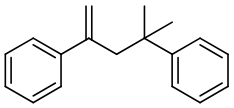
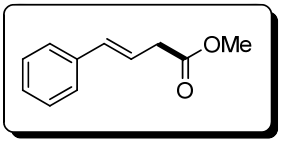

1o

B. General Procedure

General procedure for direct carbonylation of allylic C-H bond with alkenes 1. $Pd(OAc)_2$ (0.03 mol, 6.7 mg), BQ (0.6 mmol, 64.8 mg) and DDQ (0.15 mmol, 34.1 mg) were mixed with alcohol (0.5 ml)/acetonitrile (2 ml) in a glass vial or round-bottom flask equipped with a magnetic stirring bar. Then, alkene **1** was added. The mixture was stirred under a CO atmosphere (1atm) at 50 ^{o}C for 24 h. After cooling down to room temperature and concentrating in vacuum, the residue was purified by flash chromatography on silica gel to obtain the desired products **2** by using light petroleum ether/ethyl acetate (10:1, v/v) as eluent.

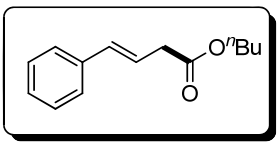
C. Analytical data for compound 2.



Methyl (*E*)-4-phenyl-3-butenoate (**2a**)[2], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.25 (dd, *J* = 1.3, 7.0 Hz, 2H), 3.71 (s, 3H), 6.26-6.33 (m, 1H), 6.48 (d, *J* = 15.9 Hz, 1H), 7.22-7.38 (m, 5 H);

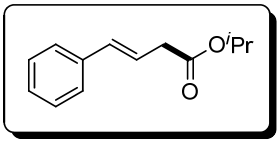
^{13}C NMR ($CDCl_3$, 100 MHz) δ 38.2, 51.9, 121.7, 126.3, 127.6, 128.6, 133.5, 136.8, 172.0 ppm.



Butyl (*E*)-4-phenyl-3-butenoate (2a')[2], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.94 (t, 3H), 1.36-1.42 (m, 2 H), 1.57-1.65 (m, 2 H), 3.24 (d, *J* = 6.8 Hz, 2H), 4.12 (t, 3H), 6.28-6.34 (m, 1H), 6.49 (d, *J* = 16.0 Hz, 1H), 7.23-7.38 (m, 5 H);

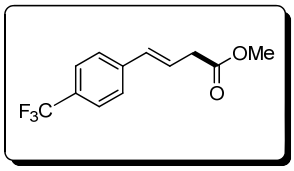
^{13}C NMR ($CDCl_3$, 100 MHz) δ 13.7, 19.1, 30.7, 38.5, 64.7, 121.9, 126.3, 127.5, 128.5, 133.3, 136.9, 171.7 ppm.



Isopropyl (*E*)-4-phenyl-3-butenoate (**2a''**)[2], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.18 (d, 6H), 3.13 (d, 2 H), 4.94-5.0 (m, 1 H), 6.18-6.26 (m, 1H), 6.41 (d, *J* = 16.0 Hz, 1H), 7.11-7.30 (m, 5 H);

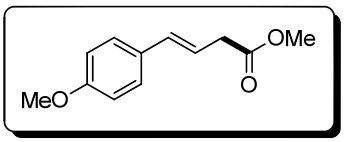
^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.8, 38.8, 68.1, 122.1, 126.3, 127.5, 128.5, 133.2, 137.0, 171.1 ppm.



Methyl (*E*)-4-(4-trifluoromethylphenyl)-3-butenoate (**2b**)[10], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.29 (d, 2H), 3.73 (s, 3 H), 6.36-6.44 (m, 1H), 6.53 (d, *J* = 16.0 Hz, 1H), 7.46 (d, *J* = 8.0 Hz, 2 H), 7.56 (d, *J* = 8.0 Hz, 5 H);

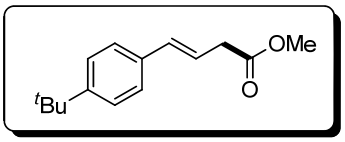
^{13}C NMR ($CDCl_3$, 100 MHz) δ□ 37.151.0, 123.5, 124.5(quartet), 127.8, 128.1, 129.9, 131.2, 139.2170.6 ppm.



Methyl (*E*)-4-(4-methoxyphenyl)-3-butenoate (**2c**)[2], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.23 (dd, *J* = 1.3, 7.0 Hz, 2H), 3.71 (s, 3H), 3.80 (s, 3 H), 6.11-6.19 (m, 1H), 6.43 (d, *J* = 15.9 Hz, 1H), 6.84 (d, *J* = 8.8 Hz, 2H); 7.30 (d, *J* = 8.8 Hz, 2H);

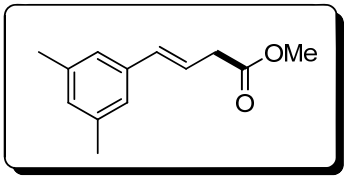
^{13}C NMR ($CDCl_3$, 100 MHz) δ□ 38.2, 51.9, 55.3, 114.0, 119.4, 127.5, 129.7, 132.9, 159.2, 172.2 ppm.



Methyl (*E*)-4-(4-*tert*-butylphenyl)-3-butenoate (**2d**) [2], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.31 (s, 9H), 3.24 (d, *J* = 7.1 Hz, 2H), 3.71 (s, 3 H), 6.21-6.29 (m, 1H), 6.47 (d, *J* = 15.9 Hz, 1H), 7.32 (d, 4H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ□ 31.3, 34.6, 38.3, 51.9, 120.9, 125.5, 126.0, 133.2, 134.1, 150.7, 172.1 ppm.

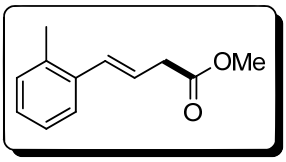


Methyl (*E*)-4-(3,5-dimethylphenyl)-3-butenoate (**2e**), colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.30 (s, 6H), 3.23 (d, *J* = 7.0 Hz, 2H), 3.71 (s, 3 H), 6.22-6.30 (m, 1H), 6.42 (d, *J* = 15.9 Hz, 1H), 6.88 (s, 1H), 6.99 (s, 2H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.2, 38.3, 51.9, 121.2, 124.2, 129.3, 133.6, 136.7, 138.0, 172.1 ppm.

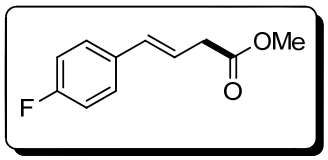
HRMS EI (m/z): calcd for $C_{13}H_{16}O_2$, 204.1155; found, 204.1150.



Methyl (*E*)-4-(2-methylphenyl)-3-butenoate (**2f**)[3], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.33 (s, 3H), 3.28 (d, *J* = 7.1 Hz, 2H), 3.72 (s, 3 H), 6.14-6.21 (m, 1H), 6.70 (d, *J* = 15.7 Hz, 1H), 7.15 (m, 3H), 7.43 (d, 1H);

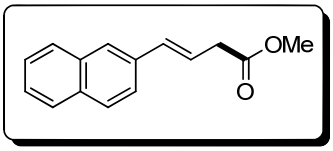
^{13}C NMR ($CDCl_3$, 100 MHz) δ 19.8, 38.5, 51.9, 122.9, 125.7, 126.1, 127.5, 130.2, 131.4, 135.2, 136.0, 172.1 ppm.



Methyl (*E*)-4-(4-fluorophenyl)-3-butenoate (**2g**)[2], pale yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.28 (d, *J* = 7.1 Hz, 2H), 3.72 (s, 3 H), 6.18-6.25 (m, 1H), 6.70 (d, *J* = 15.9 Hz, 1H), 7.15 (t, 2H), 7.43 (t, 2H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 38.1, 52.0, 115.4 (d, *J* = 1.4 Hz), 121.4 (d, *J* = 2.3 Hz), 127.8 (d, *J* = 8.9 Hz), 129.7, 132.3, 162.2 (d, *J* = 245 Hz), 172.0 ppm.



Methyl (*E*)- 3-(Naphthalen-2-yl)-3-butenoate (**2h**), colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.32 (d, *J* = 7.1 Hz, 2H), 3.74 (s, 3 H), 6.39-6.47 (m, 1H), 6.65 (d, *J* = 15.9 Hz, 1H), 7.41-7.47 (m, 2H), 7.60 (s, 1H), 7.71 (s, 1H), 7.77-7.80 (m, 3H)

^{13}C NMR ($CDCl_3$, 100 MHz) δ 38.4, 52.0, 122.0, 123.5, 125.9, 126.2, 126.3, 127.7, 128.0, 128.2, 133.0, 133.6, 133.6, 134.3, 172.1 ppm.

HRMS EI (m/z): calcd for $C_{15}H_{14}O_2$, 226.0989; found, 226.0994.

Methyl (*E*)-3-methyl-4-phenyl-3-butenoate (**2ia**)[4], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.94 (s, 3H), 3.19 (s, 2H), 3.72 (s, 3H), 6.39 (s, 1H), 7.20-7.33 (m, 5 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 18.0, 45.7, 51.9, 126.4, 128.1, 128.4, 128.9, 129.1, 129.1, 172.0 ppm.

Methyl 3-benzyl-3-butenoate (**2ib**)

^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.99 (s, 2H), 3.45 (s, 2H), 3.66 (s, 3H), 4.96 (s, 1H), 4.99 (s, 1H), 7.20-7.33 (m, 5 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 40.9, 42.7, 51.8, 115.8, 126.5, 131.5, 137.6, 138.7, 141.8, 171.8 ppm.

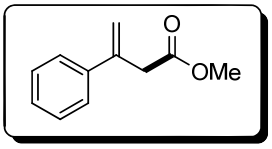
Methyl (*E*)-5-phenyl-4-pentenoate (**2ja**)[8], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.49-2.55 (m, 4H), 3.69 (s, 3H), 6.66-6.24 (m, 1H), 6.43 (d, *J* = 15.9 Hz, 1H), 7.17-7.34 (m, 5 H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 28.3, 33.8, 51.6, 126.1, 127.2, 128.4, 128.5, 131.0, 137.4, 173.4 ppm.

Methyl (*E*)-5-phenyl-3-pentenoate (**2jb**)[9]

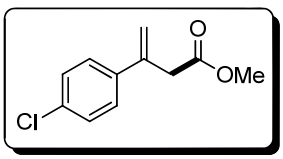
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.07 (d, *J* = 6.6 Hz, 2H), 3.38 (d, *J* = 6.4 Hz, 2H), 3.69 (s, 3H), 5.63-5.74 (m, 2H), 7.17-7.34 (m, 5 H).



Methyl 3-phenyl-3-butenoate (**2k**)[6], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.53 (s, 2H), 3.66 (s, 3H), 5.24 (s, 1H), 5.56 (s, 1H), 7.27-7.35 (m, 3H), 7.43 (d, 2H);

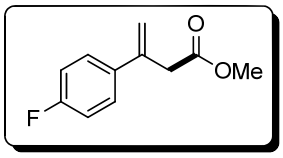
^{13}C NMR ($CDCl_3$, 100 MHz) δ 41.1, 52.0, 116.3, 125.8, 127.8, 128.4, 139.7, 140.8, 171.8 ppm.



Methyl 3-(4-chlorophenyl)-3-butenoate (**2l**)[6], pale yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.50 (s, 2H), 3.66 (s, 3H), 5.25 (s, 1H), 5.54 (s, 1H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.36 (d, *J* = 8.4 Hz, 2H);

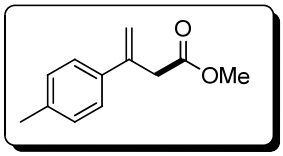
^{13}C NMR ($CDCl_3$, 100 MHz) δ 41.0, 52.1, 117.0, 125.4, 127.8, 128.4, 139.7, 140.8, 171.8 ppm.



Methyl 3-(4-fluorophenyl)-3-butenoate (**2m**)[6], yellow oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.50 (s, 2H), 3.66 (s, 3H), 5.22 (s, 1H), 5.49 (s, 1H), 7.01 (t, 2H), 7.40 (t, 2H);

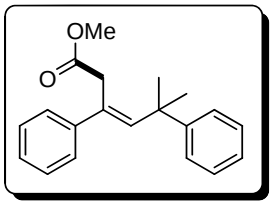
^{13}C NMR ($CDCl_3$, 100 MHz) δ 41.2, 52.0, 115.2 (d, J = 21.4 Hz) , 116.3, 127.5 (d, J = 8.0 Hz), 135.8 (d, J = 3.4 Hz), 139.9, 162.5 (d, J = 245 Hz), 171.6 ppm.



Methyl 3-(4-methylphenyl)-3-butenoate (**2n**)[8], colorless oil.

^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.34 (s, 3H), 3.51 (d, J = 0.4 Hz, 2H), 3.65 (s, 3H), 5.18 (s, 1H), 5.53 (s, 1H), 7.13 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H);

^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.1, 41.1, 52.0, 115.4, 125.6, 129.1, 136.8, 137.7, 140.6, 171.9 ppm.



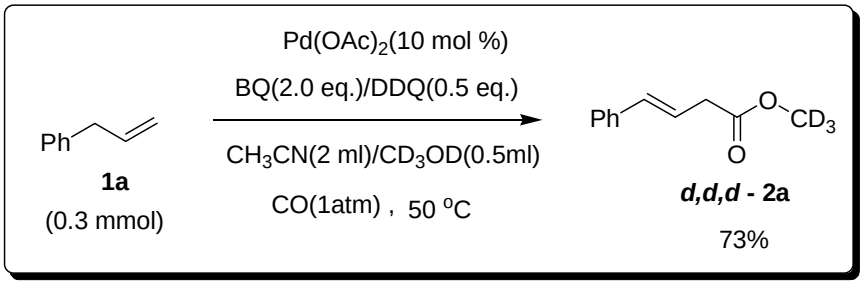
Methyl (*E*)-5-methyl-3,5-diphenyl-3-hexenoate (**3o**), yellow oil.

