SUPPORTING INFORMATION

for

Chiral Iron Complex Catalyzed Enantioselective Oxidation of Racemic Benzoins

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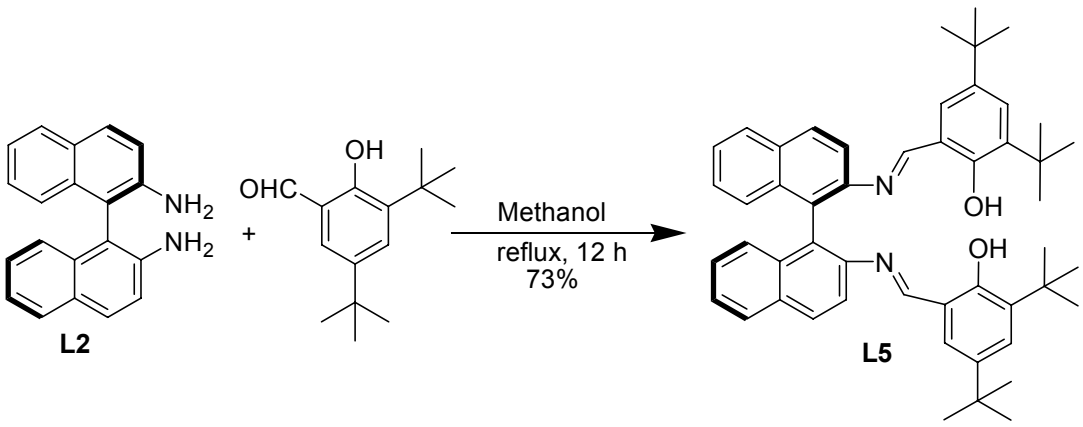
General considerations

Iron(II)acetate and 2,2,6,6-tetramethyl-piperidin-1-oxyl (TEMPO) were purchased from Sigma-Aldrich chemical company and used as received. Thin layer chromatography (TLC) was performed using Merck silica gel 60 F_{254} precoated plates (0.25 mm) and visualized by UV fluorescence quenching. Silica gel for column chromatography (particle size 100-200 mesh) purchased from SRL, India. Optical rotations were measured with Autopol IV - Rudolph Research Analytical Polarimeter. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker 400 MHz instrument. ^{1}H NMR spectra were reported relative to Me_4Si (δ 0.0 ppm) or residual $CHCl_3$ (δ 7.26 ppm). ^{13}C NMR were reported relative to $CDCl_3$ (δ 77.16 ppm). FTIR spectra were recorded on a Nicolet 6700 spectrometer and are reported in frequency of absorption (cm^{-1}). High resolution mass spectra (HRMS) were recorded on Q-Tof Micro mass spectrometer. The enantiomeric excess (%ee) of benzoins were determined by JASCO uv-2070 plus HPLC using Daicel ChiralPAK AS-H (0.46cm ϕ x 25cm) column. ^{1}H and ^{13}C NMR and HRMS Spectral data have been included for all compounds. HPLC spectra for the % *ee* determination of all optically active benzoins are given in this supporting information.

Synthesis of Ligand L5

Scheme 1



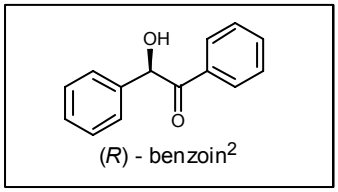
Experimental Procedure

Synthesis of L5[1]**:** In a 100 mL two neck round bottom flask equipped with reflux condenser, a mixture of **L2 (**568 mg, 2 mmol) and 3,5-di-*tert*-butylsalicylaldehyde (983 mg, 4.2 mmol) in methanol (40 mL) (10 ml of $CHCl_3$ added for better solubility of the reagents) was taken and refluxed for 12 hours. The reaction mixture was cooled to room temperature, solvent was removed under reduced pressure and the residue was purified by column chromatography on basic aluminum oxide to give **L5** (1050 mg, 73%) as a yellow compound. Mp 109-113 ^{o}C (lit.[1b] 112-114 ^{o}C); R_f 0.4 (in hexanes : ethyl acetate, 95:5 V/V); $[\alpha]^{D}_{25}$ = – 432.28 (*c* = 1 in CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.72 (s, 2H), 8.65 (s, 2H), 8.03 (d, *J* = 8.8 Hz, 2H), 7.94 (d, *J* =

8.4 Hz, 2H), 7.57 (d, *J* = 9.2 Hz, 2H), 7.41-7.46 (m, 2H), 7.33 (d, *J* = 8 Hz, 2H), 7.27-7.31 (m, 2H), 7.24 (d, *J* = 2.4 Hz, 2H), 7.04 (d, *J* = 2.4 Hz, 2H), 1.22 (s, 36H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.9, 158.3, 144.2, 139.9, 136.7, 133.5, 132.7, 129.8, 129.4, 128.3, 127.6, 126.9, 126.7, 126.6, 125.6, 118.3, 117.4, 35.0, 34.2, 31.5, 29.3; IR (Neat) 3649, 2955, 2871, 1612, 1583, 1467, 818 cm^{-1}; HRMS (*m*/*z*): $[M+1]^+$calcd for $C_{50}H_{57}N_2O_2$, 717.4420; found, 717.4423.

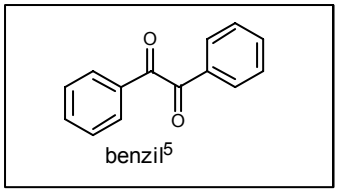
General experimental procedure for oxidative kinetic resolution

A mixture of **L5** (35.8 mg, 0.05 mmol) and iron(II)acetate (8.7 mg, 0.05 mmol) in 8 mL of hexanes was taken in a reaction tube and stirred at room temperature for 10 minutes, TEMPO (3.9 mg, 0.025 mmol) was added to the reaction mixture. After stirring for 5 minunets, benzoin (106 mg, 0.5 mmol) was added and then the reaction mixture was stirred under an O_2 atmosphere (using O_2 balloon) at 60 ^oC for 25 hours. The reaction mixture was concentrated and the resulting residue was purified by silica gel column chromatography (eluents: hexanes-ethyl acetate) to give the benzil (77 mg, yield 73%) and unreacted benzoin (22 mg, yield 21%).



(*R*) - benzoin[2]

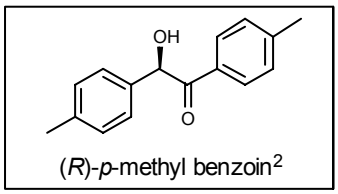
R_f 0.23 (hexanes : ethyl acetate, 90:10 v/v); $[\alpha]^D_{25}$ = – 76.0 (*c* = 1 in acetone); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88-7.93 (m, 2H), 7.48–7.53 (m, 1H), 7.23-7.41 (m, 7H), 5.95 (d, *J* = 6 Hz, 1H), 4.55 (d, *J* = 6 Hz, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.1, 139.2, 134.0, 133.7, 129.3, 129.2, 128.8, 128.7, 127.9, 76.4; IR (Neat) 3418, 1679, 1261, 1068 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd for $C_{14}H_{12}O_2Na_1$, 235.0735; found, 235.0727. The enantiomeric excess (%*ee*) was determined to be 98% by HPLC using Daicel ChiralPAK AS-H column (15% *i*-PrOH / hexanes, 1 mL/min, 220 nm): t_R (major, 13.250 min), t_R (minor, 8.500 min).



benzil[5]

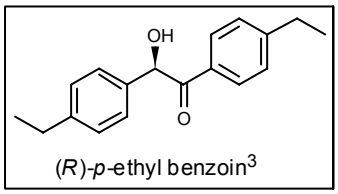
R_f 0.43; (hexanes : ethyl acetate, 90:10 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96-8.01 (m, 4H), 7.63-7.69 (m, 2H), 7.49-7.55 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ194.7, 135.0, 133.3, 130.1, 129.2; IR (Neat) 3064,1656 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd for $C_{14}H_{10}O_2Na_1$, 233.0578; found, 233.0585.

Spectral data for all other optically active benzoins



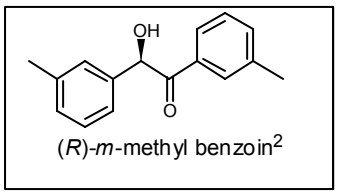
(*R*)-*p*-methyl benzoin[2]

R_f 0.13; (hexanes : ethyl acetate, 90:10 v/v): $[\alpha]^{D}_{25}$ = – 141.2 (*c* = 1 in methanol); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (d, *J* = 8.0 Hz, 2H), 7.16–7.23 (m, 4H), 7.12 (d, *J* = 8 Hz, 2H), 5.89 (d, *J* = 6.0 Hz, 1H), 4.54 (d, *J* = 6.4 Hz, 1H), 2.35 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.7, 145.0, 138.5, 136.5, 131.1, 129.9, 129.5, 129.4, 127.8, 75.9, 21.8, 21.3; IR (Neat) 3473, 2917, 1674 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd for $C_{16}H_{16}O_2Na_1$, 263.1048; found, 263.1045. The enantiomeric excess (%*ee*) was determined to be 90% by HPLC using Daicel ChiralPAK AS-H column (15% *i*-PrOH/ hexanes, 1 mL/min, 220 nm): t_R (major, 12.158 min), t_R (minor, 8.275 min).



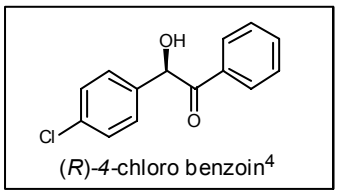
(*R*)-*p*-ethyl benzoin[3]

R_f 0.22; (hexanes: ethyl acetate, 90:10 v/v): $[\alpha]^{D}_{25}$ = – 76.6 (*c* = 1 in methanol); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.4 Hz, 2H), 7.19–7.27 (m, 4H), 7.15 (d, *J* = 8 Hz, 2H), 5.90 (d, *J* = 6 Hz, 1H), 4.55 (d, *J* = 6 Hz, 1H), 2.55-2.69 (m, 4H), 1.15-1.24 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.7, 151.1, 144.8, 136.7, 131.3, 129.6, 128.8, 128.3, 127.8, 75.9, 29.1, 28.7, 15.5, 15.0; IR (Neat) 3462, 2965, 2873, 1672, 1252, 1041, 973 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd for $C_{18}H_{20}O_2Na_1$, 291.1361; found, 291.1364. The enantiomeric excess (%*ee*) was determined to be 94% by HPLC using Daicel ChiralPAK AS-H column (15% *i*-PrOH/ hexanes, 1 mL/min, 220 nm): t_R (major, 8.333 min), t_R (minor, 6.375 min).



(*R*)-*m*-methyl benzoin[2]

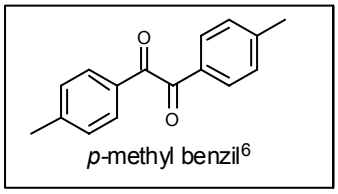
R_f 0.23; (hexanes : ethyl acetate, 90:10 v/v): $[\alpha]^{D}_{25}$ = – 127.8 (*c* = 1.0 in methanol); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (s, 1H), 7.69 (d, *J* = 7.6 Hz, 1H), 7.33 (d, *J* = 7.6 Hz, 1H), 7.17-7.27 (m, 2H), 7.11-7.15 (m, 2H), 7.07 (d, *J* = 7.6 Hz, 1H), 5.90 (d, *J* = 6 Hz, 1H), 4.52 (d, *J* = 6.4 Hz, 1H), 2.35 (s, 3H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.2, 139.1, 138.9, 138.6, 134.7, 133.6, 129.6, 129.3, 129.0, 128.5, 128.3, 126.5, 125.0, 76.2, 21.4, 21.3; IR (Neat) 3452, 2920, 1676, 1276 cm^{-1}; HRMS (*m*/*z*): $[MNa]^+$ calcd for $C_{16}H_{16}O_2Na_1$, 263.1048; found, 263.1043. The enantiomeric excess (%*ee*) was determined to be 95% by HPLC using Daicel ChiralPAK AS-H column (15% *i*-PrOH/ hexanes, 1 mL/min, 220 nm): t_R (major, 10.233 min), t_R (minor, 7.075 min).



(*R*)-*4*-chloro benzoin[4]

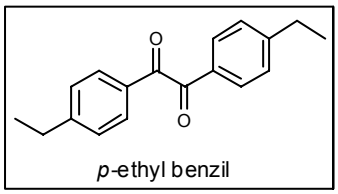
R_f 0.14; (hexanes : ethyl acetate, 90:10 v/v): $[\alpha]^{D}_{25} = -75.0$ (c = 1 in methanol); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.87 (m, 2H), 7.27-7.39 (m, 7H), 5.90 (d, J = 6.4 Hz, 1H), 4.48 (d, J = 6.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.0, 140.6, 138.8, 131.9, 130.6, 129.4, 129.2, 128.9, 127.9, 76.5; IR (Neat) 3461, 3063, 1678 cm^{-1}; HRMS (m/z): $[M]^+$ calcd for $C_{14}H_{11}ClO_2Na_1$, 269.0345; found, 269.0347. The enantiomeric excess (%*ee*) was determined to be 94% by HPLC using Daicel ChiralPAK AS-H column (15% *i*-PrOH/ hexanes, 1 mL/min, 220 nm): t_R (major, 10.833 min), t_R (minor, 7.767 min).

Spectral data for all other benzils



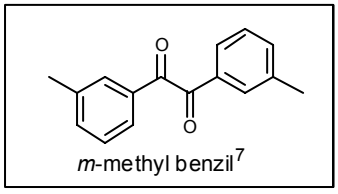
p-methyl benzil[6]

R_f 0.33; (hexanes : ethyl acetate, 90:10 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, J = 8.4 Hz, 4H), 7.30 (d, J = 8.4 Hz, 4H), 2.43 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.7, 146.2, 130.9, 130.2, 129.9, 22.1; IR (Neat) 2918, 1659 cm^{-1}; HRMS (m/z): $[MNa]^+$ calcd for $C_{16}H_{14}O_2Na_1$, 261.0891; found, 261.0892.



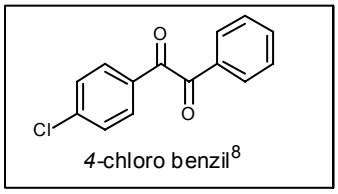
p-ethyl benzil

R_f 0.45; (hexanes : ethyl acetate, 90:10 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, J = 8.4 Hz, 4H), 7.32 (d, J = 8 Hz, 4H), 2.72 (q, J = 7.6 Hz, 4H), 1.26 (t, J = 7.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.7, 152.3, 131.0, 130.3, 128.7, 29.3, 15.2; IR (Neat) 2968, 2933, 2875, 1668 cm^{-1}; HRMS (m/z): $[M-H]^+$ calcd for $C_{18}H_{19}O_2$, 267.1385; found, 267.1388.



m-methyl benzil[7]

R_f 0.52; (hexanes : ethyl acetate, 90:10 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, J = 9.2 Hz, 4H), 7.46 (d, J = 7.2 Hz, 2H), 7.39 (t, J = 7.6 Hz, 2H), 2.40 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 195.0, 139.0, 135.7, 133.1, 130.2, 128.9, 127.2, 21.3; IR (Neat) 2922, 1667, 729 cm^{-1}; HRMS (m/z): $[M-H]^+$ calcd for $C_{16}H_{15}O_2$, 239.1072; found, 239.1070.



4-chloro benzil[8]

R_f 0.43; (hexanes : ethyl acetate, 90:10 v/v): ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91-7.99 (m, 4H), 7.65-7.71 (m, 1H), 7.48-7.56 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 194.0, 193.2, 141.7, 135.2, 133.0, 131.5, 131.3, 130.1, 129.6, 129.2; IR (Neat) 1666, 1210, 712 cm^{-1}; HRMS (*m*/*z*): $[M]^+$ calcd for $C_{14}H_9ClO_2Na_1$, 265.0189; found, 265.0187.

