Supporting Information

Silver-catalyzed the decarboxylative C(sp^2)–C(sp^3) coupling reactions via radical mechanism

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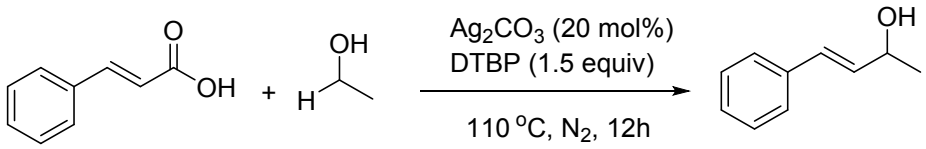
Contents

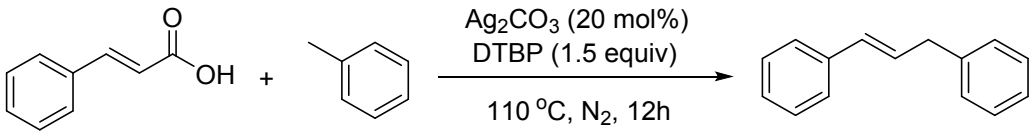
1. General information

Unless otherwise noted, all chemicals were purchased from commercial suppliers (Adamas, Aladdin, J&K etc) and used without further purification. **^{1}H NMR** and **^{13}C NMR** were recorded at ambient temperature on a 300, 400 or 500 MHz spectrometer (75, 100 or 125 MHz for ^{13}C NMR). NMR experiments are reported in δ units, parts per million (ppm), and were referenced to $CDCl_3$ (δ 7.26 or 77.0 ppm) as the internal standard. The coupling constants J are given in Hertz. Mass spectra were recorded on BRUKER AutoflexIII Smartbeam MS-spectrometer. High resolution mass spectra (HRMS) were recorded on Bruck microTof by using ESI method. Column chromatography was performed using EM silica gel 60 (300−400 mesh).

2. Synthesis and analytical data of 3 and 4



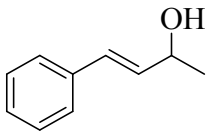
The typical experimental procedure for synthesis of *(E)*-4-phenylbut-3-en-2-ol **3a**: cinnamic acid **1a** (0.5 mmol, 74.0 mg), ethanol (1 ml), Ag_2CO_3 (0.05 mmol, 13.7 mg), DTBP (0.75 mmol, 138.0 μL) were placed into a sealed dry tube (10 mL) with a magnetic stirrer bar. The reaction was heated under a nitrogen atmosphere at 110 °C for 12 h. After the reaction finished, the catalyst was separated by centrifugation and filtration to obtain the liquid phase. The pure product was obtained by flash column chromatography on silica gel by using petroleum ether (30-60 °C) and ethyl acetate as eluents.



The typical experimental procedure for synthesis of *(E)*-prop-1-ene-1,3-diyldibenzene **4a**: cinnamic acid **1a** (0.5 mmol, 74.0 mg), Toluene (1 ml), Ag_2CO_3 (0.05 mmol, 13.7 mg), DTBP (0.75 mmol, 138.0 μL) were placed into a sealed dry tube (10 mL) with a magnetic stirrer bar. The reaction was heated under a nitrogen atmosphere at 110 °C for 12 h. After the reaction finished, the catalyst

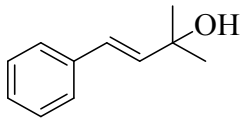
was separated by centrifugation and filtration to obtain the liquid phase. The pure product was obtained by flash column chromatography on silica gel by using petroleum ether (30-60 °C) and ethyl acetate as eluents.

Analytical data



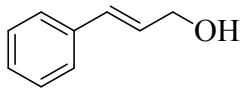
(*E*)-4-phenylbut-3-en-2-ol (**3a**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 8.0 Hz, 2H), 7.25-7.21 (m, 1H), 6.57 (d, *J* = 16.0 Hz, 1H), 6.26 (dd, *J* = 6.5, 16.0 Hz, 1H), 4.51-4.44 (m, 1H), 1.37 (d, *J* = 6.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.5, 133.5, 129.3, 128.5, 127.5, 126.4, 68.8, 23.3.



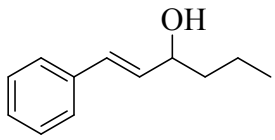
(*E*)-2-methyl-4-phenylbut-3-en-2-ol (**3b**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39-7.36 (m, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.24-7.20 (m, 1H), 6.60 (d, *J* = 16.0 Hz, 1H), 6.37 (d, *J* = 16.0 Hz, 1H), 1.42 (s, 6H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 137.4, 136.8, 128.5, 127.4, 126.38, 126.3, 71.0, 29.8.



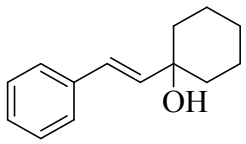
(*E*)-3-phenylprop-2-en-1-ol (**3c**)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.2 Hz, 2H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.23 (t, *J* = 7.2 Hz, 1H), 6.62 (d, *J* = 16.0 Hz, 1H), 6.40-6.31 (m, 1H), 4.31 (d, *J* = 5.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 136.6, 131.0, 128.5, 128.4, 127.6, 126.4, 63.6.



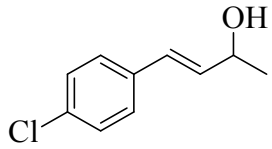
(*E*)-1-phenylhex-1-en-3-ol (**3d**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.5 Hz, 2H), 7.32 (t, *J* = 7.5 Hz, 2H), 7.24 (d, *J* = 6.0 Hz, 1H), 6.58 (d, *J* = 16.0 Hz, 1H), 6.23 (dd, *J* = 6.5, 16.0 Hz, 1H), 4.30 (q, *J* = 13.0 Hz, 1H), 1.70-1.52 (m, 2H), 1.51-1.33 (m, 2H), 0.95 (t, *J* = 6.0 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.7, 132.5, 130.1, 128.5, 127.5, 126.4, 72.8, 39.4, 18.6, 13.9.



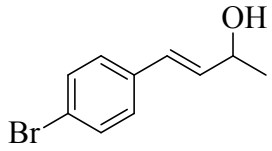
(*E*)-1-styrylcyclohexanol (**3e**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 2H), 7.23 (t, *J* = 7.0 Hz, 1H), 6.64 (d, *J* = 16.5 Hz, 1H), 6.35 (d, *J* = 16.5 Hz, 1H), 1.73-1.62 (m, 5H), 1.59-1.50 (m, 4H), 1.34-1.27 (m, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 137.4, 137.0, 128.5, 127.3, 126.9, 126.3, 71.7, 37.9, 25.4, 22.0.



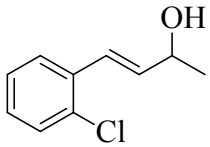
(*E*)-4-(4-chlorophenyl)-2-methylbut-3-en-2-ol (**3f**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35-7.27 (m, 4H), 6.54 (d, *J* = 16.0 Hz, 1H), 6.24 (dd, *J* = 6.5, 16.0 Hz, 1H), 4.53-4.44 (m, 1H), 1.37 (d, *J* = 6.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 135.1, 134.1, 133.2, 129.0, 128.7, 128.0, 127.6, 68.7, 23.3.



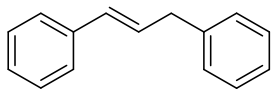
(*E*)-4-(4-bromophenyl)but-3-en-2-ol (**3g**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.5 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 6.52 (d, *J* = 16.0 Hz, 1H), 6.26 (dd, *J* = 6.5, 16.0 Hz, 1H), 4.52-4.44 (m, 1H), 1.37 (d, *J* = 6.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 135.6, 134.3, 131.6, 128.1, 127.9, 121.3, 68.7, 23.3.



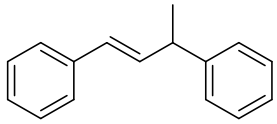
(*E*)-4-(2-chlorophenyl)but-3-en-2-ol (**3h**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52 (dd, *J* = 1.0, 7.5 Hz, 1H), 7.34 (dd, *J* = 1.0, 7.5 Hz, 1H), 7.23-7.16 (m, 2H), 6.96 (d, *J* = 16.5 Hz, 1H), 6.25 (dd, *J* = 6.5, 16.5 Hz, 1H), 4.59-4.47 (m, 1H), 1.39 (d, *J* = 6.5 Hz, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.3, 134.8, 133.1, 129.6, 128.5, 126.8, 126.7, 125.5, 68.8, 23.2.