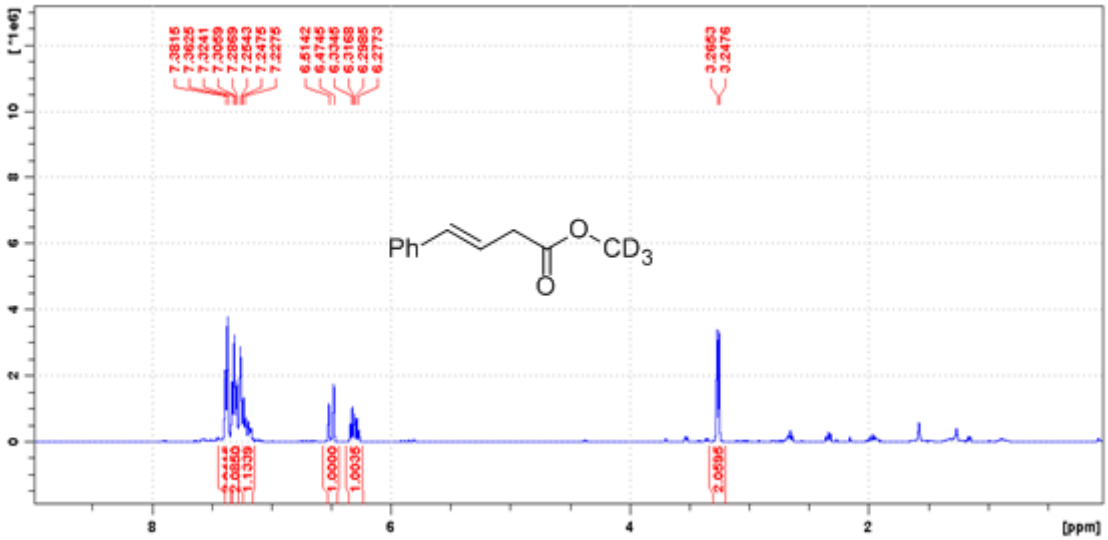
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.53 (s, 6H), 3.01 (s, 2H), 3.41 (s, 3H), 6.25 (s, 1H), 7.16-7.41 (m, 10H);

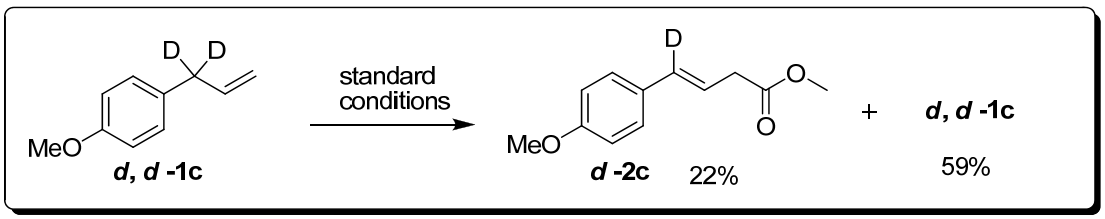
^{13}C NMR ($CDCl_3$, 100 MHz) δ 31.6, 35.7, 40.3, 51.6, 125.8, 126.2, 126.3, 127.1, 128.3, 128.4, 133.5, 142.5, 142.9, 149.5, 171.3 ppm.

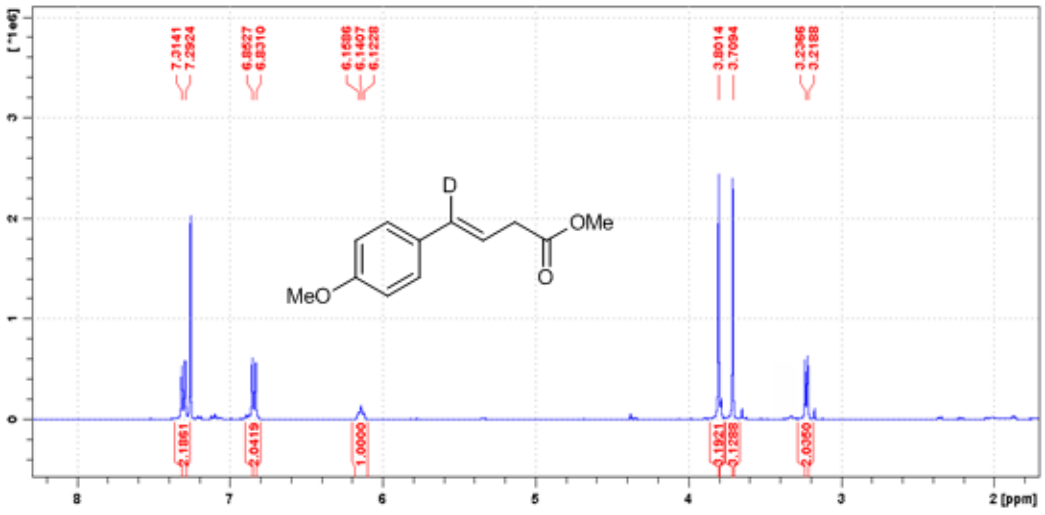
HRMS EI (m/z): calcd for $C_{20}H_{22}O_2$, 294.1618; found, 294.1620.

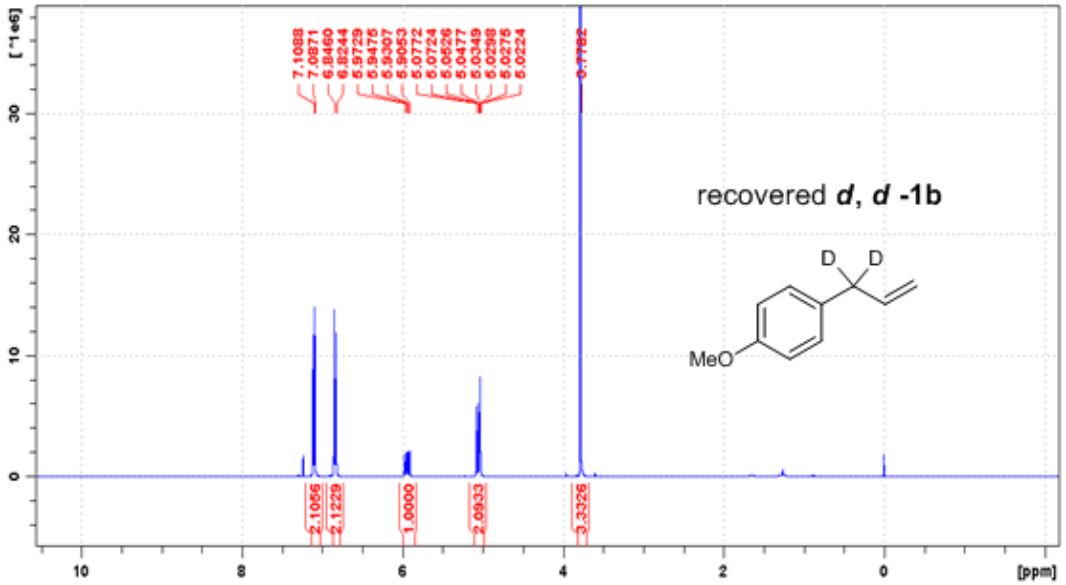
E. Mechanistic studies

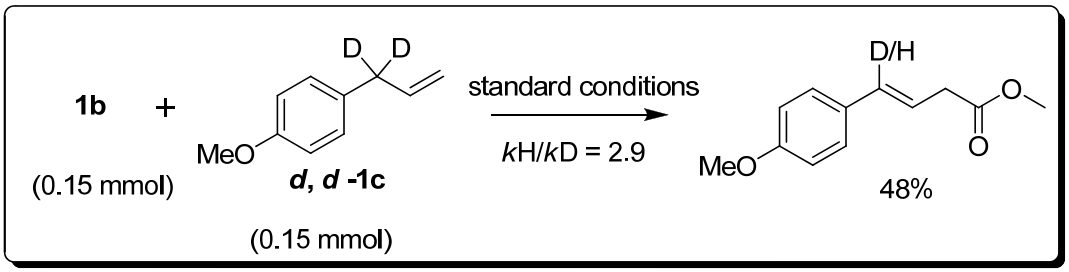


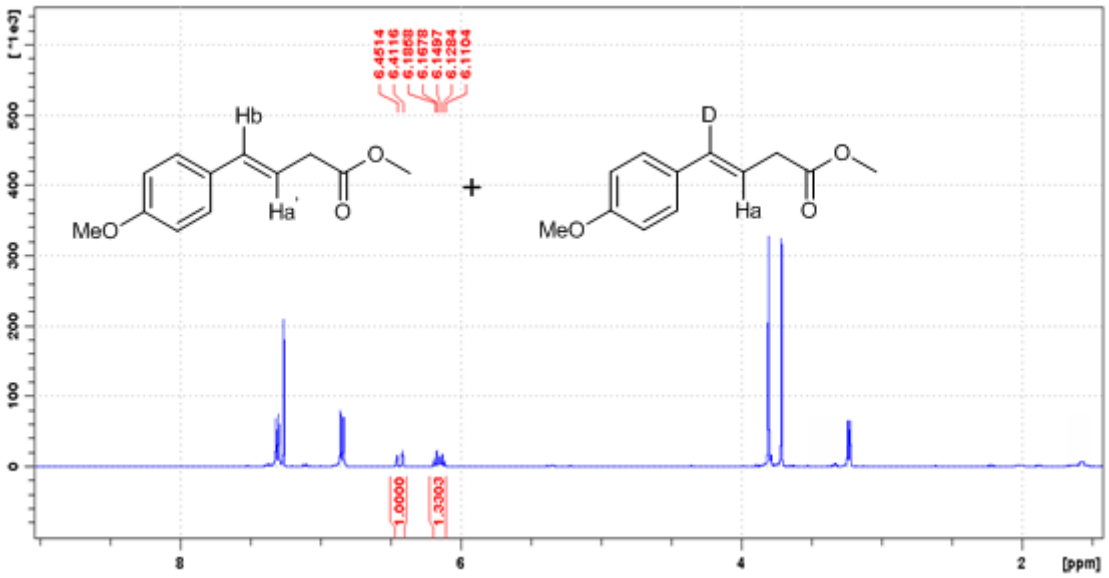


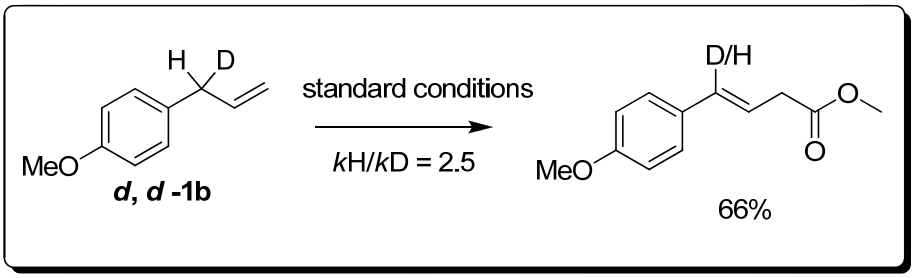


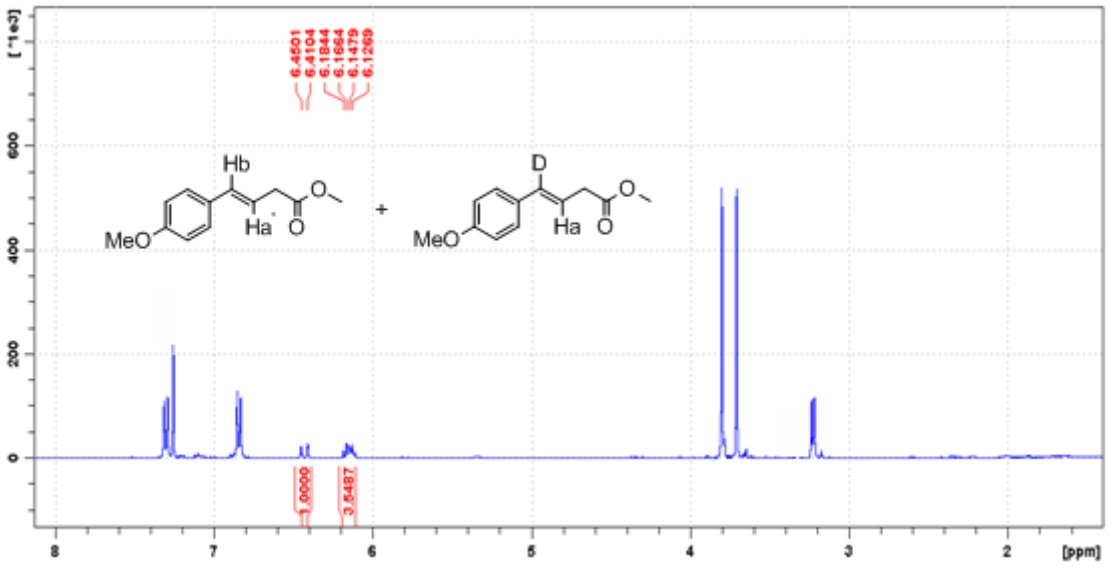



recovered d, d -1b
D
D
MeO
[ppm]


1b
+
(0.15 mmol)
D
D
MeO
d, d -1c
(0.15 mmol)
standard conditions
kH/kD = 2.9
D/H
MeO
O
O
48%


Hb
Ha
MeO
O
O
+
D
Ha
MeO
O
O
[ppm]





