A new periodic mesoporous organosilica containing diimine-phloroglucinol, Pd(II)-grafting and its excellent catalytic activity and *trans*-selectivity in C-C coupling reaction

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Supporting Information

Spectroscopic data of (^{1}H & ^{13}C NMR) of Heck-coupling products over Pd-LHMS-3:

Product, (E)-Stilbene:(Table 1, Entry 1 & 4): ^{1}H NMR(300 MHz $CDCl_3$)

δ = 7.59-7.56 (d, 4H, Ar-H) , 7.44-7.39 (t, 4H, Ar-H) , 7.35-7.18 (m,4H,Ar-H & vinyl-H) ppm.

^{13}C NMR ($CDCl_3$) : 137.4,128.7,127.6,126.6 ppm

Product, (E)-4-methoxy Stilbene (Table 1, Entry 2**):**

^{1}H NMR (300 MHz DMSO-d_6) δ = 7.57 (d, J= 4.36 Hz, 2H, ArH),7.54 (d, J=5.9 Hz, 2H, ArH), 6.96 (d, J=8.68 Hz, 2H, ArH), 7.38 (t, J=7.37 Hz, 2H, ArH), 7.25 (t, J=7.33 Hz, 1H, ArH),7.22 (d, J=16.4 Hz, 1H, Vinyl-H),7.11 (d, J=16.4 Hz, 1H, Vinyl-H), 3.77 (s, 3H, -OCH_3) ppm.

^{13}C NMR (DMSO-d_6) : δ159.4,137.8,130.1,129.1,128.5,127.6,126.5,114.6,55.62 ppm.

Product, (E)-4-nitro Stilbene (Table 1, Entry 3) :

^{1}H NMR (300 MHz DMSO-d_6) δ = 8.23 (d, J=8.57 Hz, 2H, ArH), 7.86 (d, 2H, J=8.62 Hz, ArH), 7.67 (d, J=7.56, 2H, ArH) 7.54 (d, 1H, J=16.4 Hz, Vinyl-H), 7.43-7.33(m, 4H, 3ArH, 1 Vinyl-H) ppm.

^{13}C NMR (DMSO-d_6) : δ 146.17, 143.9,136.25,133.2, 128.8,127.27,126.3,124.16 ppm.

Product, (E)-para formyl Stilbene (Table 1, Entry 5**):**

^{1}H NMR (300 MHz DMSO-d_6) δ = 7.90 (d, 2H, J=8.25 Hz, ArH), δ = 7.81 (d, 2H, J=8.25 Hz, ArH) , δ = 7.65 (d, 2H, J=7.24 Hz, ArH), 7.49-7.27 (m, 5H, 3ArH, 2 Vinyl-H) ppm.

^{13}C NMR (DMSO-d_6) : δ = 192.3,143,136.5,135,131.9,129.9,128.7,127.3,126.9 ppm

Product, (E)-4-nitro cinamic acid (Table 1, Entry 8) :

^{1}H NMR (300 MHz,DMSO-d_6) δ = 8.16 (d, 2H, J=6.9 Hz, ArH), δ = 7.86 (d, 2H, J=5.4 Hz, ArH), δ = 7.59 (d, 1H, J=9.9 Hz, Vinyl-H), δ = 6.64 (d, 1H, J=9.9 Hz, Vinyl-H) ppm.

^{13}C (DMSO-d_6) : δ = 167.5, 148.4, 141.8, 129.9, 124.4 ppm.

Product, (E)-4-methoxy cinamic acid (Table 1, Entry 7) :

^{1}H NMR (300 MHz, $CDCl_3$) : δ =7.82 (d, 1H, J= 9.6 Hz, Vinyl-H), δ= 6.48 (d, 1H, J= 9.6 Hz, Vinyl-H), δ= 7.57-7.37 (m, 4H, ArH) ppm.

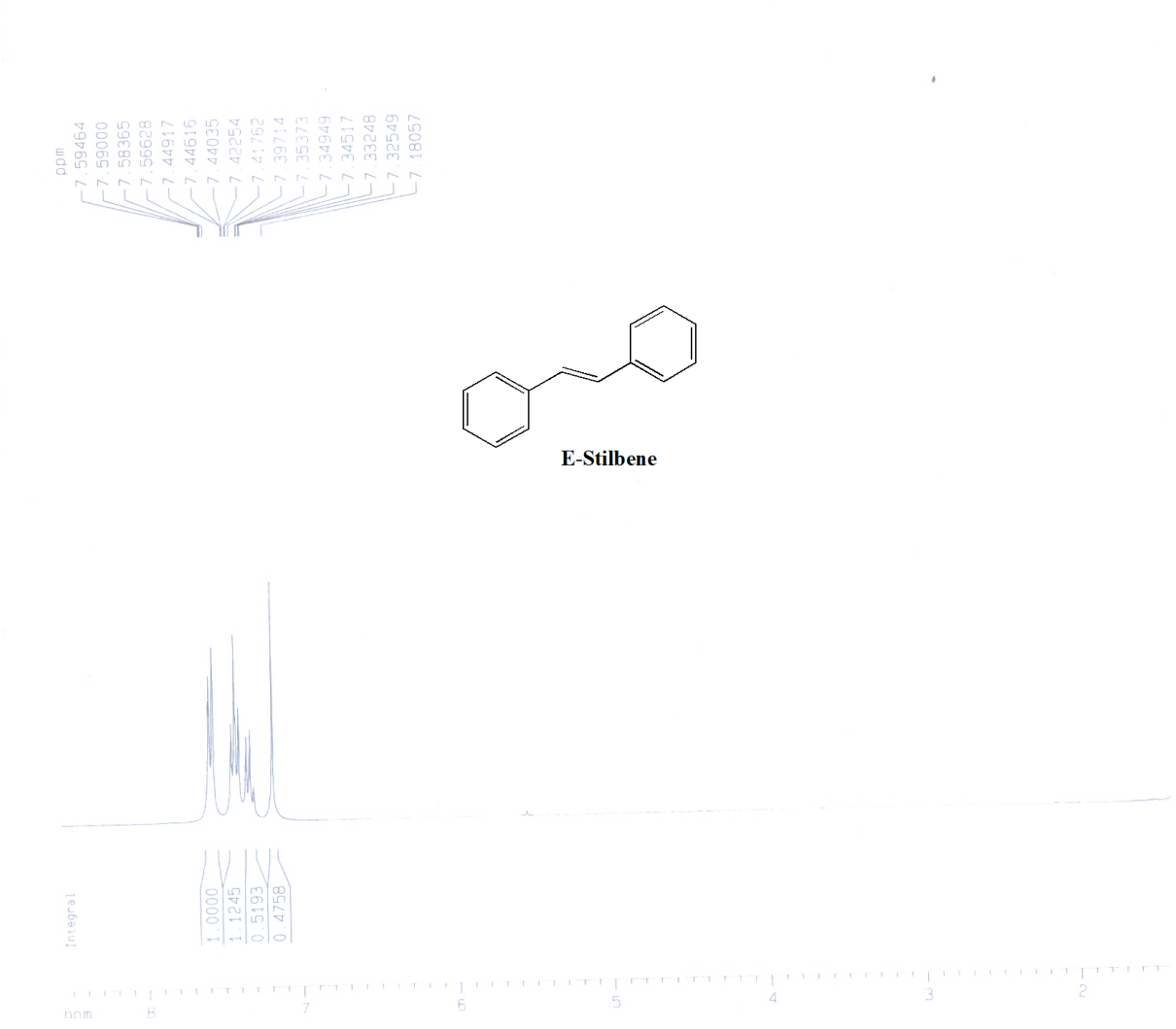
^{13}C ($CDCl_3$) : δ = 172,147,134,130,128,117,51.8 ppm.

Product, trans-cinamic acid (Table 1, Entry 6 & 9) : ^{1}H NMR (300 MHz, $CDCl_3$) : δ= 7.86 (d, 1H, J=9.6 MHz, Vinyl-H), δ= 7.60 (d, 2H, ArH), δ= 7.46 (m, 3H, ArH), δ= 6.52 (d, J= 9.6 MHz, 1H, Vinyl-H) δ= 11.77 (s, 1H, COOH) ppm.