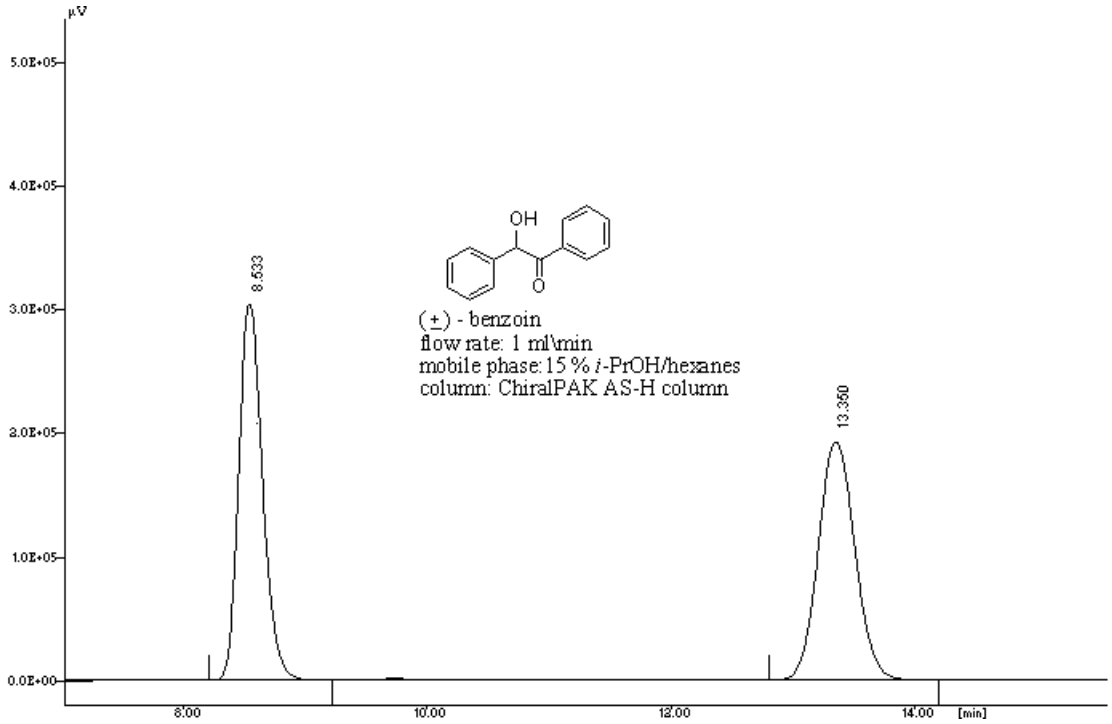
Calculation of selectivity (*s*) = Calculation of selectivity (k_{fast}/k_{slow}) was accomplished by using the method of kagan.[9]

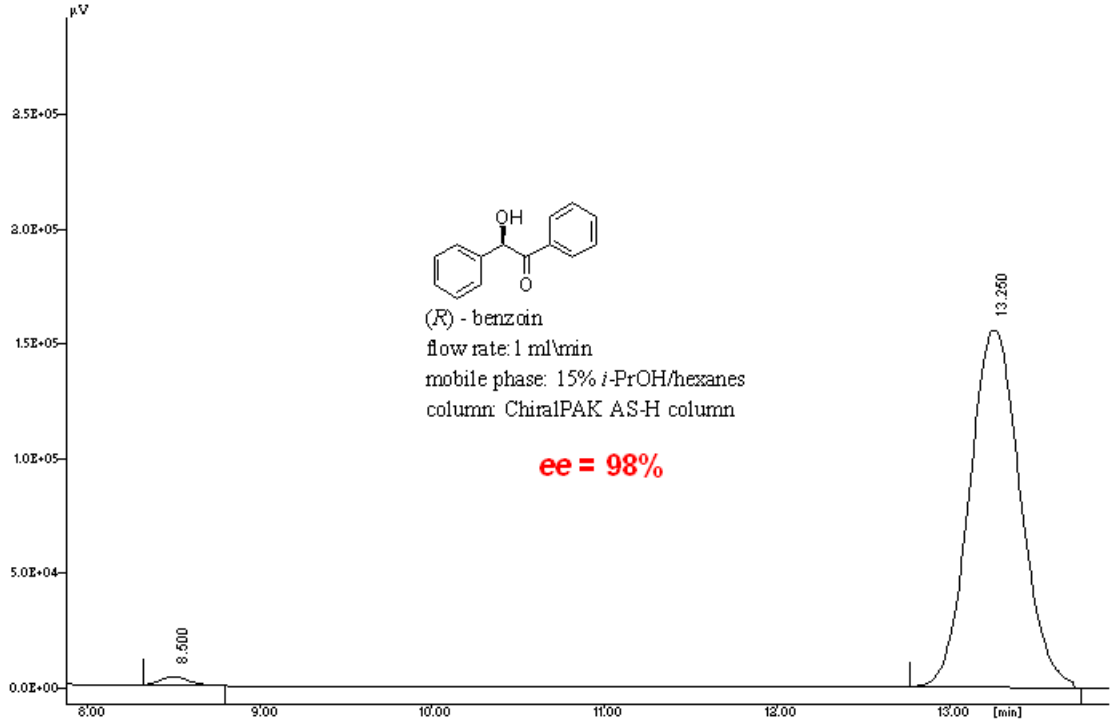
$$s = \frac{k_{fast}}{k_{slow}} = \frac{\ln [(1 - C)(1 - ee)]}{\ln [(1 - C)(1 + ee)]}$$

s = selectivity factor
C = conversion
ee = enantiomeric excess of recovered alcohol