F. References

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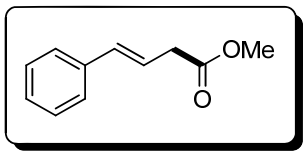
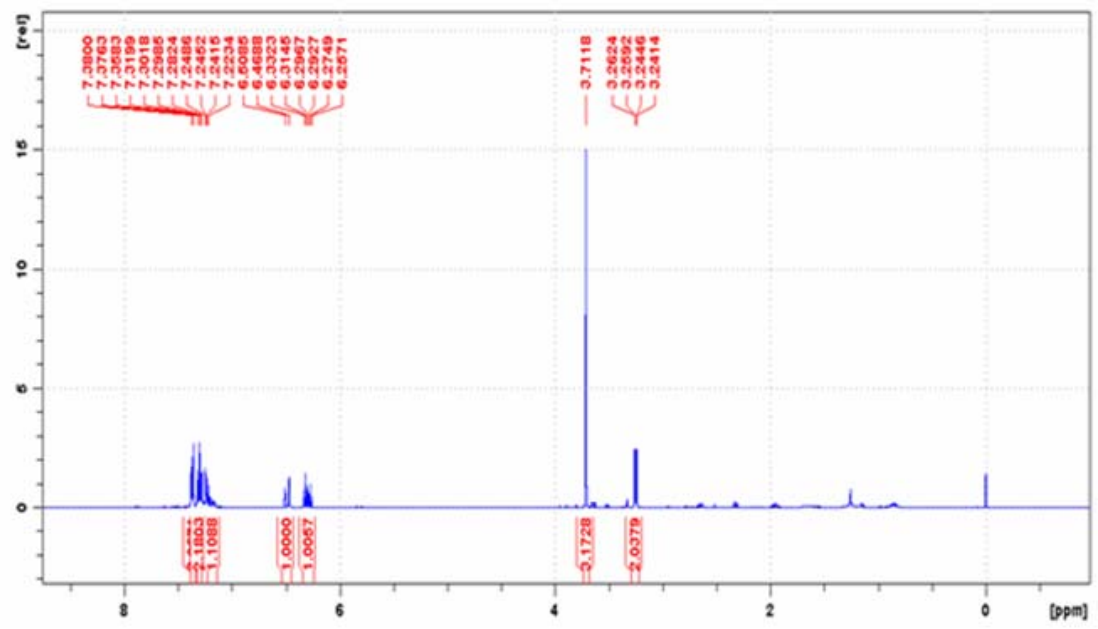
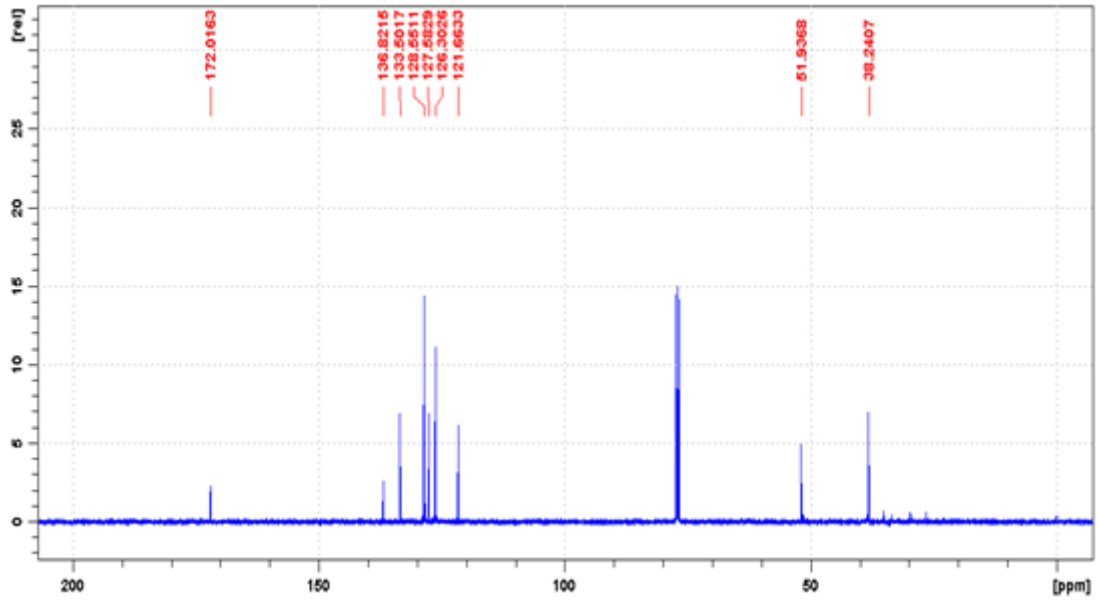
(9) Pak, C. S.; Lee, E.; Lee, G. H. *J. Org. Chem.* **1993**, *58*, 1523.
(10) Bulugahapitiya, P.; Landais, Y.; Parra-Rapado, L.; Planchenault, L.; Weber, V. *J. Org. Chem.* **1997**, *62*, 1630.

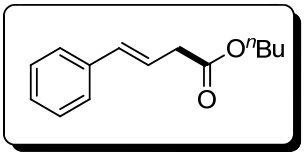
F. NMR Spectra



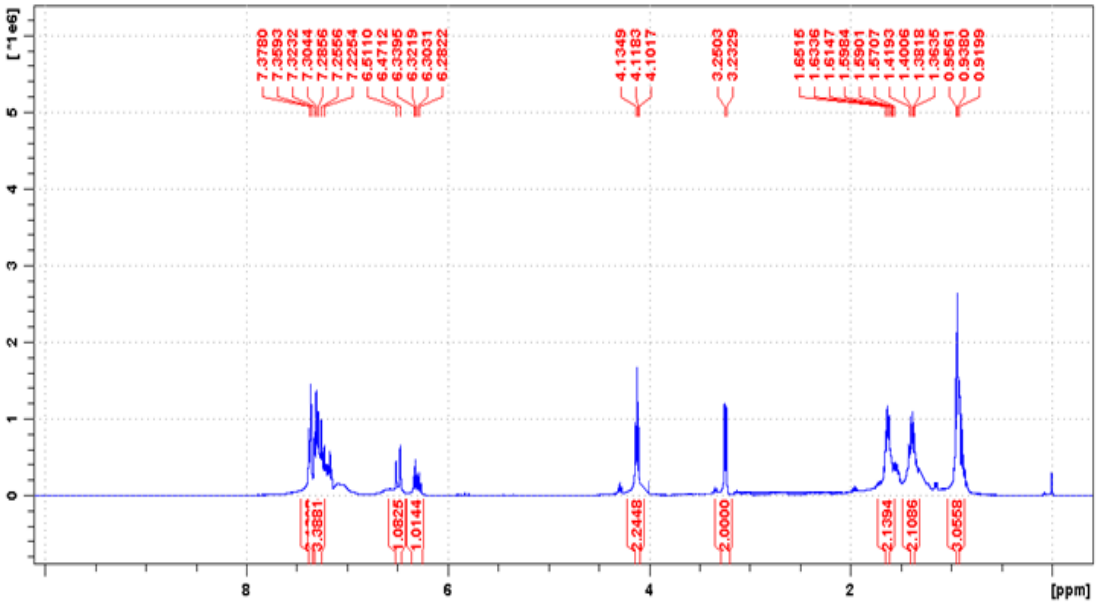
2a






O^{n}Bu
O

2a'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6.3395
6.3219
6.3031
6.2822
4.1349
4.1183
4.1017
3.2503
3.2329
1.6515
1.6336
1.6147
1.5984
1.5901
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1.4193
1.4006
1.3818
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0.9380
0.9199
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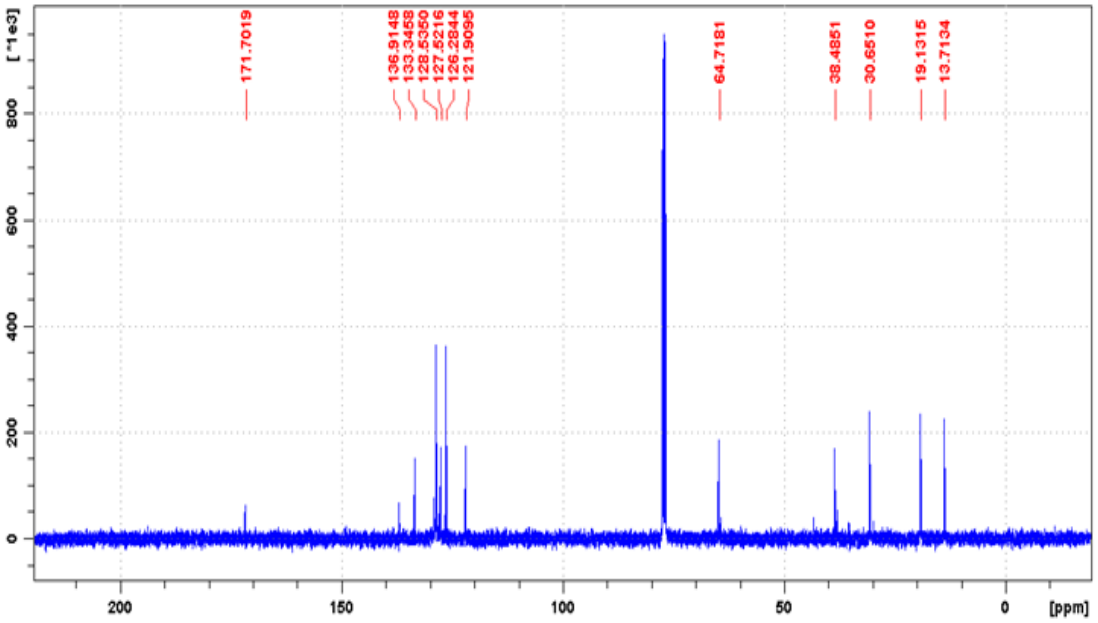
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30.6510
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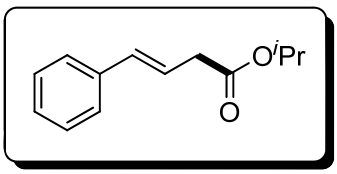
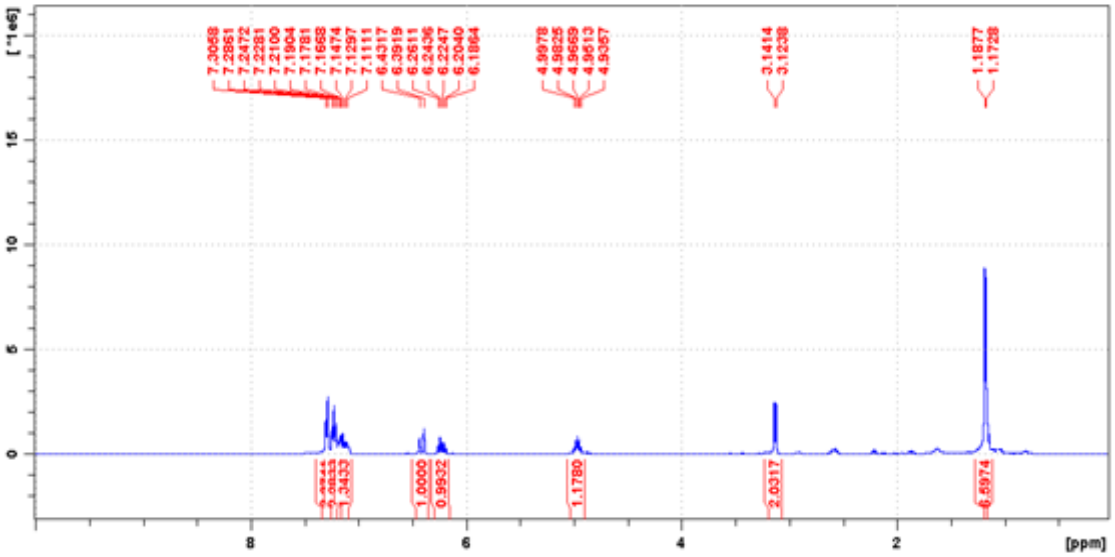
13.7134
[ppm]