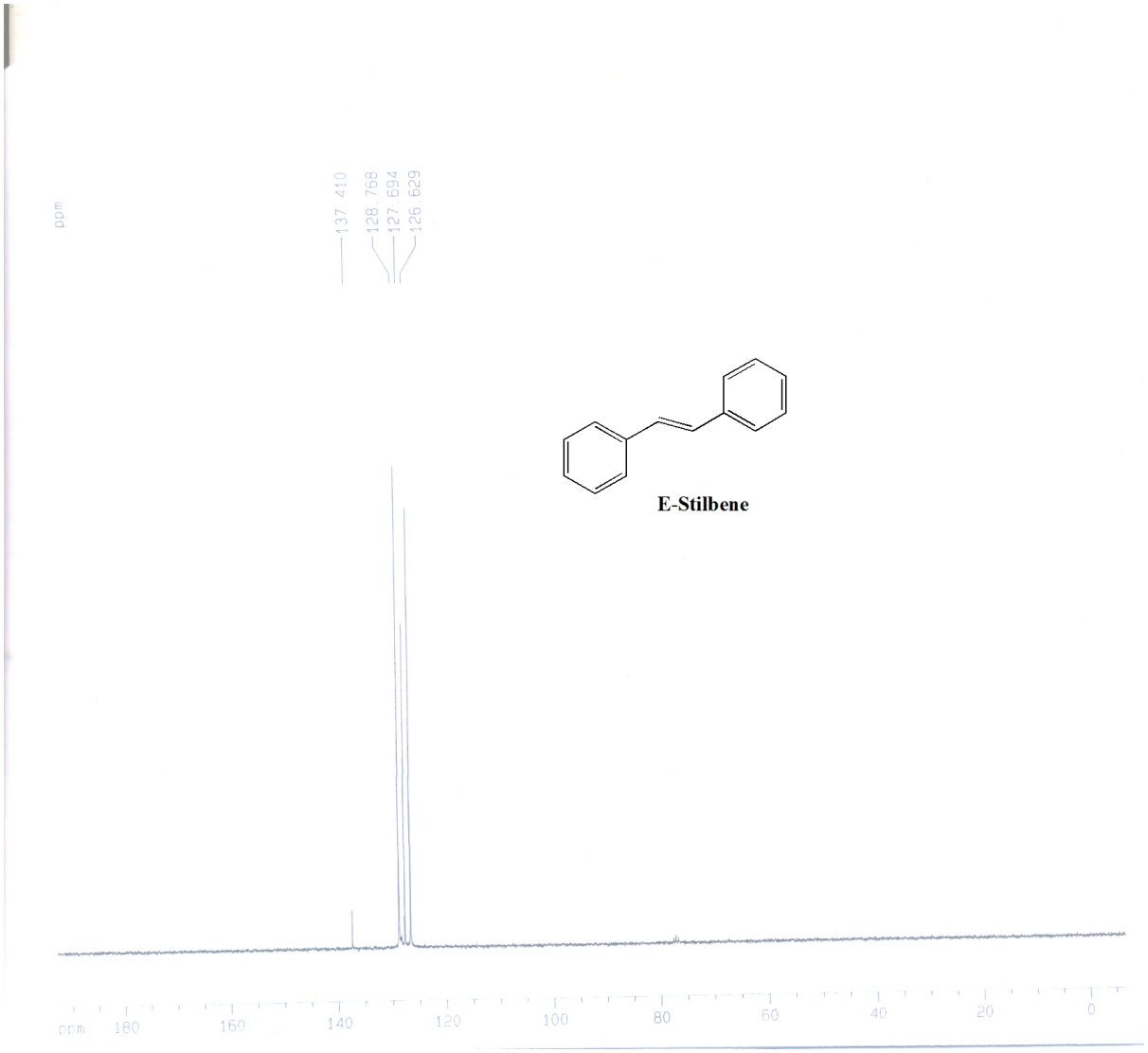
^{13}C NMR ($CDCl_3$, 300MHz) : δ= 172,147,134,130,128,117 ppm.

1. Reference :

(S1, S4 ^{1}H NMR)

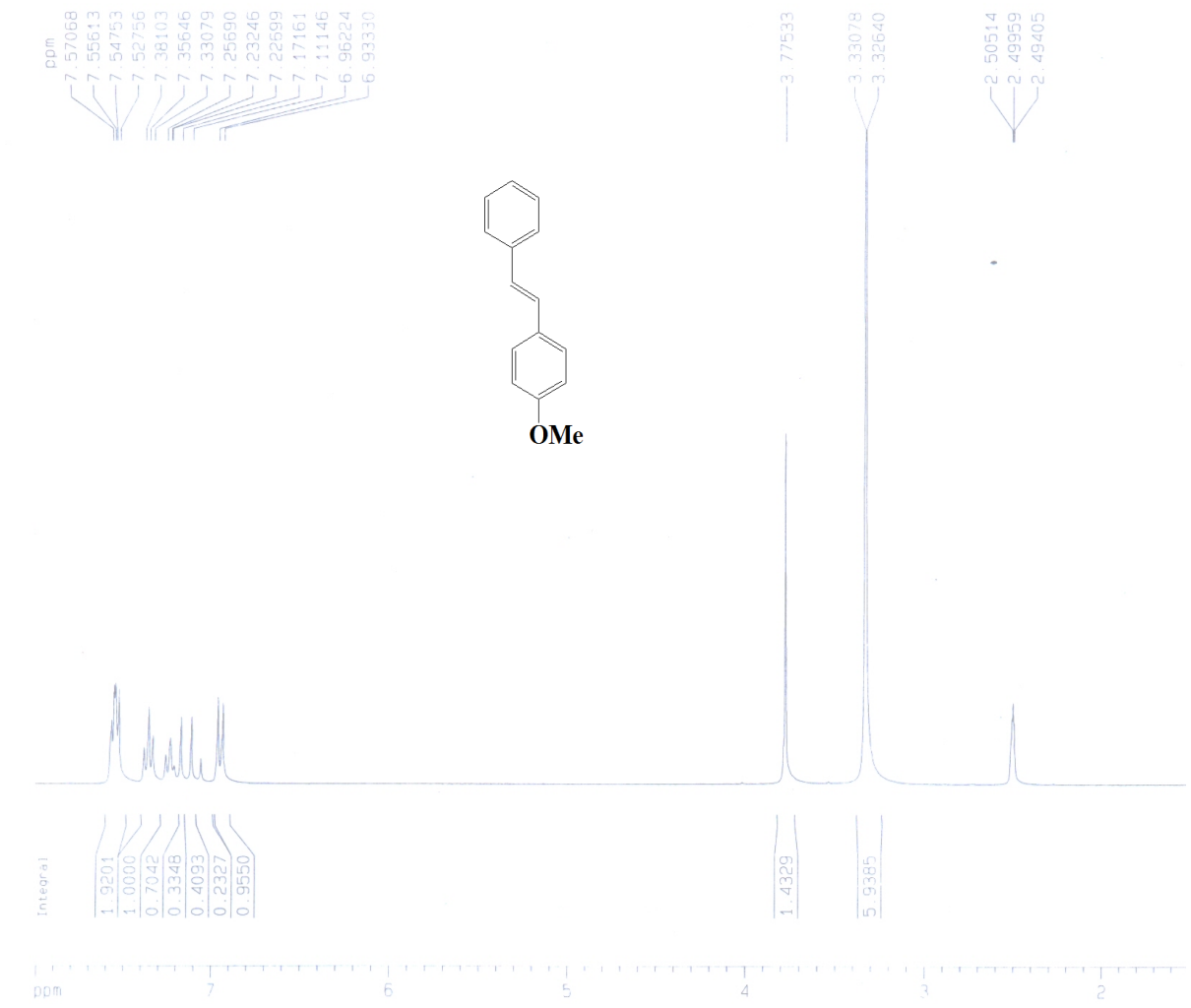


(S1, S4 ^{13}C NMR)