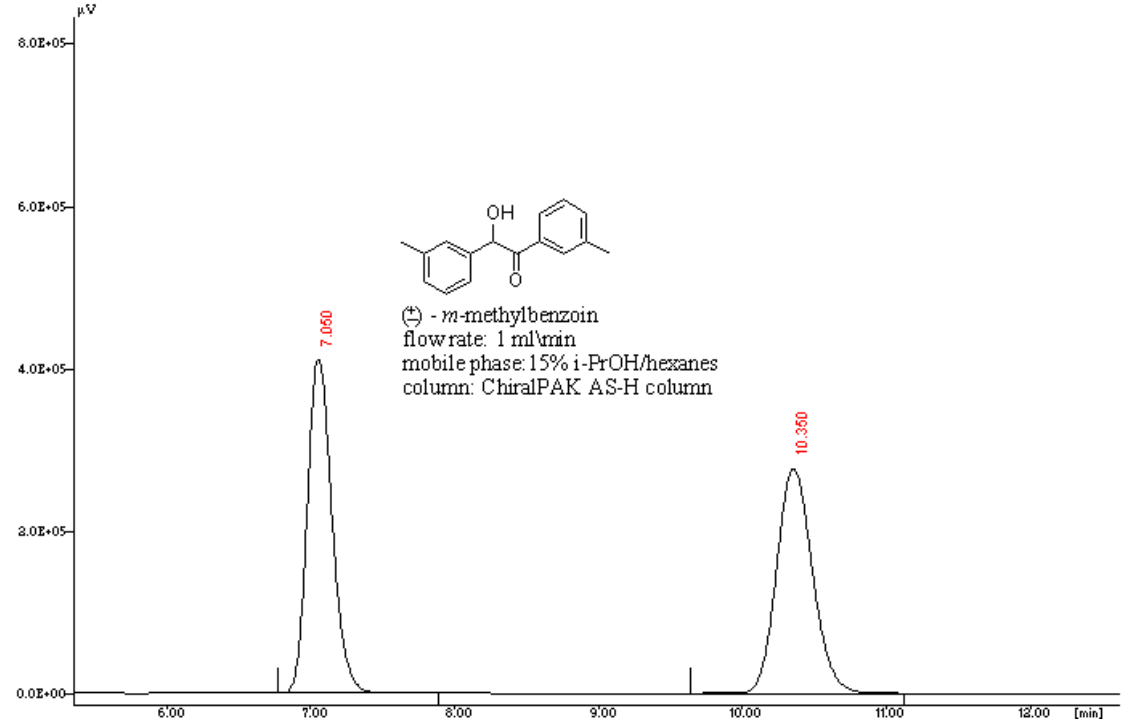
HPLC spectras for all benzoins

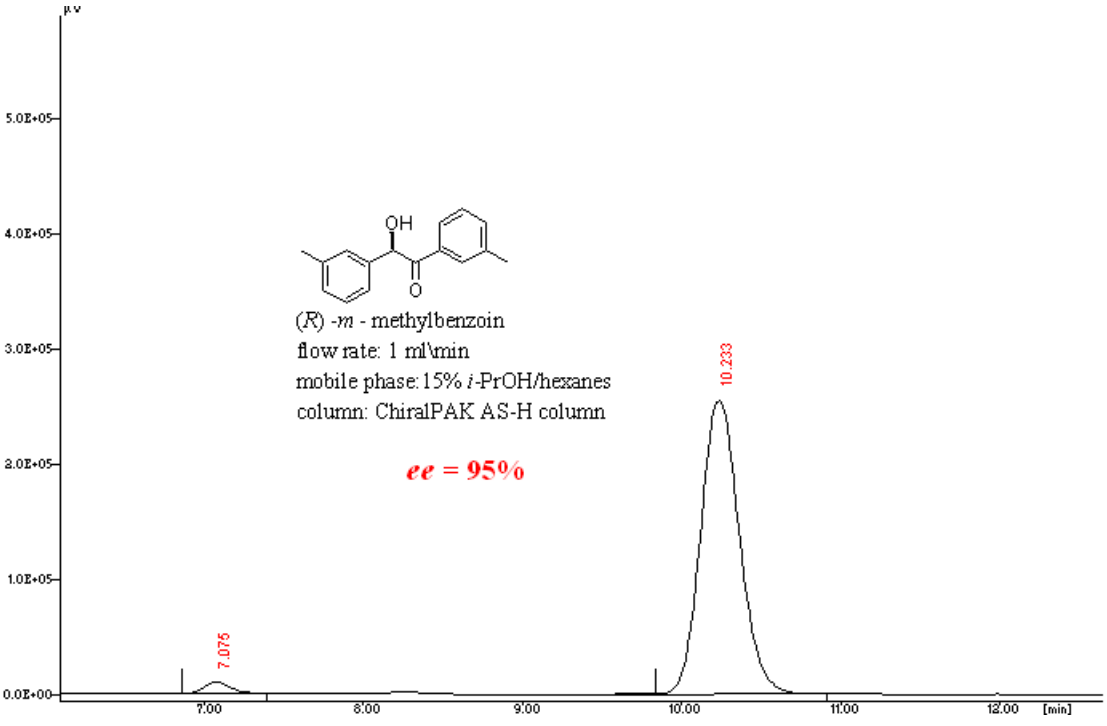
Benzoin



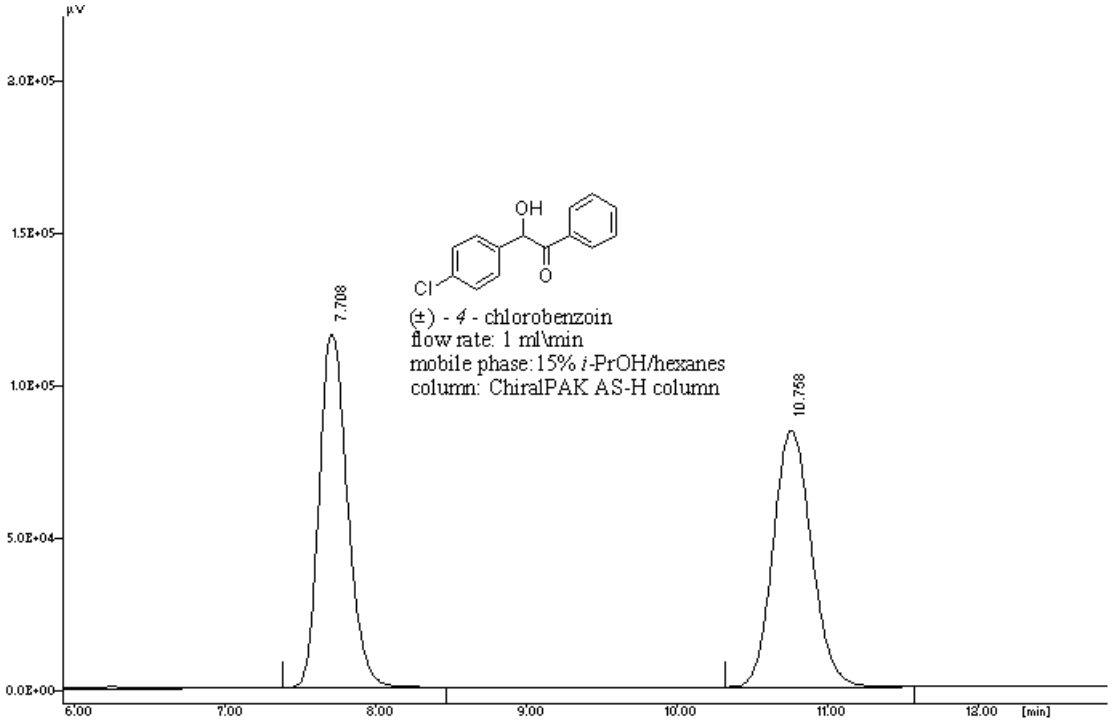


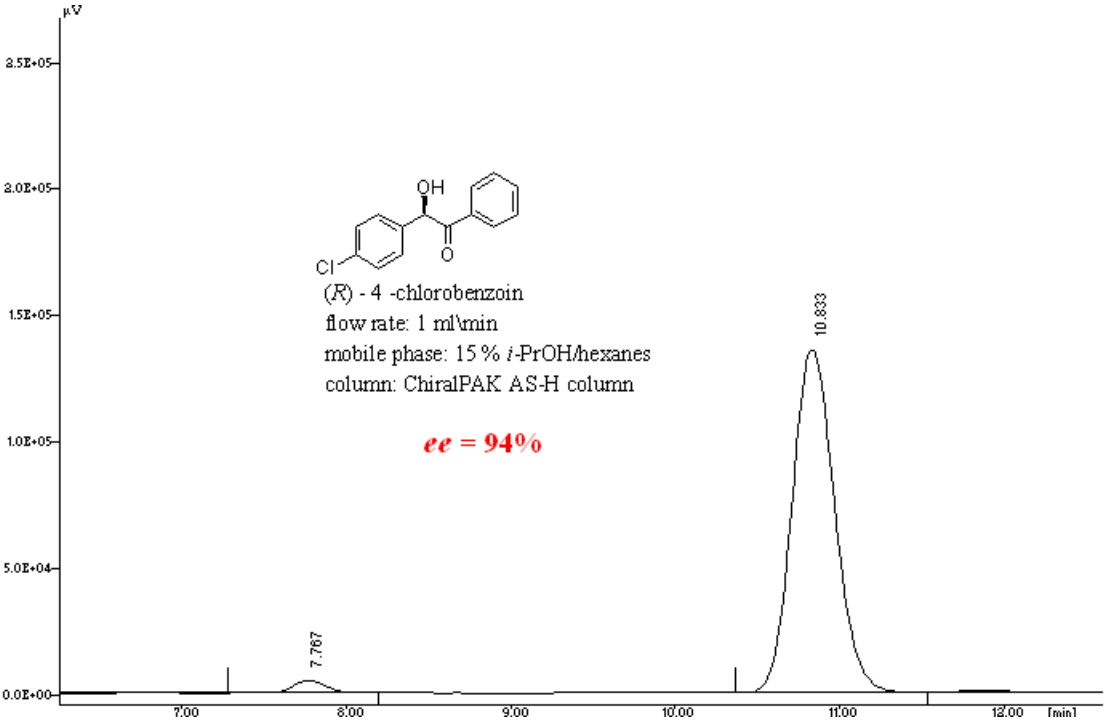
m-methylbenzoin



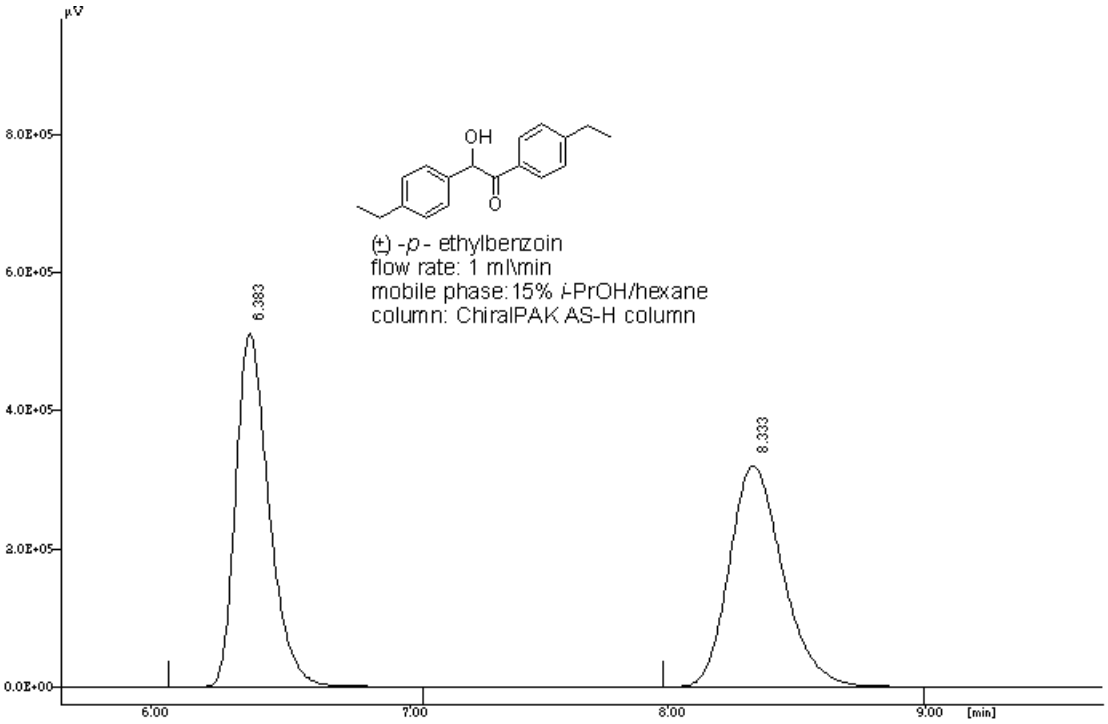


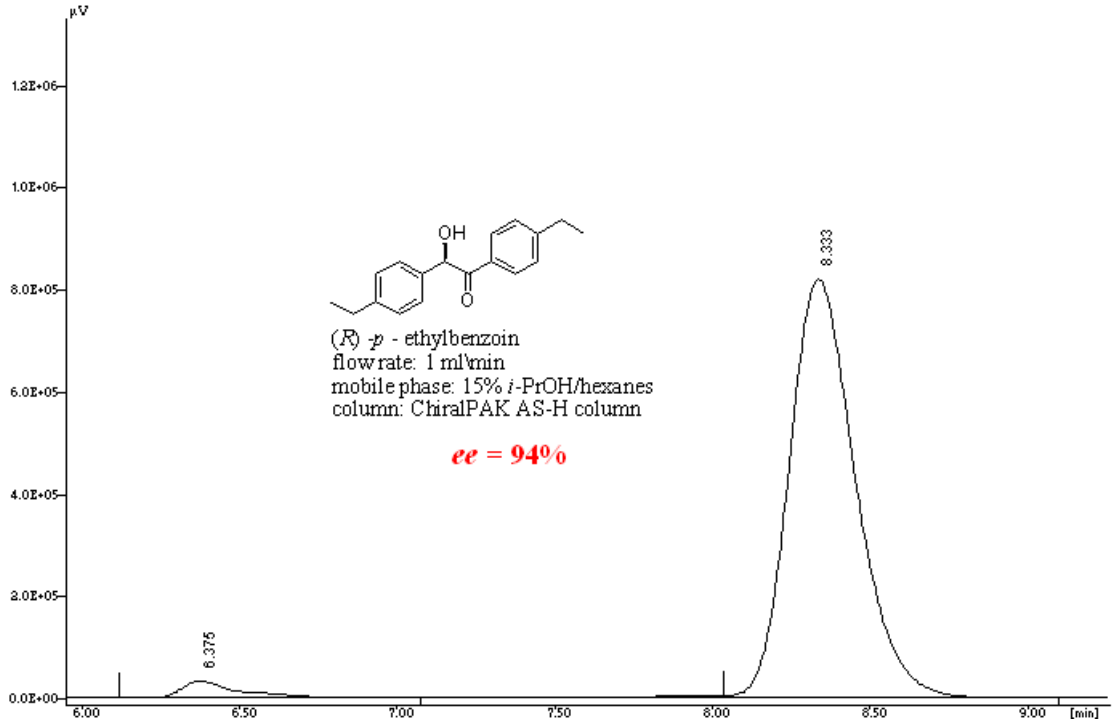
4- Chlorobenzoin



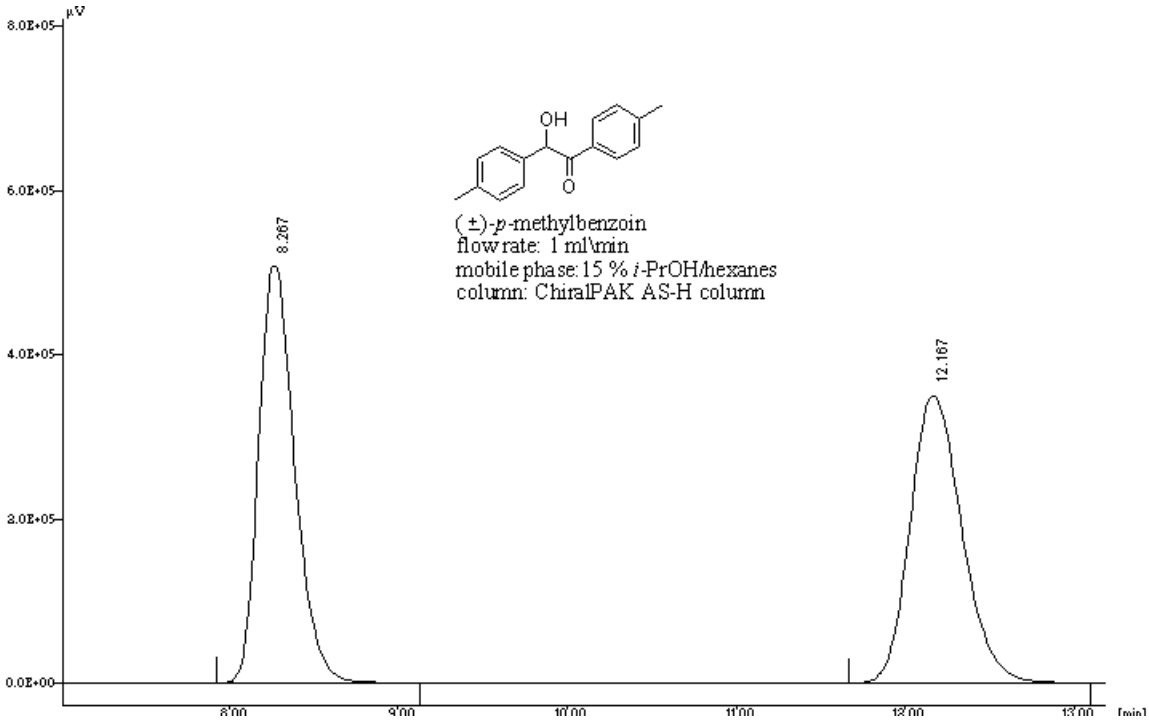


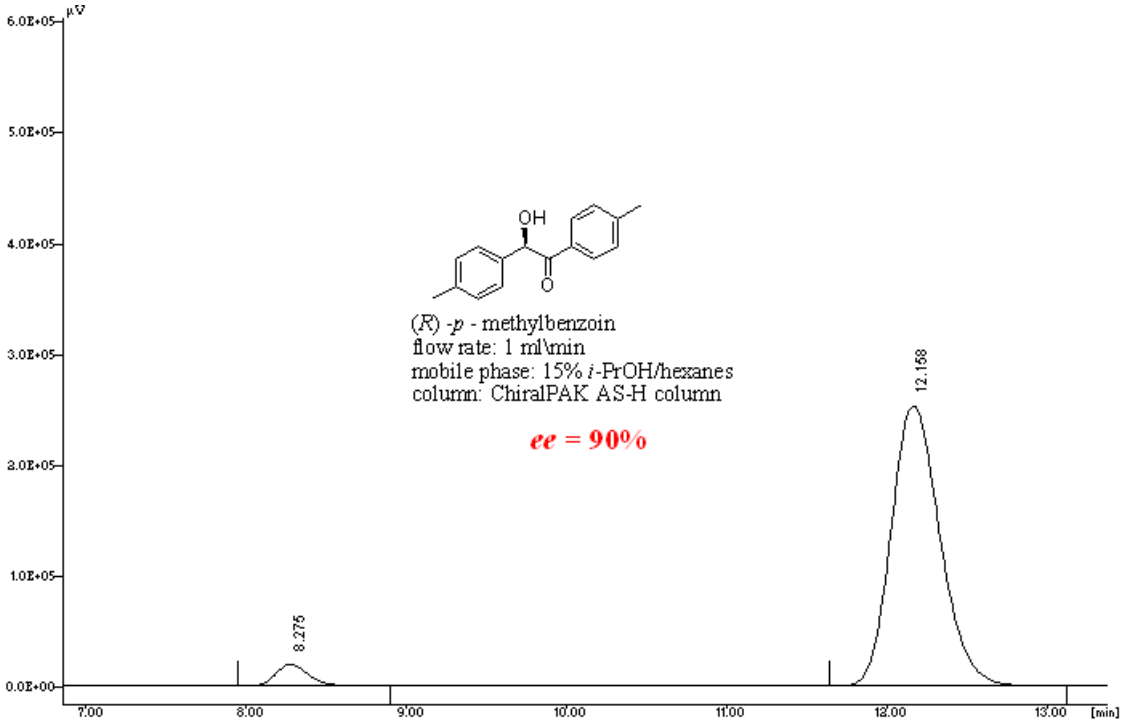
p-ethylbenzoin



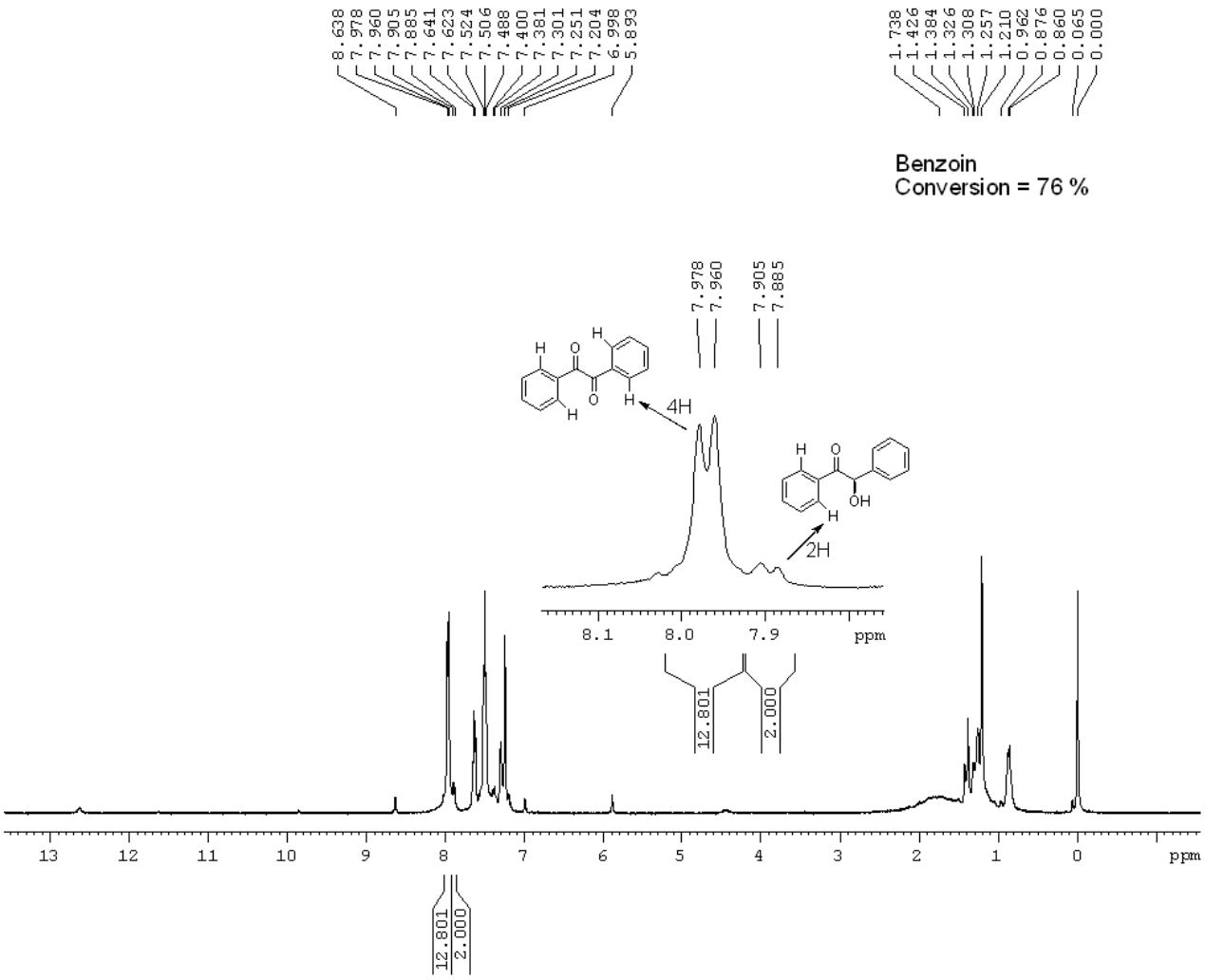


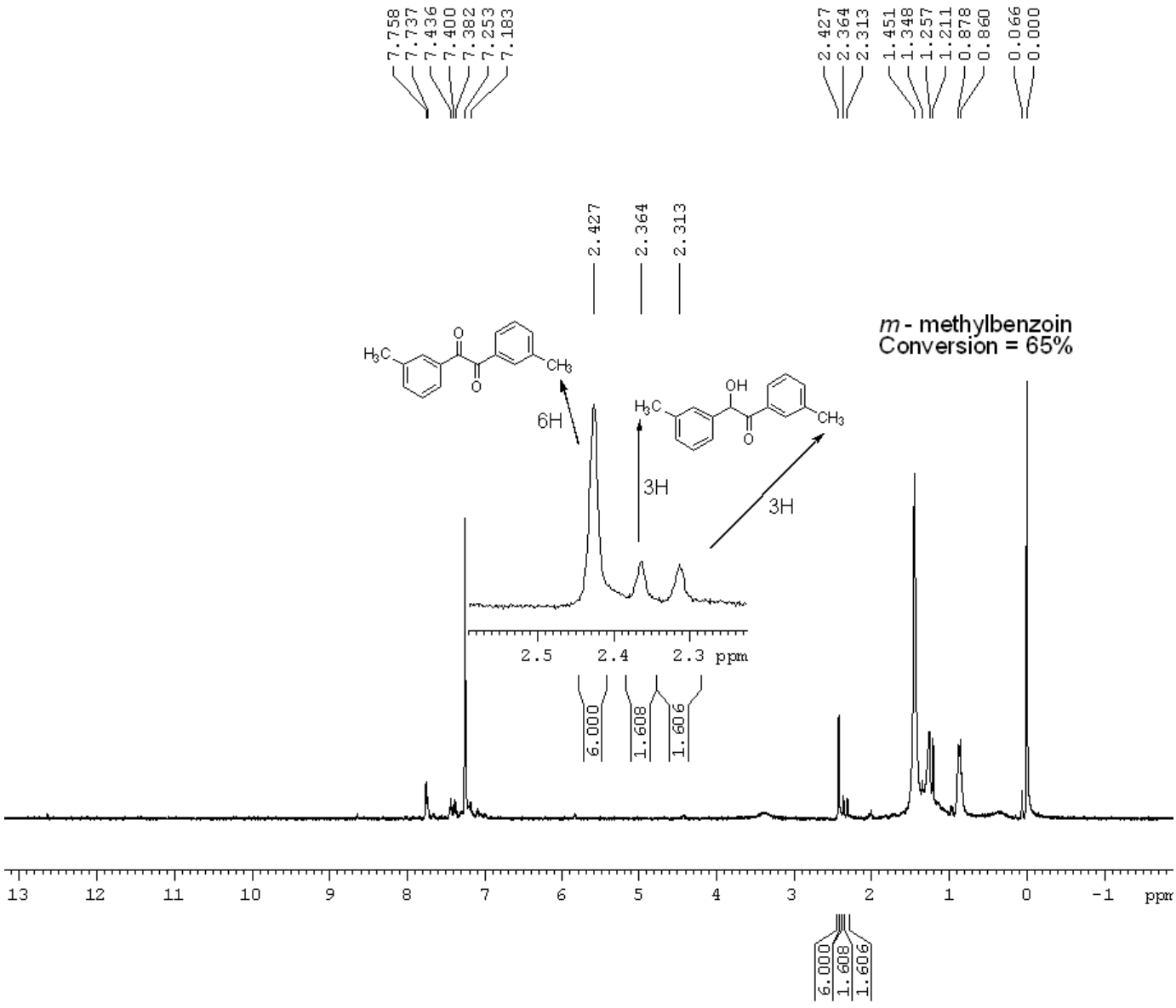
p-methylbenzoin





Calculation of conversion C (%) using ^{1}H NMR

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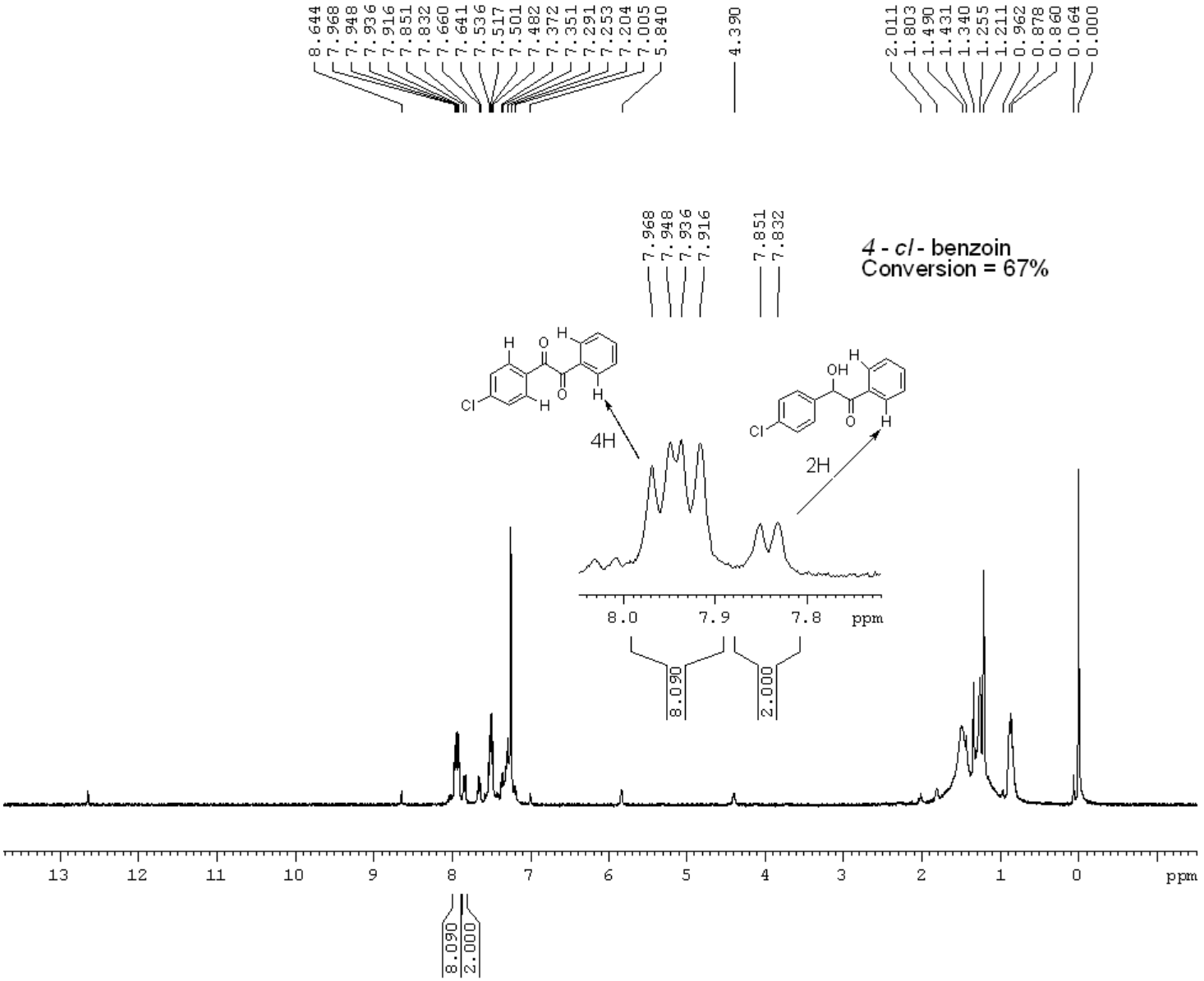
7.737
7.436
7.400
7.382
7.253
7.183
2.427
2.364
2.313
1.451
1.348
1.257
1.211
0.878
0.860
0.066
0.000
m - methylbenzoin
Conversion = 65%
6H
3H
3H
OH
2.5
2.4
2.3 ppm
6.000
1.608
1.606
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm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7.936
7.916
7.851
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7.660