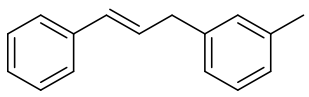
(*E*)-prop-1-ene-1,3-diyldibenzene (**4a**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42-7.38 (m, 2H), 7.38-7.32 (m, 3H), 7.31-7.27 (m, 3H), 7.27-7.21 (m, 2H), 6.52 (d, *J* = 16.0 Hz, 1H), 6.44-6.37 (m, 1H), 3.60 (d, *J* = 7.0 Hz, 2H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 140.1, 137.4, 131.0, 129.2, 128.6, 128.4, 127.0, 126.16, 126.1, 39.3.



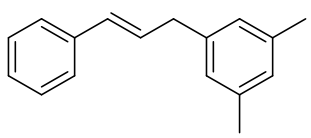
(*E*)-*but*-1-ene-1,3-diyldibenzene (4b)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.15 (m, 10H), 6.45-6.32 (m, 2H), 3.69-3.57 (m, 1H), 7.14 (d, J = 7.2 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 145.6, 137.5, 135.2, 128.4, 127.2, 127.0, 126.19, 126.1, 42.5, 21.1.



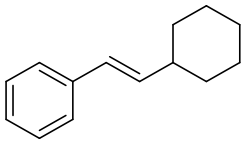
(*E*)-1-cinnamyl-3-methylbenzene (**4c**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 (d, *J* = 9.0 Hz, 2H), 7.24 (t, *J* = 9.5 Hz, 2H), 7.17 (d, *J* = 9.0 Hz, 2H), 7.01 (t, *J* = 8.5 Hz, 3H), 6.45 (d, *J* = 16.0 Hz, 1H), 6.38-6.27 (m, 1H), 3.50 (d, *J* = 8.0 Hz, 2H), 2.31 (s, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 140.1, 138.1, 137.5, 130.9, 129.47, 129.4, 128.5, 128.4, 127.1, 126.9, 126.1, 125.7, 39.3, 21.4.



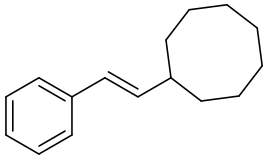
(*E*)-1-cinnamyl-3,5-dimethylbenzene (**4d**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.0 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.20 (t, *J* = 7.5 Hz, 1H), 6.86 (s, 3H), 6.46 (d, *J* = 16.0 Hz, 1H), 6.38-6.30 (m, 1H), 3.48 (d, *J* = 7.0 Hz, 2H), 2.29 (s, 6H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 140.1, 138.1, 137.6, 130.9, 129.5, 128.5, 127.9, 127.1, 126.5, 126.2, 39.3, 21.3.



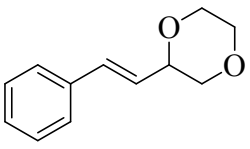
(*E*)-(2-cyclohexylvinyl)benzene (**4e**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (d, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 8.0 Hz, 2H), 7.20-7.14 (m, 1H), 6.35 (d, *J* = 16.0 Hz, 1H), 6.19 (dd, *J* = 7.0, 16.0 Hz, 1H), 2.17-2.07 (m, 1H), 1.84-1.72 (m, 4H), 1.71-1.63 (m, 1H), 1.35-1.26 (m, 2H), 1.22-1.12 (m, 3H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 138.0, 136.8, 128.4, 127.2, 126.7, 125.9, 41.1, 32.9, 26.1, 26.0.



(*E*)-styrylcyclooctane (4f)

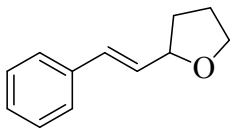
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 7.6 Hz, 2H), 7.25 (t, *J* = 8.0 Hz, 2H), 7.17 (t, *J* = 7.2 Hz, 1H), 6.33 (d, *J* = 16.0 Hz, 1H), 6.22 (dd, *J* = 7.2, 15.6 Hz, 1H), 2.38 (s, 1H), 1.80-1.65 (m, 4H), 1.61-1.50 (m, 10H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 138.1, 137.8, 128.4, 126.7, 126.6, 125.9, 41.3, 31.8, 27.4, 25.9, 25.0.



(*E*)-2-styryl-1,4-dioxane (**4g**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J* = 7.5 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.24 (d, *J* = 6.5 Hz, 1H), 6.70 (d, *J* = 16.0 Hz, 1H), 6.10 (dd, *J* = 6.5, 16.0 Hz, 1H), 4.25 (t, *J* = 7.5 Hz, 1H), 3.91-3.78 (m, 3H), 3.73 (d, *J* = 11.0 Hz,

1H), 3.70-3.62 (m, 1H), 3.42 (t, J = 5.5 Hz, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.3, 132.6, 128.5, 127.8, 126.4, 125.0, 76.0, 70.9, 66.5, 66.2.



(*E*)-3-styryltetrahydrofuran (**4h**)

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42-7.38 (m, 2H), 7.35-7.30 (m, 2H), 7.27-7.22 (m, 1H), 6.63 (d, J = 16.5 Hz, 1H), 6.26 (dd, J = 6.5, 16.5 Hz, 1H), 4.54-4.47 (m, 1H), 4.03-3.96 (m, 1H), 3.90-3.83 (m, 1H), 2.20-2.11 (m, 1H), 2.07-1.93 (m, 2H), 1.79-1.69 (m, 1H); **^{13}C NMR** (125 MHz, $CDCl_3$) δ 136.8, 130.5, 130.4, 128.4, 127.4, 126.4, 79.6, 68.1, 32.3, 25.8.