E-Stilbene

(S2, ^{1}H NMR)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7.17161
7.11146
6.96224
6.93330
3.77533
3.33078
3.32640
2.50514
2.49959
2.49405
OMe
Integral
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1.0000
0.7042
0.3348
0.4093
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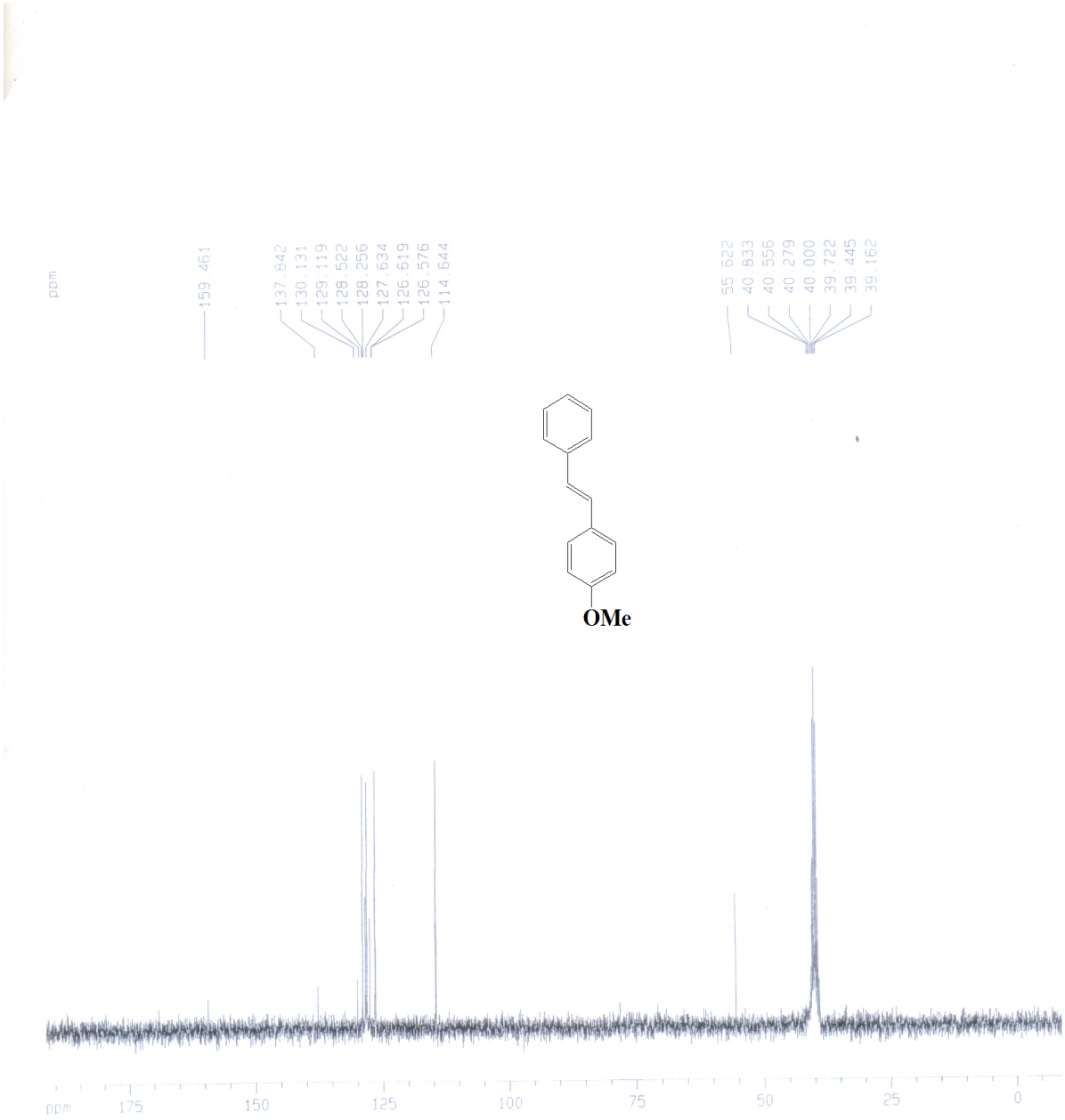
ppm
7
6
5
4
3
2

(S2, ^{13}C NMR)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40.556
40.279
40.000
39.722
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ppm
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75
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25
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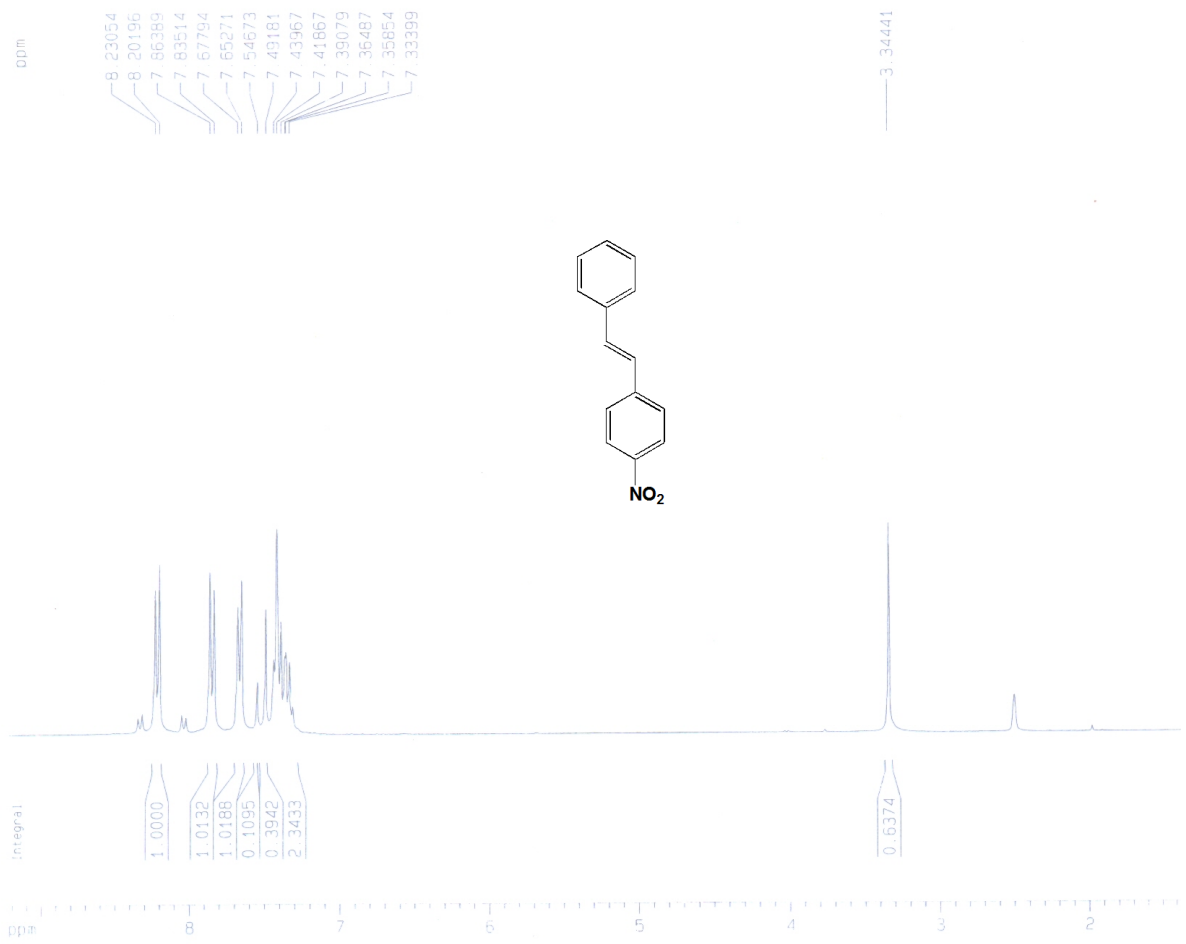
(S3, ^{1}H NMR)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7.36487
7.35854
7.33399
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NO2
Integral
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6
5
4
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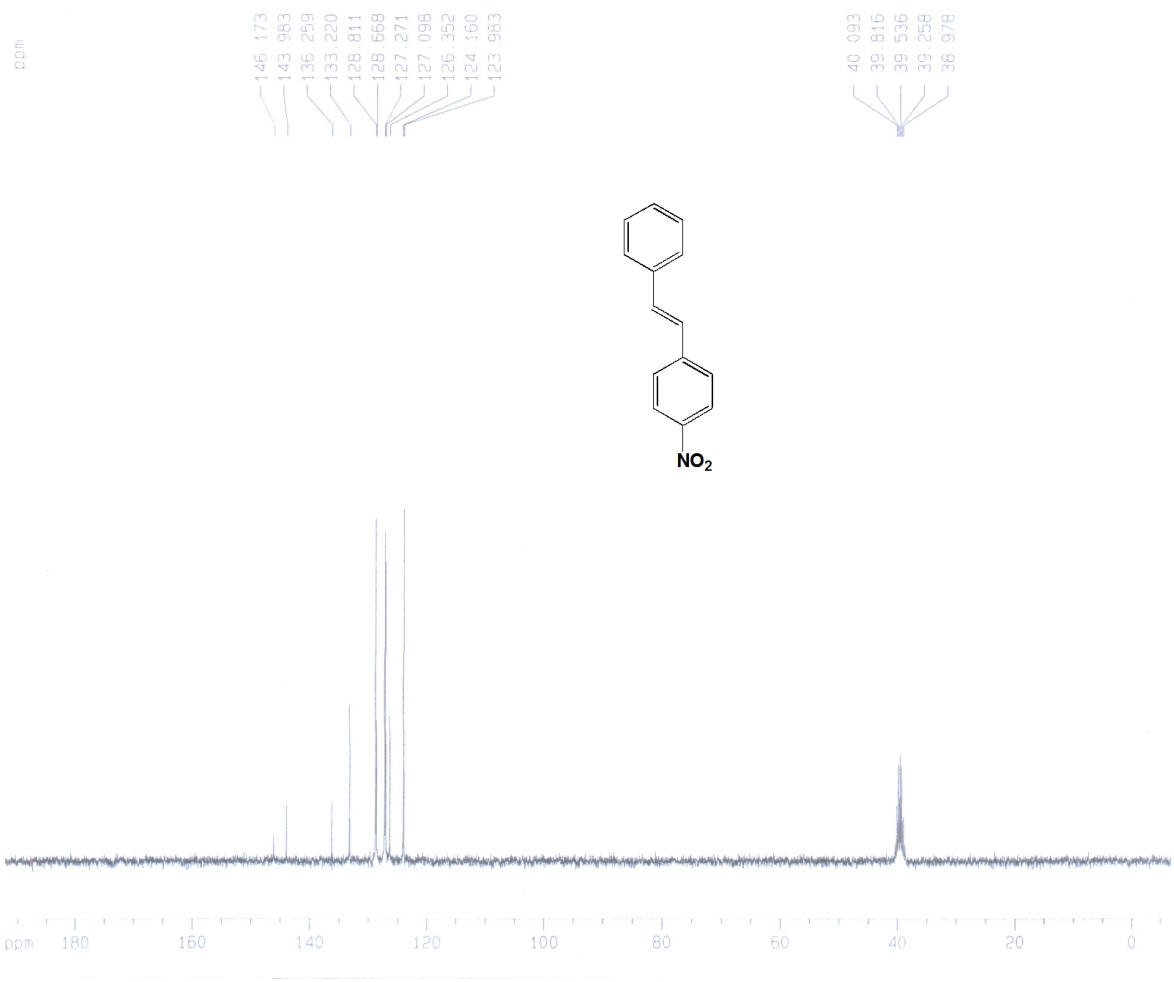
2