7.641
7.536
7.517
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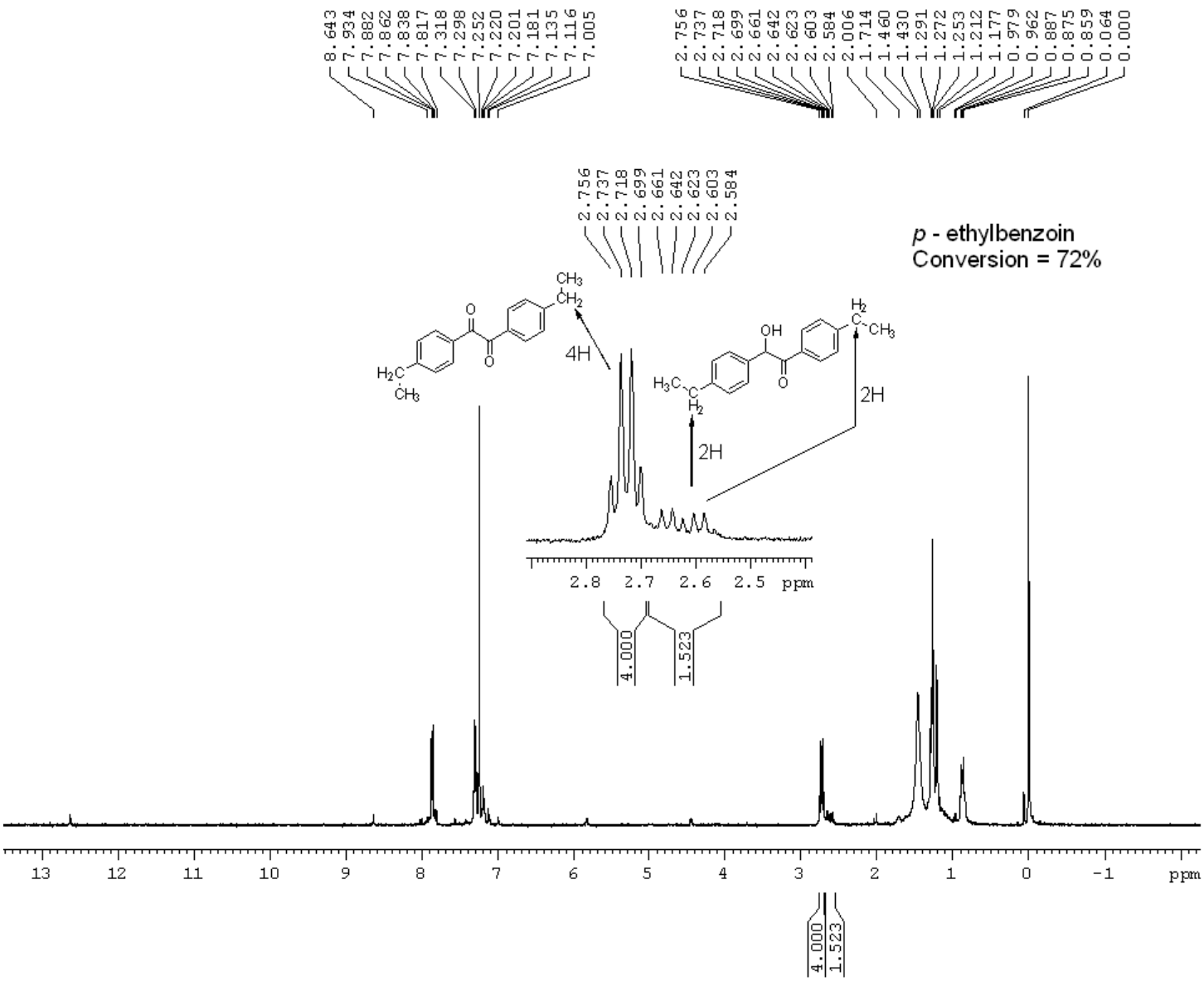
7.482
7.372
7.351
7.291
7.253
7.204
7.005
5.840
4.390
2.011
1.803
1.490
1.431
1.340
1.255
1.211
0.962
0.878
0.860
0.064
0.000
4 - cl - benzoin
Conversion = 67%
4H
2H
8.0
7.9
7.8
ppm
8.090
2.000
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm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7.862
7.838
7.817
7.318
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7.220
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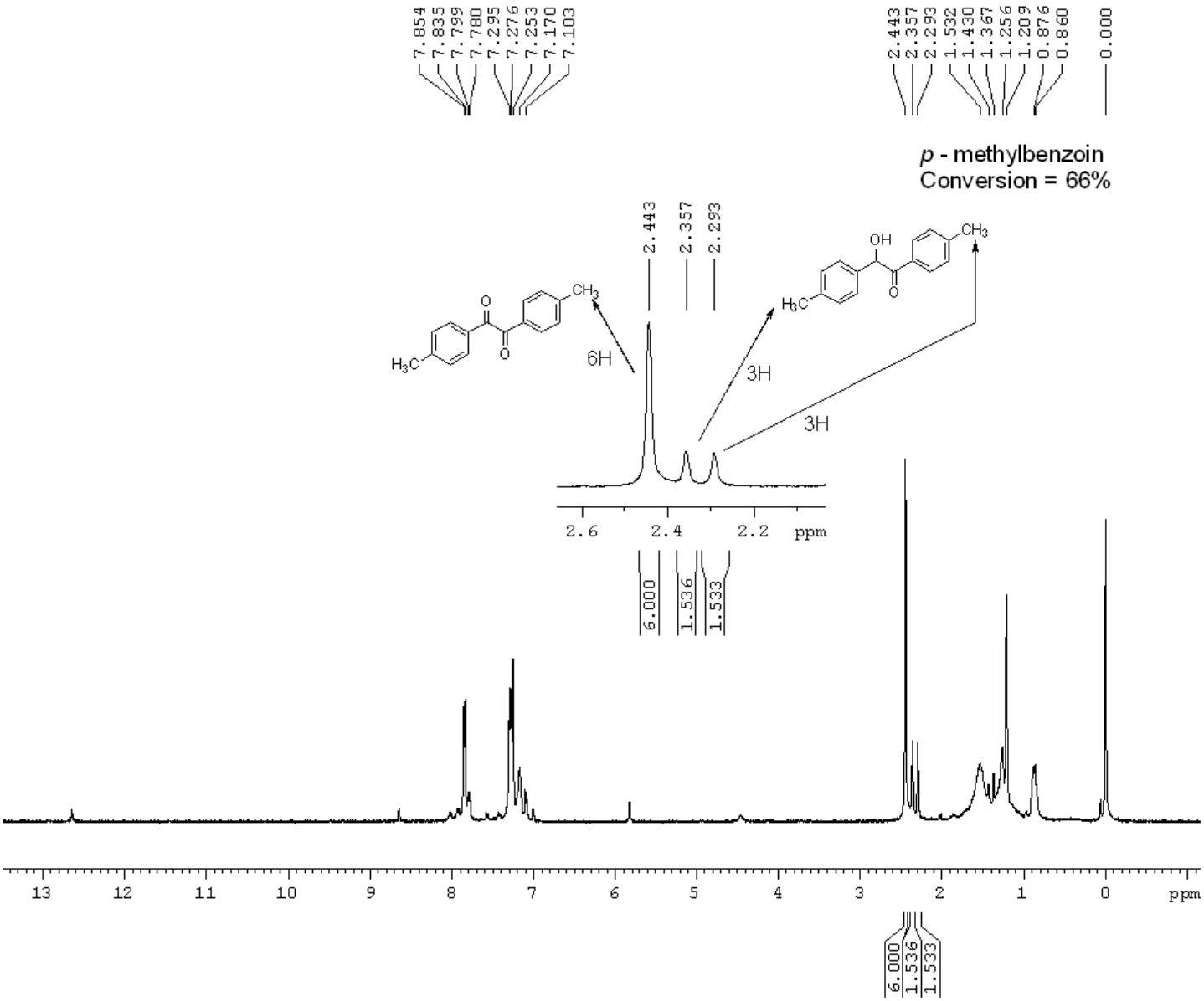
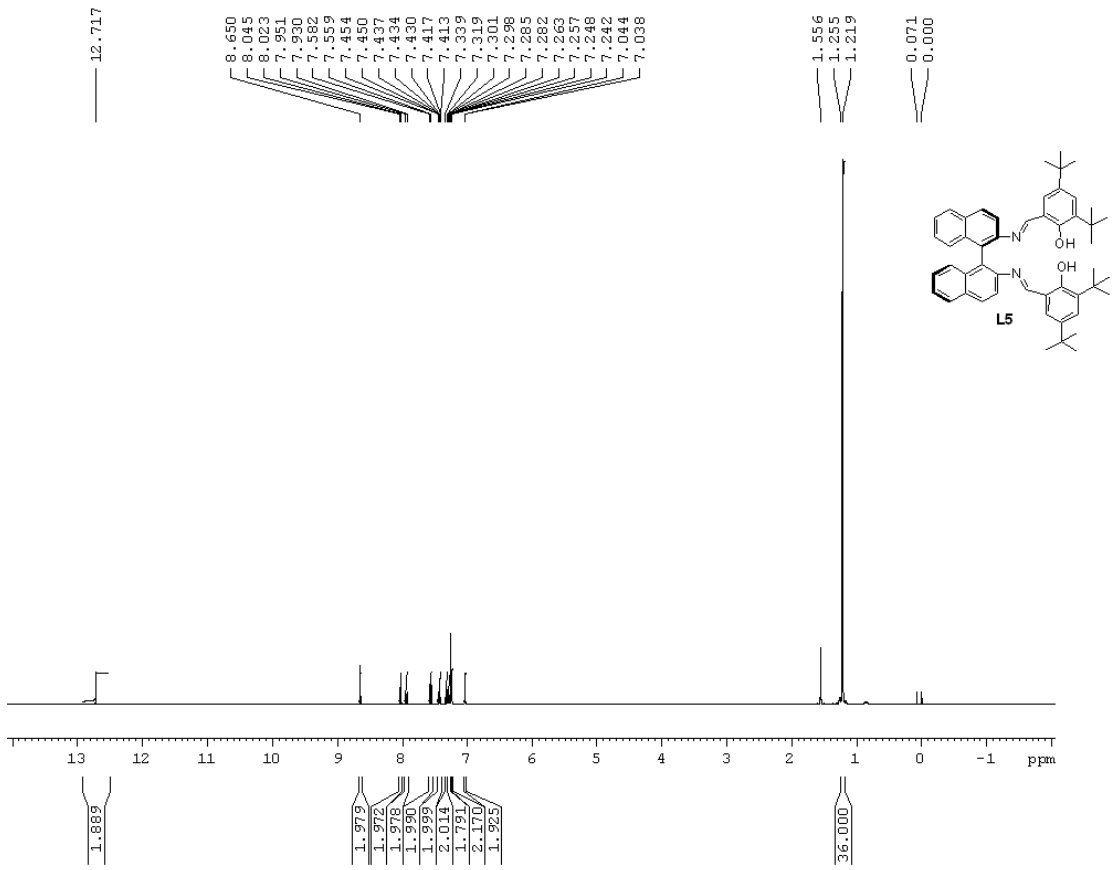
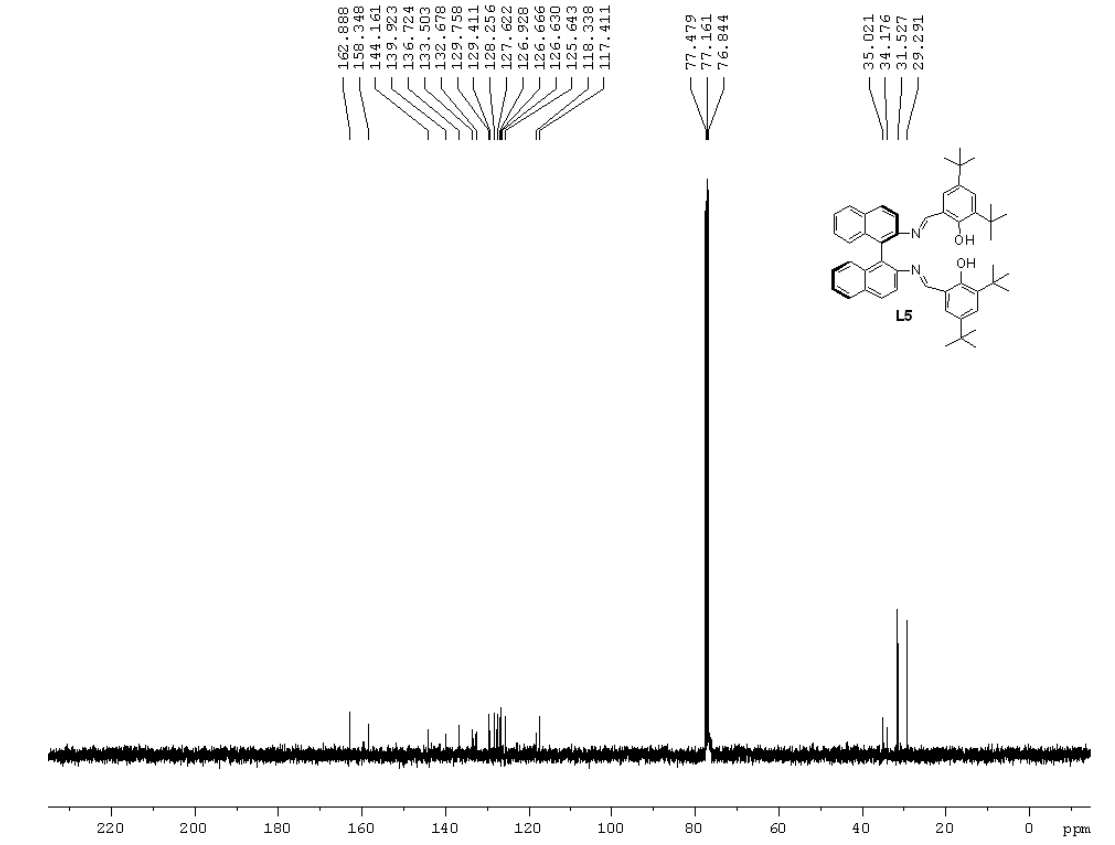
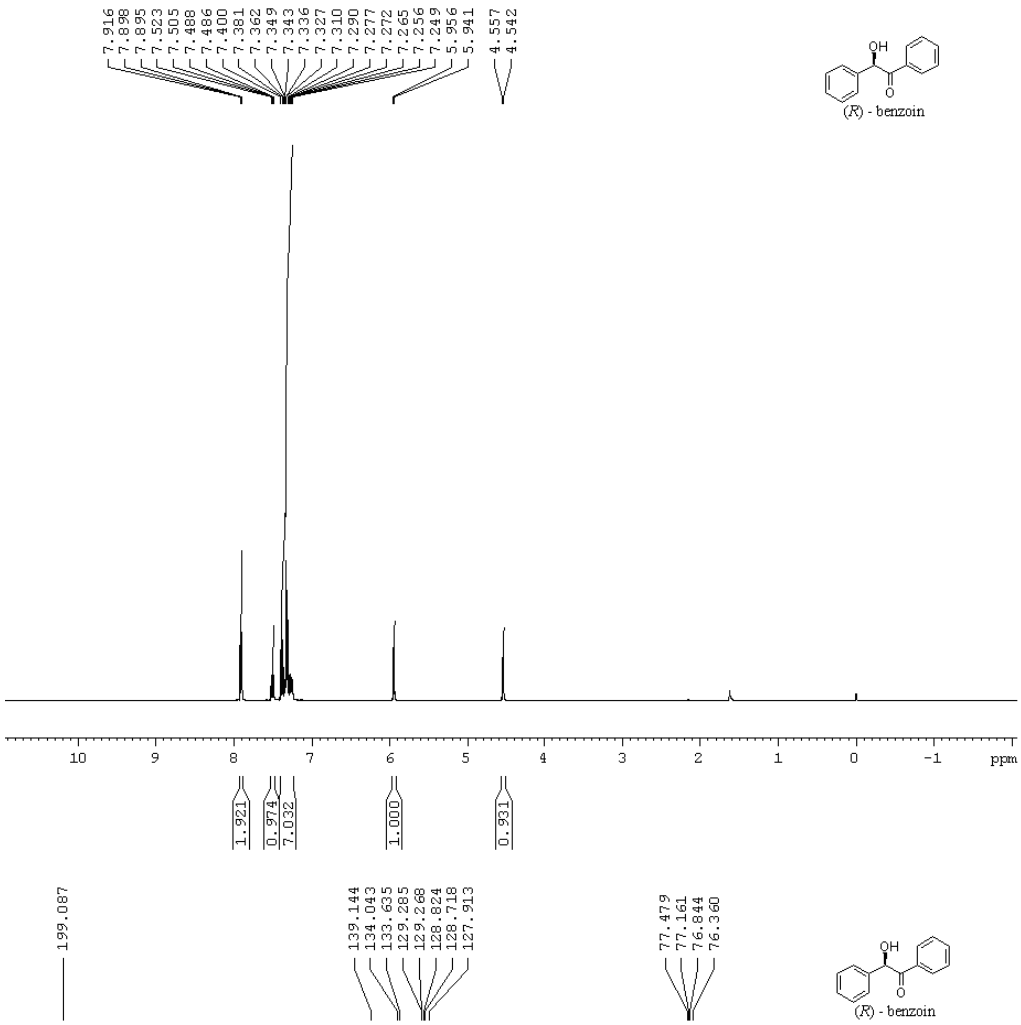
7.116
7.005
2.756
2.737
2.718
2.699
2.661
2.642
2.623
2.603
2.584
2.006
1.714
1.460
1.430
1.291
1.272
1.253
1.212
1.177
0.979
0.962
0.887
0.875
0.859
0.064
0.000
p - ethylbenzoin
Conversion = 72%
4H
2H
2H
2.8 2.7 2.6 2.5 ppm
4.000
1.523
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
4.000
1.523