O^{i}Pr
O

2a''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7.1781
7.1688
7.1474
7.1297
7.1111
6.4317
6.3919
6.2511
6.2436
6.2247
6.2040
6.1864
4.9978
4.9825
4.9669
4.9513
4.9357
3.1414
3.1238
1.1877
1.1728
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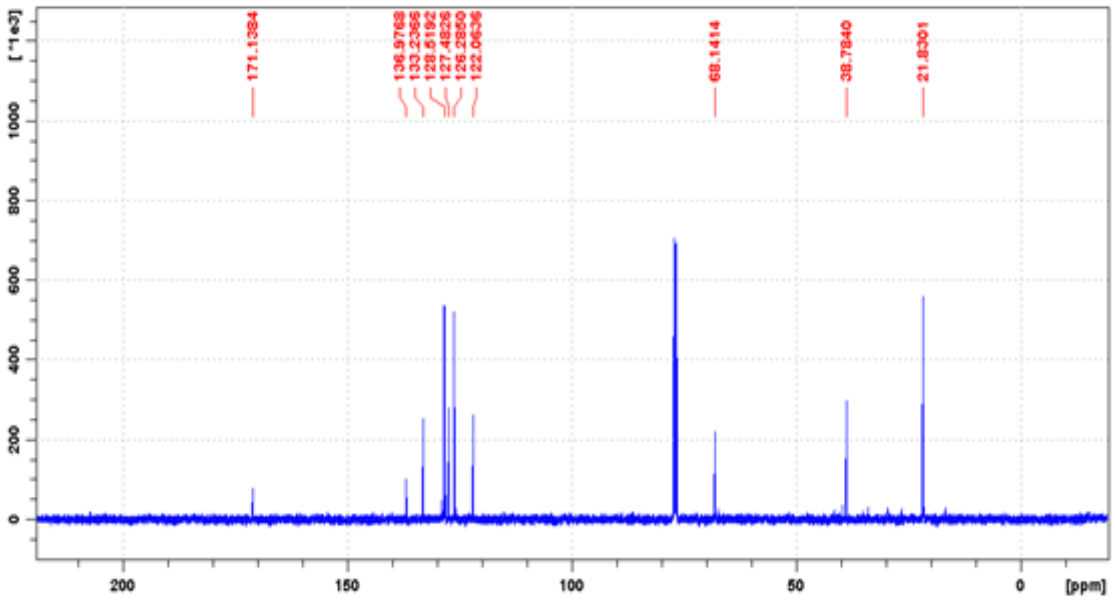
1.1780
2.0317
6.5974
8
6
4
2
[ppm]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100
50
0
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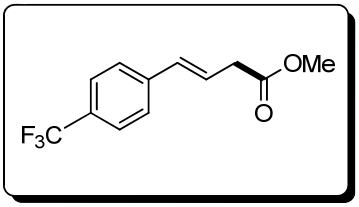
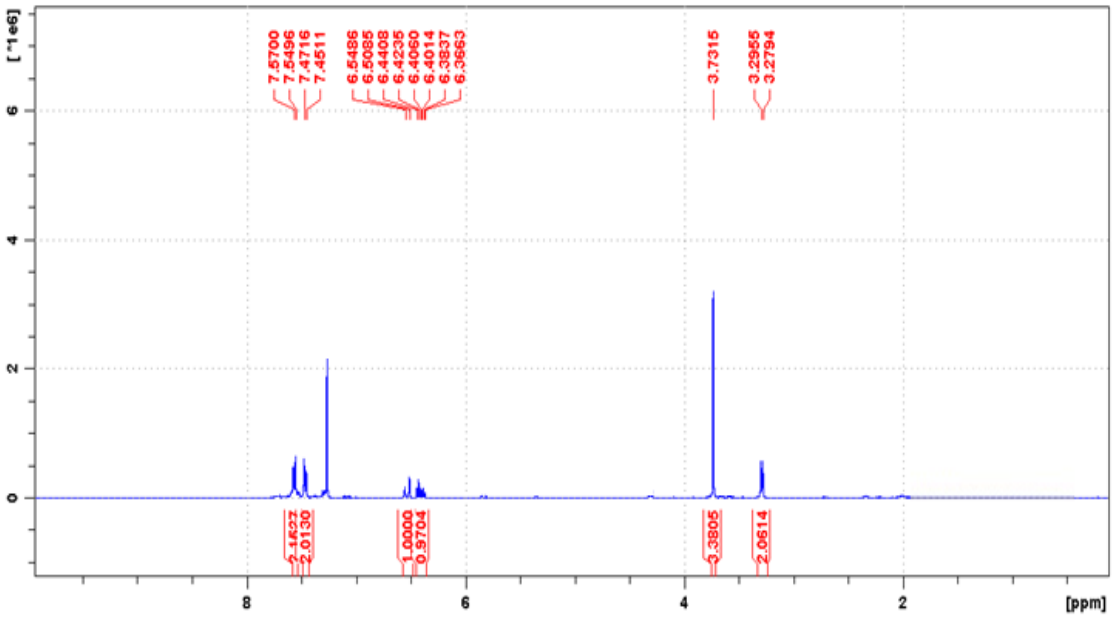

OMe
O
F3C

2b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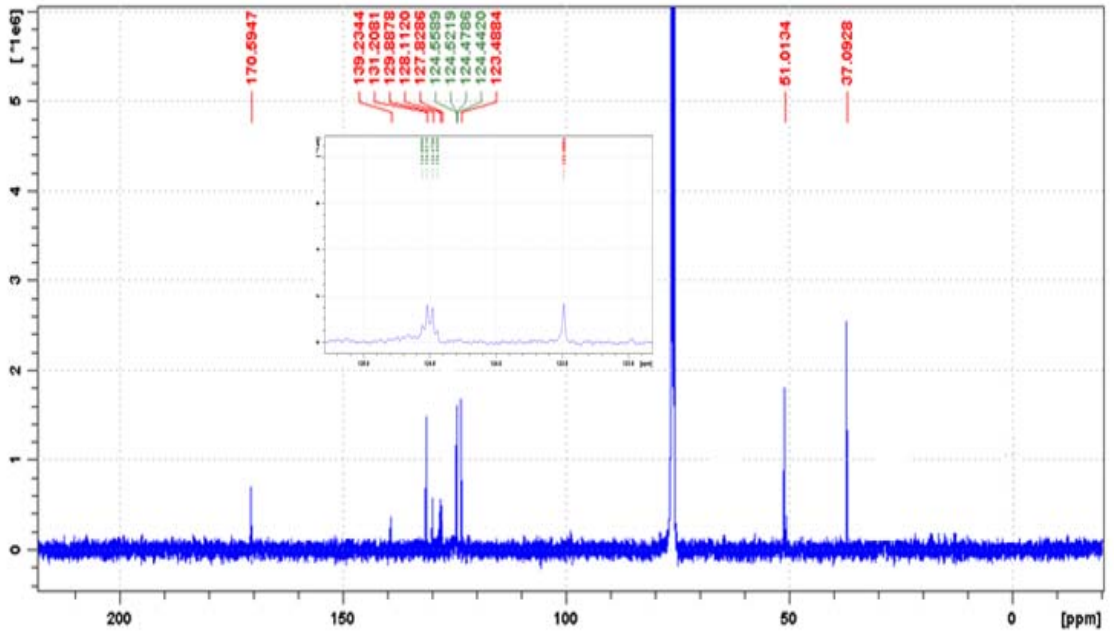
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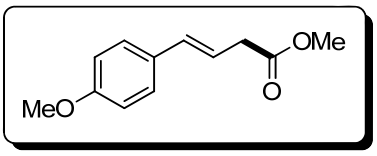
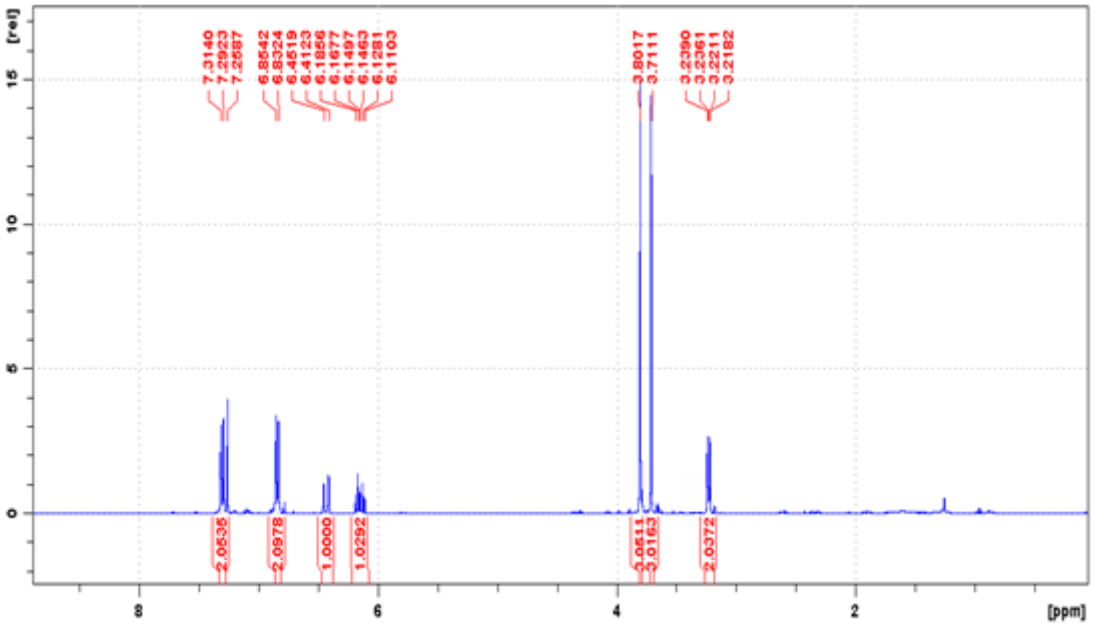
[ppm]


OMe
O
MeO

2c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3.7111
3.2390
3.2361
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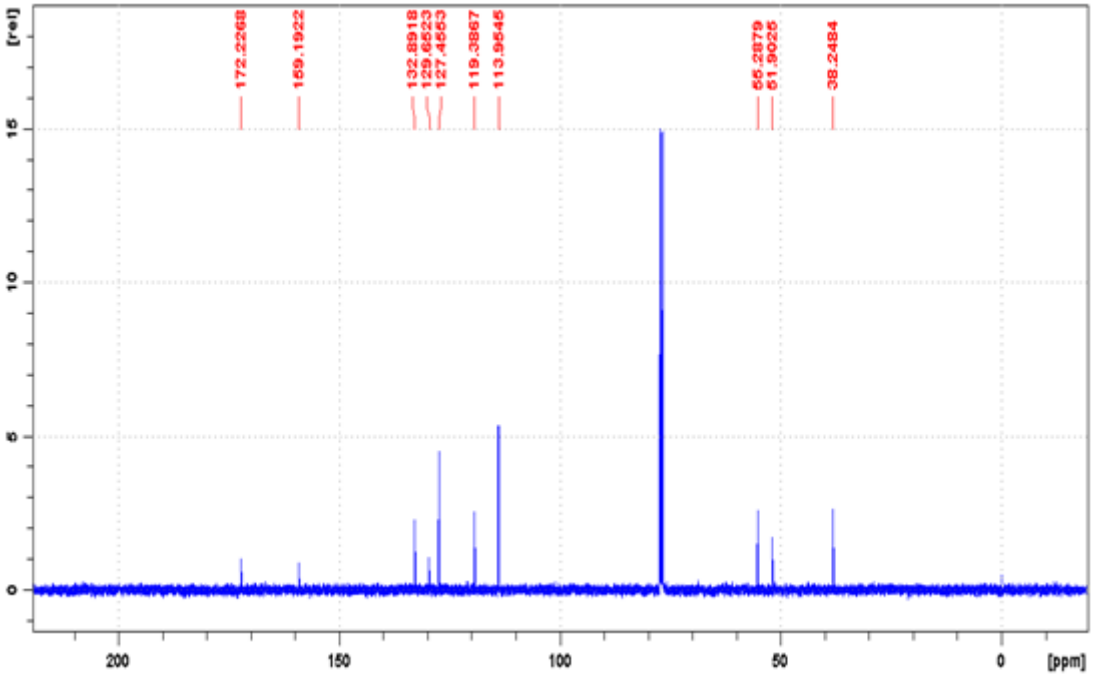
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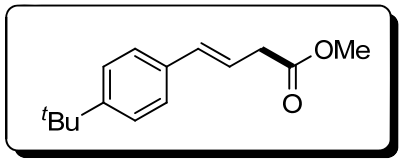
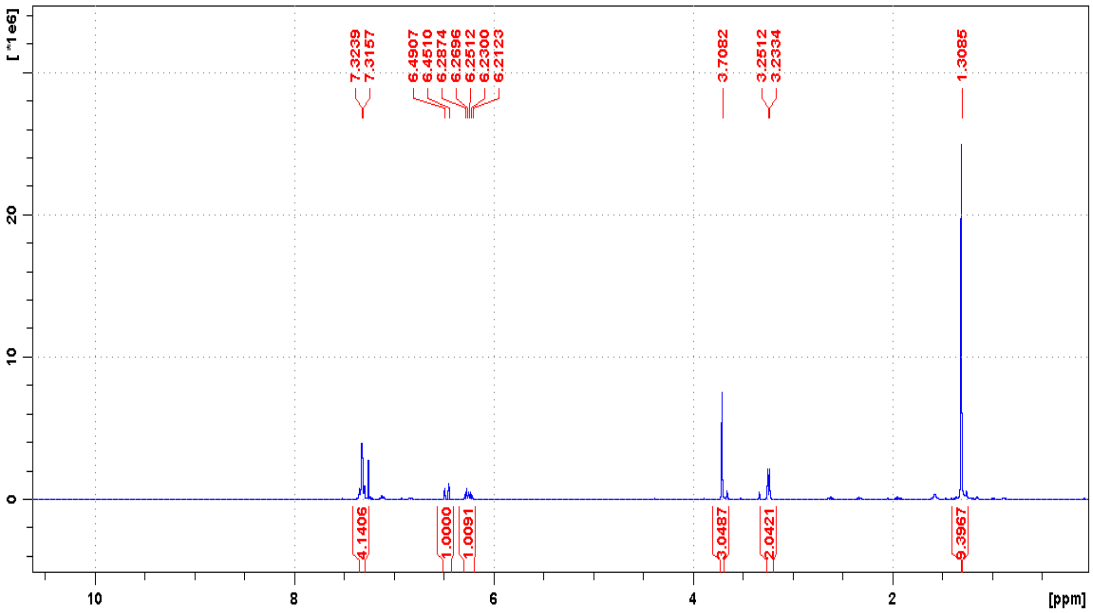