(S3, ^{13}C NMR)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39.816
39.536
39.258
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NO2
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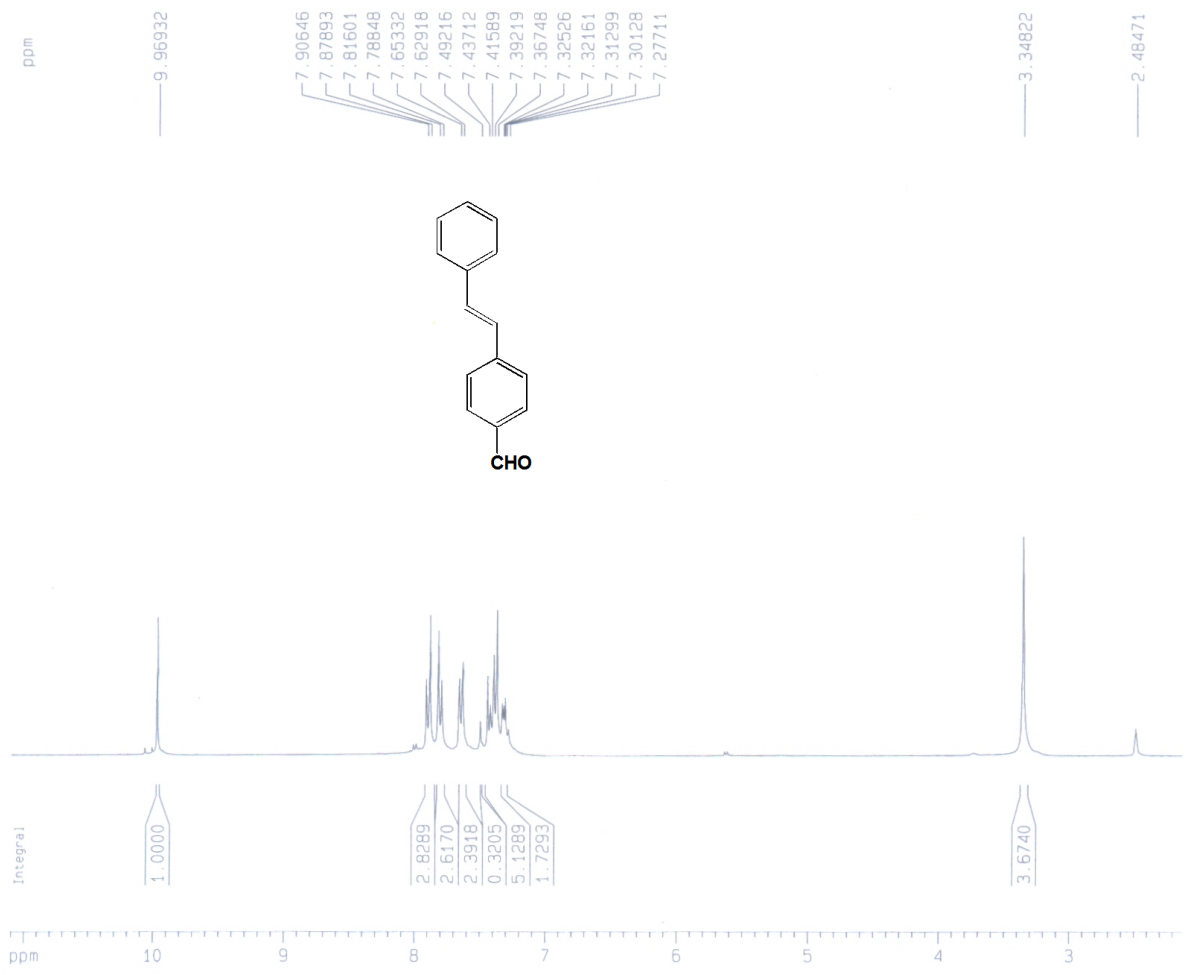
(S5, ^{1}H NMR)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7.32526
7.32161
7.31299
7.30128
7.27711
3.34822
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Integral
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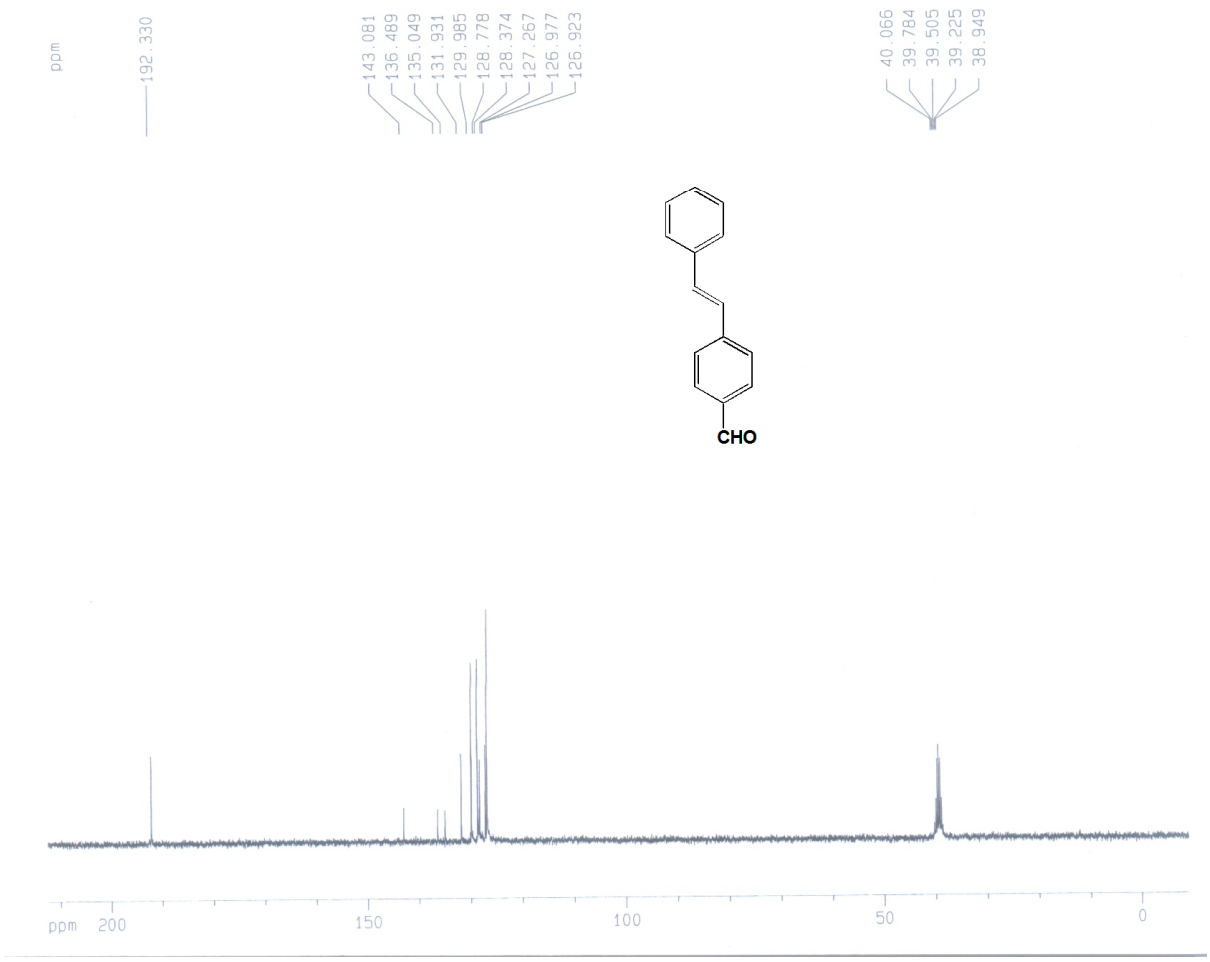
8
7
6
5
4
3