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3 References

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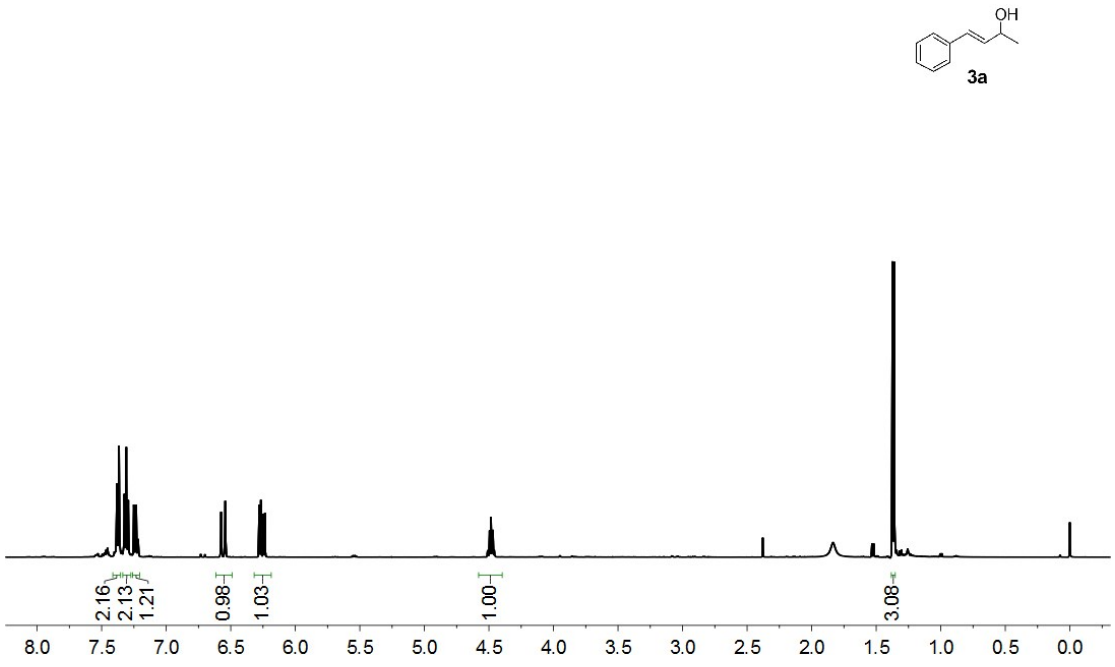
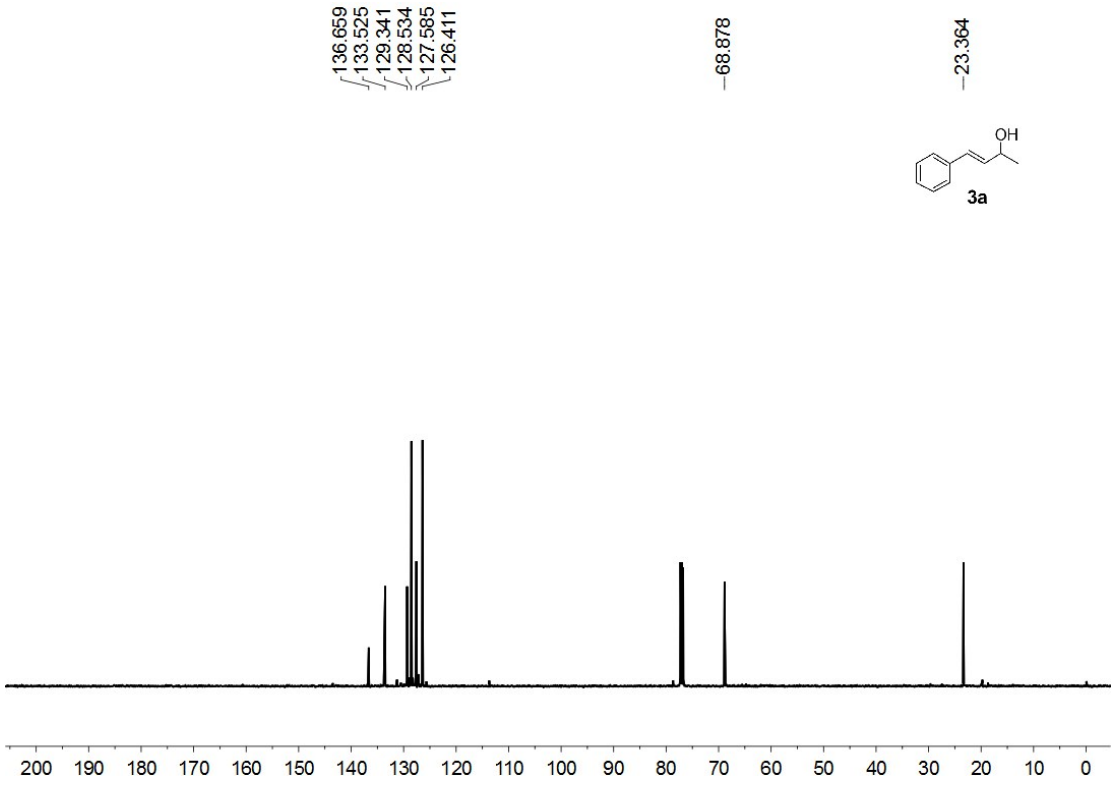
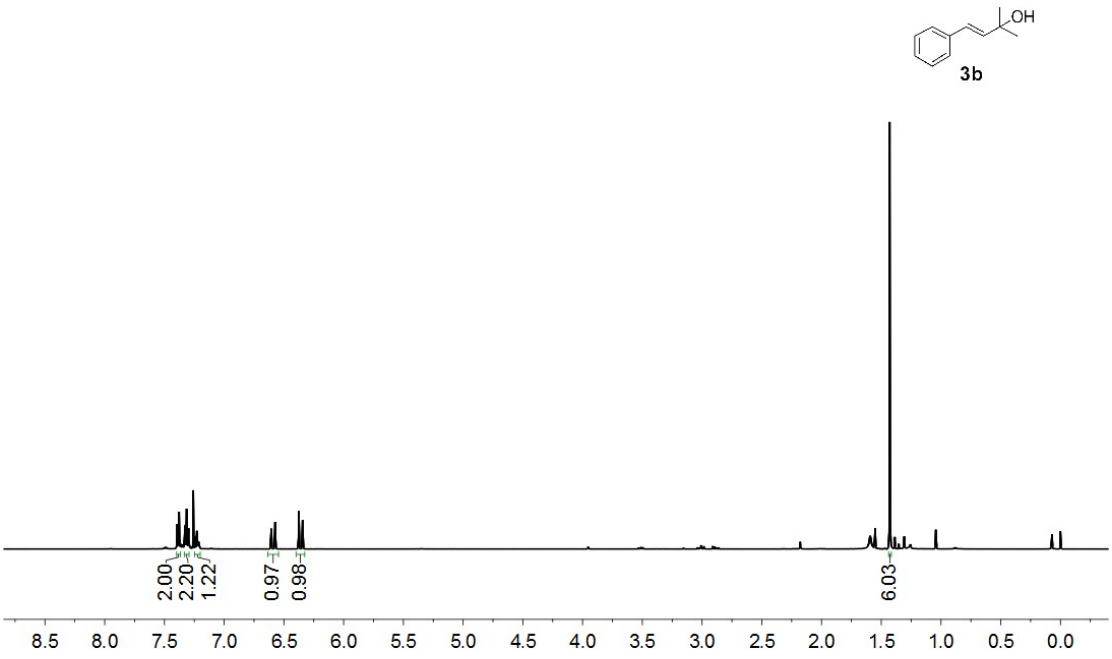
(6) J. Zhao, H. Fang, J. Han and Y. Pan, *Beilstein J. Org. Chem.*, 2013, **9**, 1718.

4 NMR spectra copies



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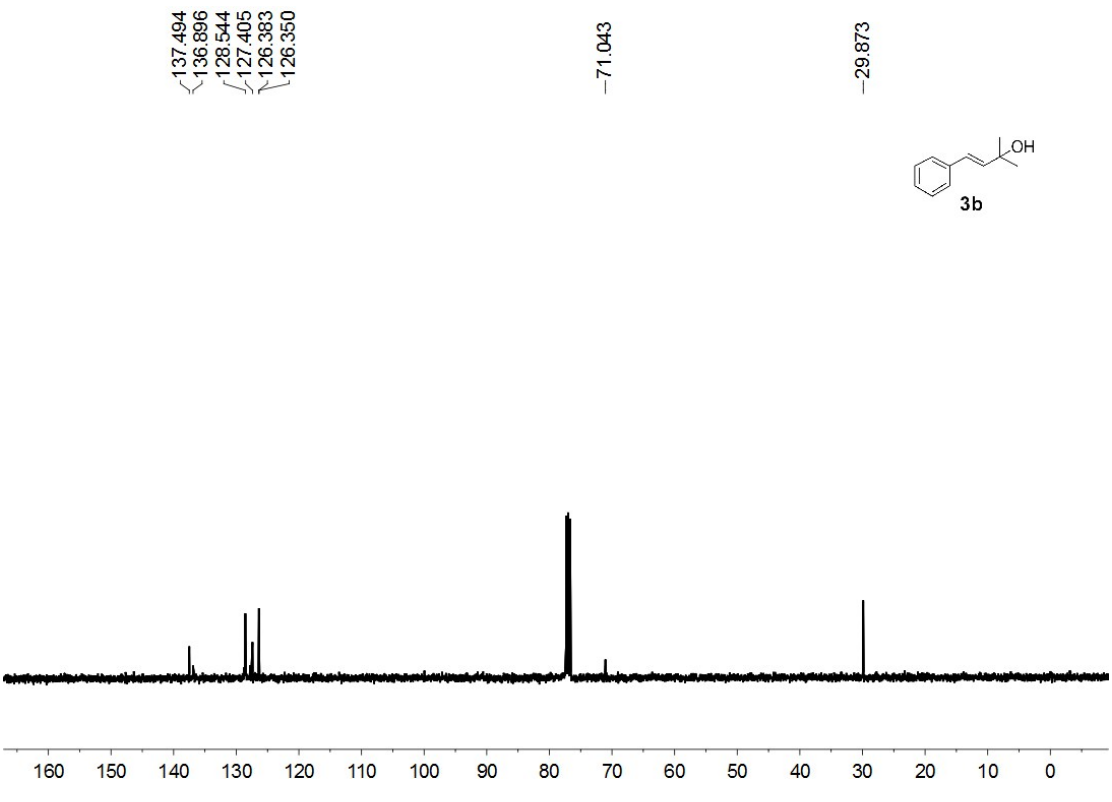
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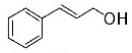
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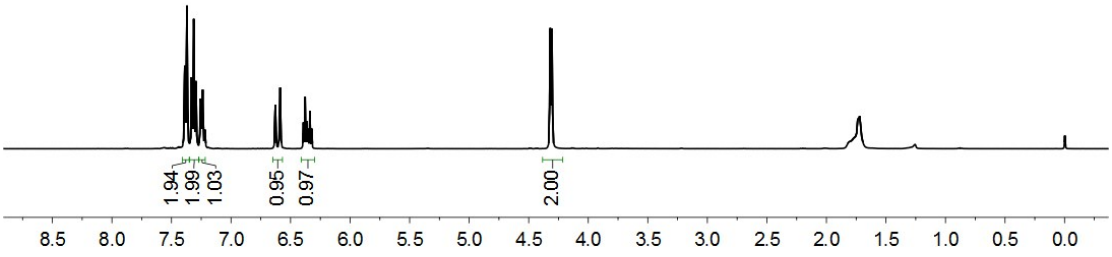
3c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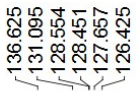
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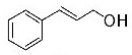
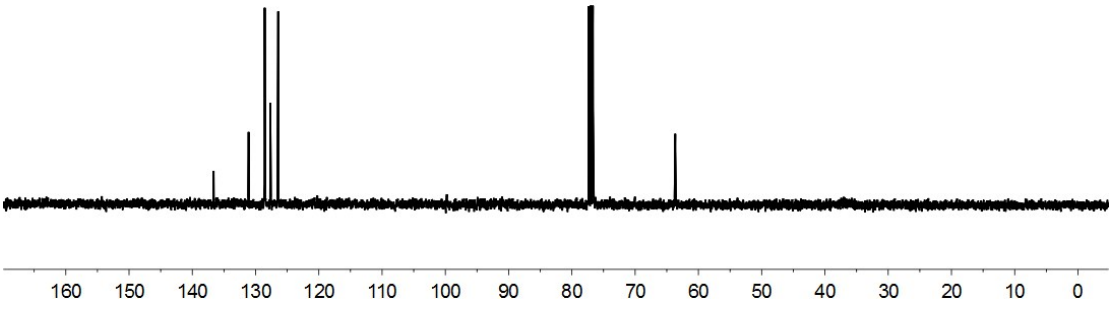
63.661


OH

3c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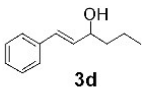
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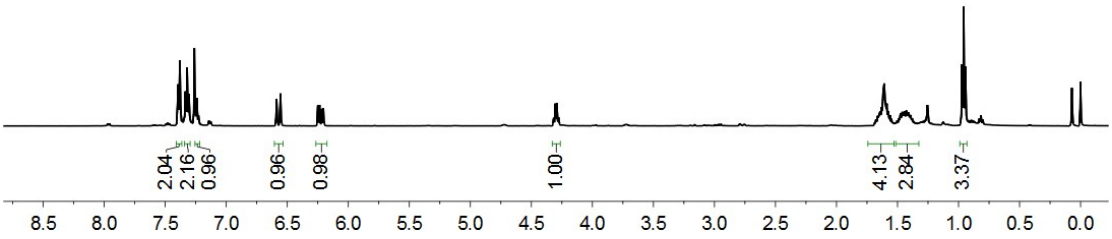
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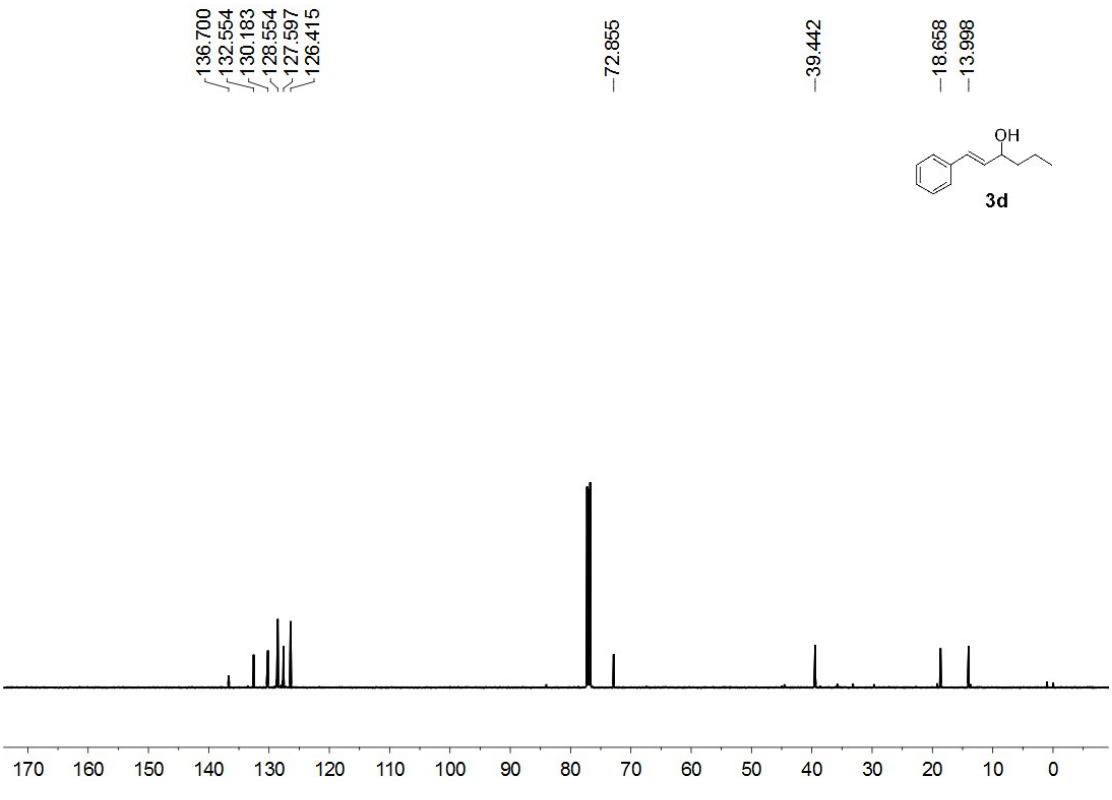
2.16
0.96
0.96
0.98
1.00
4.13
2.84
3.37
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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128.554
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39.442
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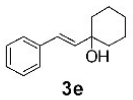
13.998
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

OH

3d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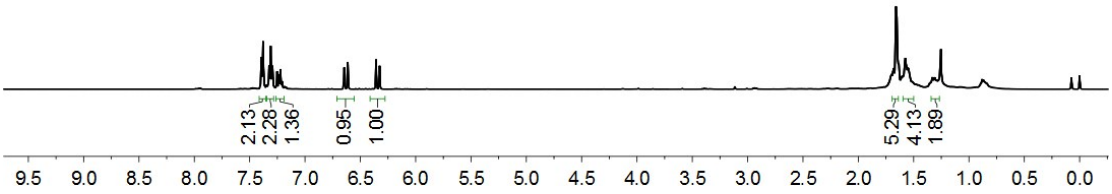
3e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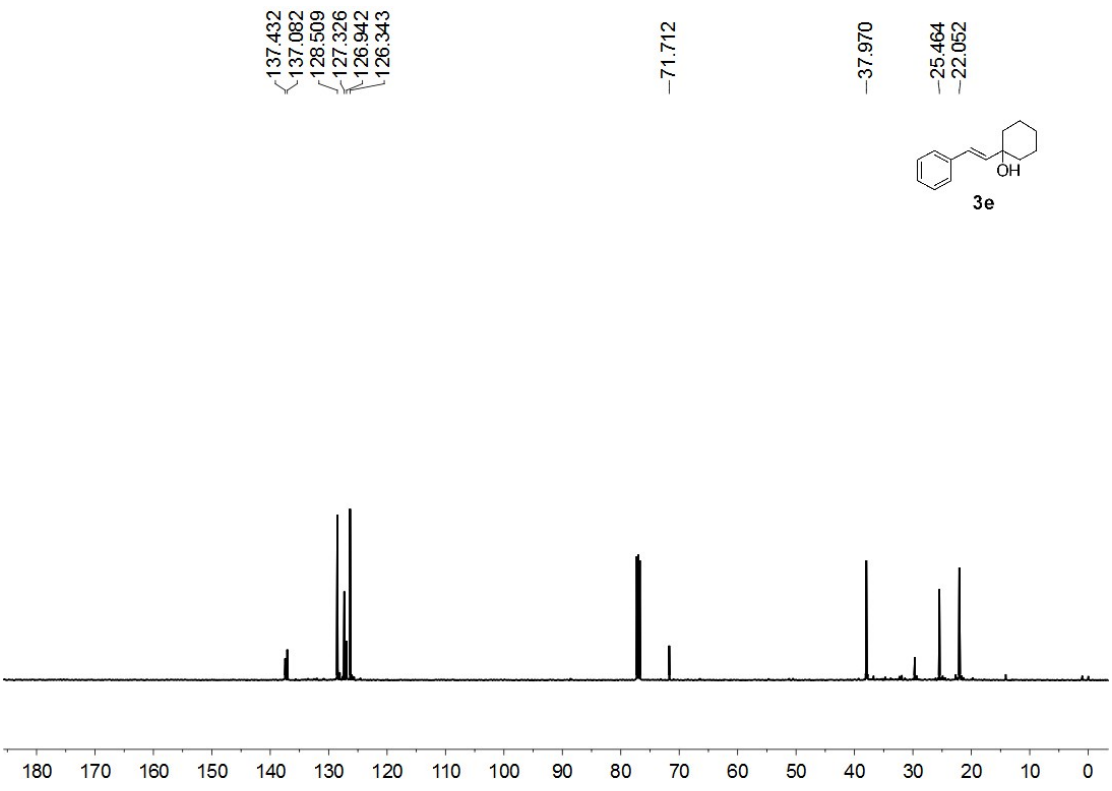
2.28
1.36
0.95
1.00
5.29
4.13
1.89
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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127.326
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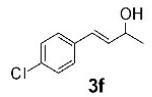
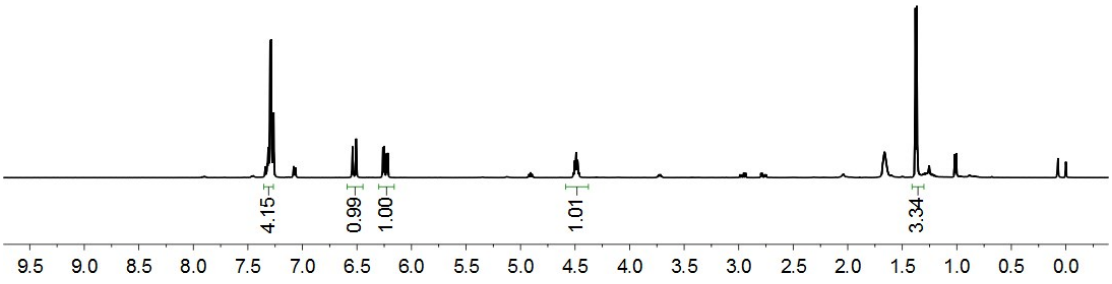
71.712
37.970
25.464
22.052
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

OH

3e


OH
Cl

3f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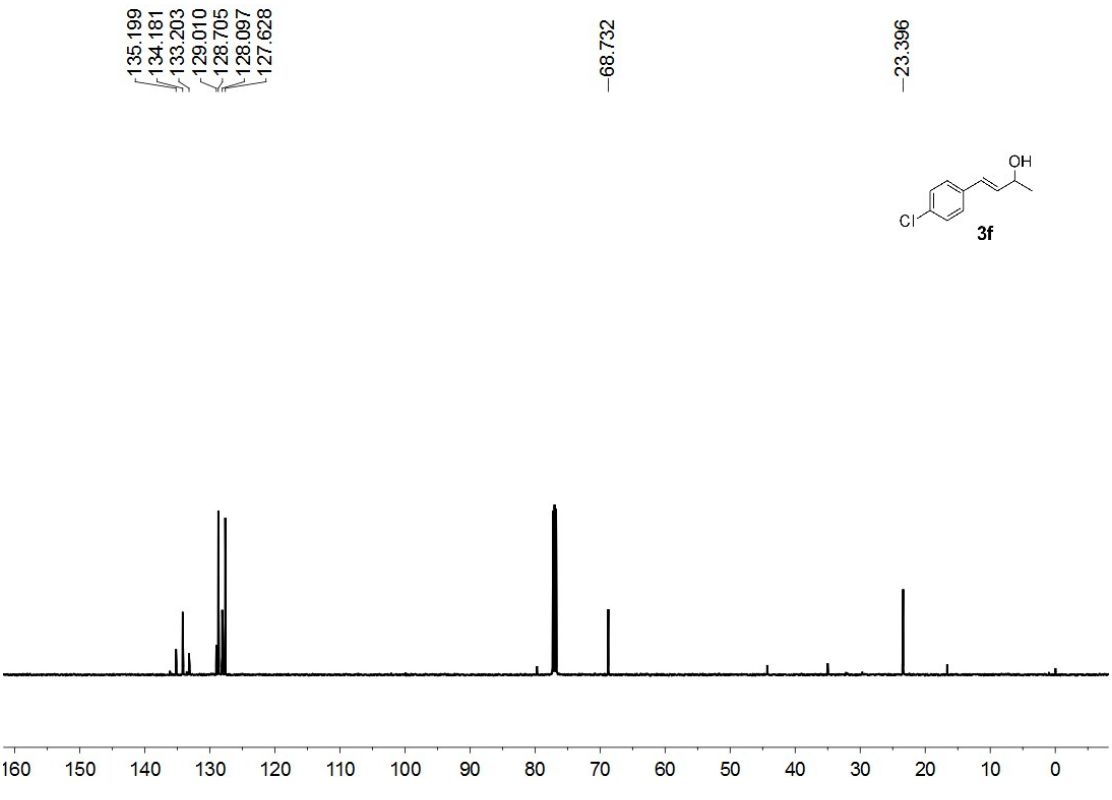
0.99
1.00
1.01
3.34
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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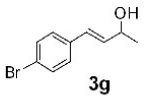
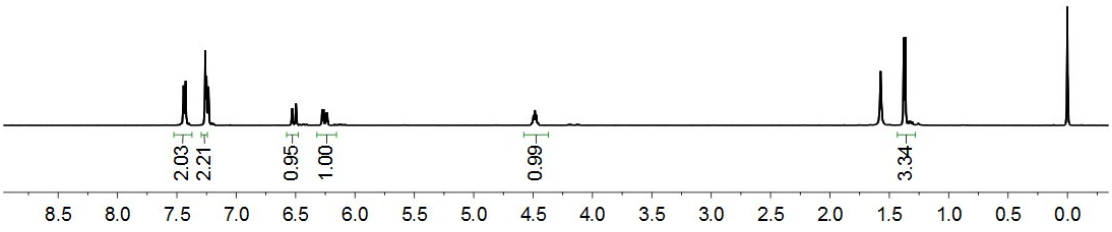
128.097
127.628
68.732
23.396
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

OH
Cl

3f


OH
Br

3g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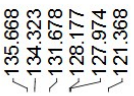
2.21
0.95
1.00
0.99
3.34
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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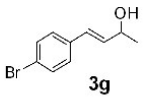
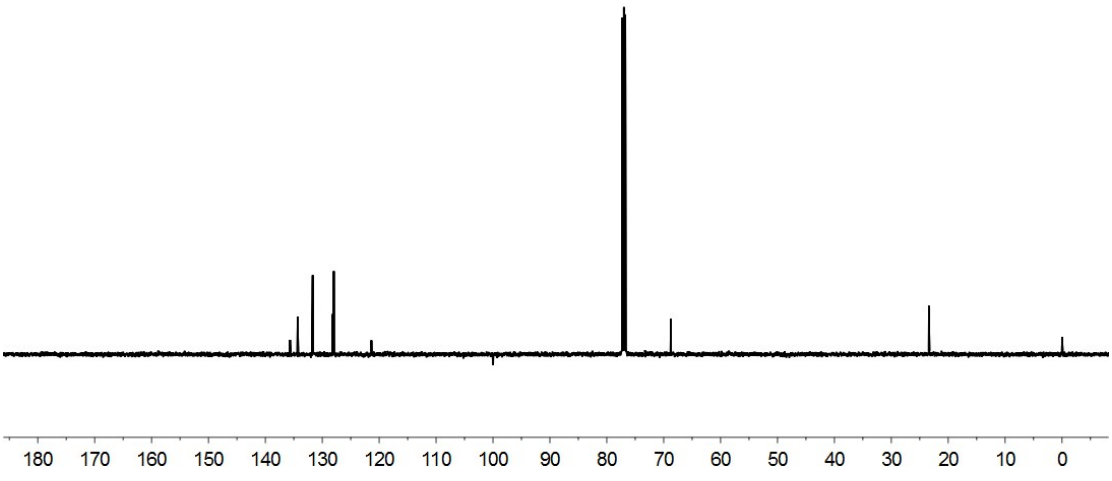
131.678
128.177
127.974
121.368


68.751


23.395


OH
Br

3g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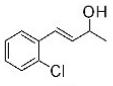
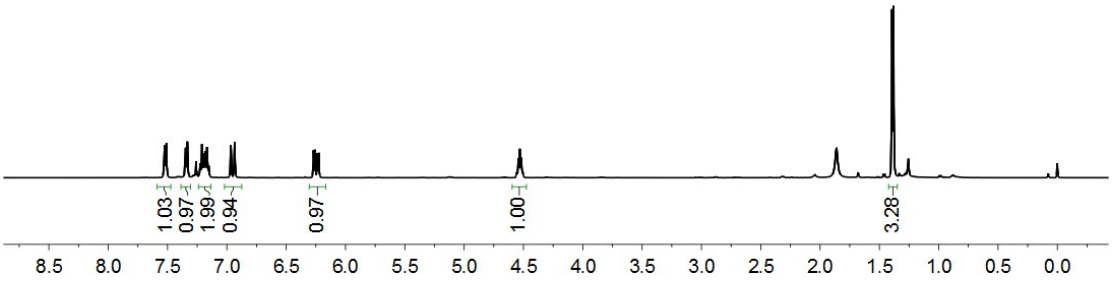

OH
Cl

3h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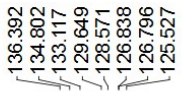
0.97
1.99
0.94
0.97
1.00
3.28
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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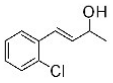
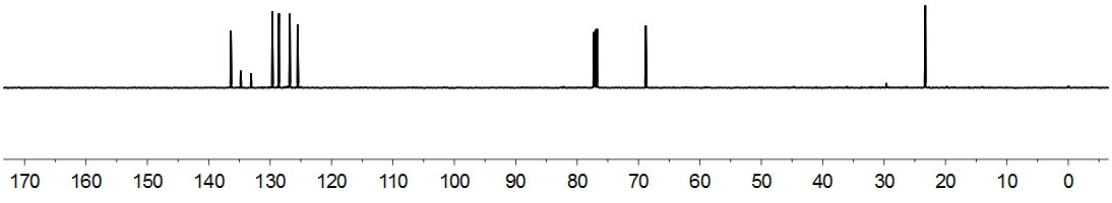
126.796
125.527


68.825


23.285


OH
Cl

3h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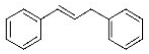
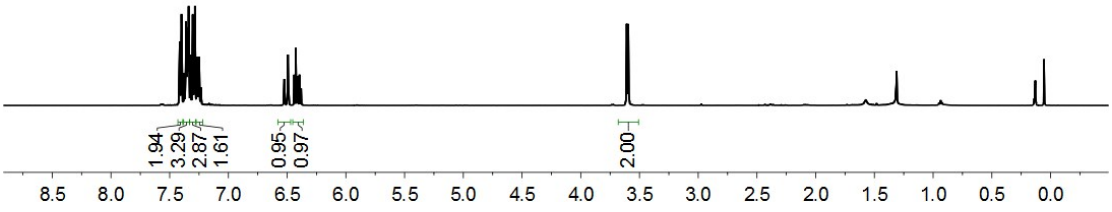


4a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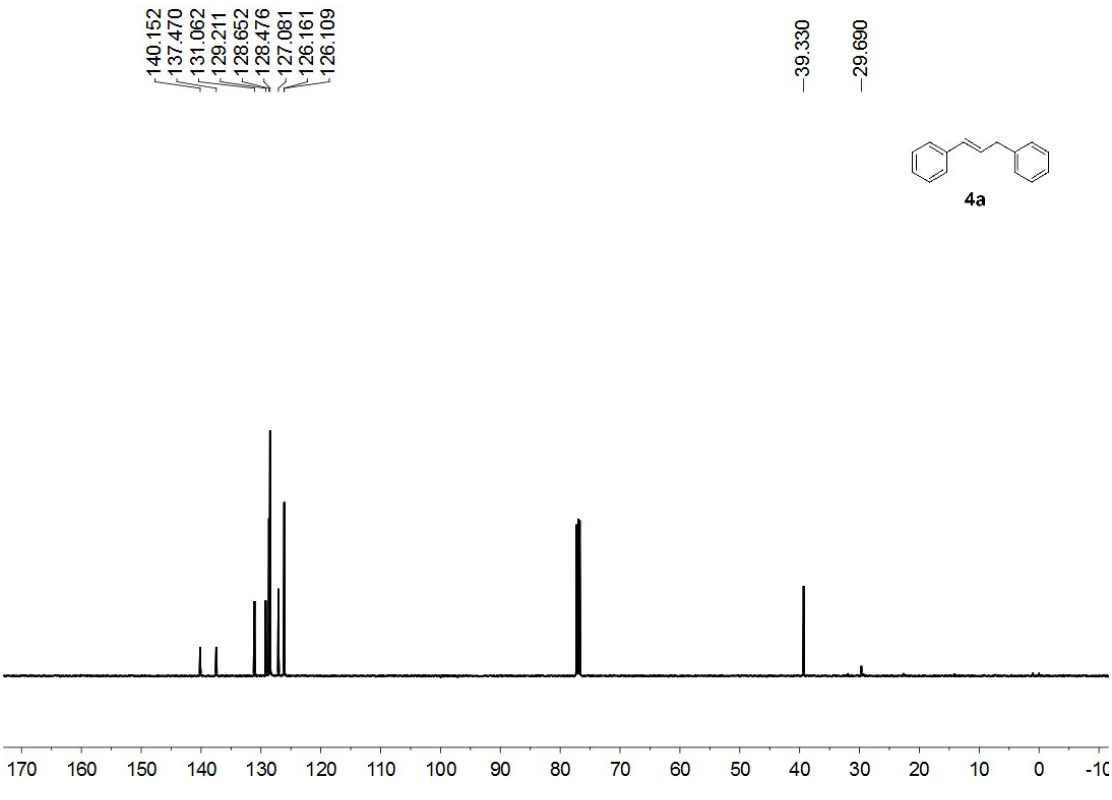
3.29
2.87
1.61
0.95
0.97
2.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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39.330
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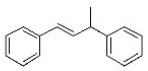
4a



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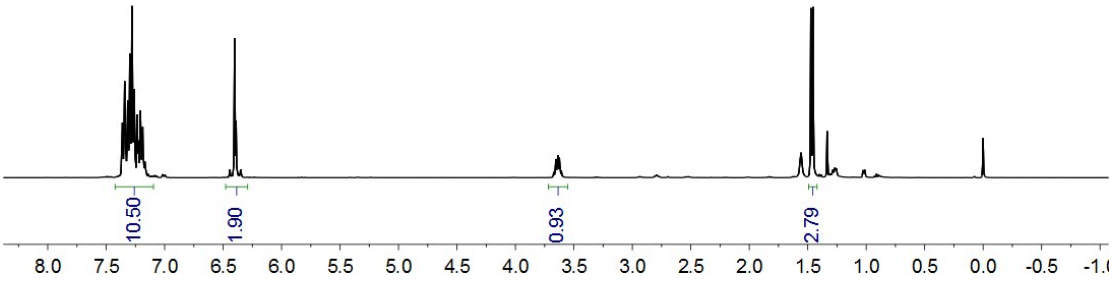
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1.90
0.93
2.79
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5


145.600
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127.019
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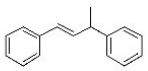
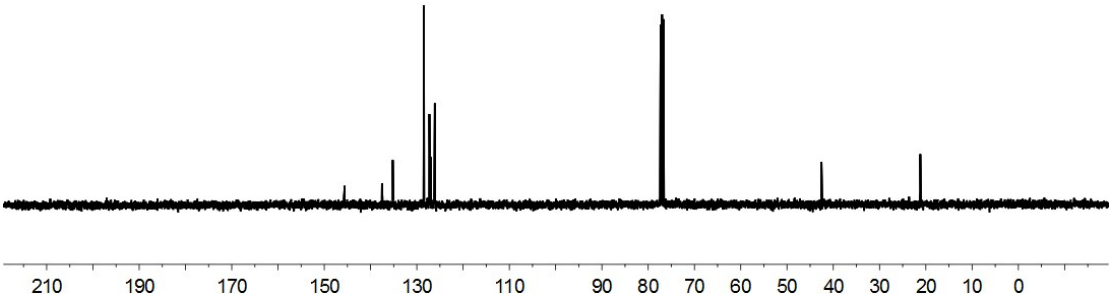
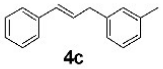
126.117


42.539
21.194

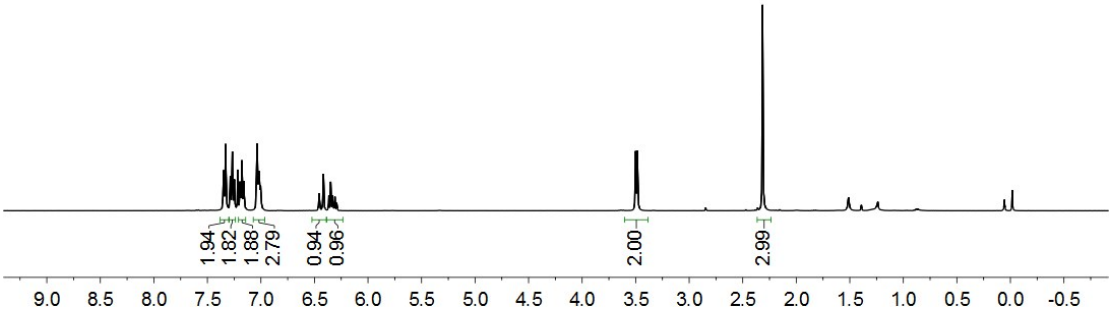


4b


210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0



4c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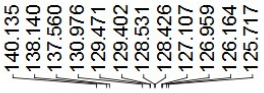
1.82
1.88
2.79
0.94
0.96
2.00
2.99
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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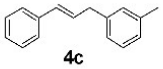
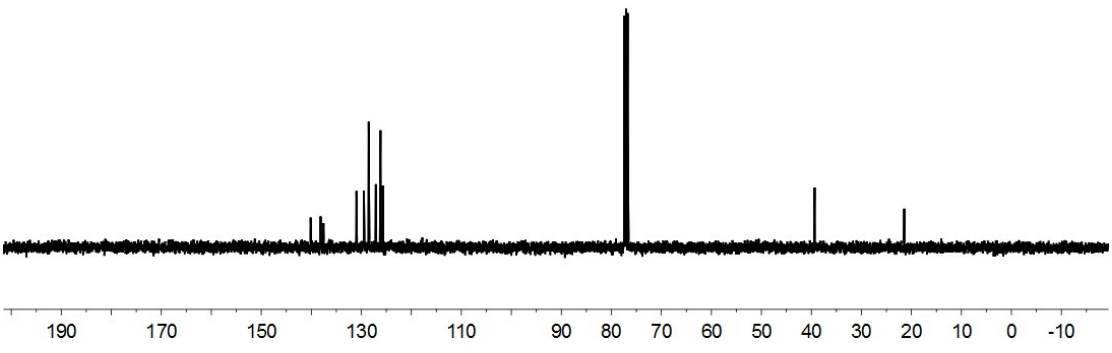
127.107
126.959
126.164
125.717


39.348
21.436



4c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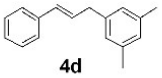

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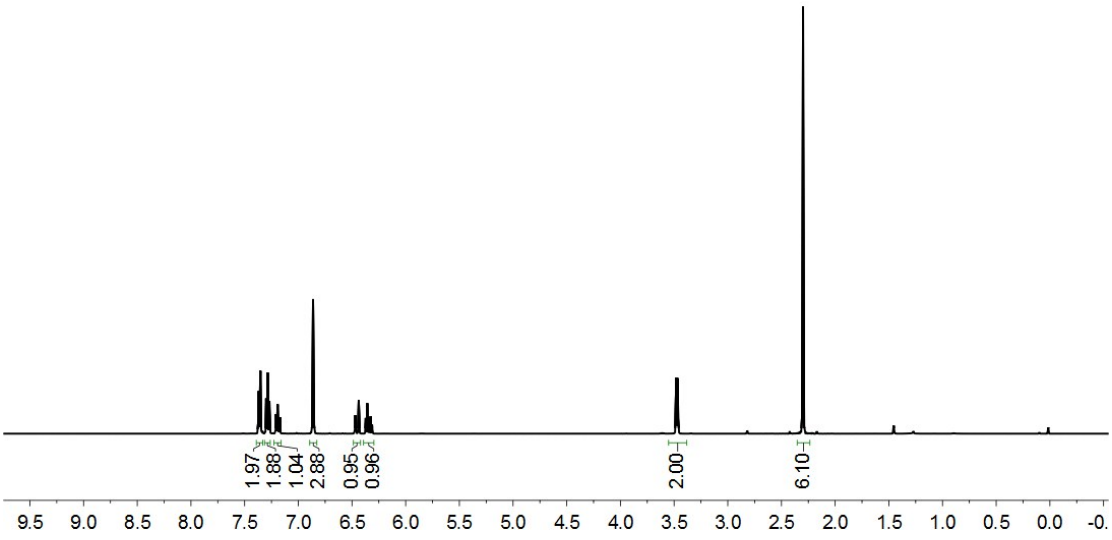
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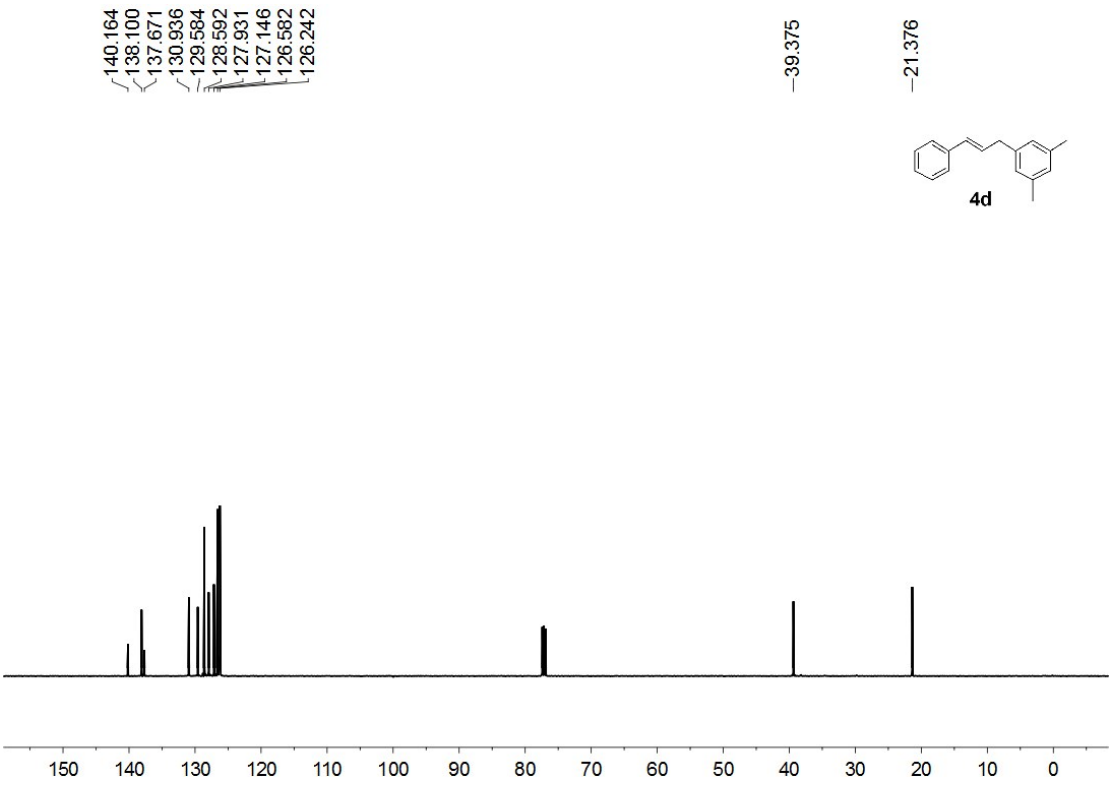
1.88
1.04
2.88
0.95
0.96
2.00
6.10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.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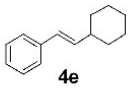
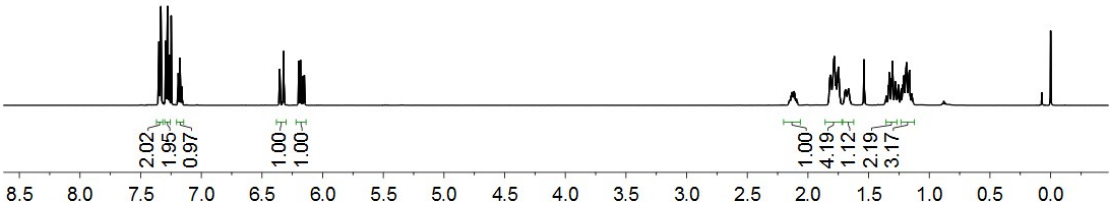
130.936
129.584
128.592
127.931
127.146
126.582
126.242
39.375
21.376
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

4d



4e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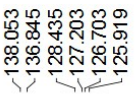
1.95
0.97
1.00
1.00
1.00
4.19
1.12
2.19
3.17
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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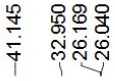
126.703
125.919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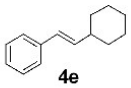
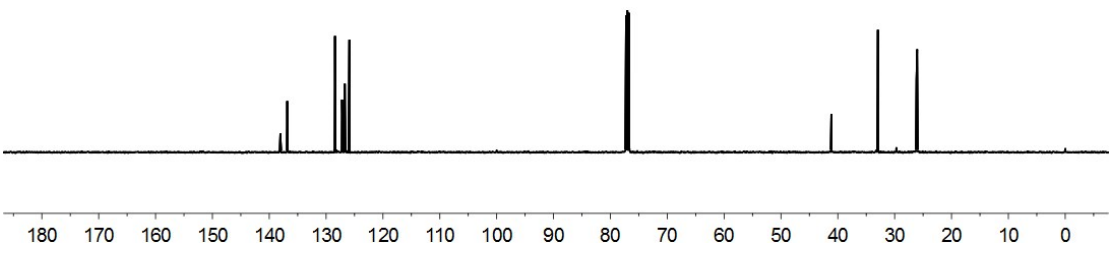
32.950
26.169
26.040



4e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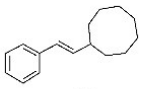
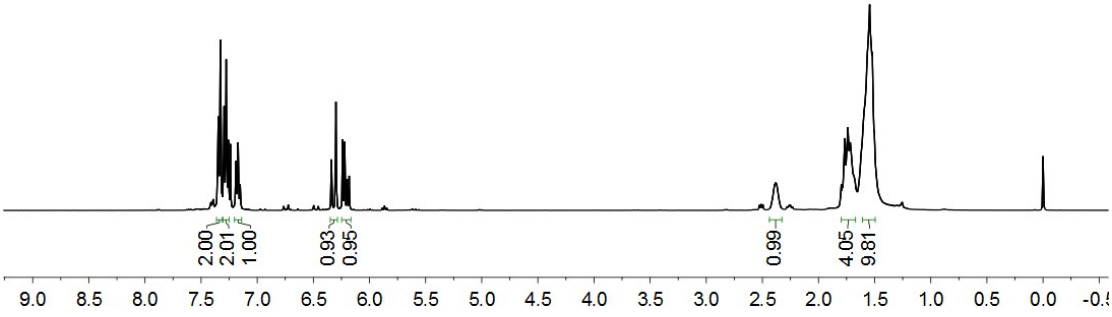
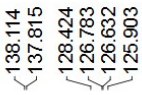


4f


2.00
2.01
1.00
0.93
0.95
0.99
4.05
9.81
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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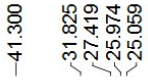
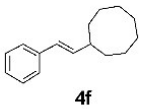

41.300
31.825
27.419
25.974
25.059



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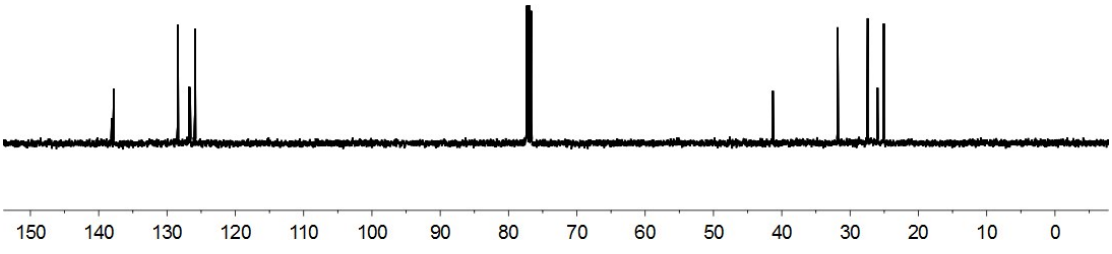
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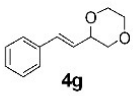

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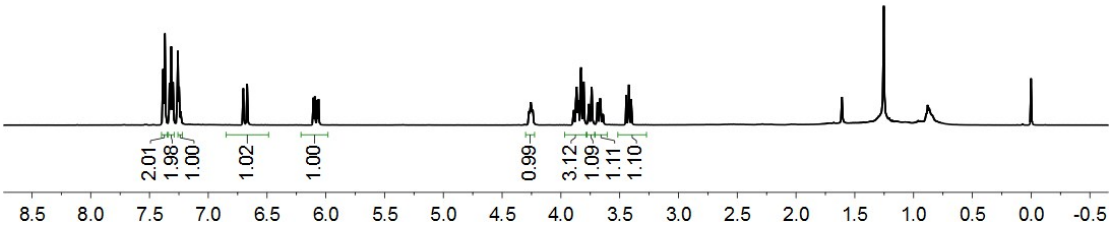
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1.00
1.02
1.00
0.99
3.12
1.09
1.11
1.10
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5


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132.645
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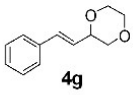
66.587
66.283



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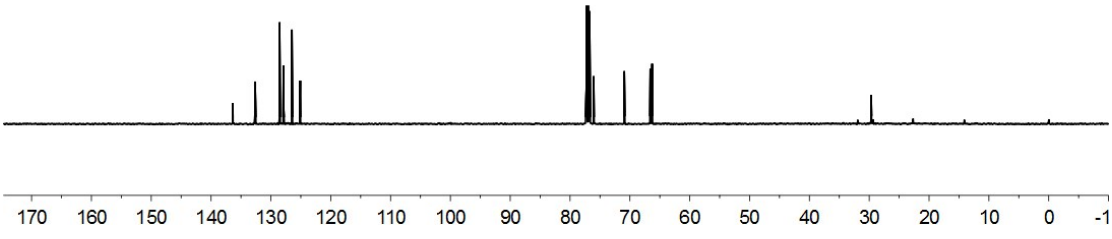
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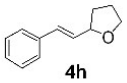

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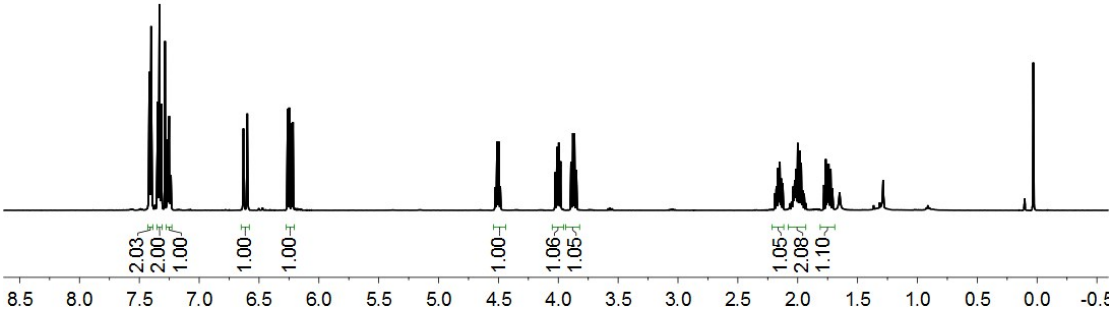
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1.00
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1.00
1.06
1.05
1.05
2.08
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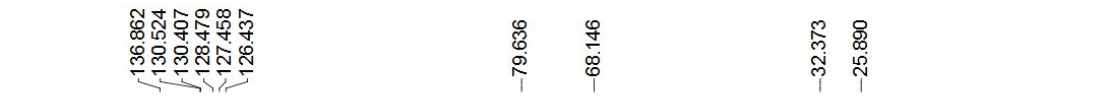
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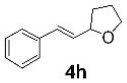
25.890



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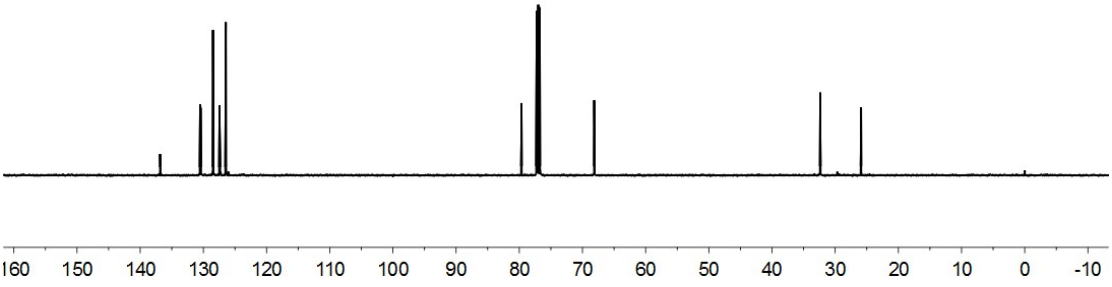
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