OMe
O
tBu

2d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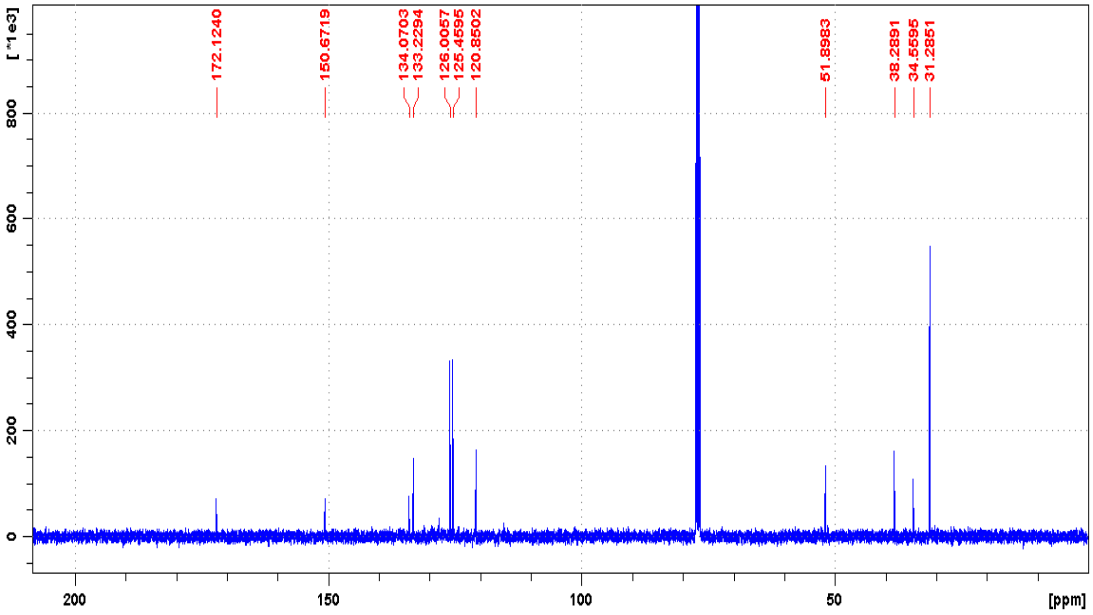
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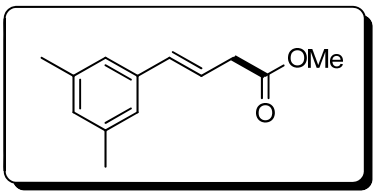
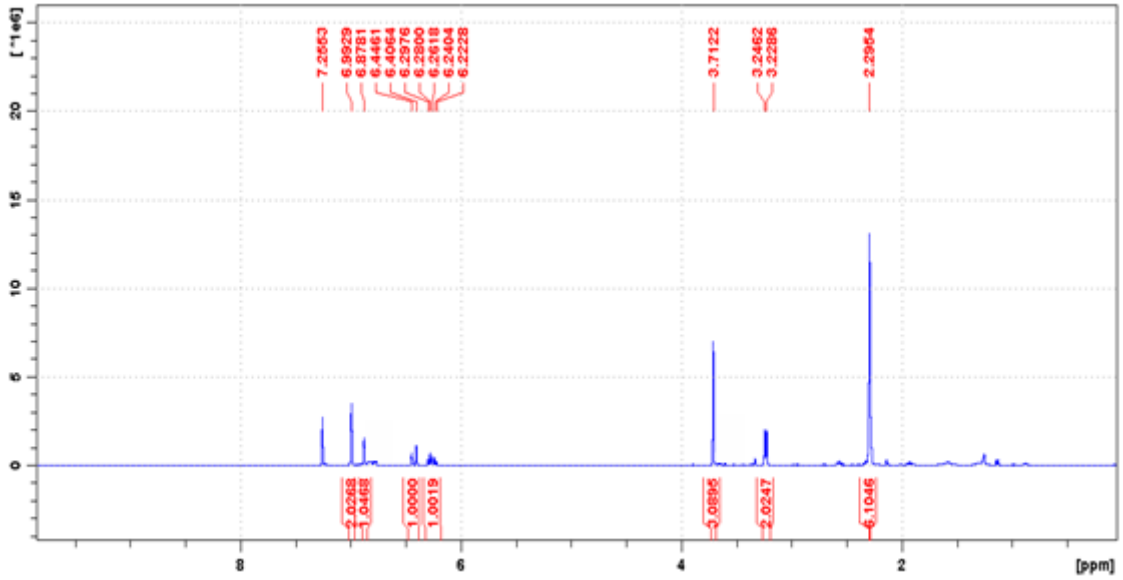

OMe
O

2e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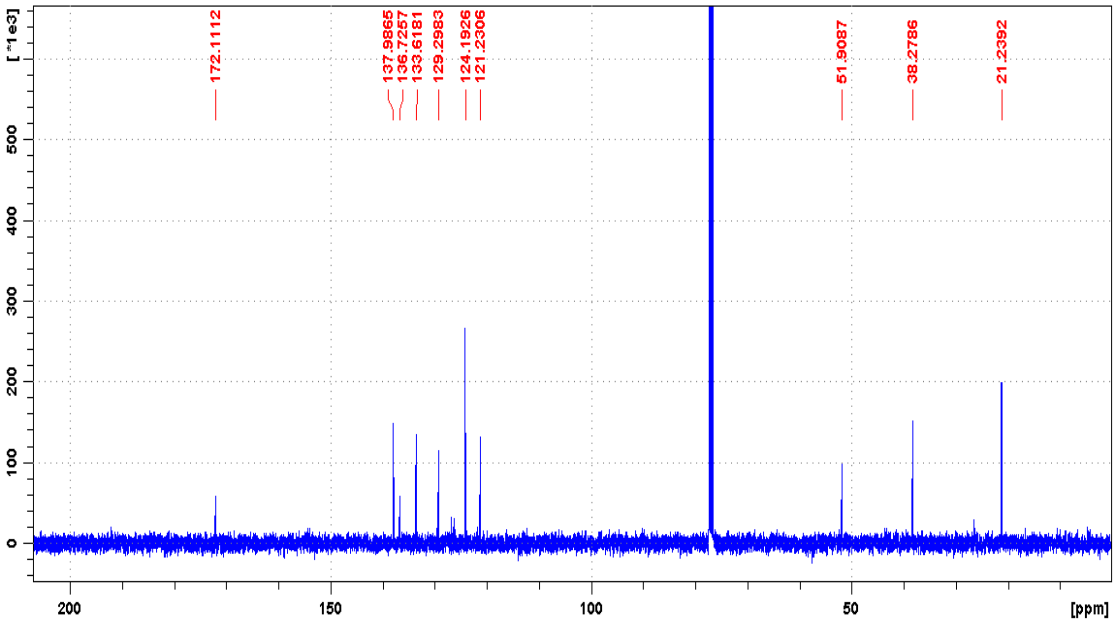
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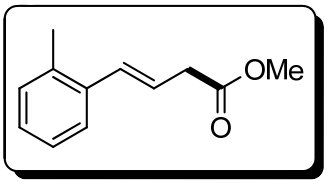
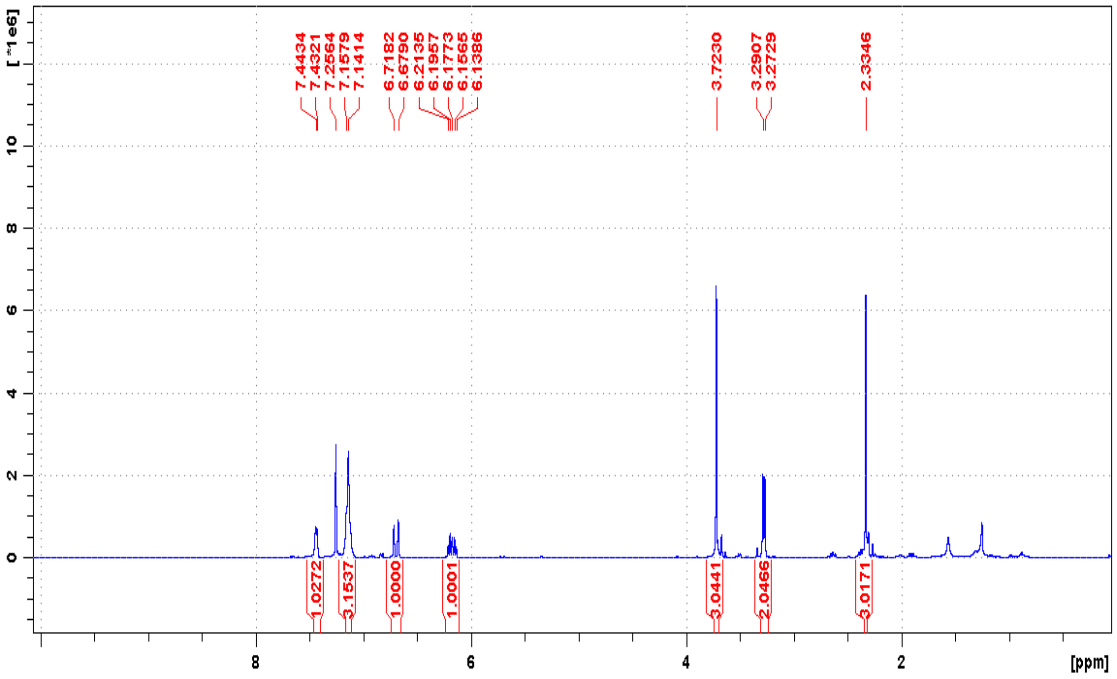

OMe
O

2f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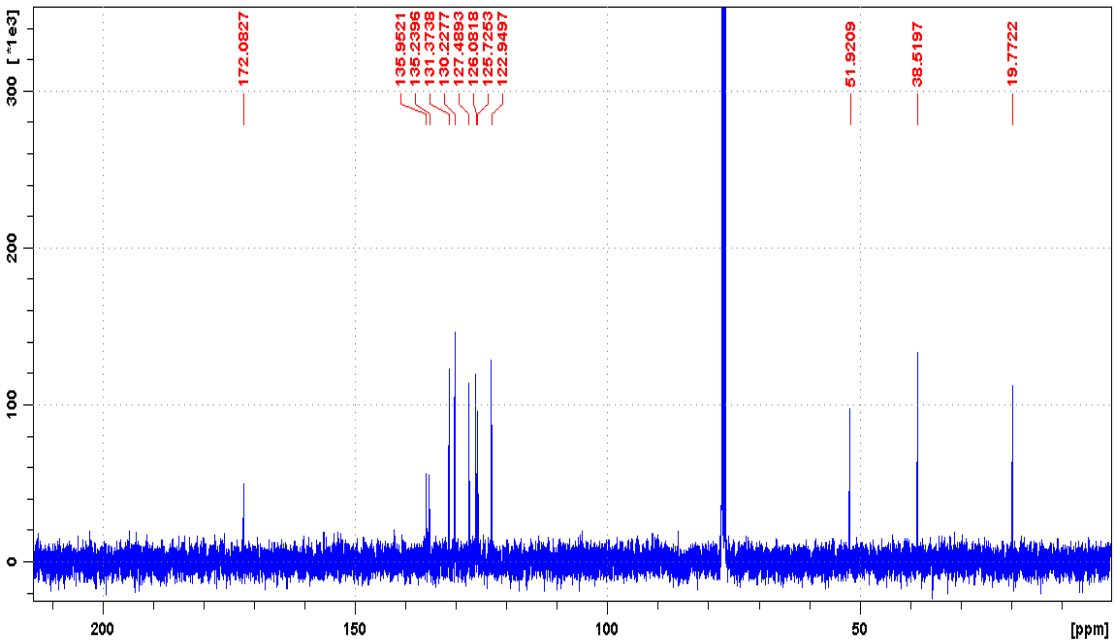
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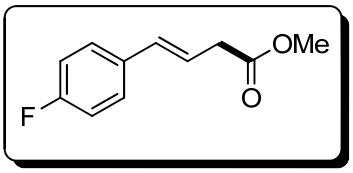
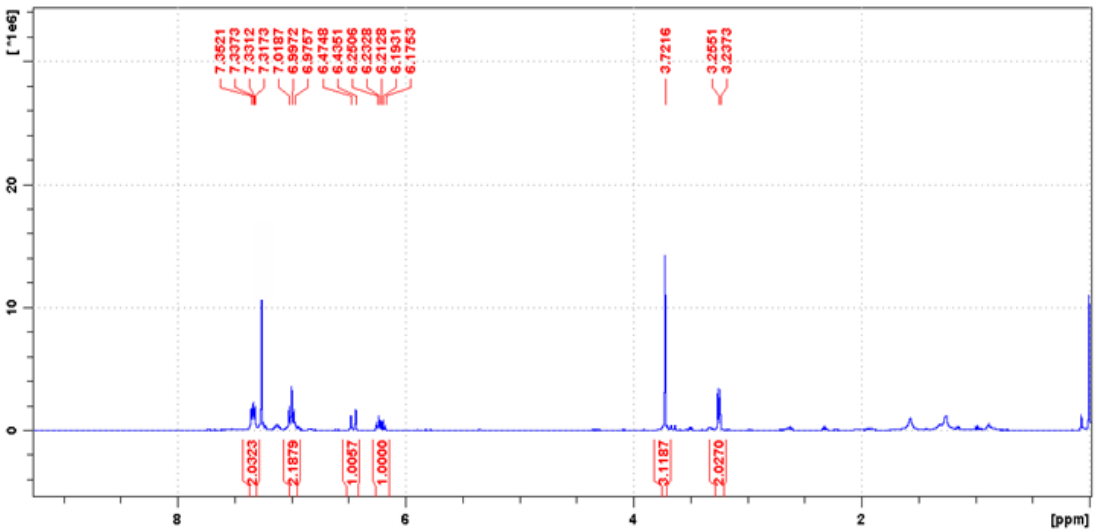