(S5, ^{13}C NMR)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ppm
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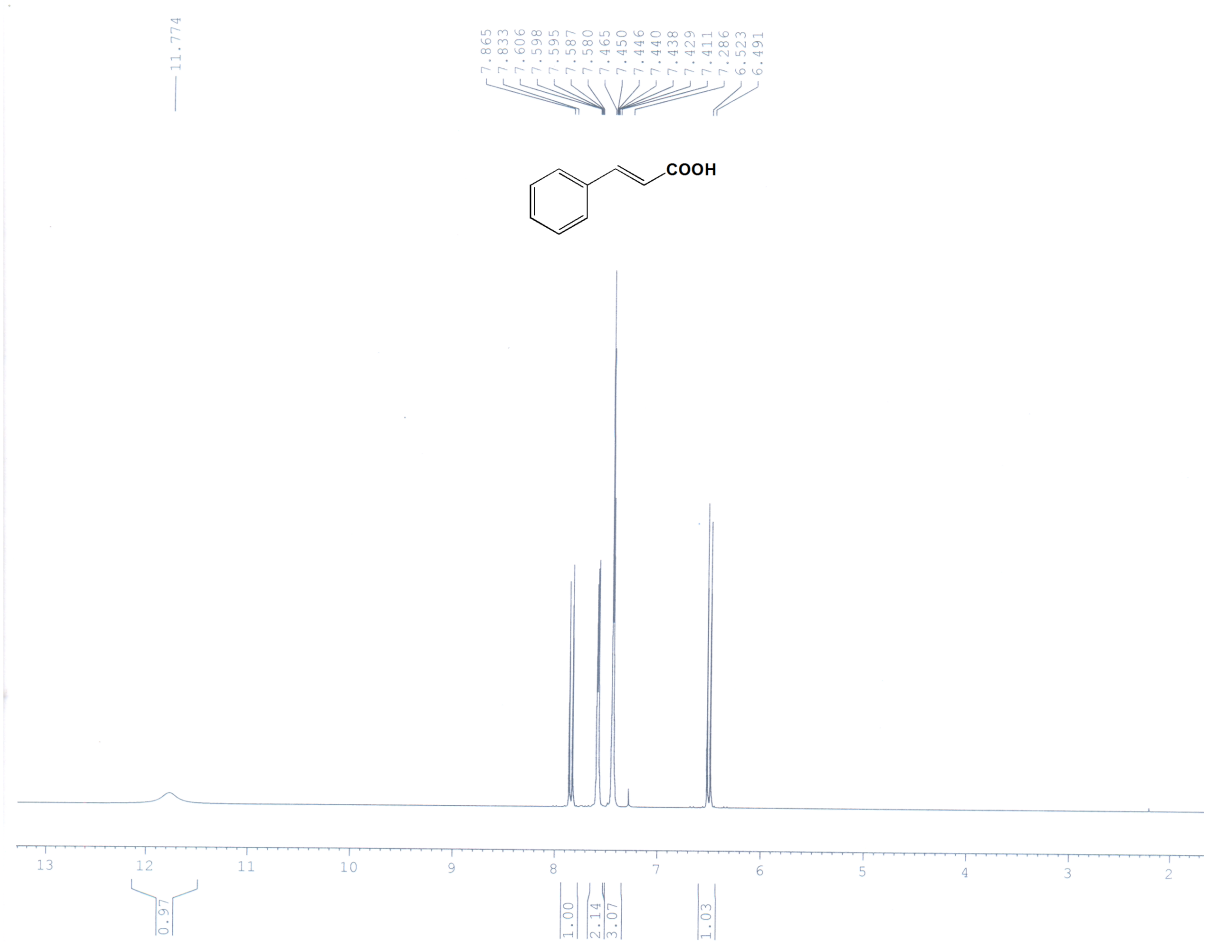
150
100
50
0