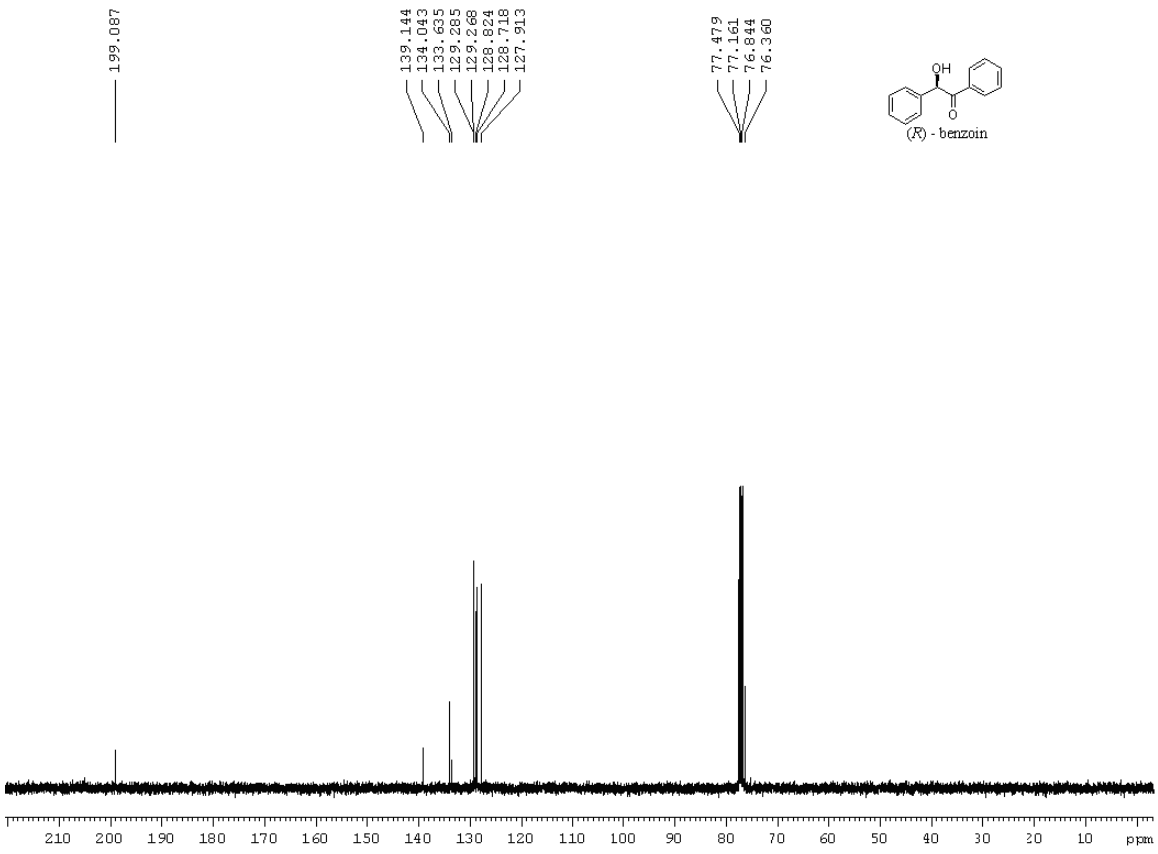


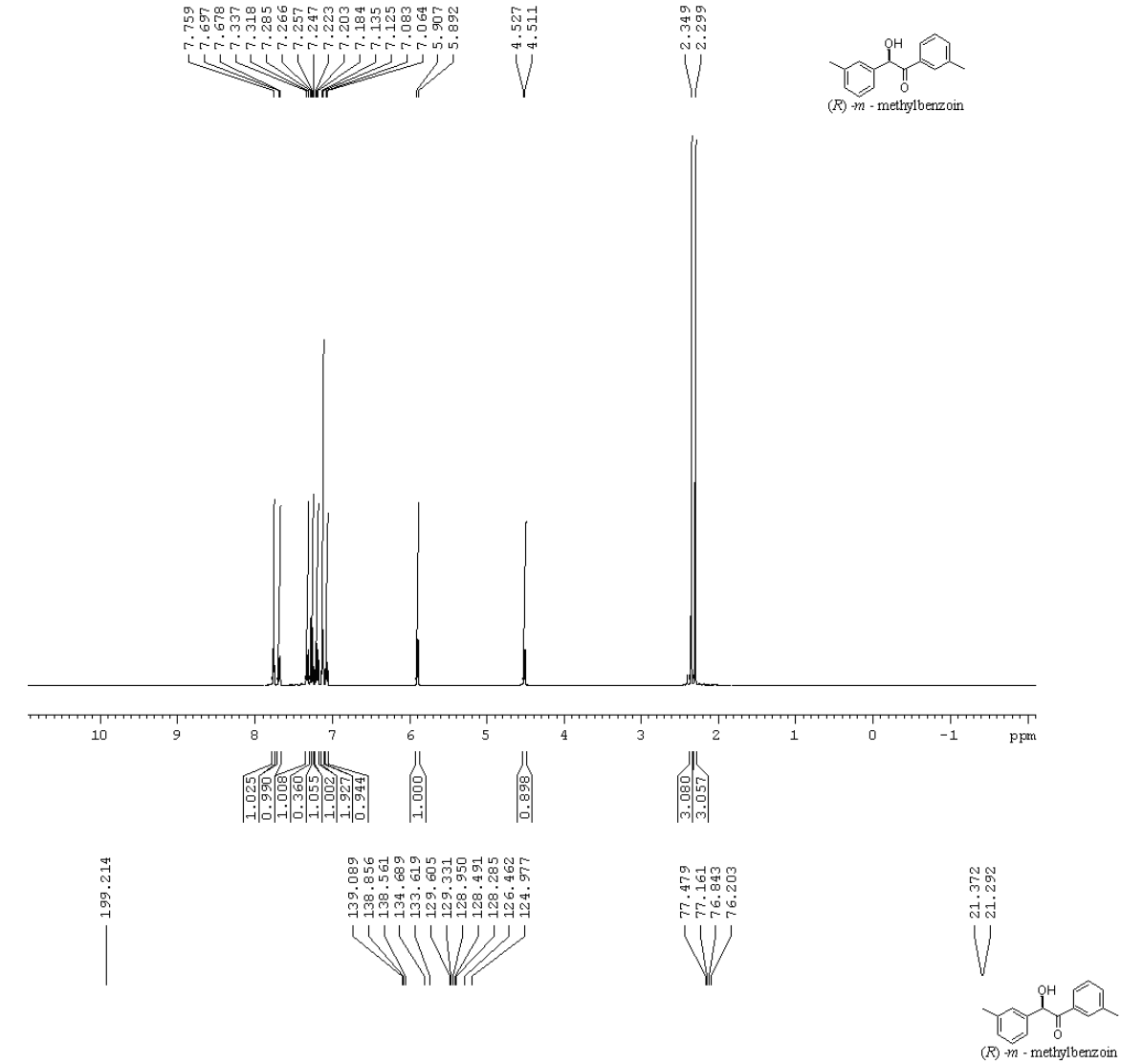
^{1}H and ^{13}C spectra for all compounds

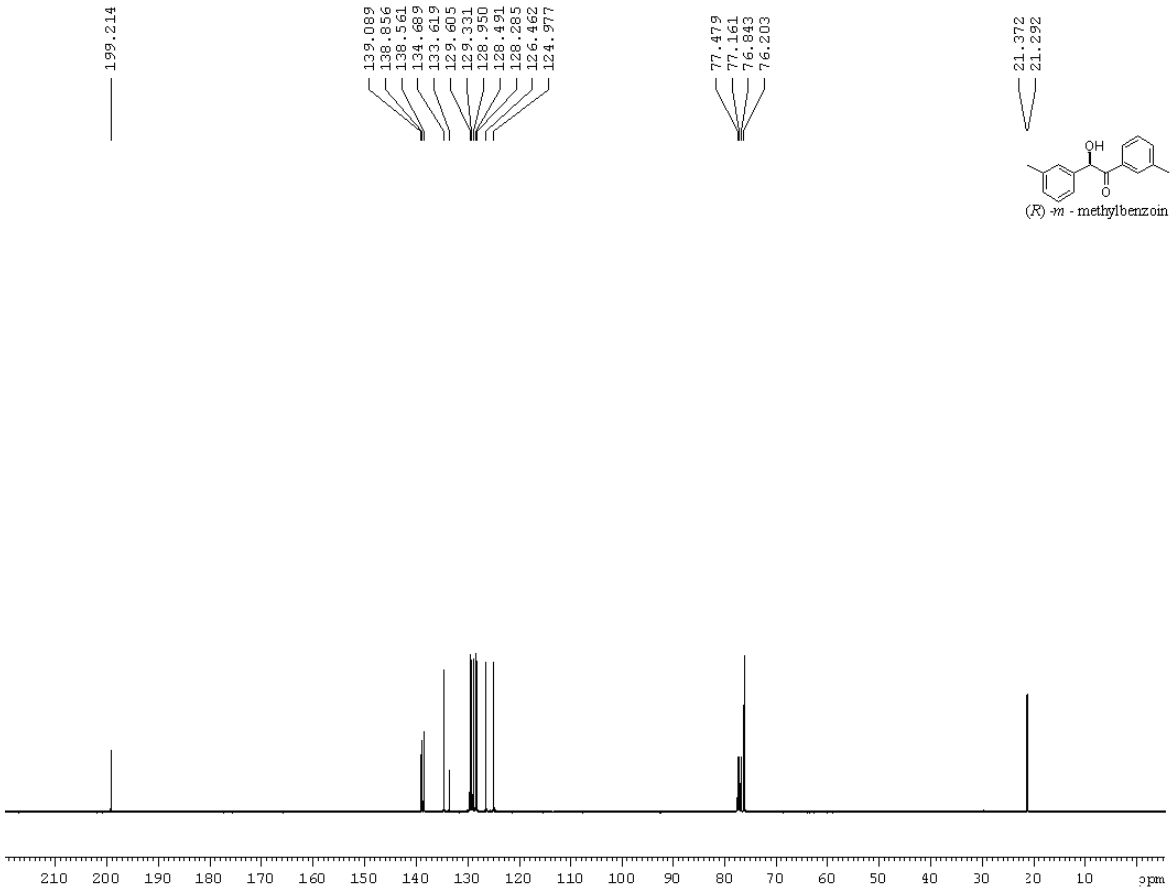

12.717
8.650
8.045
8.023
7.951
7.930
7.582
7.559
7.454
7.450
7.437
7.434
7.430
7.417
7.413
7.339
7.319
7.301
7.298
7.285
7.282
7.263
7.257
7.248
7.242
7.044
7.038
1.556
1.255
1.219
0.071
0.000
OH
OH
L5
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.889
1.979
1.972
1.978
1.990
1.999
2.014
1.791
2.170
1.925
36.000