OMe
O
F

2g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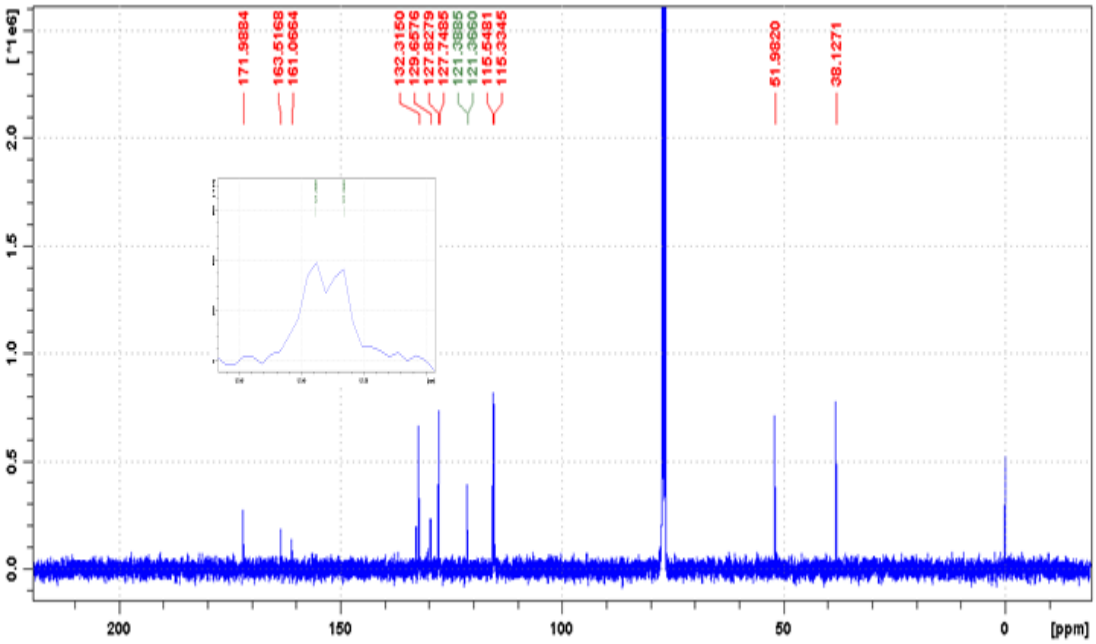
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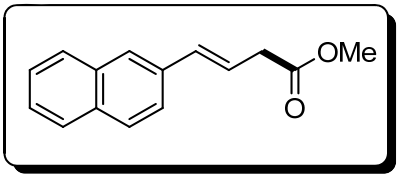
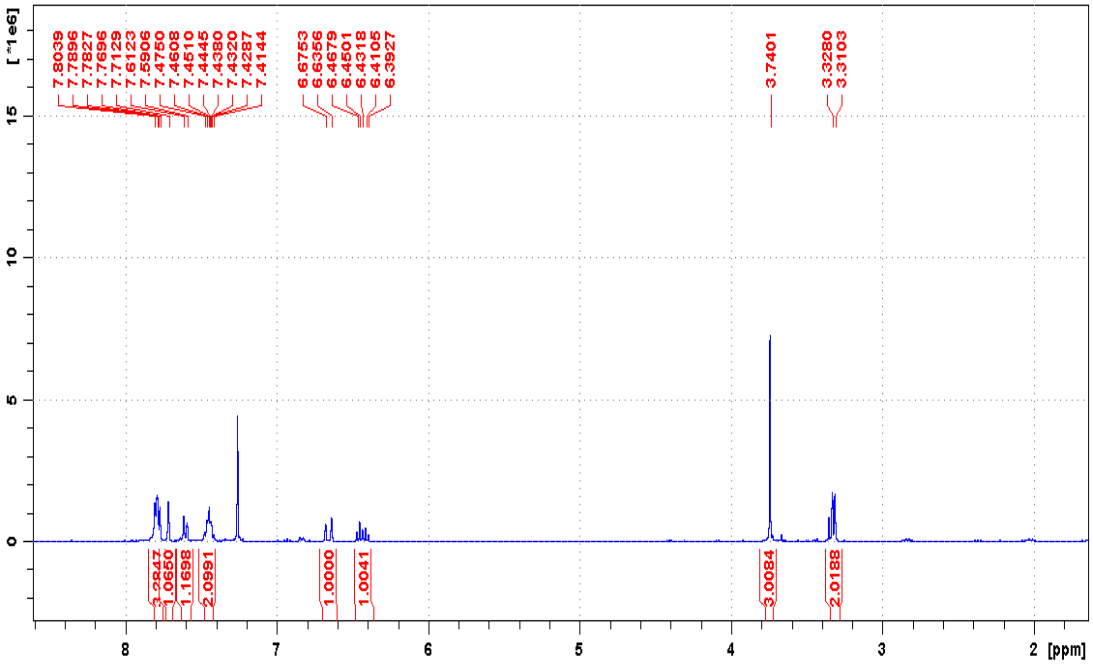

OMe
O

2h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7.4320
7.4287
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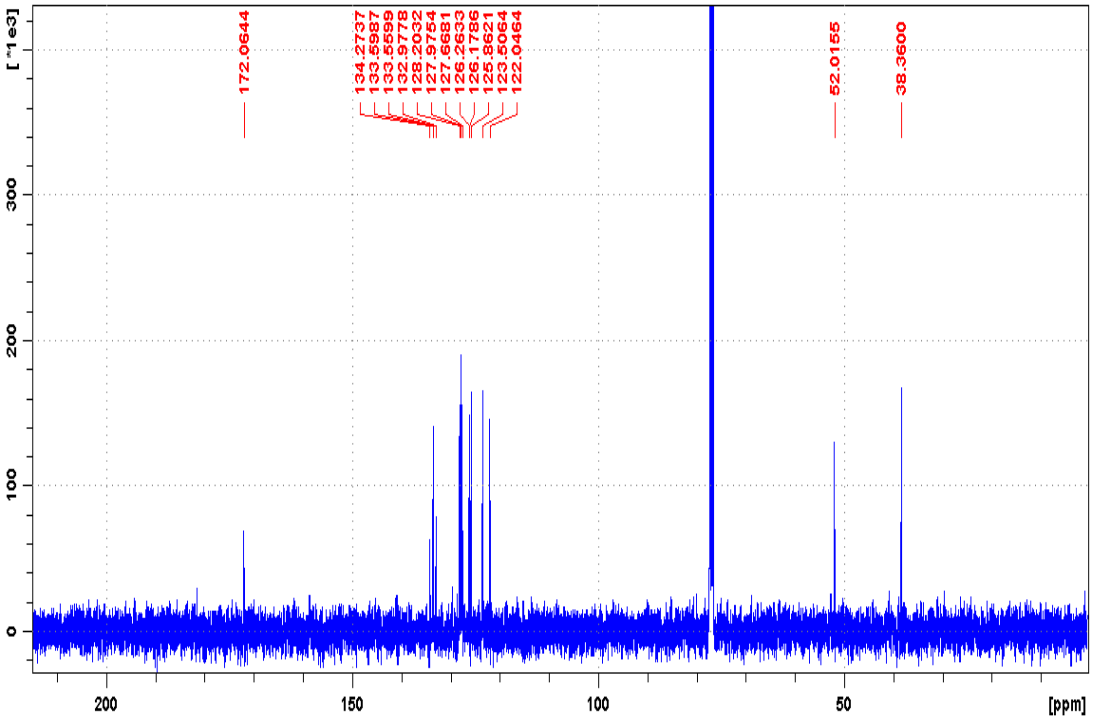
6.6356
6.4679
6.4501
6.4318
6.4105
6.3927
3.7401
3.3280
3.3103
3.0847
3.0650
1.1698
2.0991
1.0000
1.0041
3.0084
2.0188
8
7
6
5
4
3
2 [ppm]


[*1e3]
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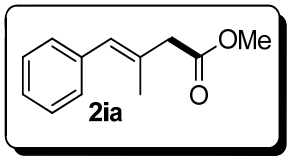
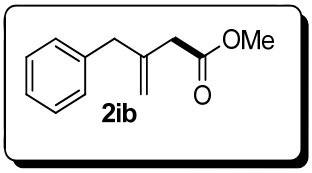
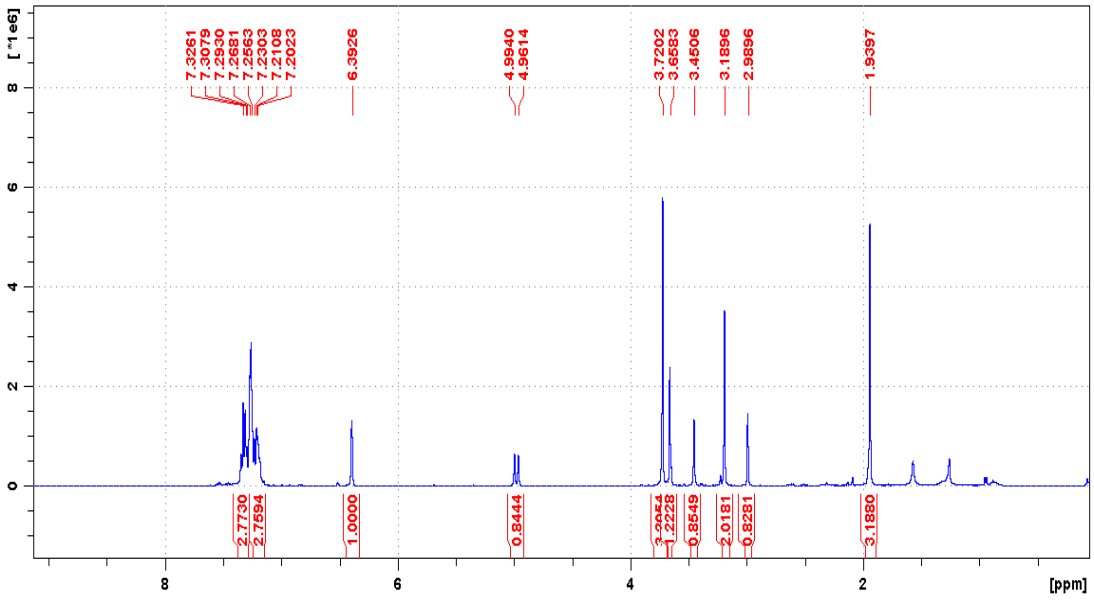
200
100
0
172.0644
134.2737
133.5987
133.5599
132.9778
128.2032
127.9764
127.6681
126.2633
126.1786
125.8621
123.5064
122.0464
52.0155
38.3600
200
150
100
50
[ppm]

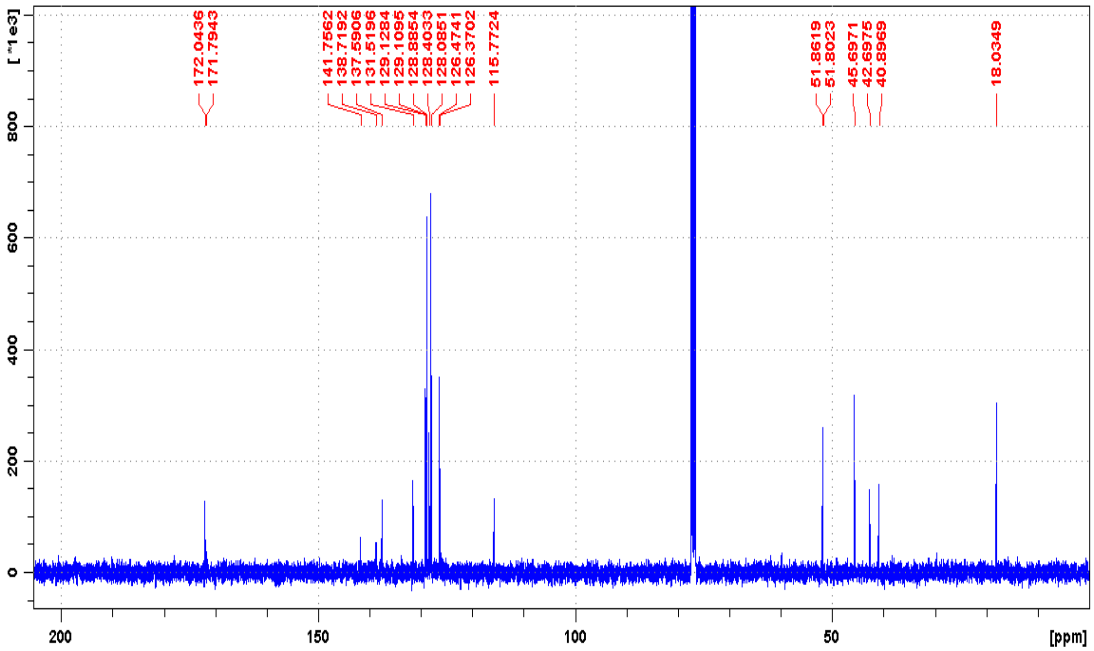


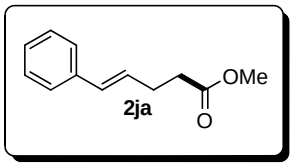
Mixed with



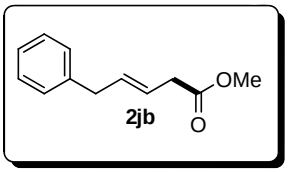
2ia : 2ib= 2.5:1



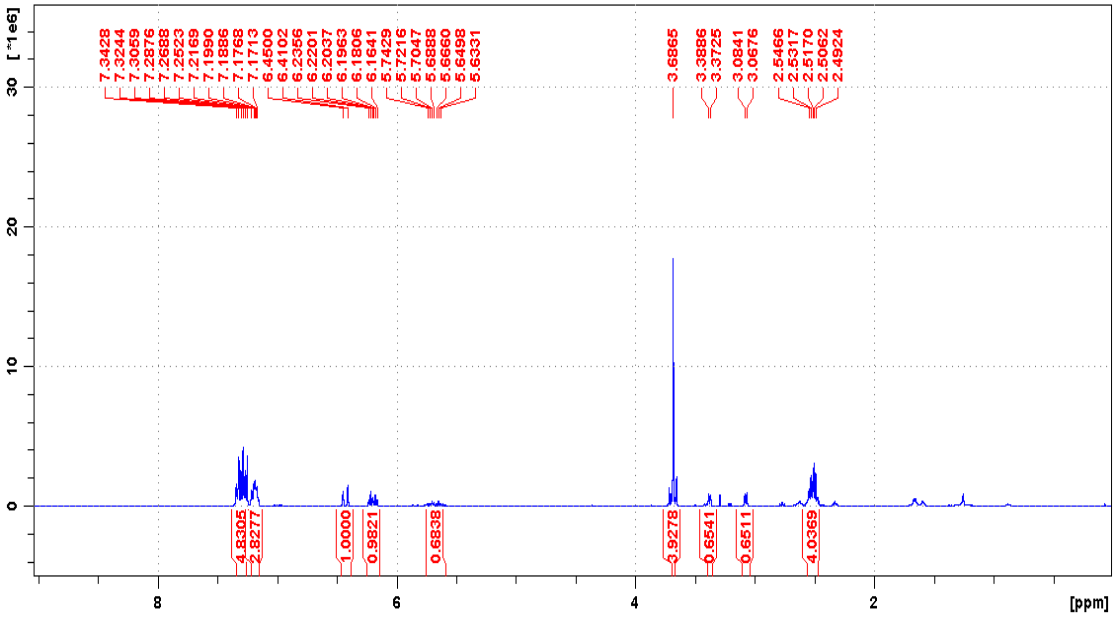


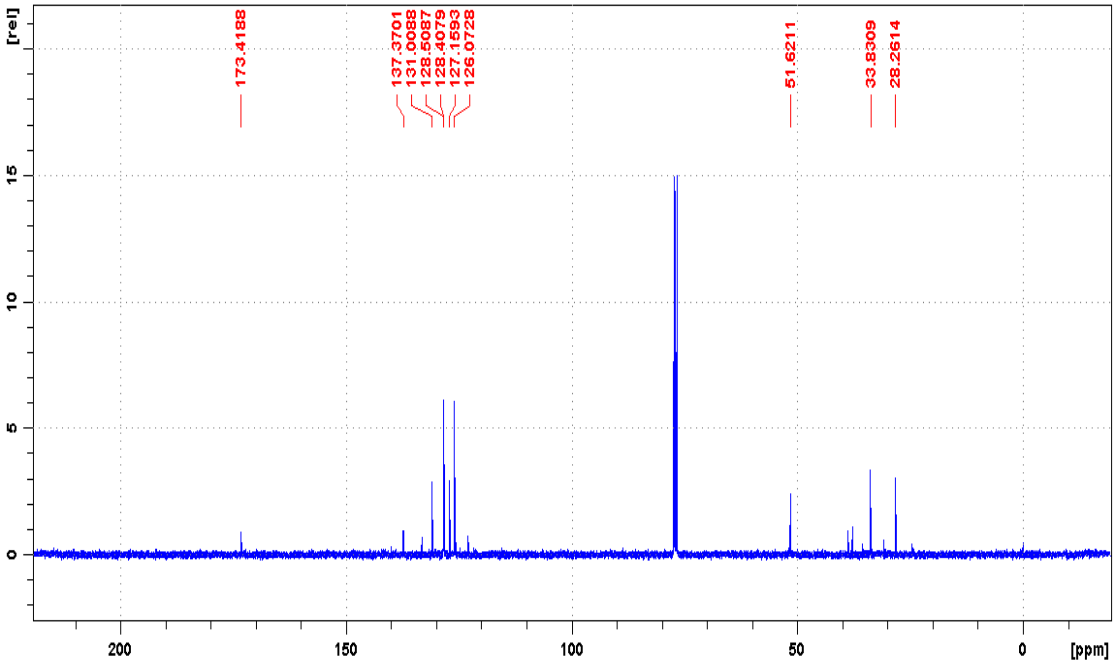


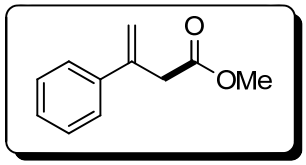
Mixed with



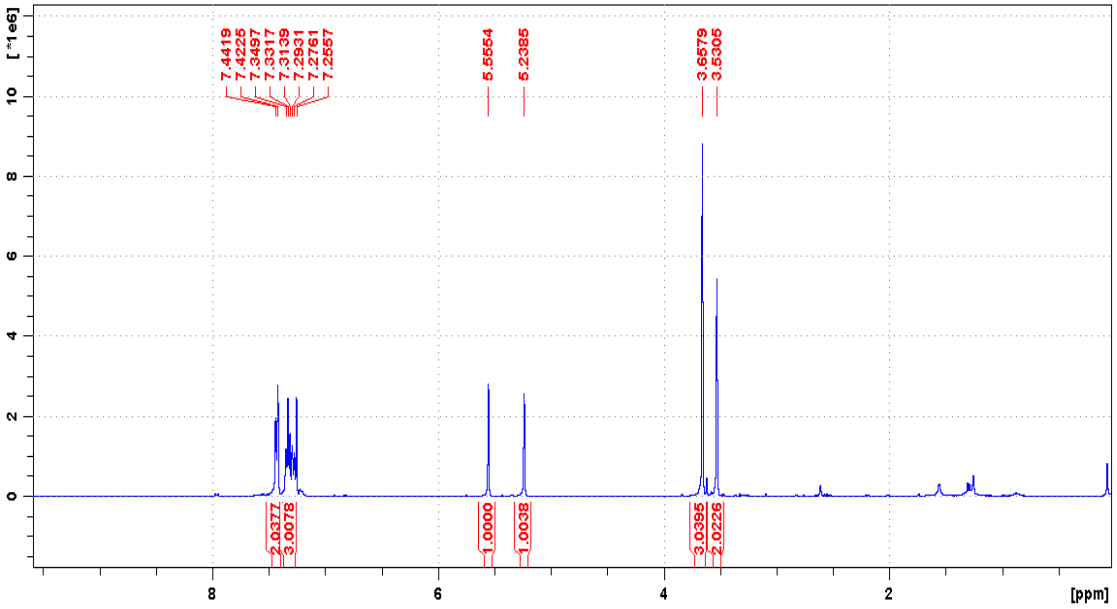
2ja : 2jb= 3:1






O
OMe

2k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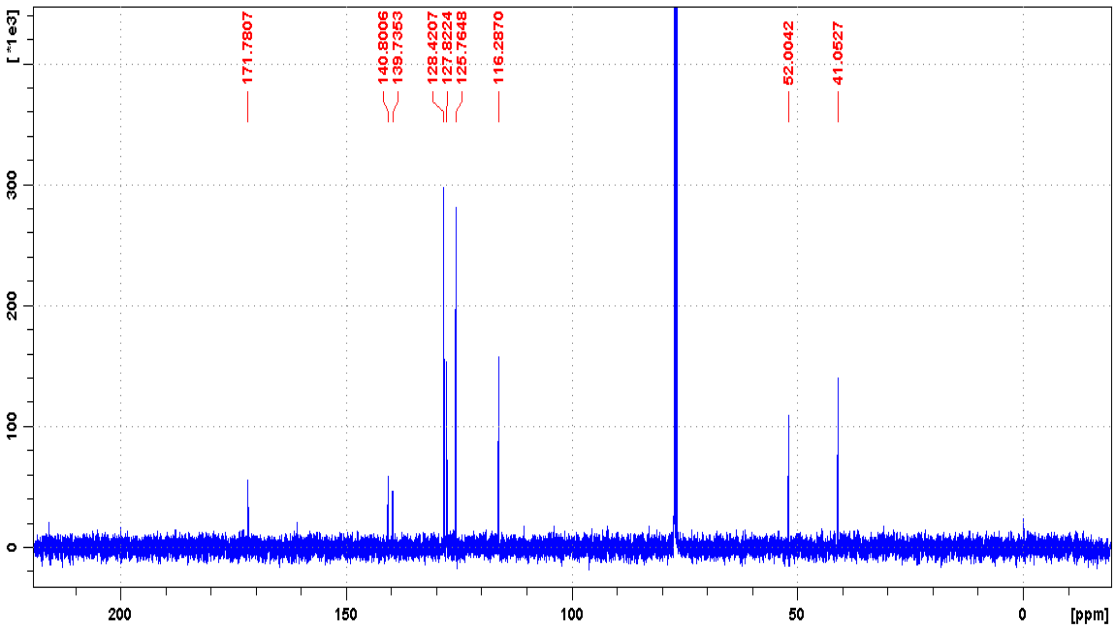
7.4225
7.3497
7.3317
7.3139
7.2931
7.2761
7.2557
5.5554
5.2385
3.6579
3.5305
2.0377
3.0078
1.0000
1.0038
3.0395
2.0226
[ppm]


[*1e3]
171.7807
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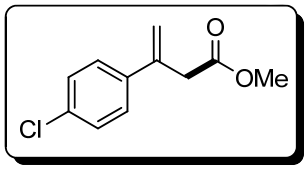
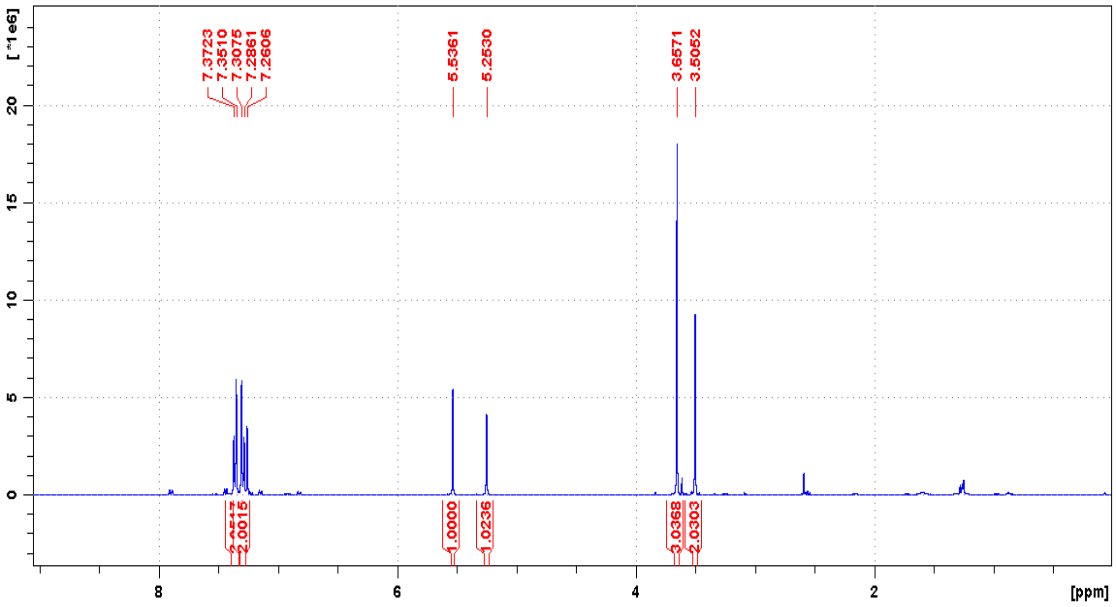
139.7353
128.4207
127.8224
125.7648
116.2870
52.0042
41.0527
[ppm]


O
OMe
Cl

2l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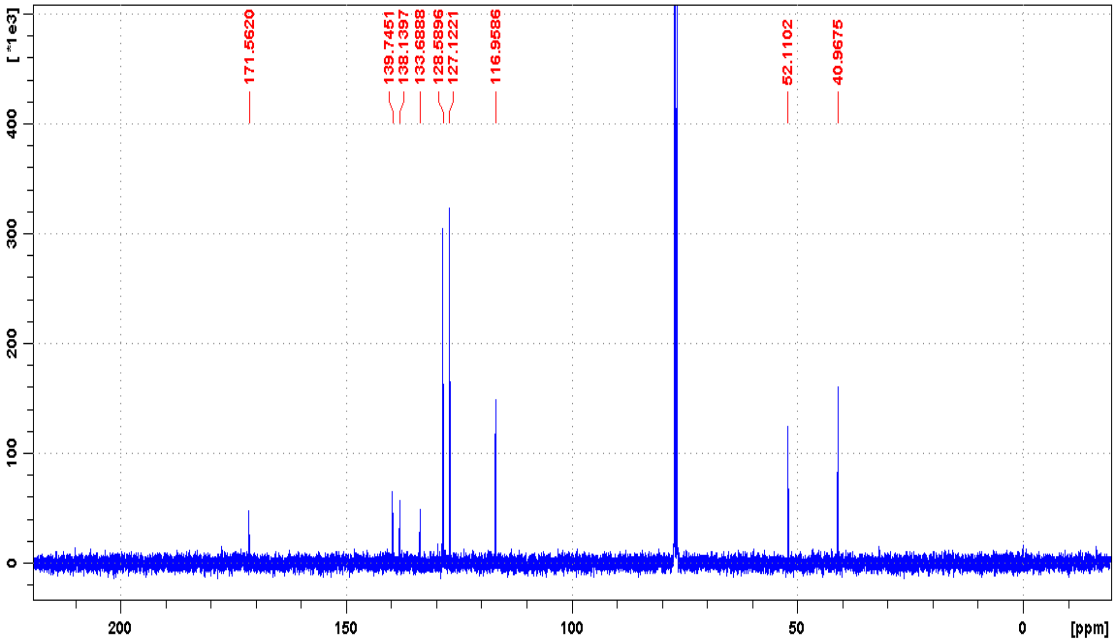
7.3510
7.3075
7.2861
7.2606
5.5361
5.2530
3.6571
3.5052
1.0000
1.0236
3.0368
2.0303
2.0015
[ppm]


[*1e3]
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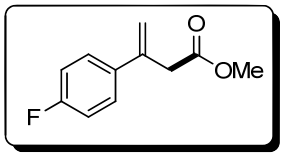
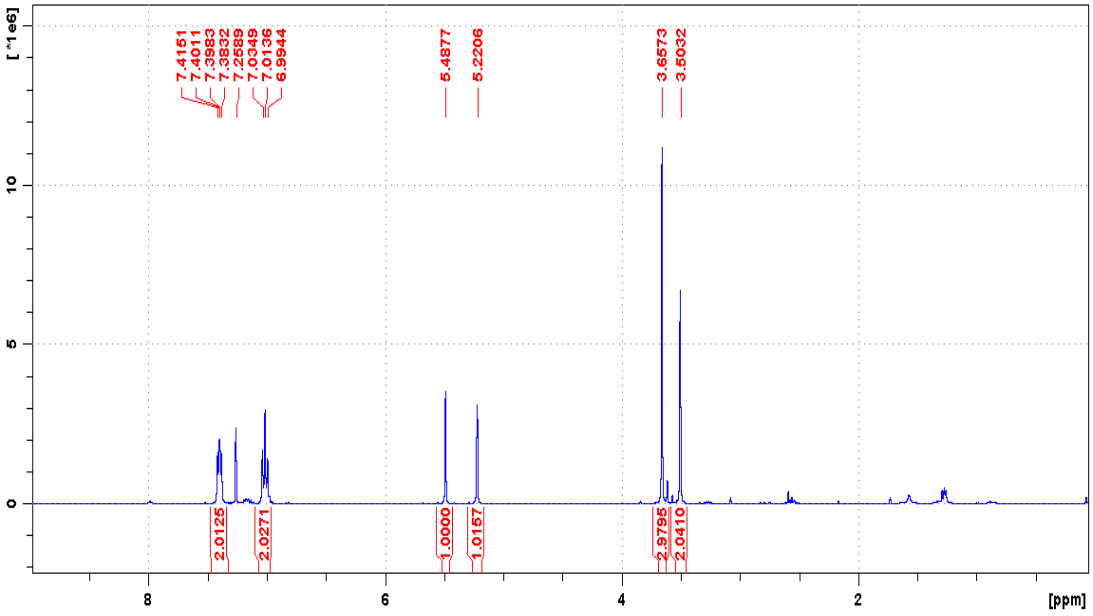
138.1397
133.6888
128.5896
127.1221
116.9586
52.1102
40.9675
[ppm]


O
OMe
F

2m


[*1e6]
10
5
0
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7.4011
7.3983
7.3832
7.2589
7.0349
7.0136
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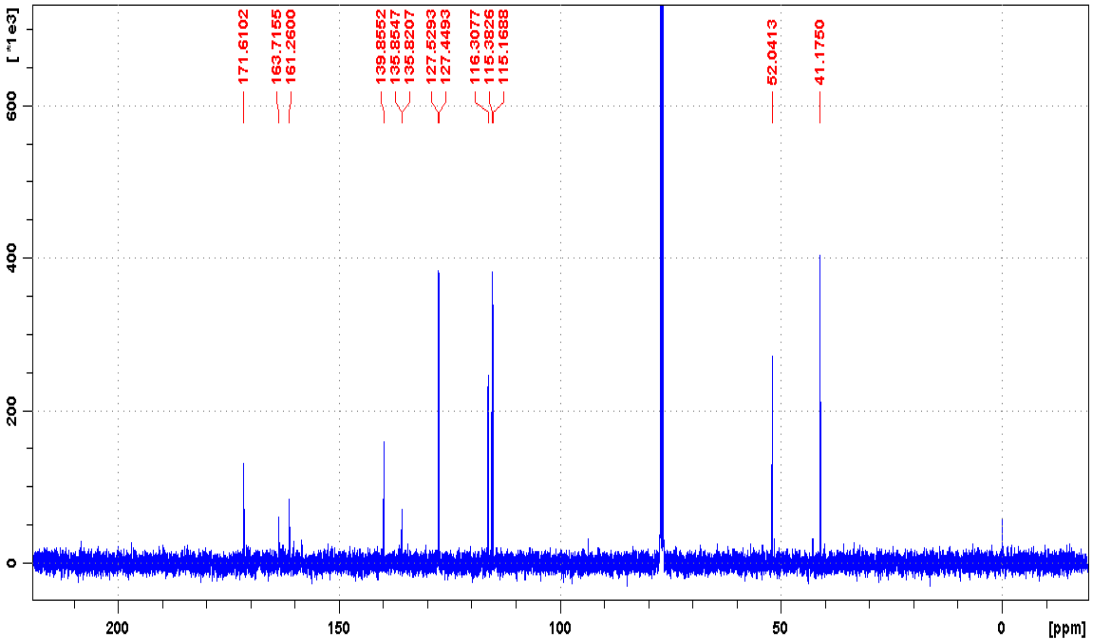
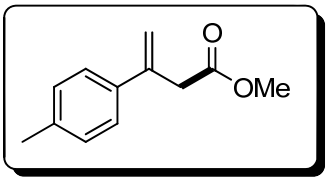
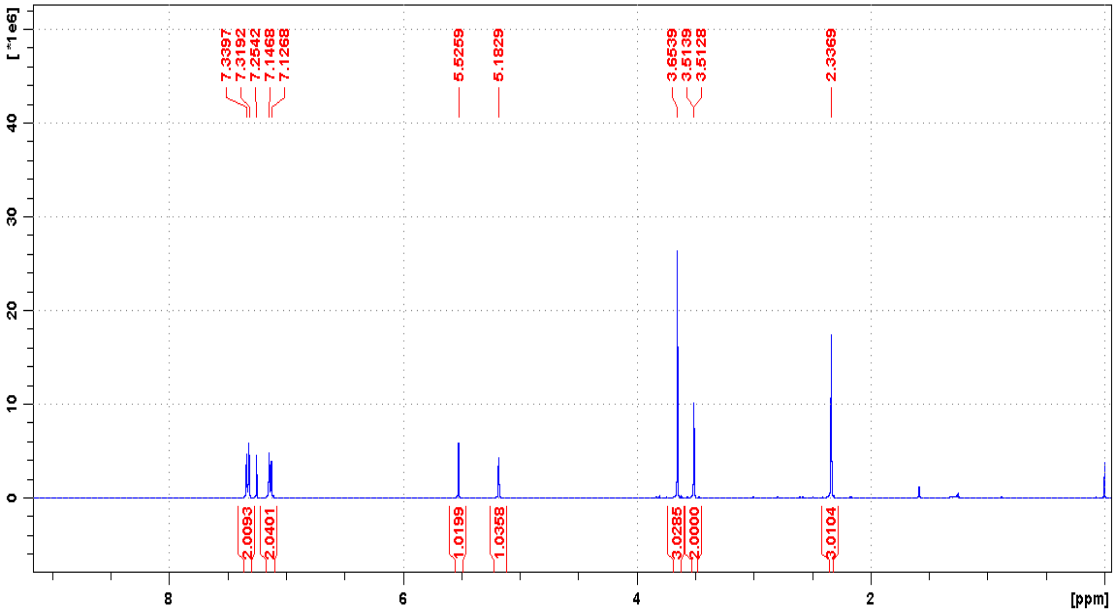
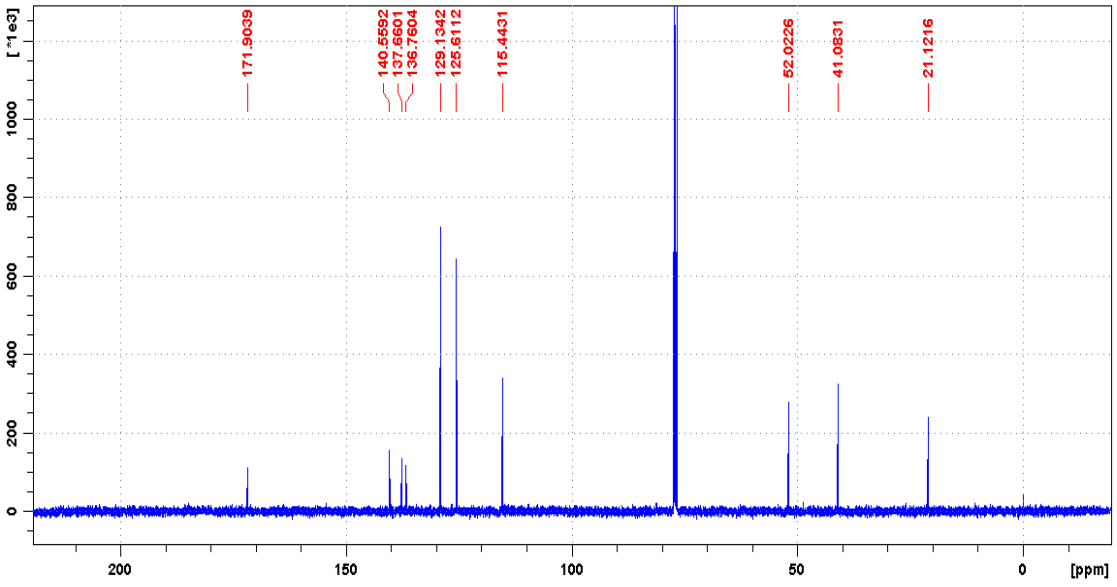
5.4877
5.2206
3.6573
3.5032
2.0125
2.0271
1.0000
1.0157
2.9795
2.0410
8
6
4
2
[ppm]


[*1e3]
600
400
200
0
171.6102
163.7155
161.2600
139.8562
135.8547
135.8207
127.5293
127.4493
116.3077
115.3226
115.1688
52.0413
41.1750
200
150
100
50
0
[ppm]


O
OMe

2n


[*1e6]
7.3397
7.3192
7.2542
7.1468
7.1268
5.5259
5.1829
3.6539
3.5139
3.5128
2.3369
2.0093
2.0401
1.0199
1.0358
3.0285
2.0000
3.0104
[ppm]


[*1e3]
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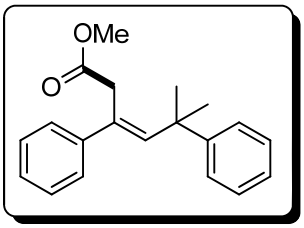
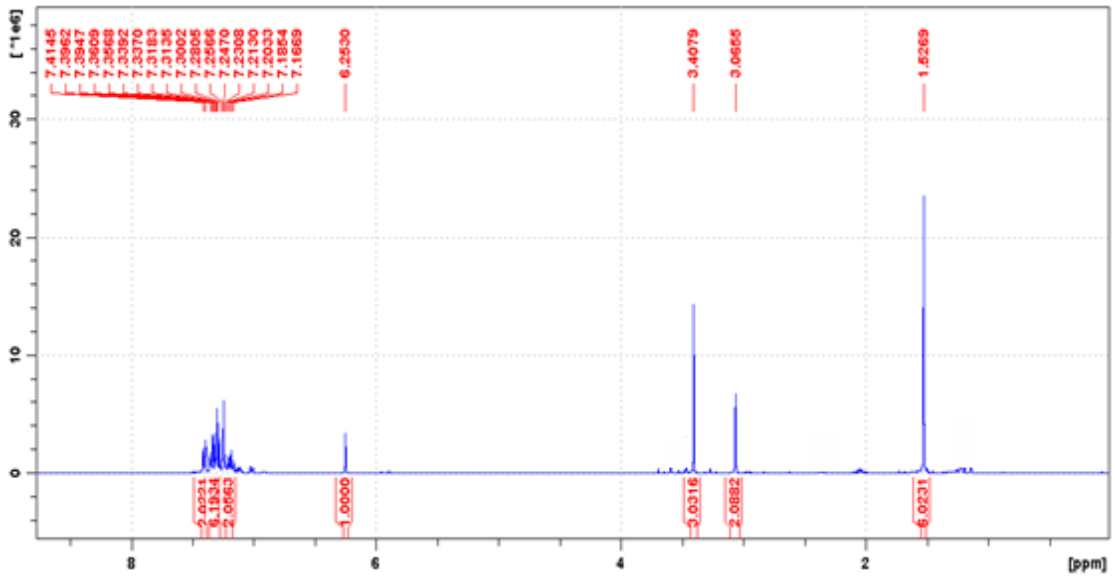
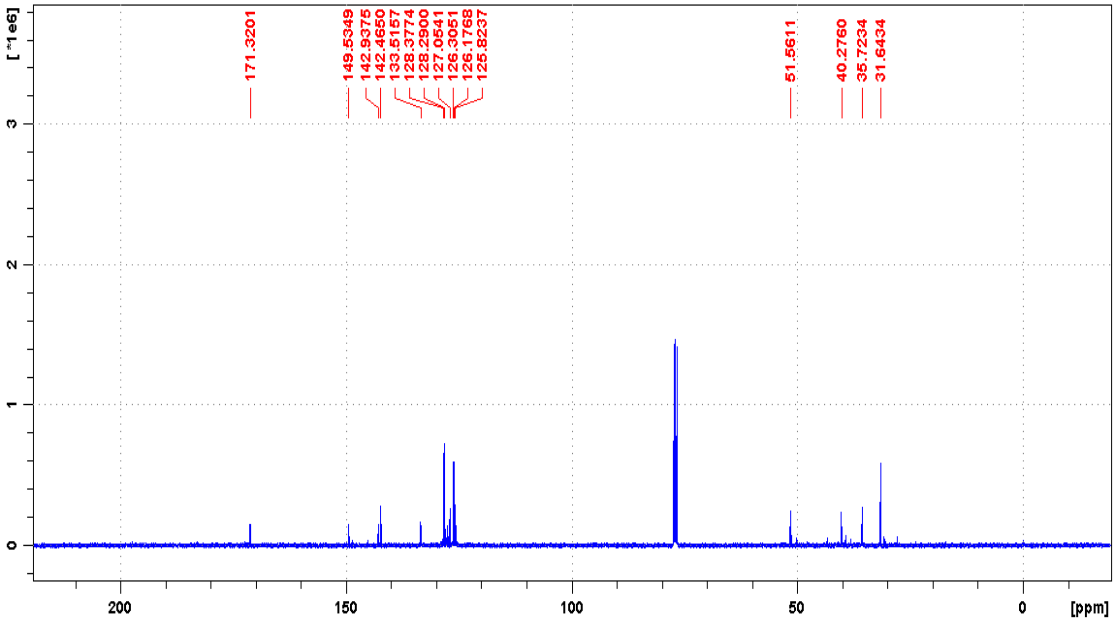
136.7604
129.1342
125.6112
115.4431
52.0226
41.0831
21.1216
[ppm]


OMe
O

2o


[*1e6]
7.4145
7.3962
7.3947
7.3609
7.3568
7.3392
7.3370
7.3183
7.3135
7.3002
7.2805
7.2566
7.2470
7.2308
7.2130
7.2033
7.1854
7.1669
6.2530
3.4079
3.0665
1.5269
2.0231
6.1934
2.0563
1.0000
3.0316
2.0882
6.0231
[ppm]


[*1e6]
171.3201
149.5349
142.9375
142.4650
133.5157
128.3774
128.2900
127.0541
126.3051
126.1768
125.8237
51.5611
40.2760
35.7234
31.6434
[ppm]