(S6, S9, ^{1}H NMR)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7.429
7.411
7.286
6.523
6.491
COOH
13
12
11
10
9
8
7
6
5
4
3
2
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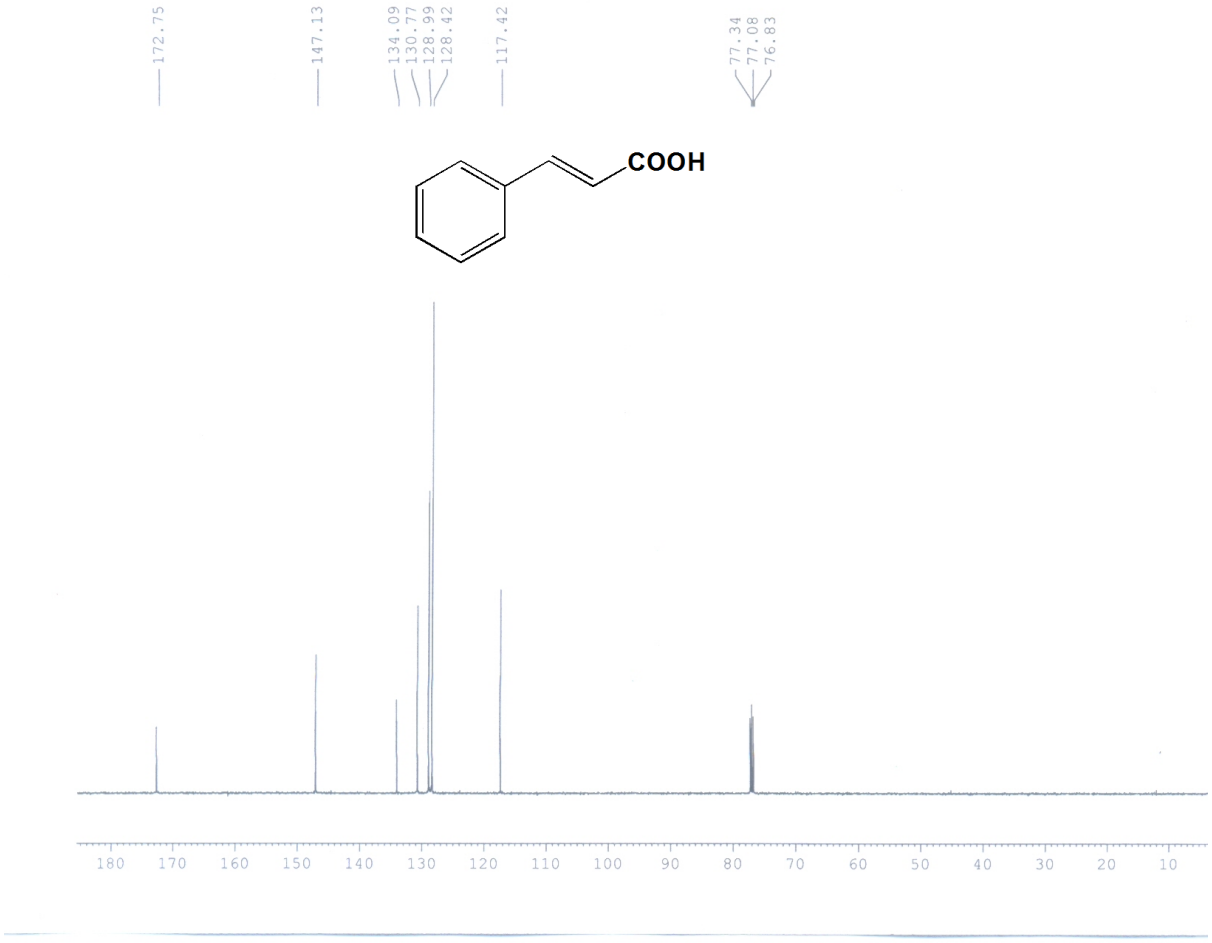
1.00
2.14
3.07
1.03

(S6, S9, ^{13}C NMR)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134.09
130.77
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128.42
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77.34
77.08
76.83
COOH
180
170
160
150
140
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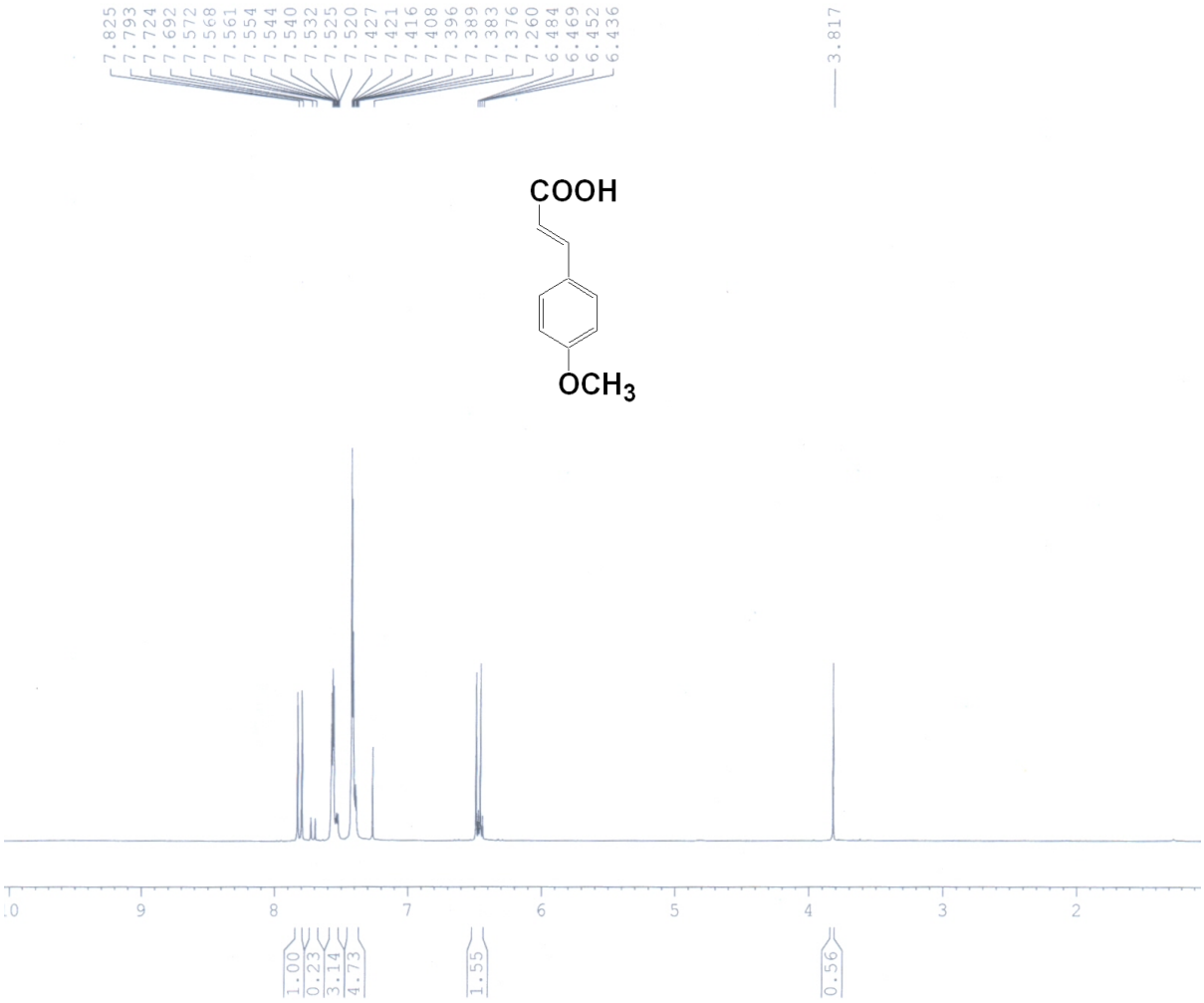
120
110
100
90
80
70
60
50
40
30
20
10

(S7, ^{1}H NMR)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7.427
7.421
7.416
7.408
7.396
7.389
7.383
7.376
7.260
6.484
6.469
6.452
6.436
3.817
COOH
OCH3
10
9
8
7
6
5
4
3
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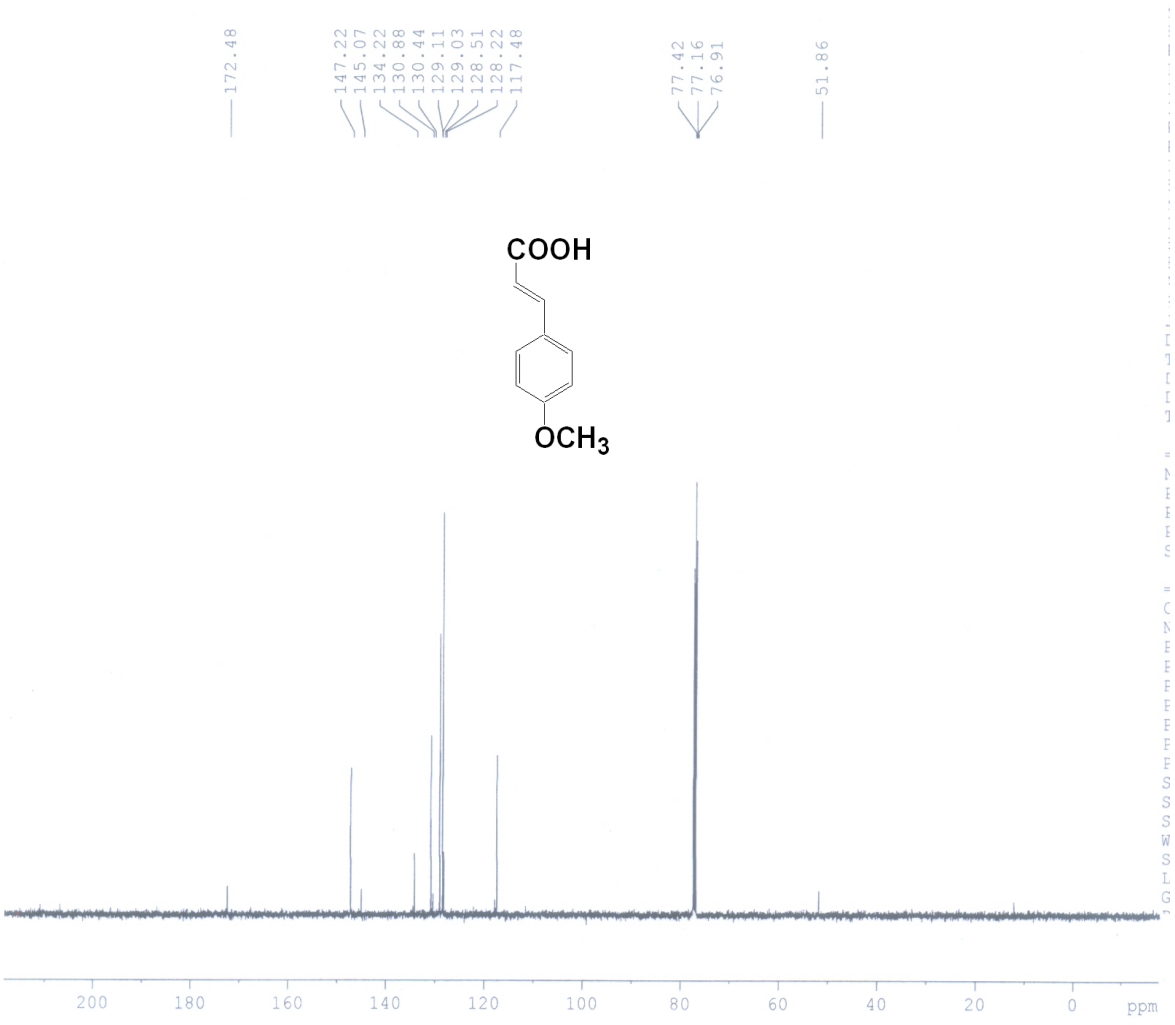
0.56