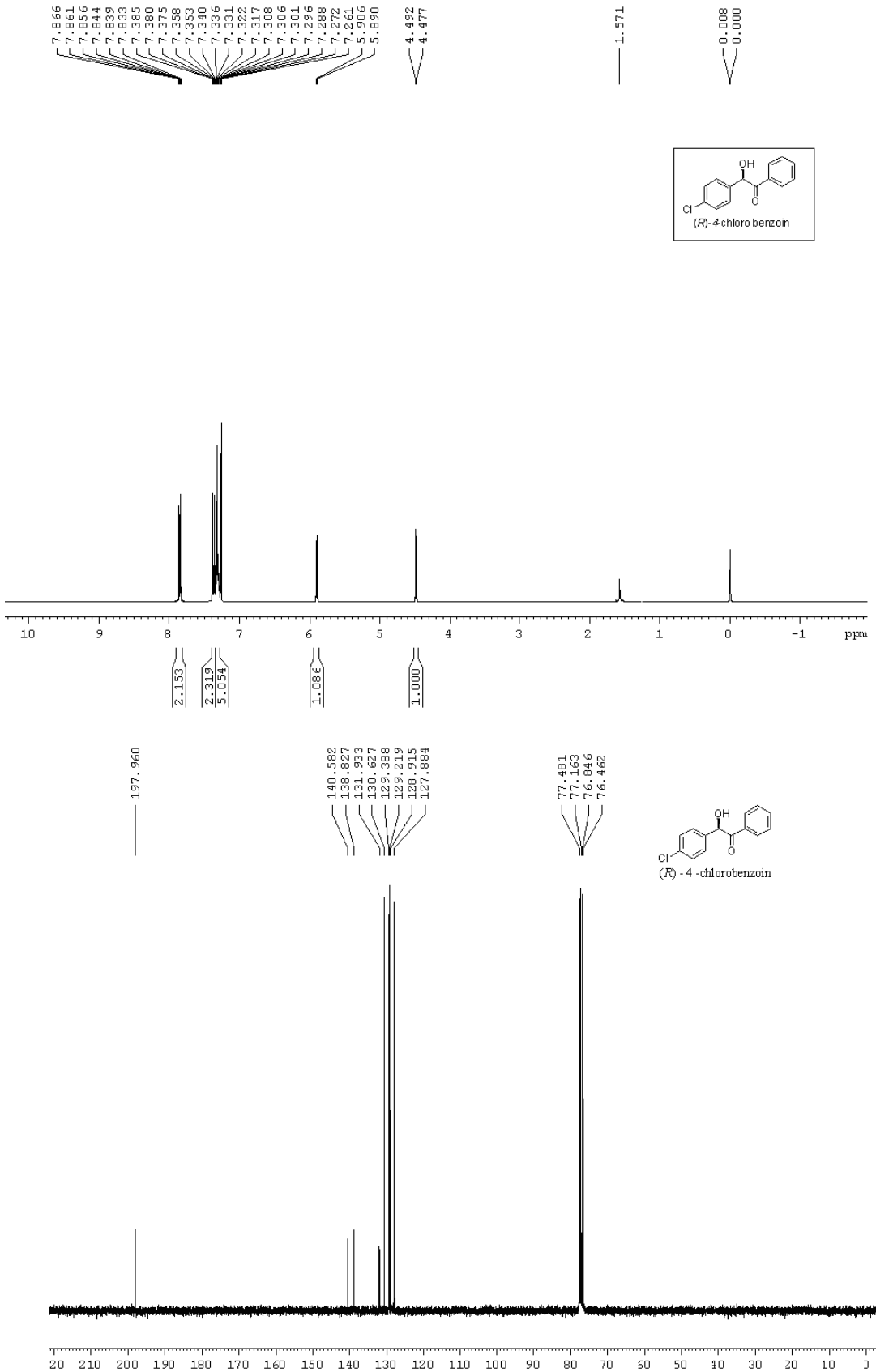

162.888
158.348
144.161
139.923
136.724
133.503
132.678
129.758
129.411
128.256
127.622
126.928
126.666
126.630
125.643
118.338
117.411
77.479
77.161
76.844
35.021
34.176
31.527
29.291
OH
OH
L5
220
200
180
160
140
120
100
80
60
40
20
0
ppm

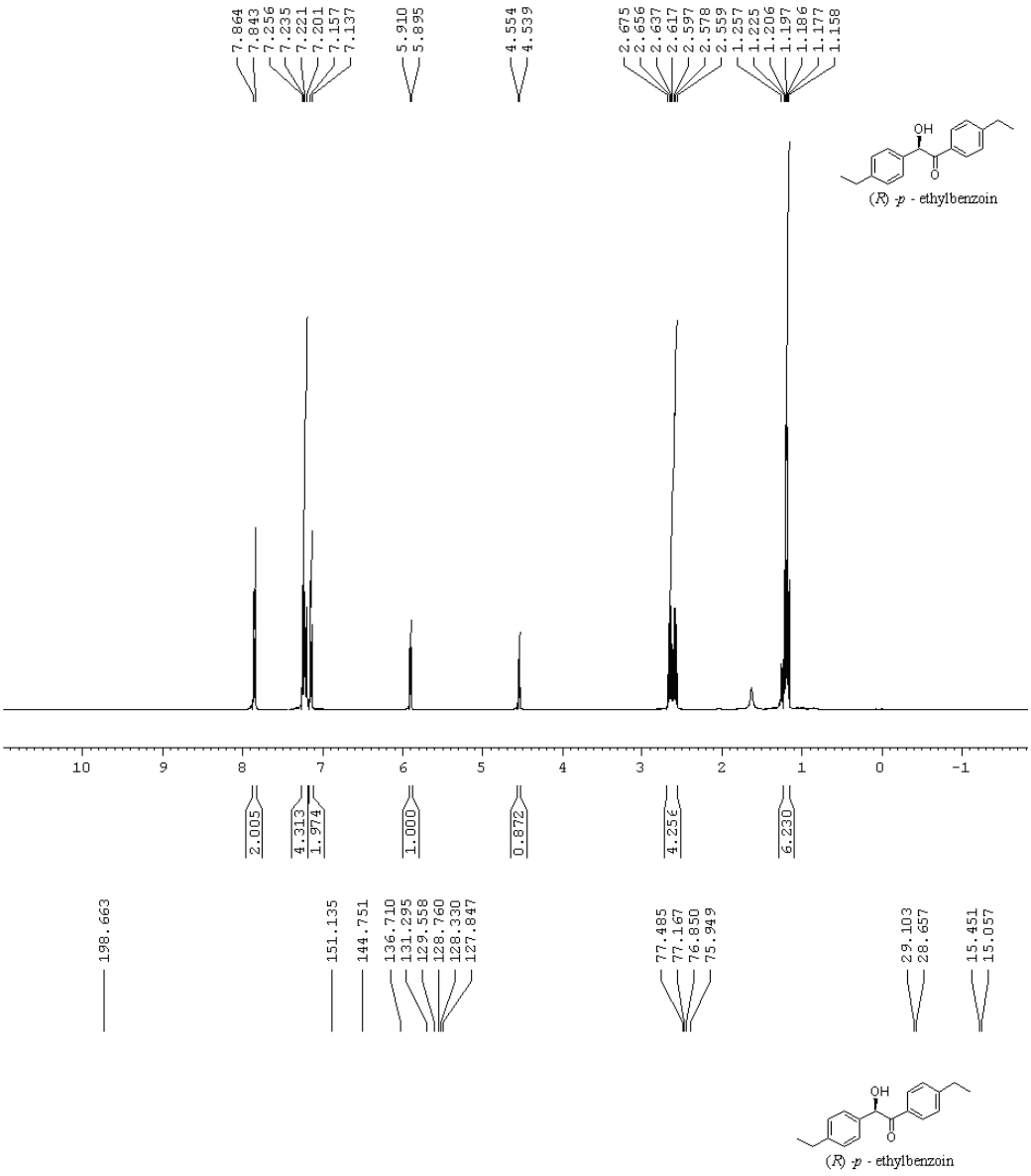

7.916
7.898
7.895
7.523
7.505
7.488
7.486
7.400
7.381
7.362
7.349
7.343
7.336
7.327
7.310
7.290
7.277
7.272
7.265
7.256
7.249
5.956
5.941
4.557
4.542
OH
O
(R) - benzoin
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.921
0.974
7.032
1.000
0.931

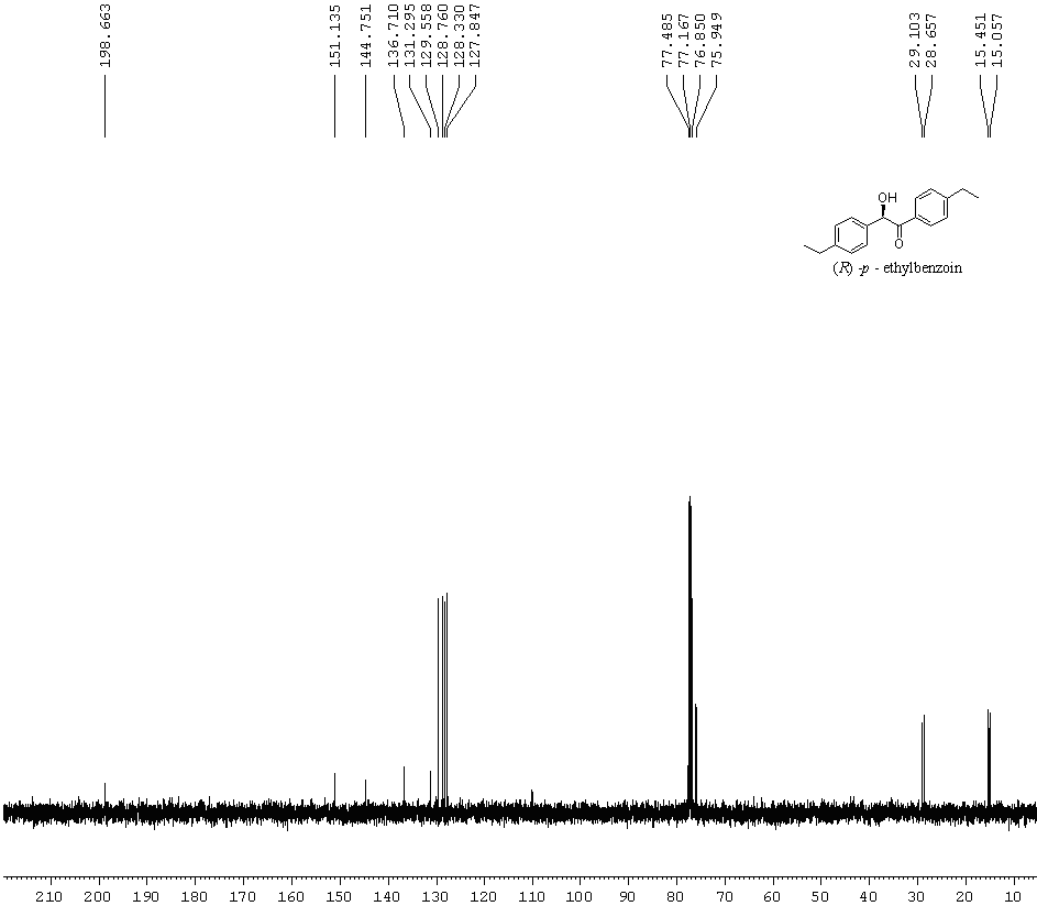

199.087
139.144
134.043
133.635
129.285
129.268
128.824
128.718
127.913
77.479
77.161
76.844
76.360
OH
O
(R) - benzoin
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

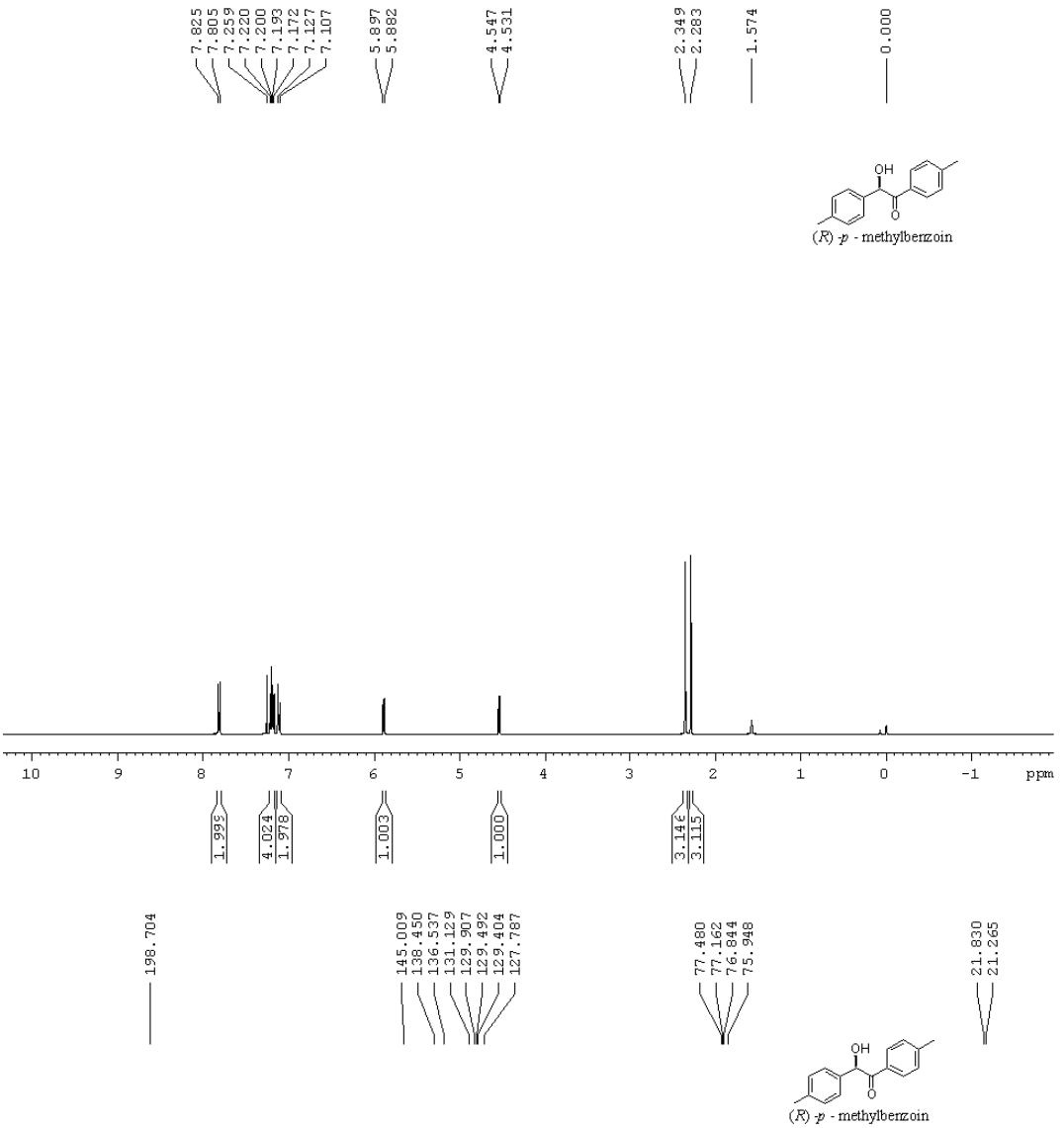

7.759
7.697
7.678
7.337
7.318
7.285
7.266
7.257
7.247
7.223
7.203
7.184
7.135
7.125
7.083
7.064
5.907
5.892
4.527
4.511
2.349
2.299
OH
O
(R) -m - methylbenzoin
ppm
1.025
0.990
1.008
0.360
1.055
1.002
1.927
0.944
1.000
0.898
3.060
3.057

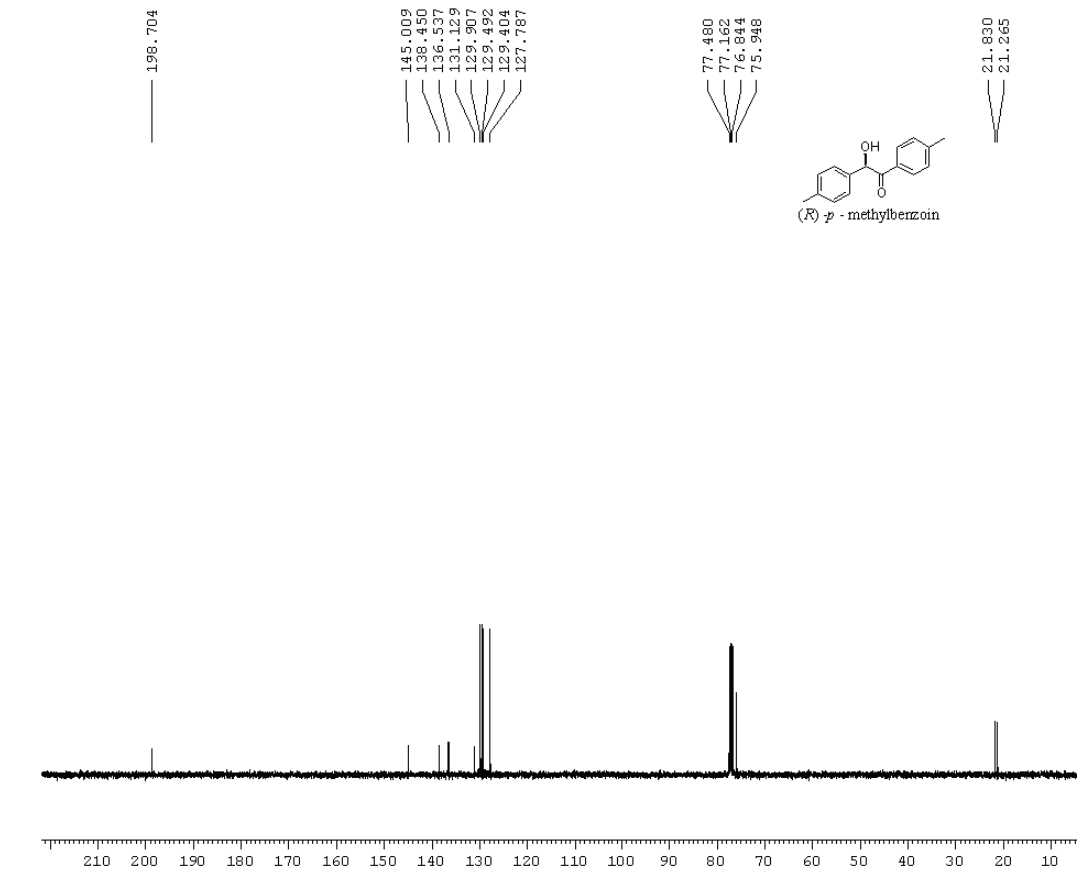

199.214
139.089
138.856
138.561
134.689
133.619
129.605
129.331
128.950
128.491
128.285
126.462
124.977
77.479
77.161
76.843
76.203
21.372
21.292
OH
O
(R) -m - methylbenzoin
ppm

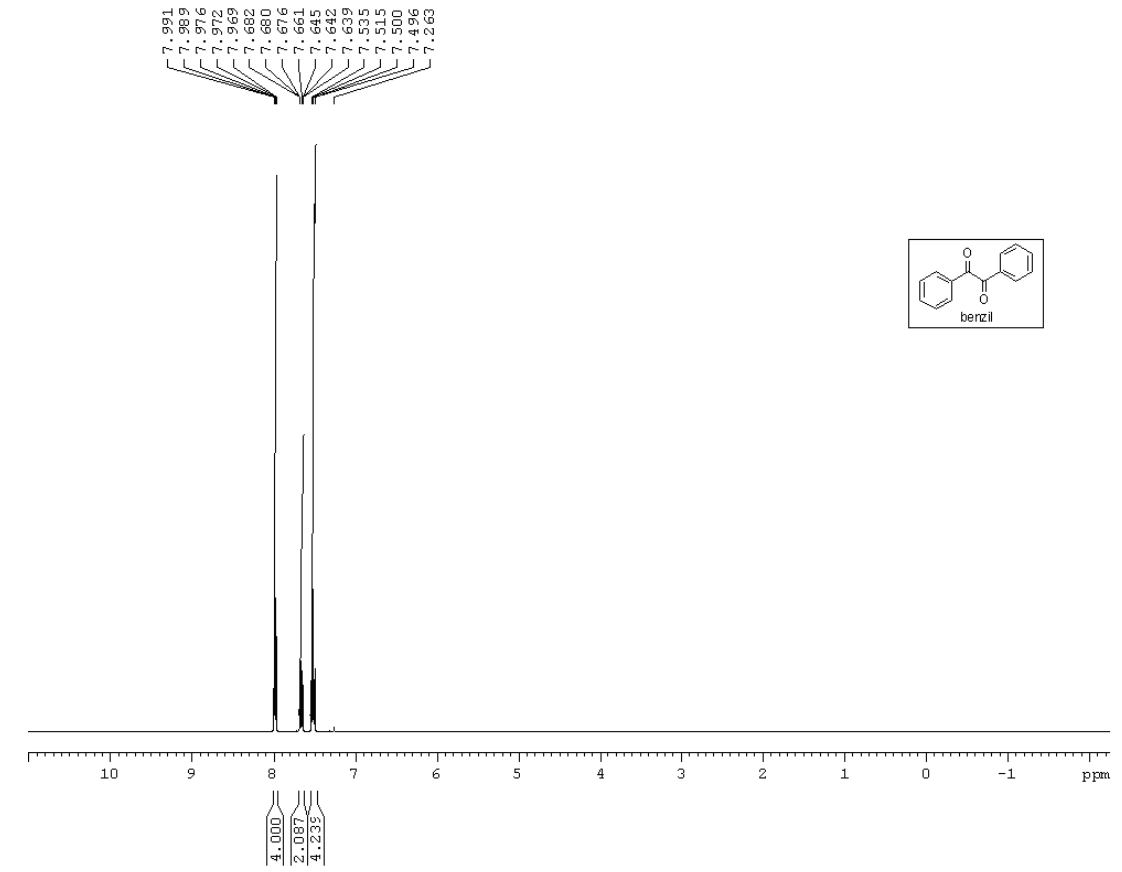

7.866
7.861
7.856
7.844
7.839
7.833
7.385
7.380
7.375
7.358
7.353
7.340
7.336
7.331
7.322
7.317
7.308
7.306
7.301
7.296
7.288
7.272
7.261
5.906
5.890
4.492
4.477
1.571
0.008
0.000
OH
Cl
O
(R)-4-chloro benzoin
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.153
2.319
5.054
1.084
1.000
197.960
140.582
138.827
131.933
130.627
129.388
129.219
128.915
127.884
77.481
77.163
76.846
76.462
OH
Cl
O
(R) - 4 -chlorobenzoin
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

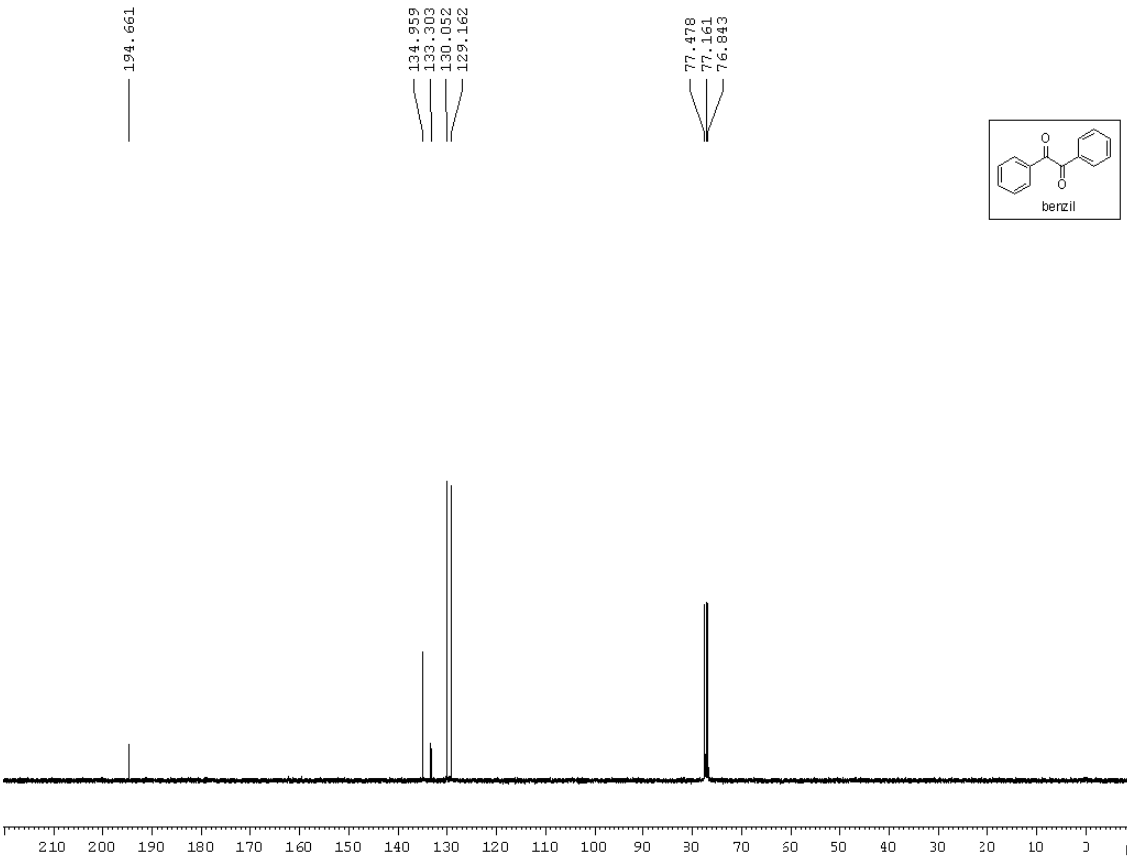

7.864
7.843
7.256
7.235
7.221
7.201
7.157
7.137
5.910
5.895
4.554
4.539
2.675
2.656
2.637
2.617
2.597
2.578
2.559
1.257
1.225
1.206
1.197
1.186
1.177
1.158
OH
O
(R) -p - ethylbenzoin
2.005
4.313
1.974
1.000
0.872
4.256
6.230

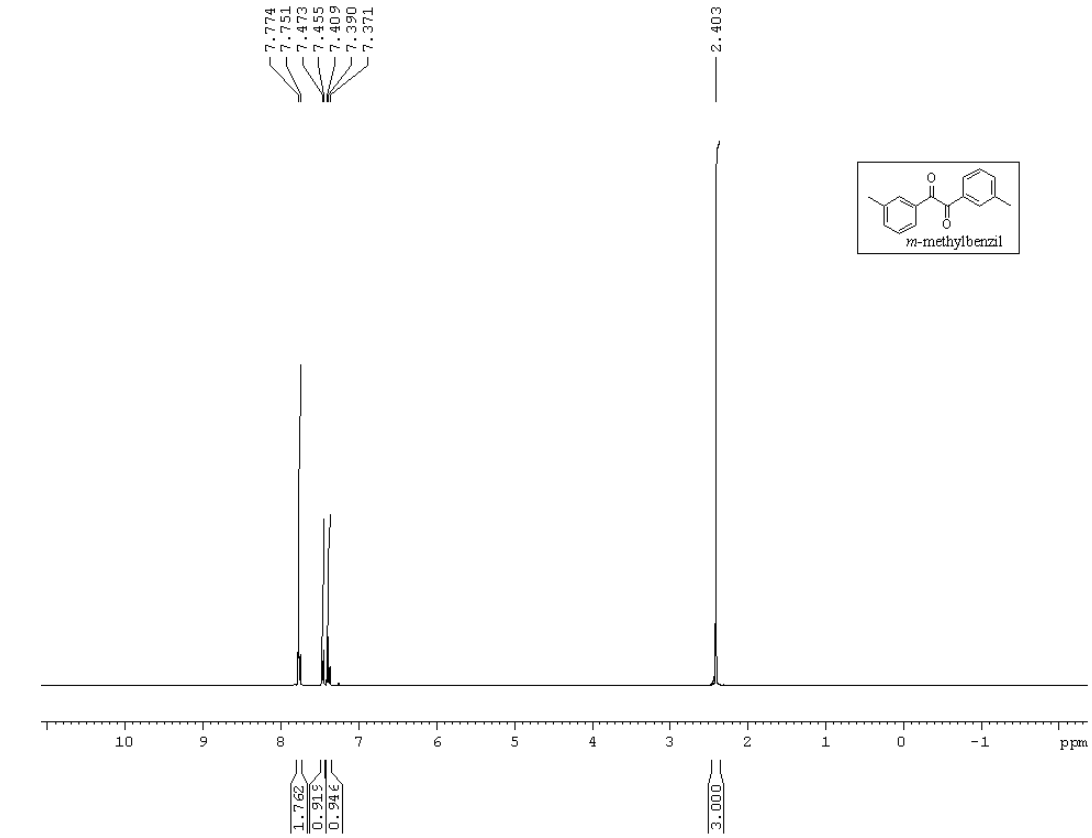

198.663
151.135
144.751
136.710
131.295
129.558
128.760
128.330
127.847
77.485
77.167
76.850
75.949
29.103
28.657
15.451
15.057
OH
O
(R) -p - ethylbenzoin

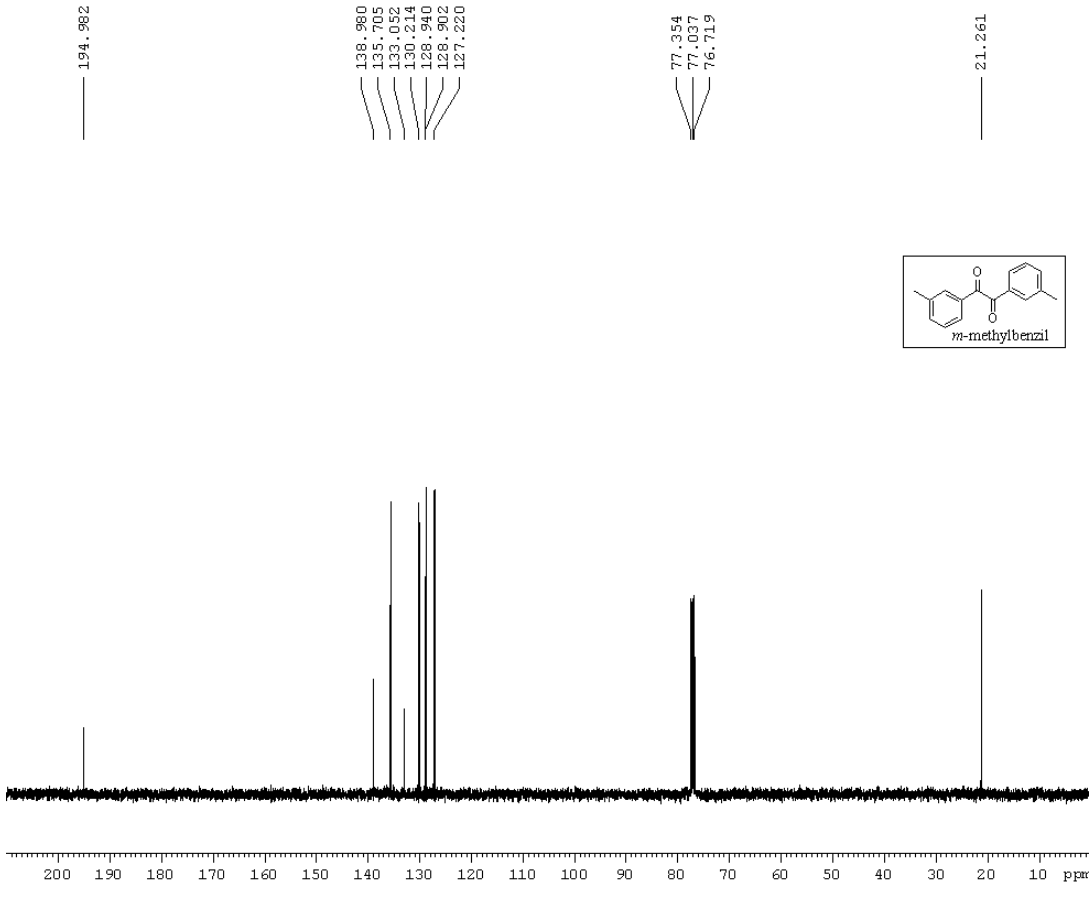

7.825
7.805
7.259
7.220
7.200
7.193
7.172
7.127
7.107
5.897
5.882
4.547
4.531
2.349
2.283
1.574
0.000
OH
O
(R) -p - methylbenzoin
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.995
4.024
1.978
1.003
1.000
3.146
3.115

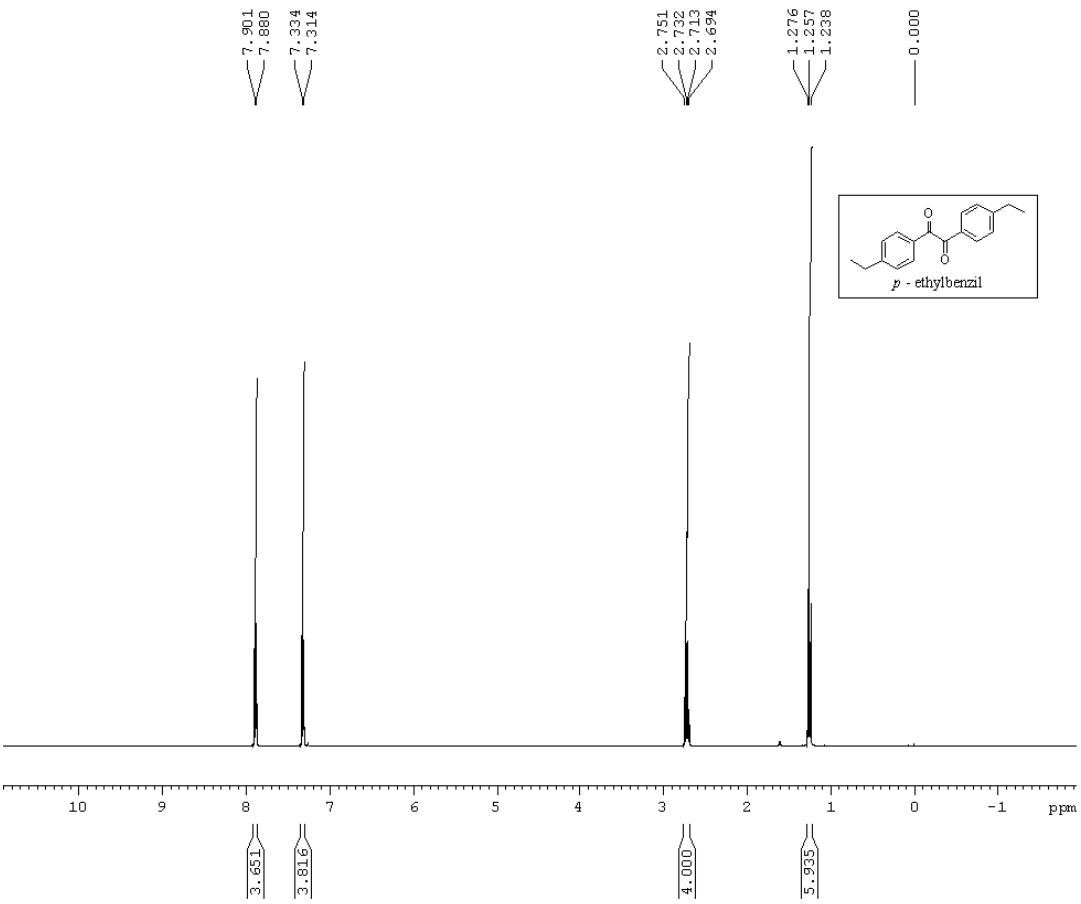

198.704
145.009
138.450
136.537
131.129
129.907
129.492
129.404
127.787
77.480
77.162
76.844
75.948
21.830
21.265
OH
O
(R) -p - methylbenzoin
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

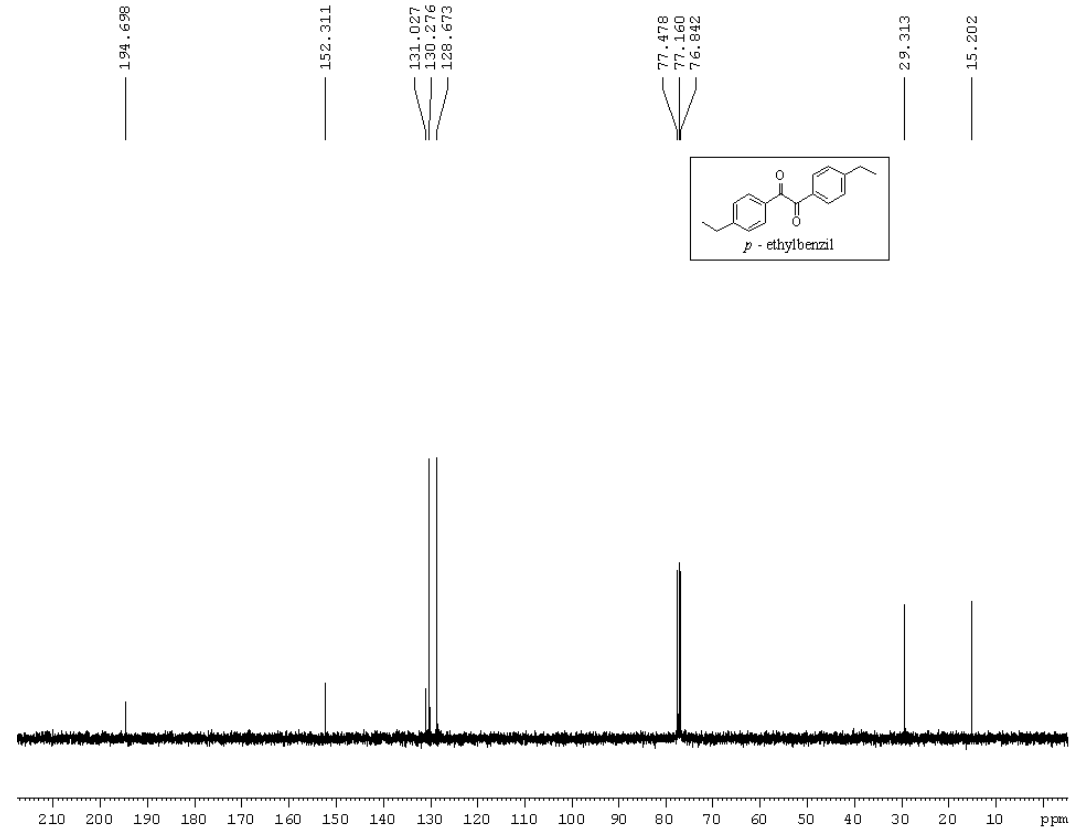

7.991
7.989
7.976
7.972
7.969
7.682
7.680
7.676
7.661
7.645
7.642
7.639
7.535
7.515
7.500
7.496
7.263
benzil
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.000
2.087
4.235

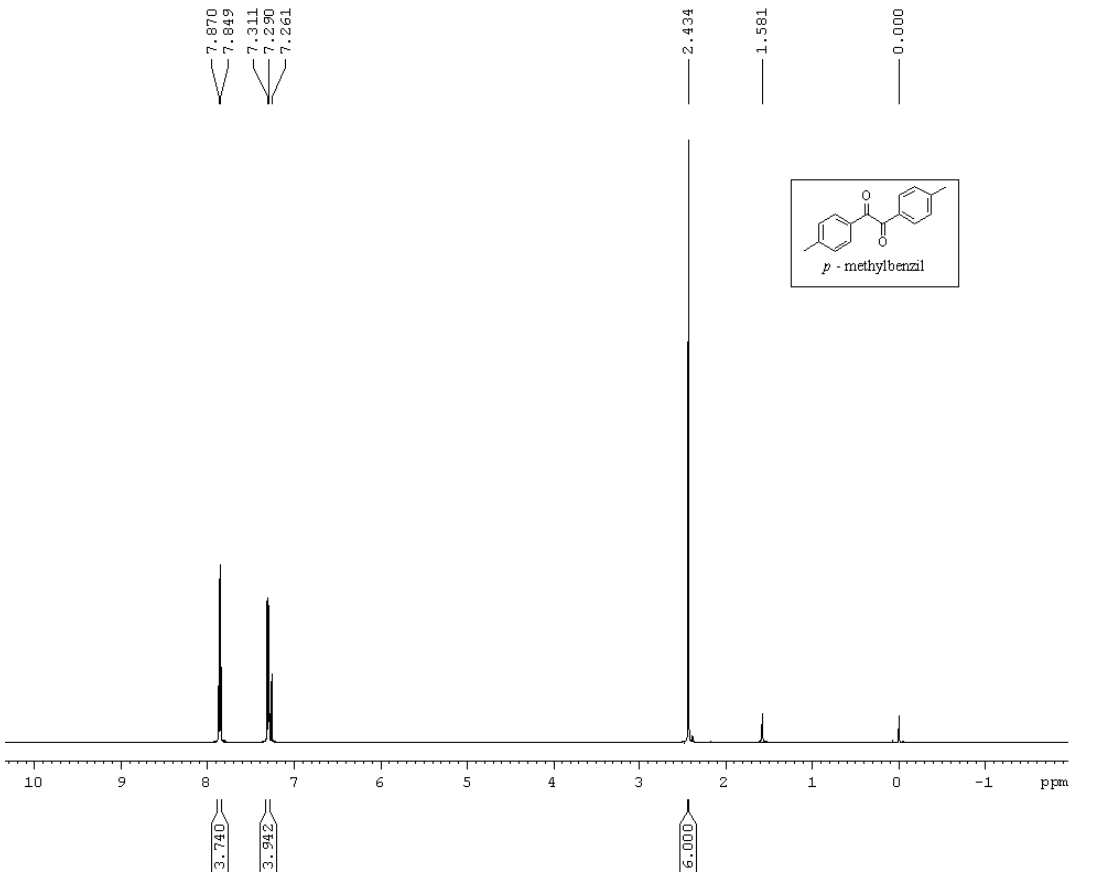

194.661
134.959
133.303
130.052
129.162
77.478
77.161
76.843
benzil
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

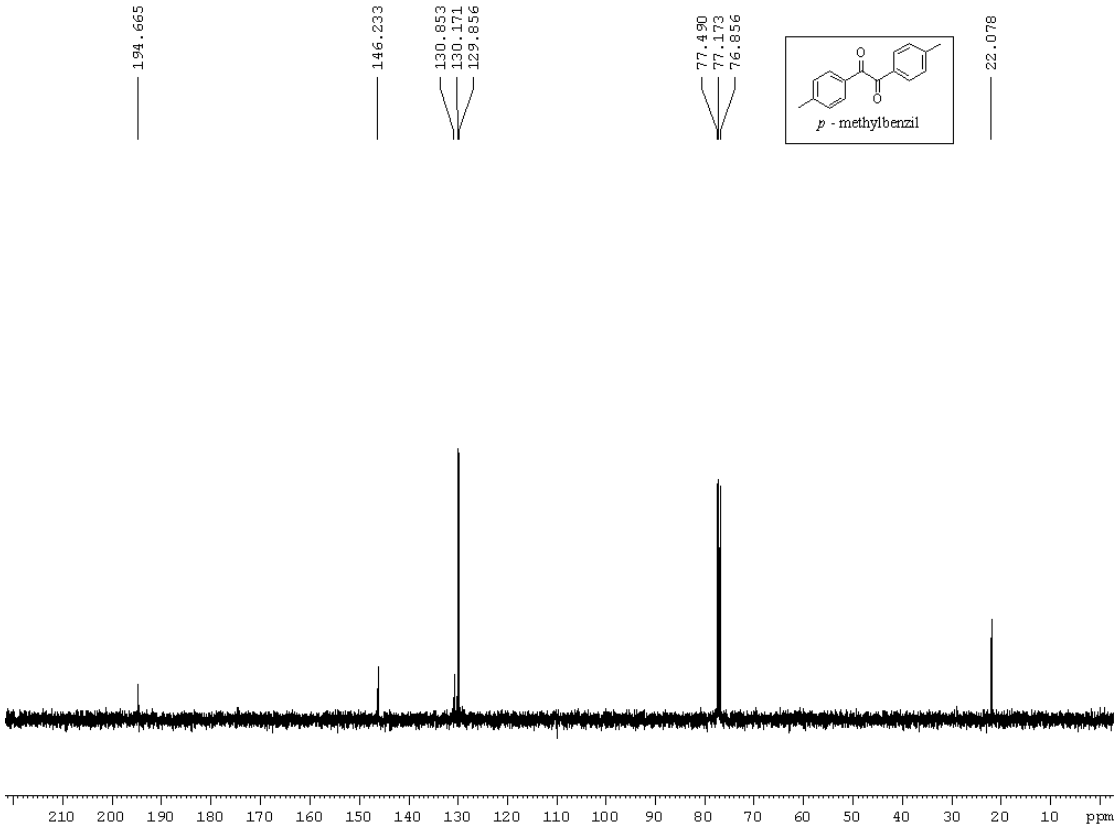

7.774
7.751
7.473
7.455
7.409
7.390
7.371
2.403
m-methylbenzil
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.762
0.915
0.94€
3.000

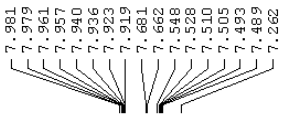

194.982
138.980
135.705
133.052
130.214
128.940
128.902
127.220
77.354
77.037
76.719
21.261
m-methylbenzil
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

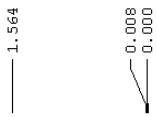

7.901
7.880
7.334
7.314
2.751
2.732
2.713
2.694
1.276
1.257
1.238
0.000
p - ethylbenzil
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
3.651
3.816
4.000
5.935

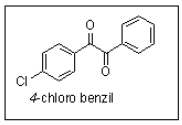

194.698
152.311
131.027
130.276
128.673
77.478
77.160
76.842
29.313
15.202
p - ethylbenzil
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

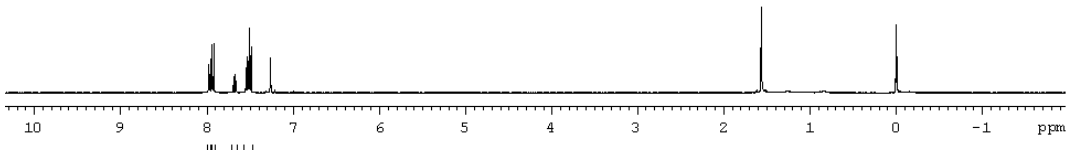

7.870
7.849
7.311
7.290
7.261
2.434
1.581
0.000
p - methylbenzil
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
3.740
3.942
6.000


194.665
146.233
130.853
130.171
129.856
77.490
77.173
76.856
22.078
p - methylbenzil
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

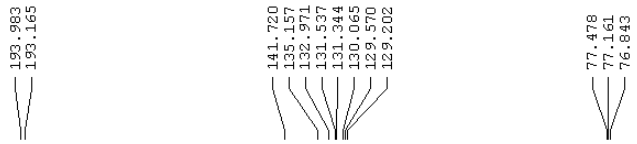

7.981
7.979
7.961
7.957
7.940
7.936
7.923
7.919
7.681
7.662
7.548
7.528
7.510
7.505
7.493
7.489
7.262

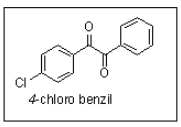

1.564
0.008
0.000

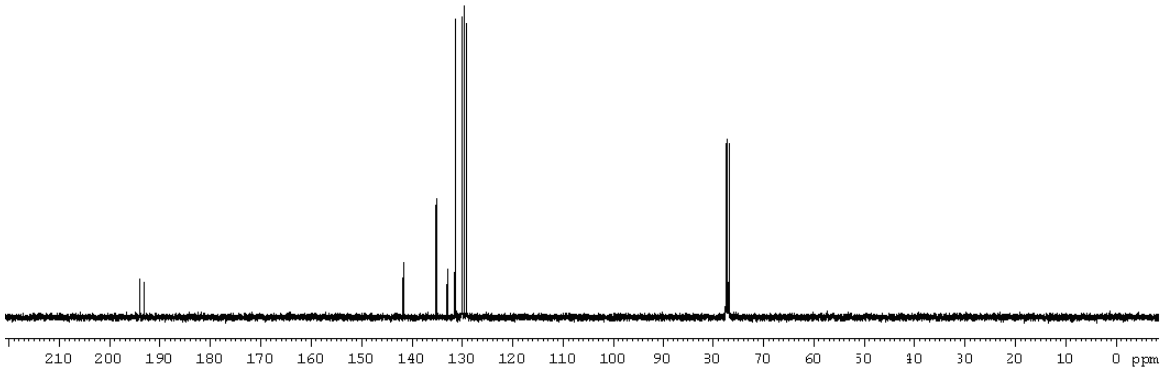

O
O
Cl
4-chloro benzil


10
9
8
7
6
5
4
3
2
1
0
-1
ppm


1.989
1.990
1.000
4.141


193.983
193.165
141.720
135.157
132.971
131.537
131.344
130.065
129.570
129.202
77.478
77.161
76.843


O
O
Cl
4-chloro benzil


210
200
190
180
170
160
150
140
130
120
110
100
90
30
70
60
50
40
30
20
10
0 ppm

References

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