(S7 ^{13}C NMR)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145.07
134.22
130.88
130.44
129.11
129.03
128.51
128.22
117.48
77.42
77.16
76.91
51.86
COOH
OCH3
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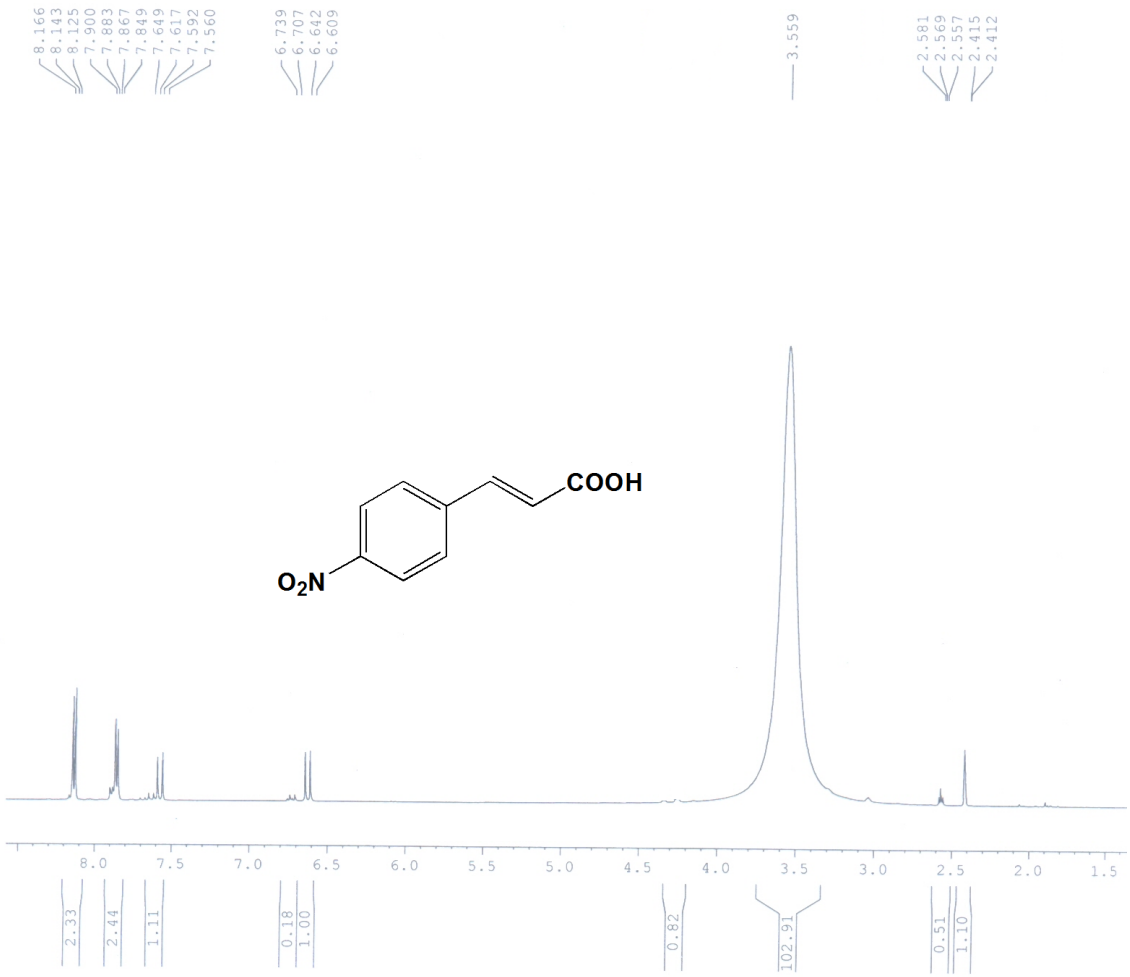
180
160
140
120
100
80
60
40
20
0
ppm

(S8, ^{1}H NMR)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7.560
6.739
6.707
6.642
6.609
3.559
2.581
2.569
2.557
2.415
2.412
COOH
O2N
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
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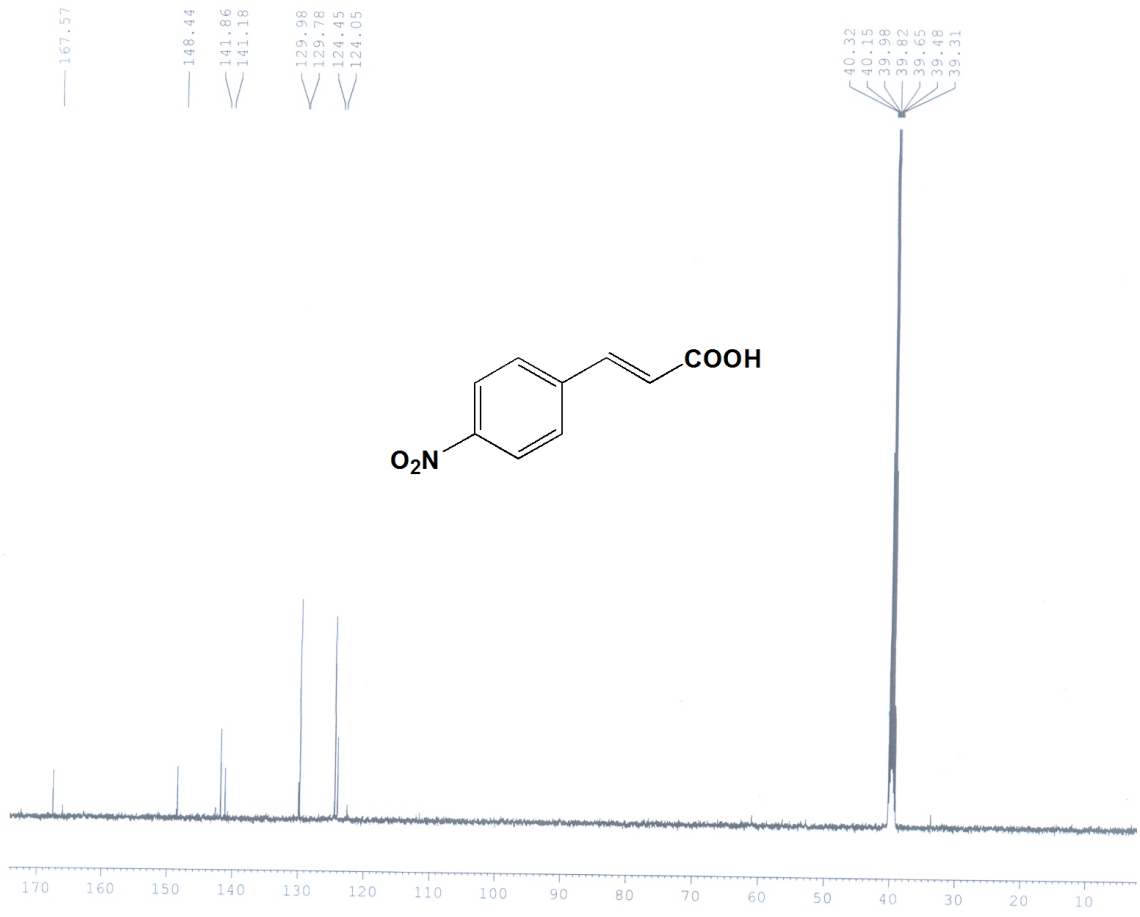
(S8, ^{13}C NMR)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39.98
39.82
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39.48
39.31
COOH
O2N
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150
140
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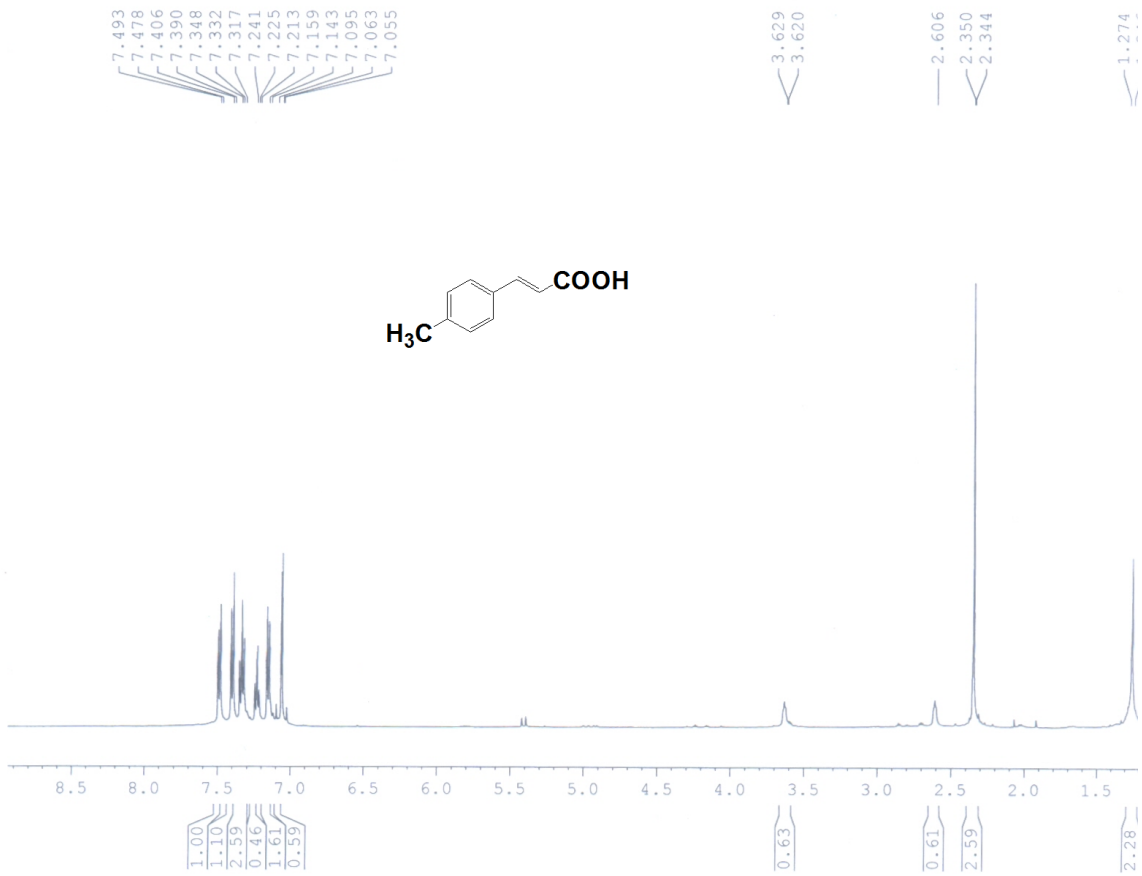
120
110
100
90
80
70
60
50
40
30
20
10

(S10 , ^{1}H NMR)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7.348
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7.143
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7.063
7.055
3.629
3.620
2.606
2.350
2.344
1.274
COOH
H3C
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
1.00
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0.59
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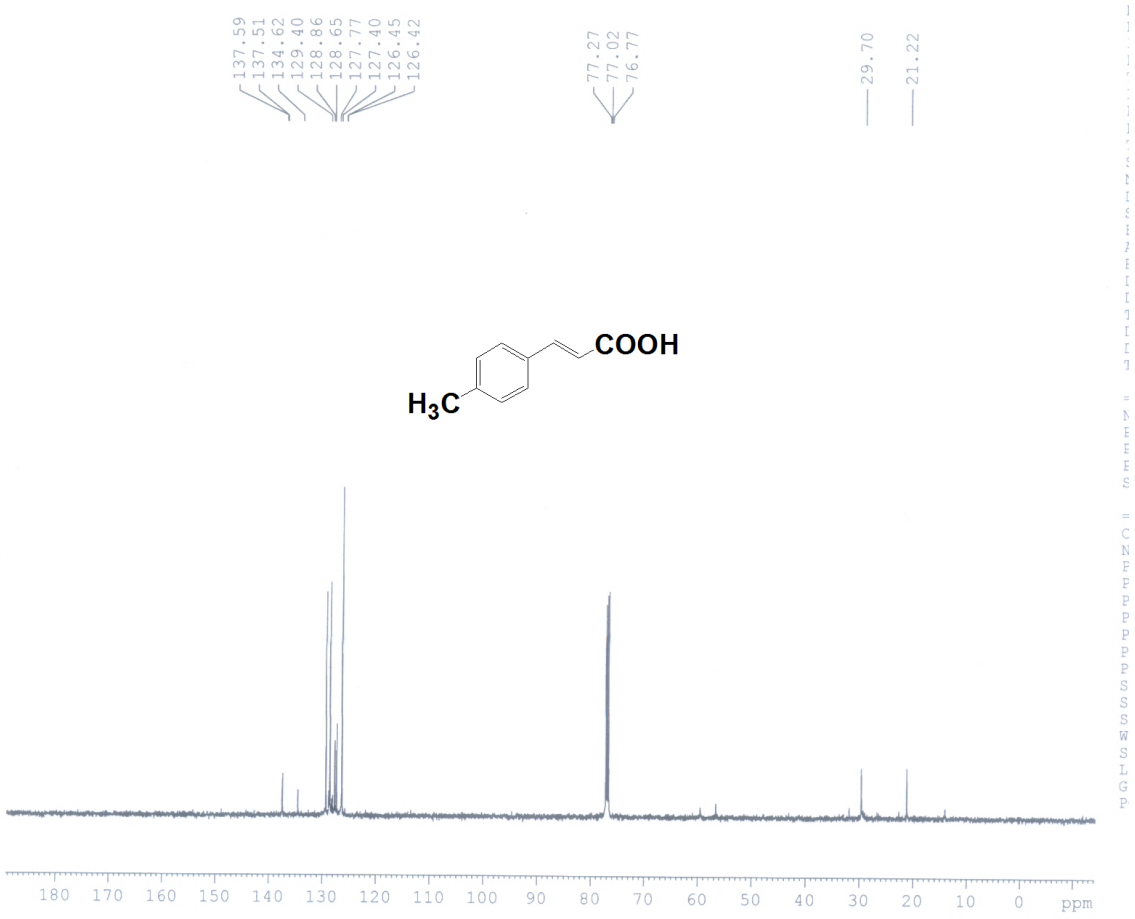
(S10, ^{13}C NMR)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128.65
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126.42
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77.02
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29.70
21.22
COOH
H3C
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm