An efficient and practical aerobic oxidation of benzylic methylenes by recyclable N-hydroxyimide

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Supplementary Information

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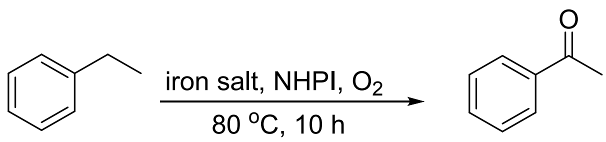
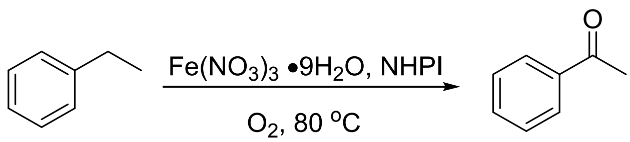
Table S1. Screen of iron salts[a]



Entry	Catalyst	Conversion (%)[b]
1	Iron tristearate	78
2	$Fe(acac)_3$	73
3	$Fe(NH_4)_2(SO_4)_2$	trace
4	$FeOH(CH_3COO)_2$	80
5	$FeCl_3$	trace
6	$Fe(NO_3)_3{\cdot}9H_2O$	83
7	$Fe_2(SO4)_3$[c]	69

[a]Reaction conditions: 2 mmol ethylbenzene and 10 mol % metal salts, 10 mol % NHPI in 2 mL AcOH are stirred at 100℃ oil bath for 10 h.
[b]Conversion is determined by GC.
[c]5 mol% $Fe_2(SO_4)_3$ was added.

Table S2. Screen of solvents[a]



Entry	Solvent		Conversion (%)b
1	AcOH		23
2	Chlorobenzene		trace
3	Cyclohexanone		9
4	EtOAc[c]		11
5	EtOH[c]		<5
6	CH_3CN		41
7	PhCN		57
8	DMF		trace
9	DMSO		<5
10	Toluene		19
11	n-Butanol		<5
12	t-Butanol		<5

[a]2 mmol ethylbenzene, 5 mol % metal salts, 5 mol % NHPI in 2 mL solvents are stirred at 80 ℃ oil bath for 10 h.
[b]Conversion is determined by GC.
[c]Recation mixture was stirred at refluxing condition.

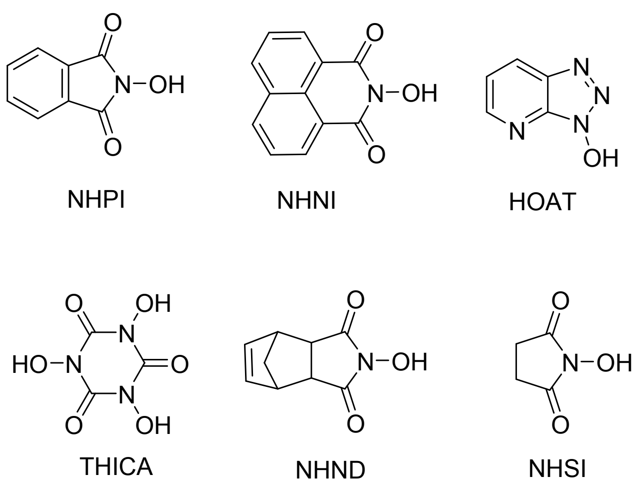
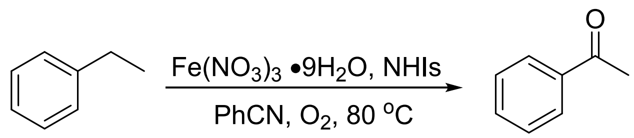


Figure S1. Structure of screened NHIs

Table S3. Screen of NHIs[a]



Entry	NHIs	Conversion (%)[b]
1	NHPI	99
2	NHNI	44
3	NHND	11
4	NHSI	97
5	HOAT	<5
6	THICA	38

[a] Reaction conditions: 2 mmol ethylbenzene and 5 mol % metal salts，5 mol % NHIs in 2 mL PhCN are stirred in 90 ℃ oil bath for 10 h.

[b] Conversion is determined by GC with diphenyl ether as internal standard.

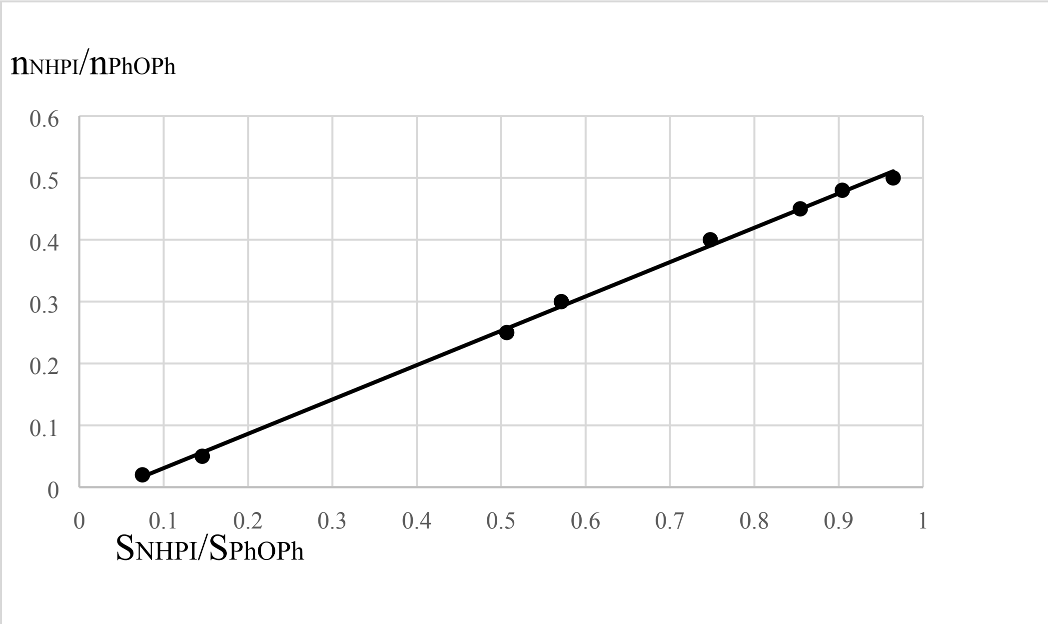


Figure S2. Linear regression line and regression equation of NHPI/internal standard when molar raion of NHPI : PhOPh = 0.02 - 0.50)

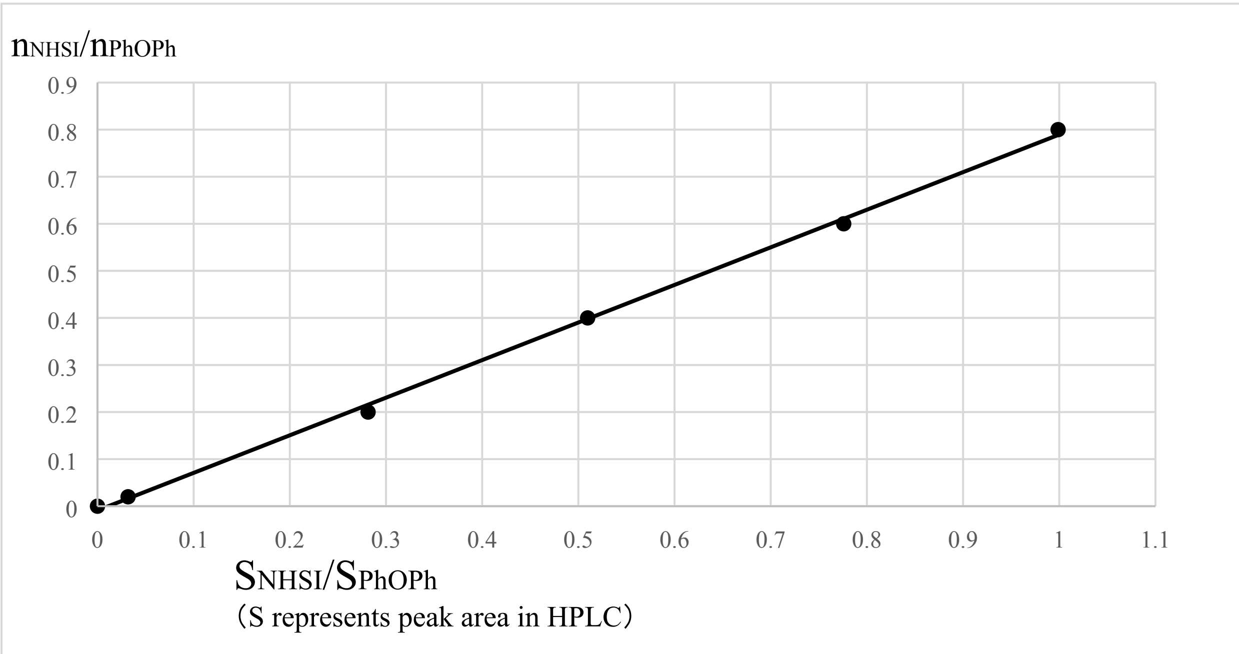
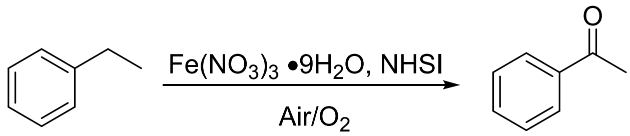


Figure S3. Linear regression line and regression equation of NHSI/internal standard when molar ratio of NHSI : PhOPh = 0.00 - 0.80)

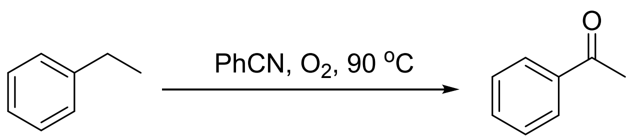
Table S4. Further optimization of $Fe(NO_3)_3$/NHSI/O_2 system[a]



Entry	$Fe(NO_3)_3·9H_2O$ (mol%)	NHSI (mol%)	PhCN (mL)	T (°C)	Oxygen Source[b]	Time (h)	Yield[c] (%)
1	5	5	2	90	A	10	93
2	3	3	2	90	A	18	93
3	1	1	2	90	A	24	57
4	1	1	2	100	A	48	68
5	1	2	2	100	A	48	77
6	2	2	2	100	A	24	83
7	1	3	2	100	A	12	86
8	1	3	2	90	A	18	86
9	1	3	2	80	A	48	63
10	1	3	1	90	A	16	86
11	1	3	0.5	90	A	48	61
12	1	3	1	90	B	14	90
13	1	3	1	90	C	16	90
14[d]	1	3	1	90	B	16	89

[a]Reaction conditions: 2 mmol ethylbenzene, $Fe(NO_3)_3·9H_2O$ and NHSI in PhCN are stirred in 90 °C oil bath.
[b]Oxygen source condition: A: Air (open system); B: O_2 Balloon without purging; C: O_2 balloon with purging (replacing original air in system with pure O_2).
[c]Determined by GC with diphenyl ether as internal standard.
[d]20 mmol% KPF_6 was added as additive.

Table S5. Controlled experiments of EB oxidation[a]



Entry	Catalyst	Additive	Conversion[b]
1	5 mol% $Fe(NO_3)_3$ 5 mol% NHSI	\	98
2[c]	5 mol% $Fe(NO_3)_3$ 5 mol% NHSI	\	98
3	2 mol% $Fe(NO_3)_3$	\	79

	5 mol% NHSI		
4	5 mol% NHSI	\	54
5	5 mol% $Fe(NO_3)_3$	\	trace
6	5 mol% $Fe(NO_3)_3$ 1 mol% NHSI	\	21
7	2.5 mol% $Fe_2(SO_4)_3$ 5 mol% NHSI	\	71
8	15 mol% $NaNO_3$ 5 mol% NHSI	\	57
9	5 mol% $Fe(NO_3)_3$ 5 mol% NHSI	5 mol% BHT	79
10	5 mol% $Fe(NO_3)_3$ 5 mol% NHSI	20 mol% BHT	53
11	5 mol% $Fe(NO_3)_3$ 5 mol% NHSI	100 mol% BHT	trace

[a]Reaction conditions: 2 mmol ethylbenzene, catalyst, additive in 1 mL PhCN with O_2 balloon are stirred in 90 ^oC oil bath for 24 h.
[b]Conversion was determined by GC.
[c]Reaction flask was tightly wrapped by aluminum foil to avoid any visible light.

Product characterization

Benzophenone[1] **(2a).** Compound **2a** was obtain as white solid (357 mg, 98% yield). m. p. 48-49 ^oC (lit.[1] 47-49 ^oC) ^1H NMR (500 MHz, Chloroform-*d*) δ 7.84 – 7.82 (m, 4H), 7.60 (tt, 2H, *J* = 7.5, 1.5 Hz), 7.52 – 7.48 (m, 4H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ196.8, 137.6, 132.4, 130.1, 128.3.

4-Chlorobenzophenone[2] **(2b).** Compound **2b** was obtain as white solid (359 mg, 83% yield). m. p. 75-76 ^oC (lit.[2] 75-77 ^oC) ^1H NMR (500 MHz, Chloroform-*d*) δ 7.80-7.76 (m, 4H), 7.62 (tt, *J* =7.5 Hz, 1.5 Hz, 1H), 7.53-7.47 (m, 4H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 195.5, 138.9, 137.3, 135.9, 132.7, 131.5, 129.9, 128.7,128.4.

9-Fluorenone[1] **(2c).** Compound **2c** was obtain as yellow solid (349 mg, 97% yield). m. p. 83-84 ^oC (lit.[5] 84-86 ^oC) ^1H NMR (500 MHz, Chloroform-*d*) δ 7.66 (dt, *J* = 7.5 Hz, 1 Hz, 2H), 7.51 (dt, *J* = 7.5 Hz, 1 Hz, 2H), 7.48 (dt, *J* = 7.5,1.5 Hz, 2H), 7.29 (td, *J* = 7.5 Hz, 1.5 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 193.9, 144.4, 134.7, 134.1, 129.0, 124.3, 120.3.

9-Xanthenone[3][4] **(2d).** Compound **2d** was obtain as white solid (384 mg, 98% yield). m. p. 170-172 ^oC (lit.[3] 168-170 ^oC) ^1H NMR (500 MHz, Chloroform-*d*) δ 8.33 (dd, *J* = 8.0 Hz, 1.5 Hz, 2H), 7.72 – 7.69 (m, 2H), 7.47 (d, *J* = 8.5 Hz, 2H), 7.38 – 7.35 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 177.1, 156.1, 134.7, 126.7, 123.9, 121.8, 117.9.

1-[4-(4-Pyridinyl)phenyl]ethenone[5] **(2e).** Compound **2e** was obtain as off-white solid (362 mg, 99% yield). m. p. 71-72 ^oC (lit.[5] 71-72 ^oC) ^1H NMR (600 MHz, Chloroform-*d*) δ 8.82 (d, *J* = 6 Hz, 2H), 7.83 (d, *J* = 7.2 Hz, 2H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.59 (d, *J* = 6 Hz, 2H), 7.53 (t, *J* = 7.8 Hz, 2H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 195.1, 150.3, 144.4, 135.9, 133.6, 130.1, 128.7, 122.9.

1-Tetralone[1] **(2f).** Compound **2f** was obtain as light yellow liquid (289 mg, 99% yield). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.98 (dd, *J* = 8.0 Hz, 1.5Hz, 1H), 7.40 (td, *J* = 7.5 Hz, 1.5Hz, 1H), 7.24(t, *J* = 7.5 Hz, 1H), 7.19 (d, *J* = 7.5 Hz, 1H), 2.90 (t, *J* = 6.5 Hz, 1H), 2.59 (t, *J* = 6.5 Hz, 1H), 2.10-2.03 (m,1H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 197.9, 144.2, 133.1, 132.4, 128.6, 126.8, 126.3, 38.9, 29.4, 23.1.

1-Indanone[1] **(2g).** Compound **2g** was obtain as light yellow solid (230 mg, 87% yield). m. p. 39-41 ^{o}C (lit.[1] 40-42 ^{o}C) ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.76 (d, *J* =7.5Hz, 1H), 7.59 (td, *J* =7.5 Hz, 1.0 Hz, 1H), 7.49 (dt, *J* = 8 Hz, 1.0 Hz, 1H), 7.39-7.36 (m, 1H), 3.15 (t, *J* = 6 Hz, 2H), 2.70 (t, *J* = 6 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 207.1, 155.1, 137.0, 134.6, 127.2, 126.7, 123.7, 36.2, 25.8.

3,4-dihydro-1H-2-benzopyran-1-one[6] **(2h).** Compound **2h** was obtain as light yellow liquid. ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 7.5 Hz, 1H), 7.51 (tt, *J* = 7.5 Hz, 1.2 Hz, 1H), 7.35 (t, *J* = 7.5Hz, 1H), 7.24 (d, *J* = 7.5 Hz, 1H), 4.50 (t, *J* = 5.5 Hz, 2H), 3.03 (t, *J* = 6.0 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 165.0, 139.5, 133.5, 130.1, 127.5, 127.2, 125.1, 67.2, 27.6.

Acetophenone[1] **(2i).** Compound **2i** was obtain as light yellow liquid (204 mg, 85% yield). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.93-7.91 (m, 2H), 7.52 (tt, *J* = 7.5, 1.5 Hz, 1H), 7.43 – 7.40 (m, 2H), 2.55 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 198.06, 137.09, 133.09, 128.55, 128.28, 26.54.

n-Butyrophenone[6] **(2j).** Compound **2j** was obtain as light yellow liquid (269 mg, 91% yield). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.95-7.93 (m, 2H), 7.51 (tt, *J* = 7.5 Hz, 1.5 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 2H), 2.91 (t, *J* = 7.5 Hz, 2H), 1.75 (sext, *J* = 7.5 Hz, 2H), 0.98 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 200.02, 136.92, 132.61, 129.31, 40.23, 17.53, 13.64.

2'-Bromoacetophenone[7] **(2k).** Compound **2k** was obtain as light yellow liquid (394 mg, 99% yield). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.57 (dd, *J* = 8 Hz, 1 Hz, 1H), 7.43 (dd, *J* = 7.5 Hz, 2 Hz, 1H), 7.33 (td, *J* = 7.5 Hz, 1 Hz, 1H), 7.26 (td, *J* = 8 Hz, 1.5Hz, 1H), 2.59 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 201.1, 141.3, 133.7, 131.7,128.8, 127.3, 118.7, 30.1.

4'-Bromoacetophenone[1] **(2l).** Compound **2l** was obtain as light yellow solid (386 mg, 97% yield). m. p. 51-52 ^{o}C (lit.[1] 48-50 ^{o}C) ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 8.5 Hz, 1H), 7.61 (d, *J* =8.5 Hz, 1H), 2.59 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 197.0, 135.9, 131.9, 129.8, 128.3, 26.5.

2-Bromoacetophenone[8] **(2m).** Compound **2m** was obtain as off-white solid (394 mg, 99% yield). m. p. 46-47 ^{o}C (lit.[8] 46-48 ^{o}C) ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.01-7.99 (m, 2H), 7.62 (tt, *J* = 7.5 Hz, 1.5Hz, 1H), 7.53-7.49 (m, 2H), 4.47 (s, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 191.3, 134.0, 133.9, 128.9, 30.9.

4'-Cyanoacetophenone[9] **(2n).** Compound **2n** was obtain as white solid (200 mg, 69% yield). m. p. 57-59 ^{o}C (lit.[21] 59-60 ^{o}C) ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.05 (d, *J* = 8 Hz, 2H), 7.78 (d, *J* = 8.5 Hz, 2H), 2.65 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 196.5, 139.9, 132.5, 128.7, 117.9, 116.4, 26.7.

4’-Nitroacetophenone[1] **(2o).** Compound **2o** was obtain as yellow solid (280 mg, 85 % yield). m. p. 78-79 ^{o}C (lit.[1] 80-81 ^{o}C) ^{1}H NMR (500 MHz, Chloroform-*d*) δ

8.30 (dt, J = 9, 2 Hz, 2H), 8.10 (dt, J = 9, 2 Hz, 2H), 2.68 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 196.3, 150.3, 141.4, 129.3, 123.8, 26.9.

4’-Ethylacetophenone[10] **(2p).** Compound **2p** was obtain as light yellow liquid (97 mg, 33 % yield).^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.89 (d, J = 8.0 Hz, 2H), 7.28 (d, J = 8.0 Hz, 2H), 2.71 (q, J = 7.5 Hz, 2H), 2.58 (s, 3H), 1.26 (t, J = 7.5 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 197.8, 150.0, 134.9, 128.5, 128.0, 28.9, 26.5, 15.1.

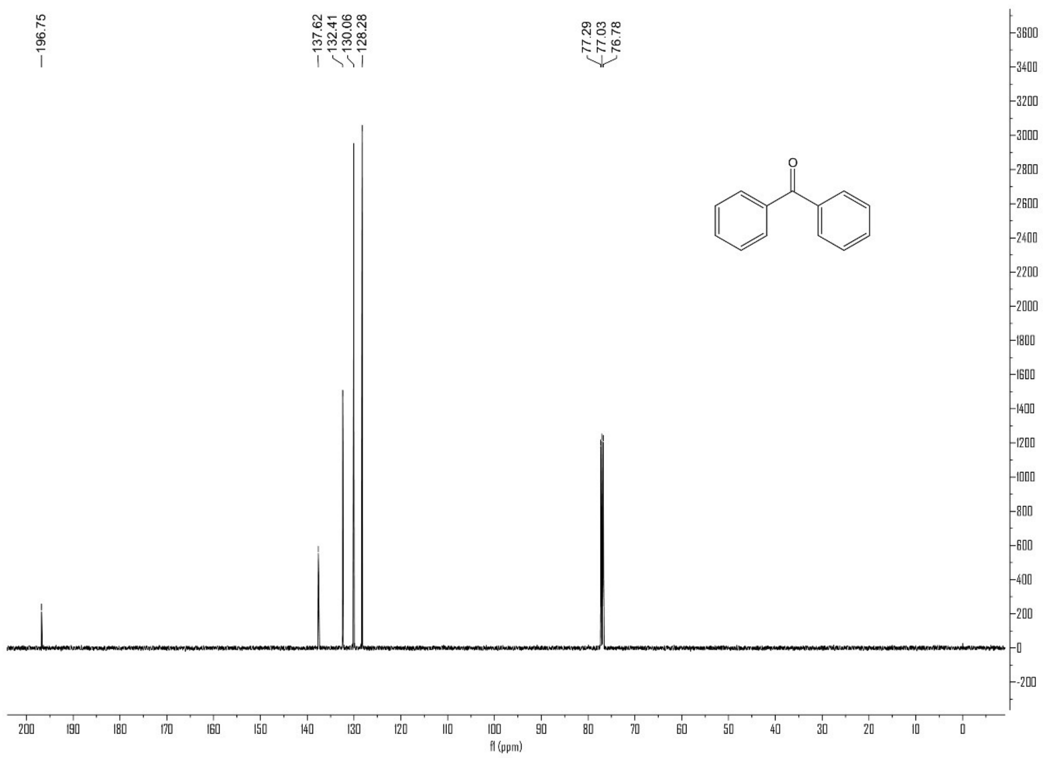
4'-Methylacetophenone[11] **(2q).** Compound **2q** was obtain as light yellow liquid (99 mg, 37 % yield). ^{1}H NMR (500 MHz, Chloroform-d) δ 7.84 (d, J = 8.5 Hz, 2H), 7.24 (d, J = 8 Hz, 2H), 2.55, (s, 3H), 2.39 (s, 3H). ^{13}C NMR (150 MHz, Chloroform-d) δ 197.6, 143.7, 134.6, 129.1, 128.3, 26.3, 21.5.

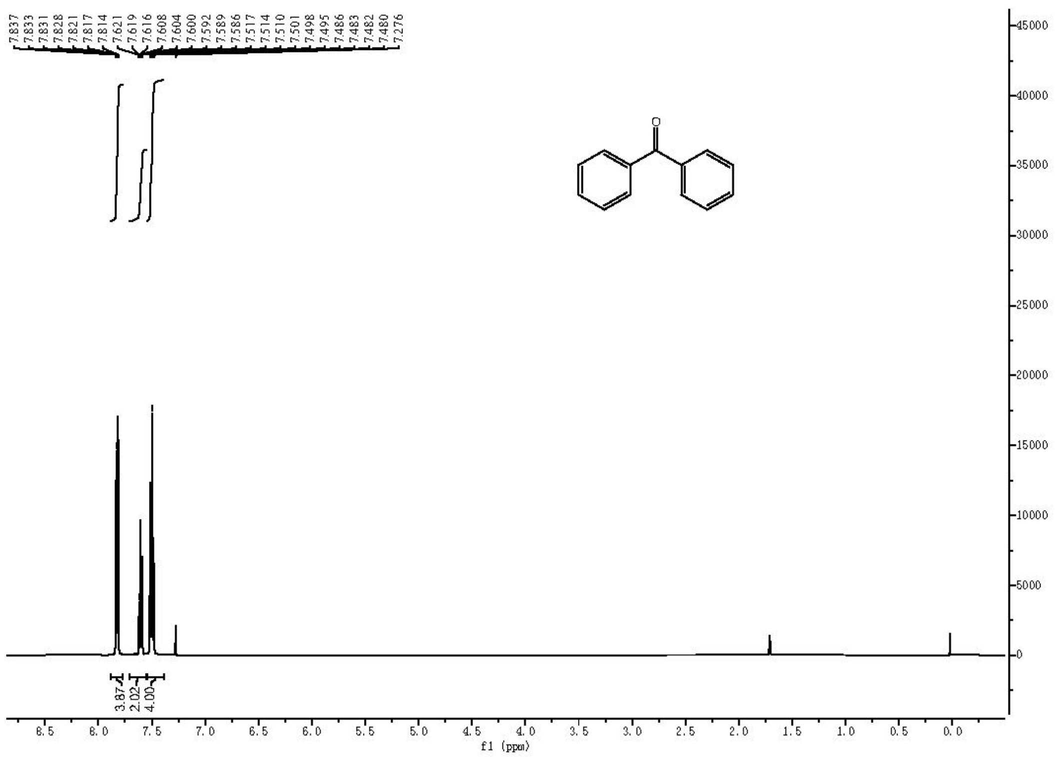
4’-Acetoxyacetophenone[12][13] **(2r).** Compound **2r** was obtain as off-white solid (238 mg, 67 % yield). m. p. 51-52 ^{o}C (lit.[12] 52-54 ^{o}C). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.89-7.83 (m, 2H), 7.10-7.05 (m, 2H), 2.45 (s, 1H), 2.19 (s, 1H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 196.4, 168.5, 154.1, 134.4, 129.6, 121.5, 26.6, 20.7.

4'-Methoxyacetophenone[11] **(2s).** Compound **2s** was obtain as light yellow liquid (105 mg, 35 % yield). ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.75-7.72 (m, 2H), 6.75-6.72 (m, 2H), 3.66-3.65 (m, 3H), 2.35-2.34 (m, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 196.4, 163.3, 130.4, 113.5, 55.2, 26.1.

NMR Spectrum of products

2a:





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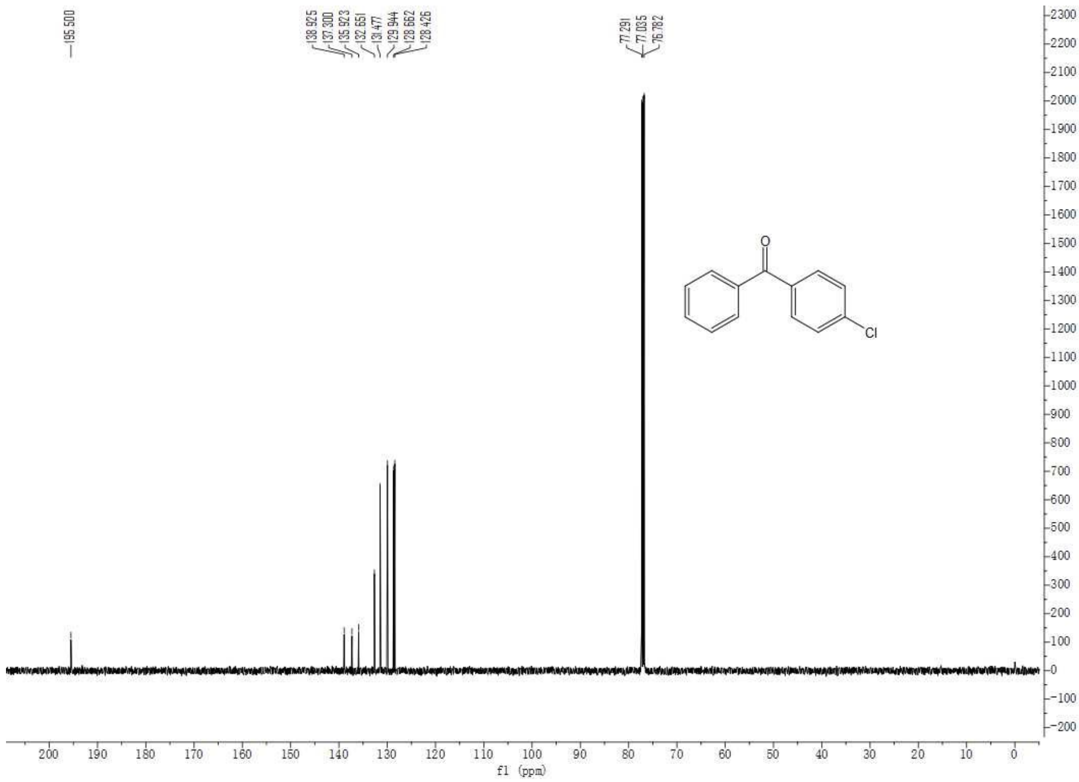
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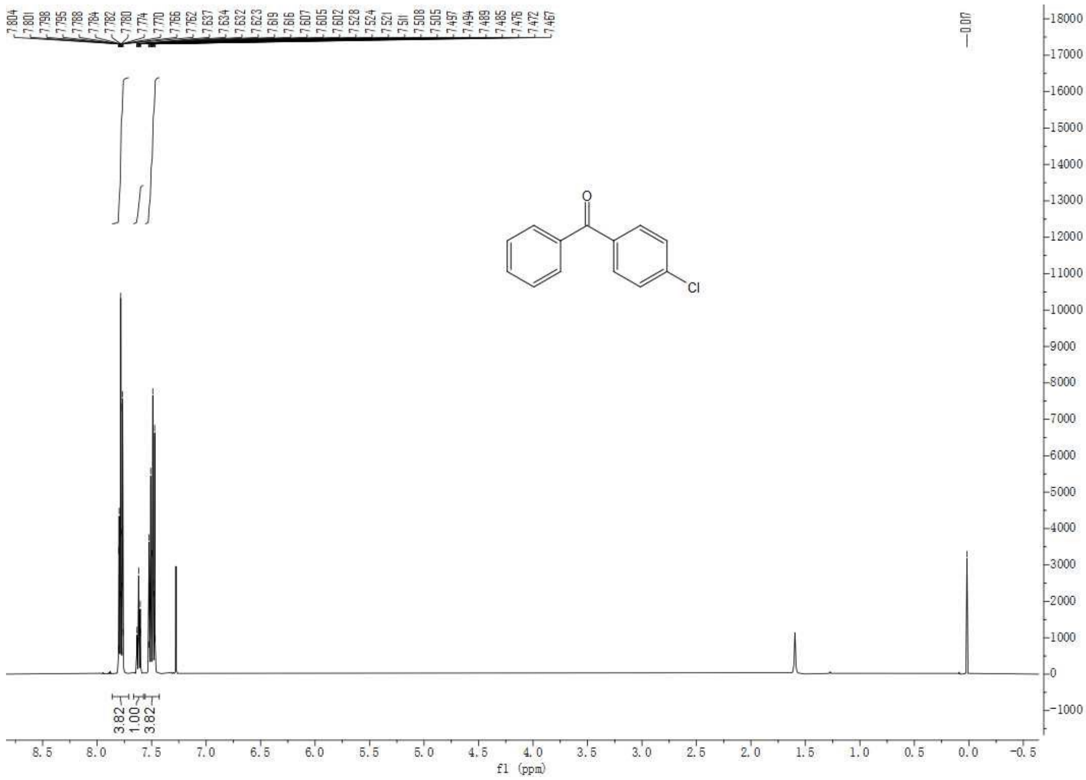
76.782
O
Cl
fl (ppm)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7.637
7.634
7.632
7.623
7.619
7.616
7.607
7.605
7.602
7.528
7.524
7.521
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7.505
7.497
7.494
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Cl
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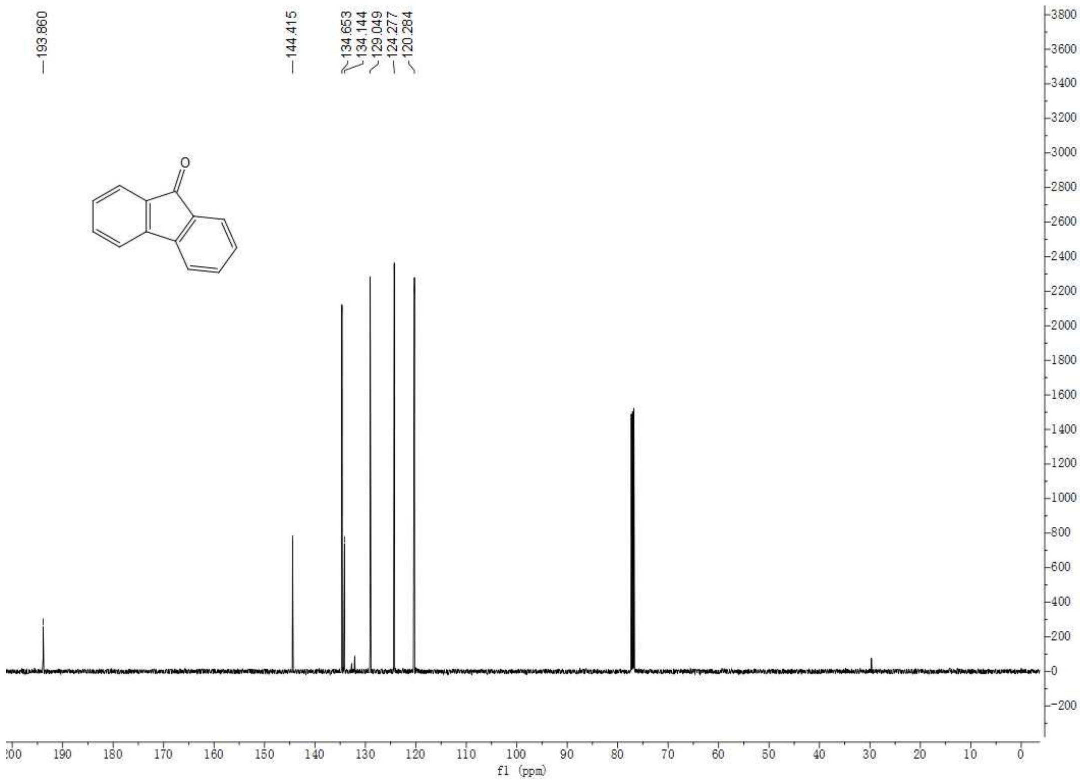
3.82
fl (ppm)

2c: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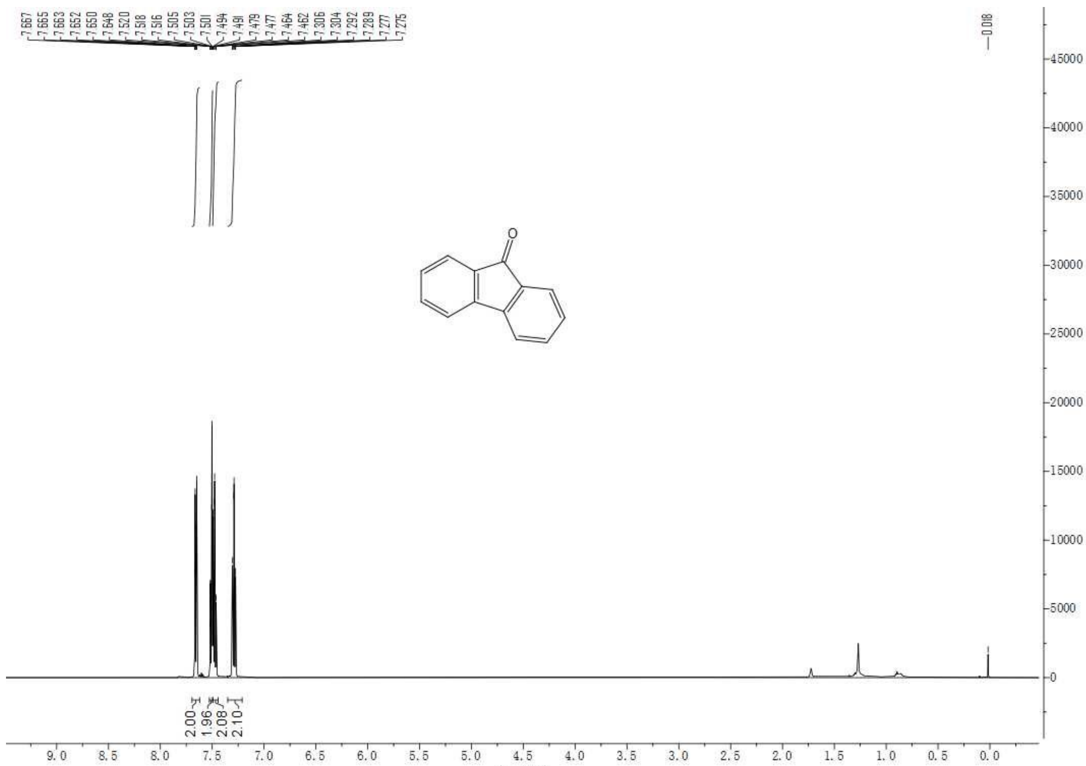
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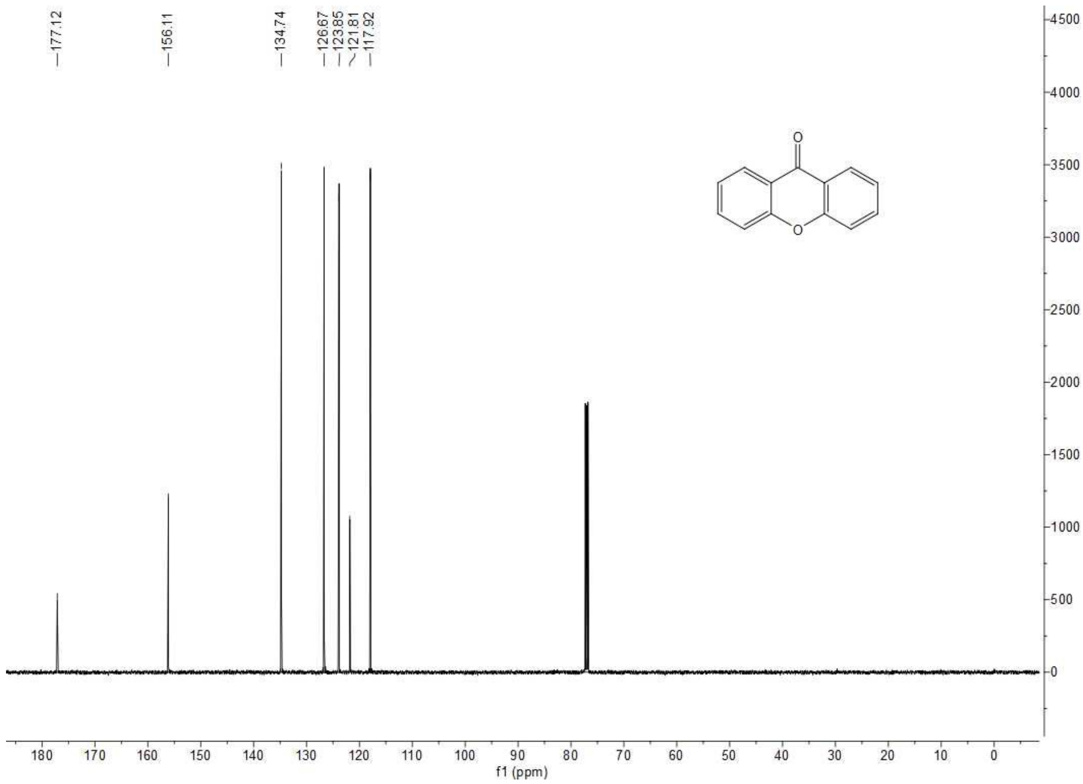
fl (ppm)

2d: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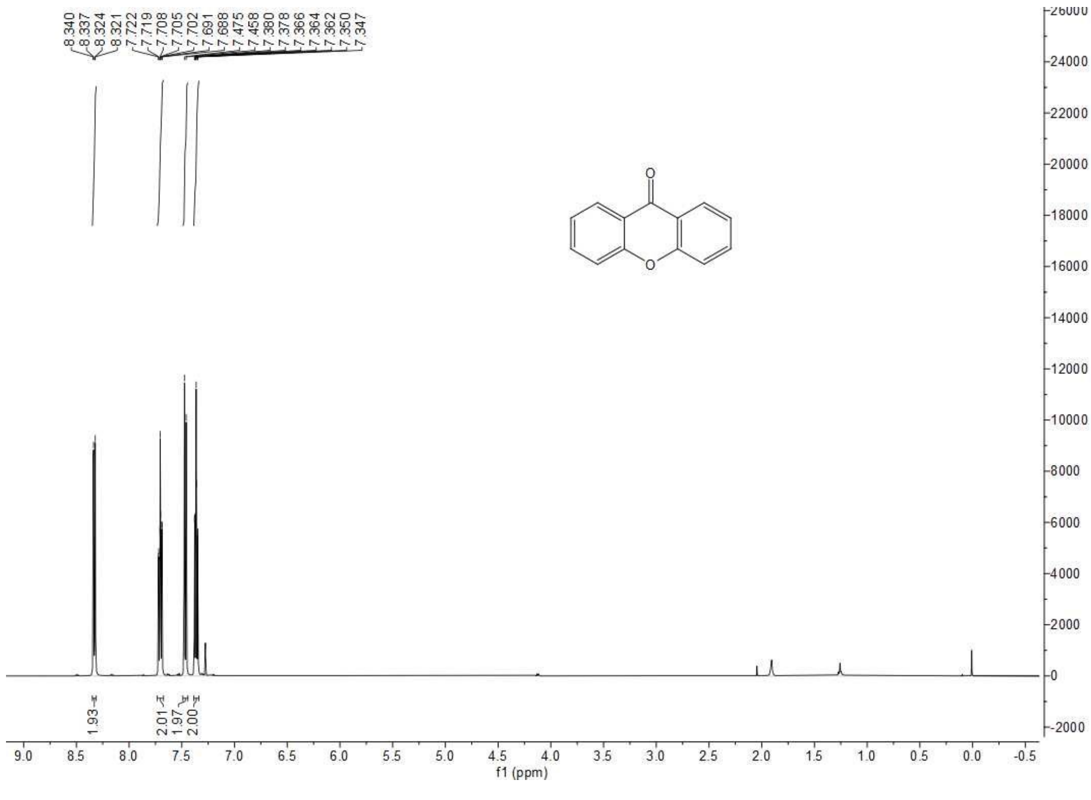
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7.380
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7.347
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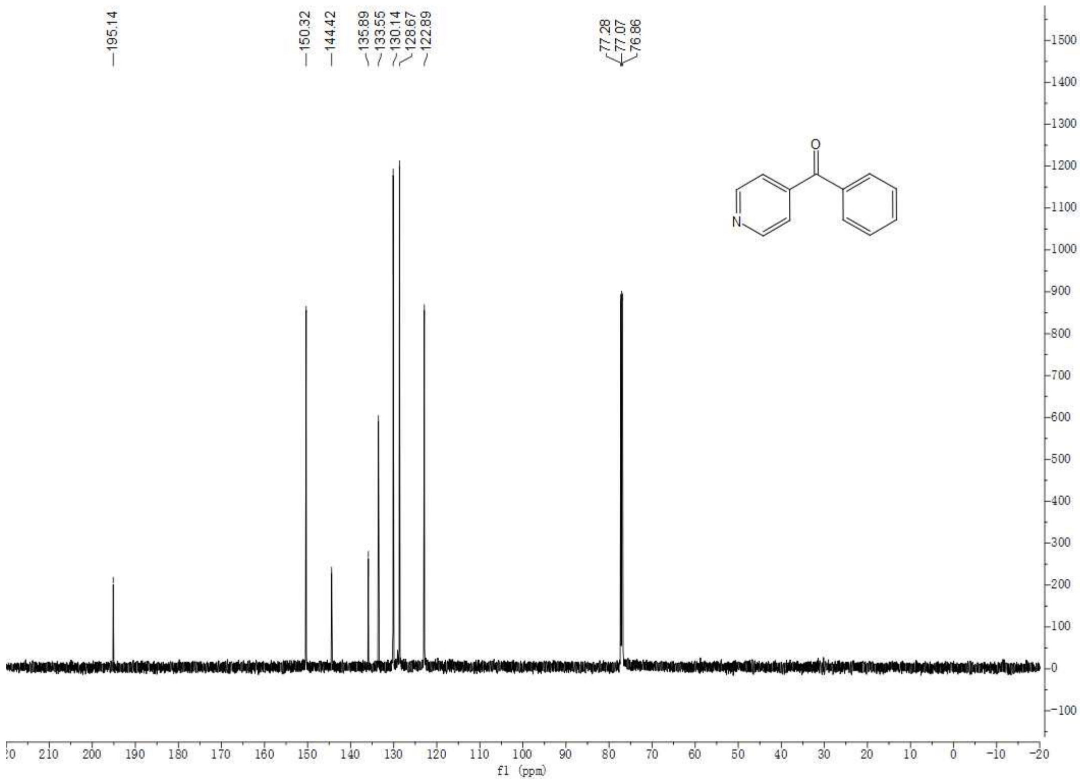
1.97
2.00
f1 (ppm)

2e: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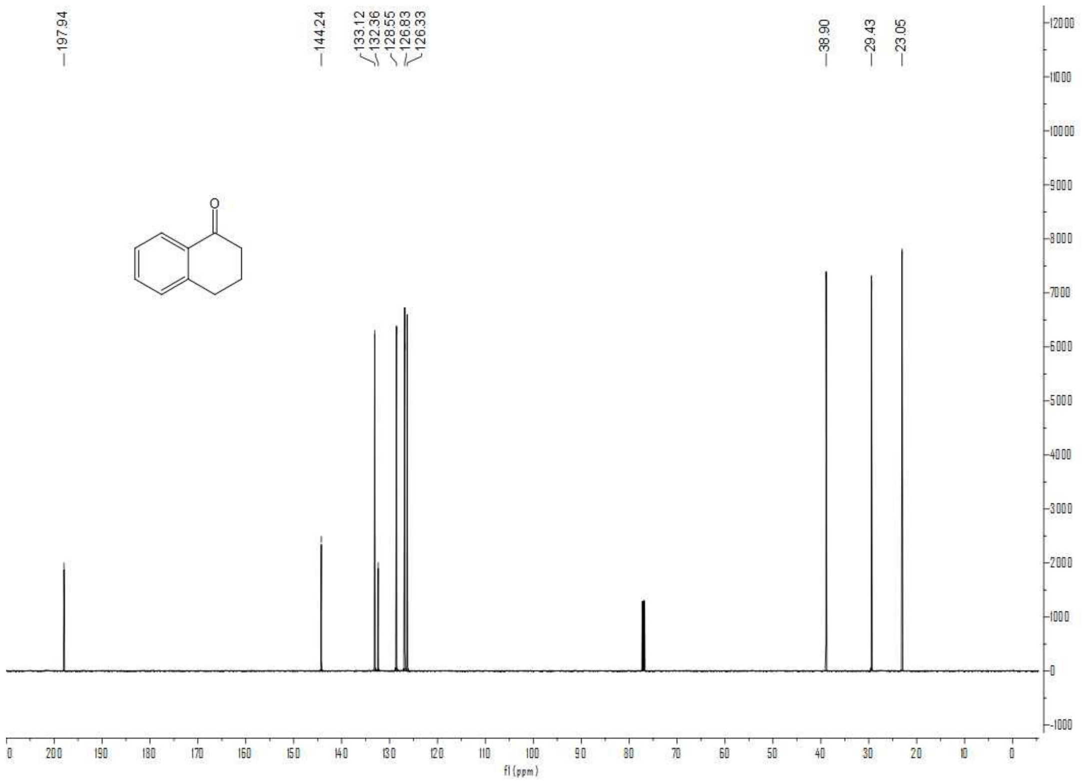

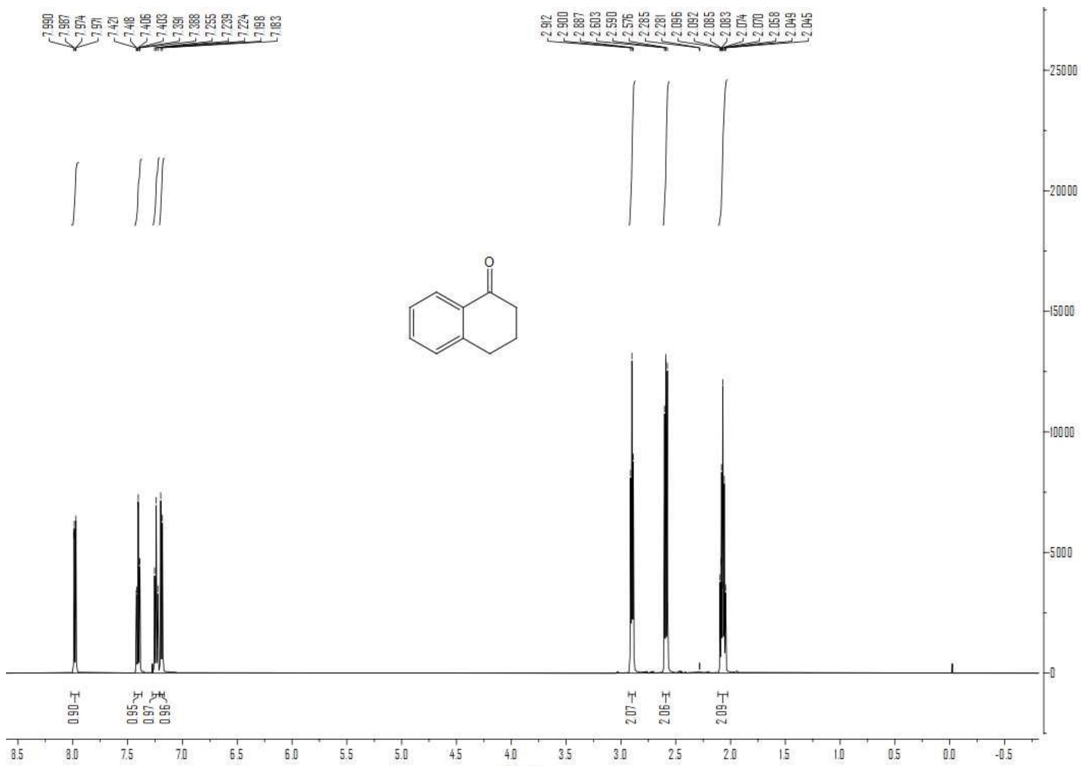

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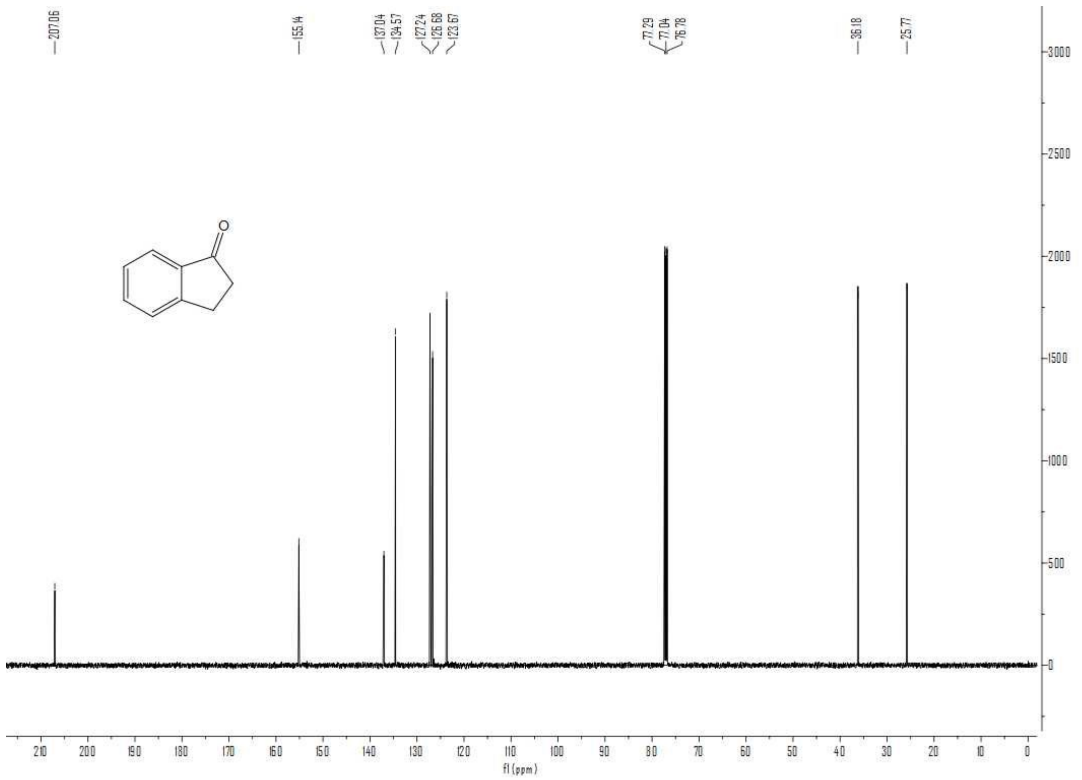
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134.57
127.24
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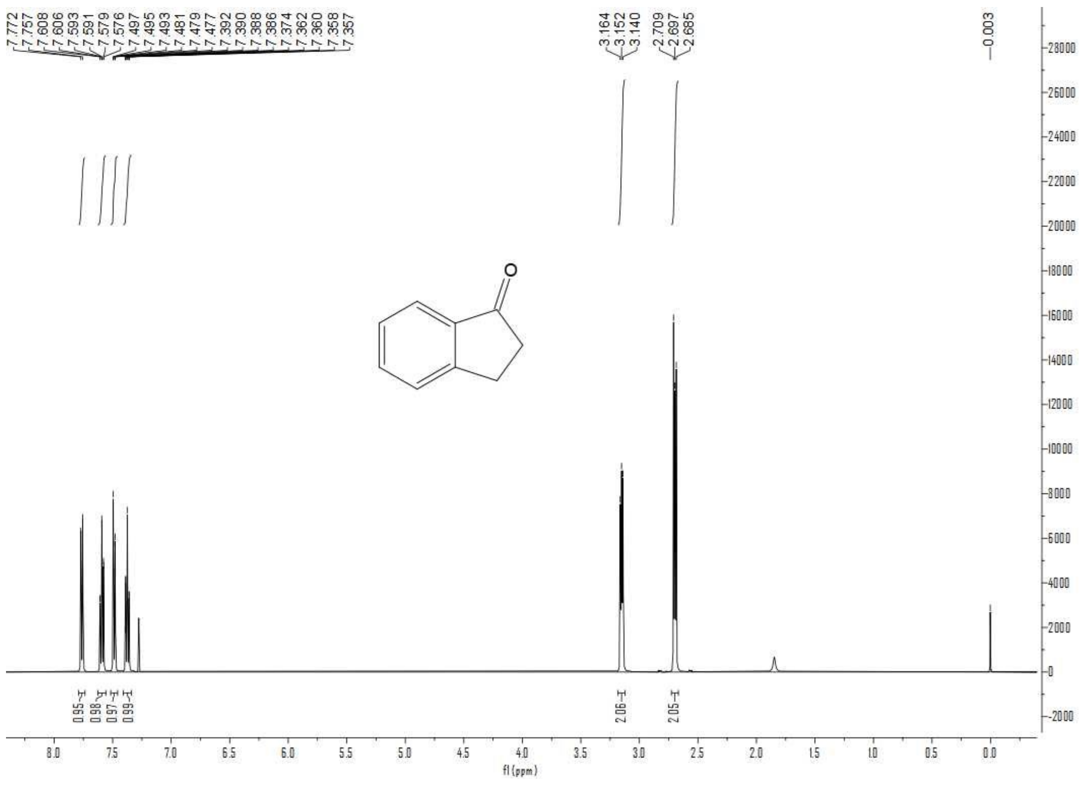
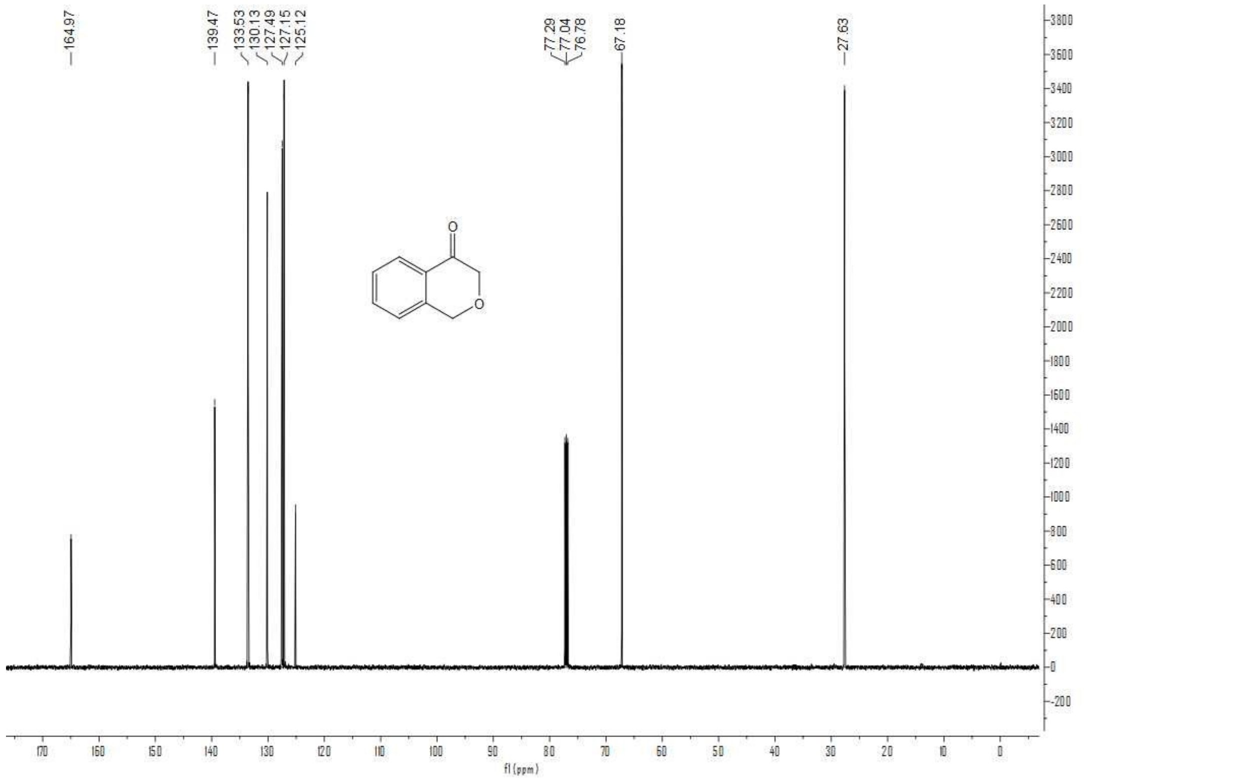
O
f1 (ppm)


7.772
7.757
7.608
7.606
7.593
7.591
7.579
7.576
7.497
7.495
7.493
7.481
7.479
7.477
7.392
7.390
7.388
7.386
7.374
7.362
7.360
7.358
7.357
3.164
3.152
3.140
2.709
2.697
2.685
-0.003
O
0.95
0.98
0.97
0.99
2.06
2.05
f1 (ppm)

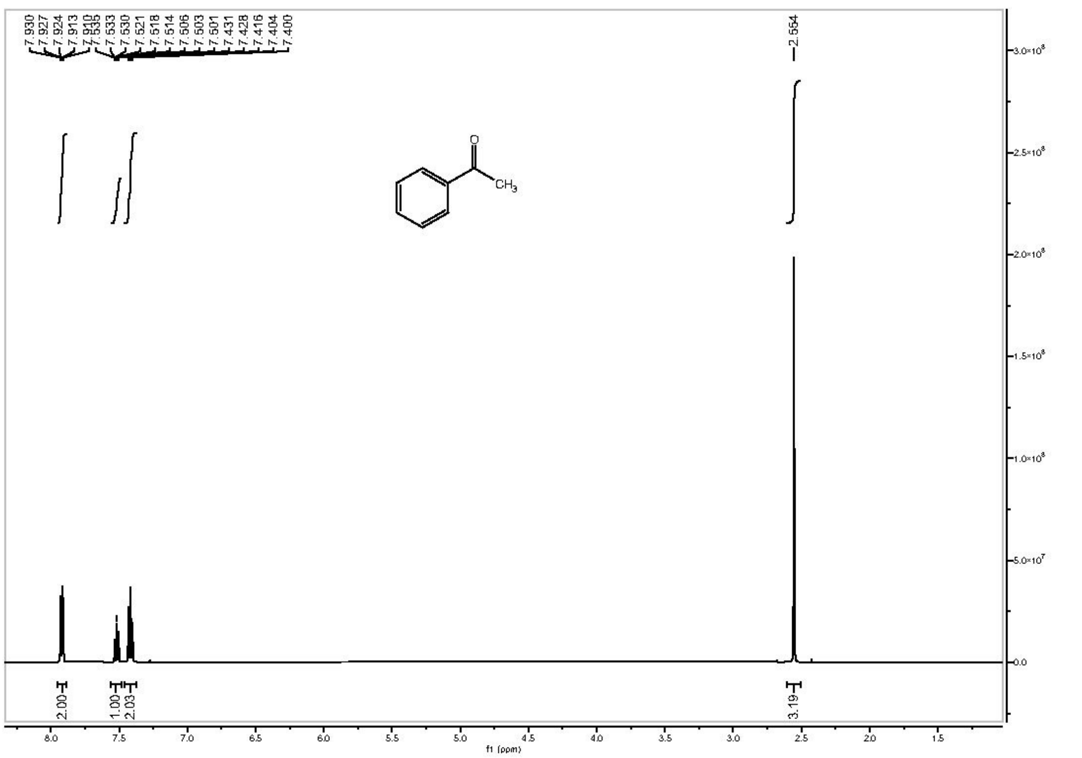
2h:



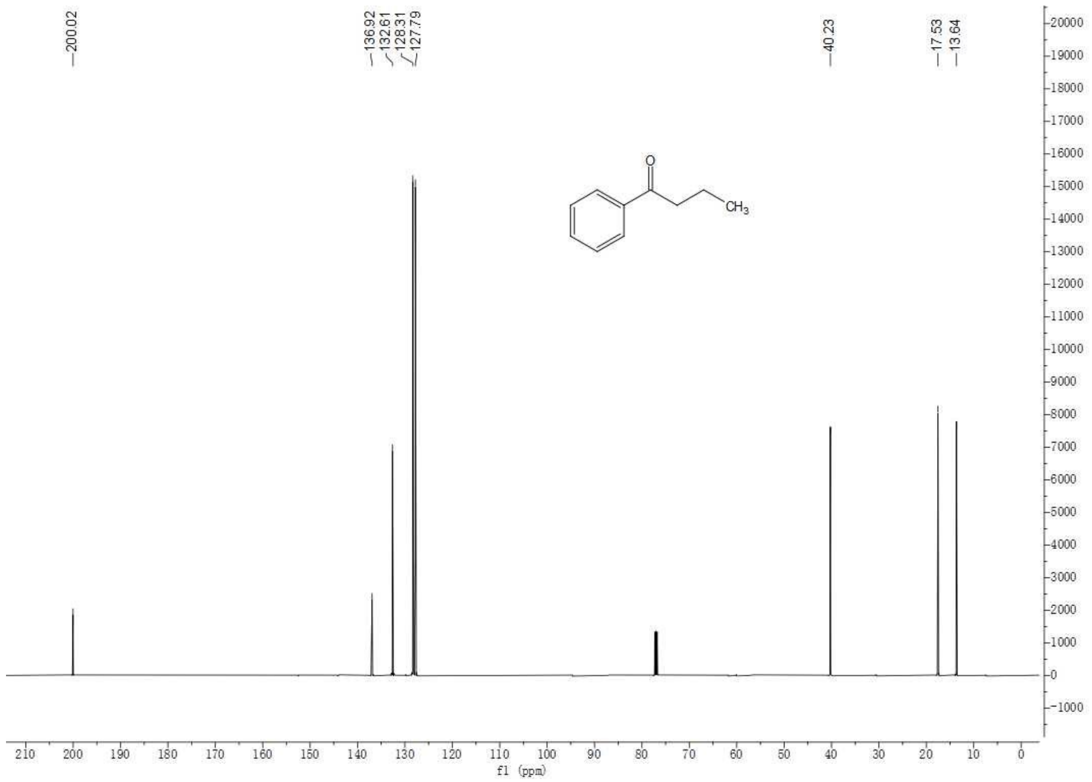
164.97
139.47
133.53
130.13
127.49
127.15
125.12
77.29
77.04
76.78
67.18
27.63
f1 (ppm)

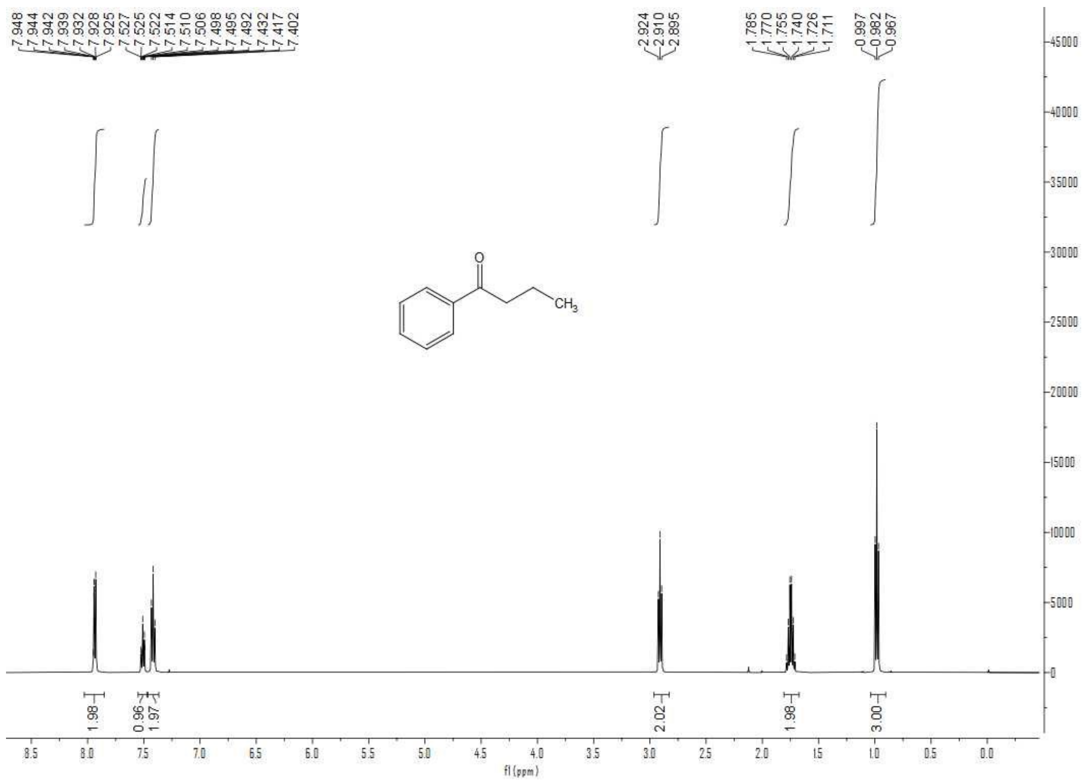
8.048
8.033
4.507
4.496
4.485
3.041
3.029
3.017
1.00
1.06
1.07
1.06
2.27
2.29

2i:

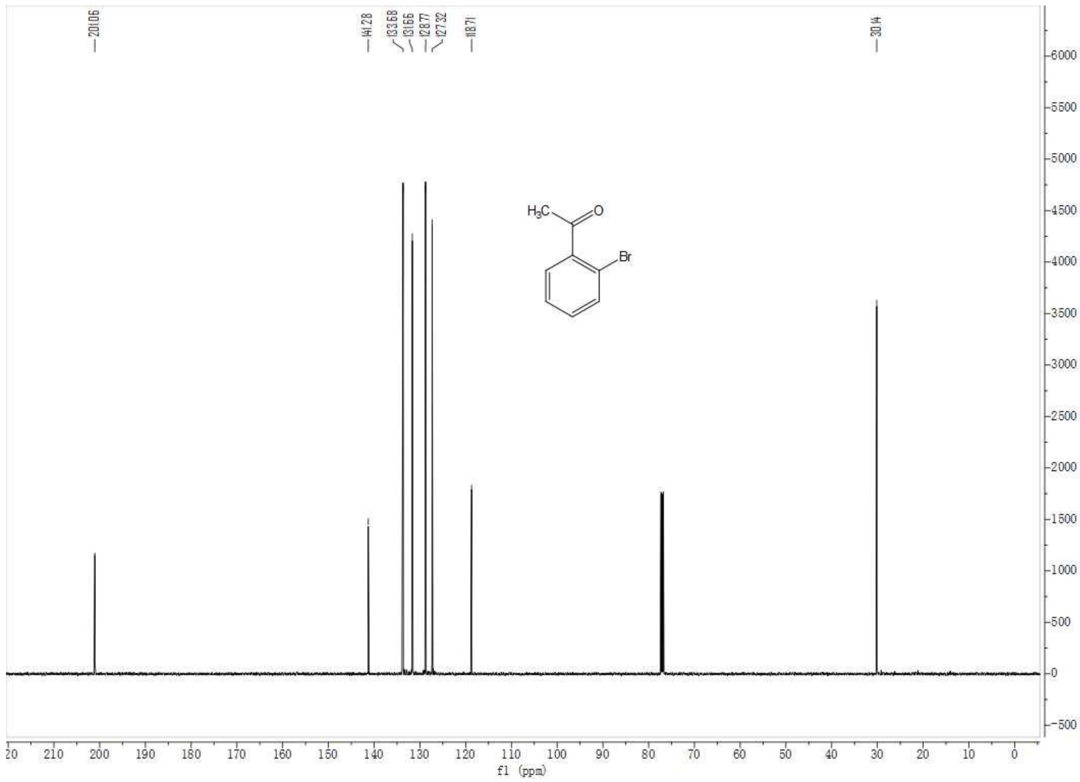


2j:

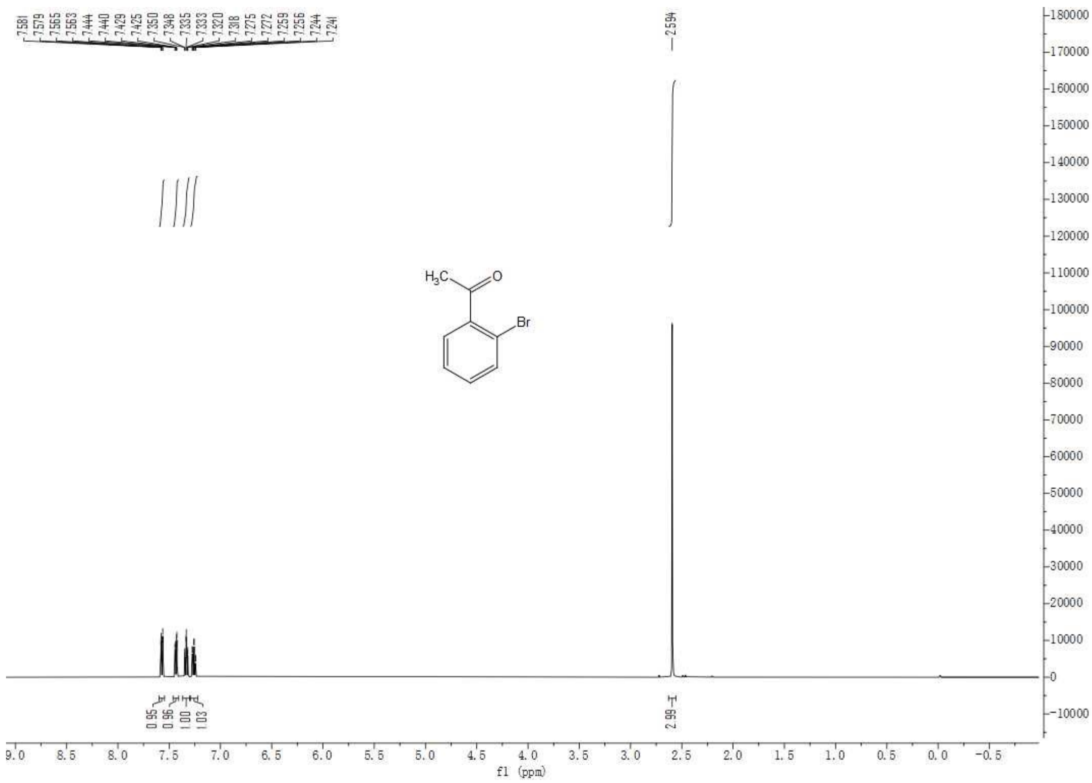




2k:

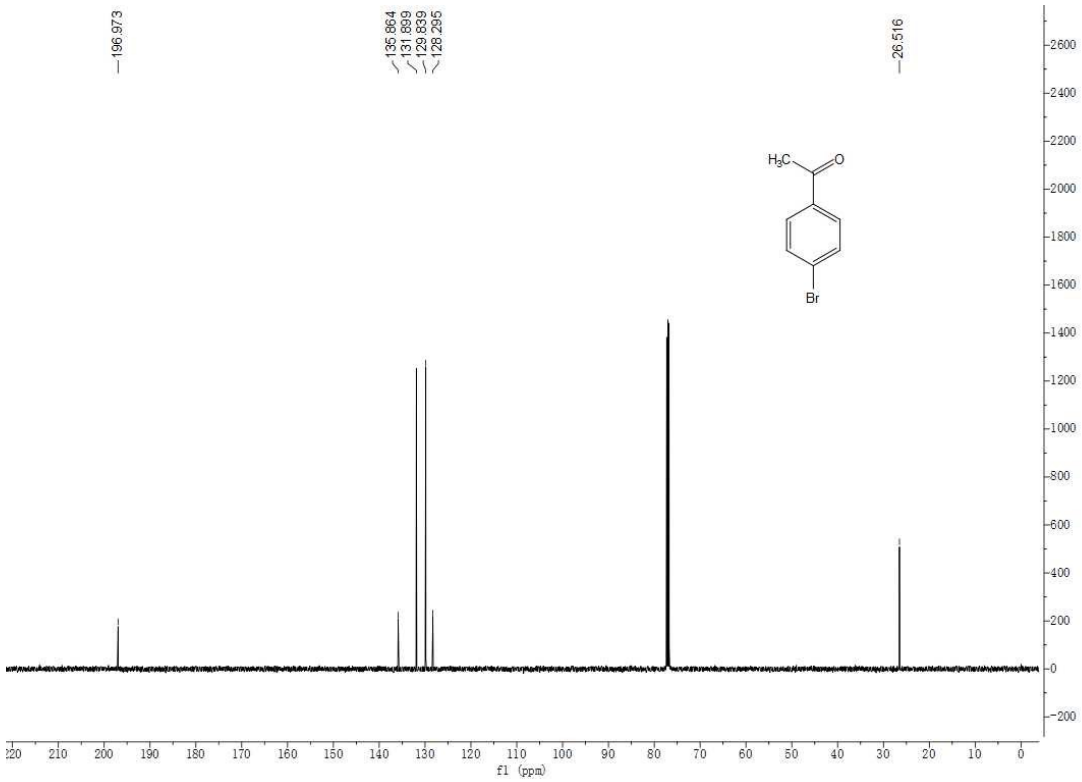


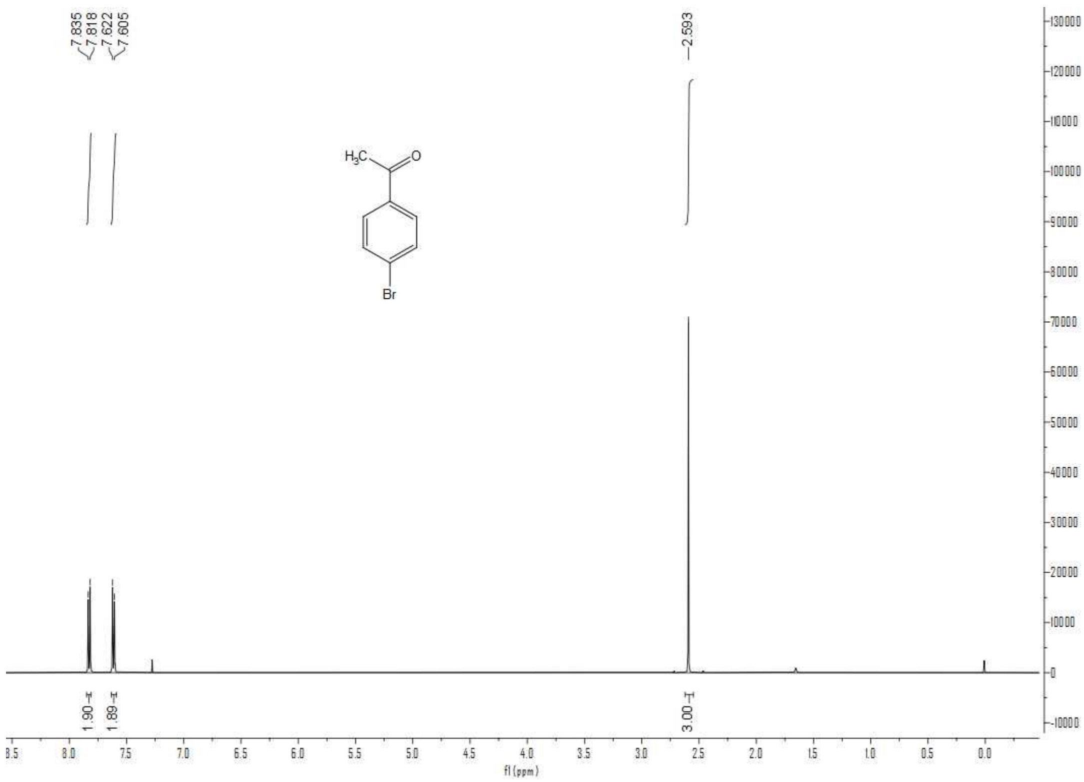
201.06
141.28
133.68
131.66
128.77
127.32
118.71
30.14
H_3C
O
Br
fl (ppm)



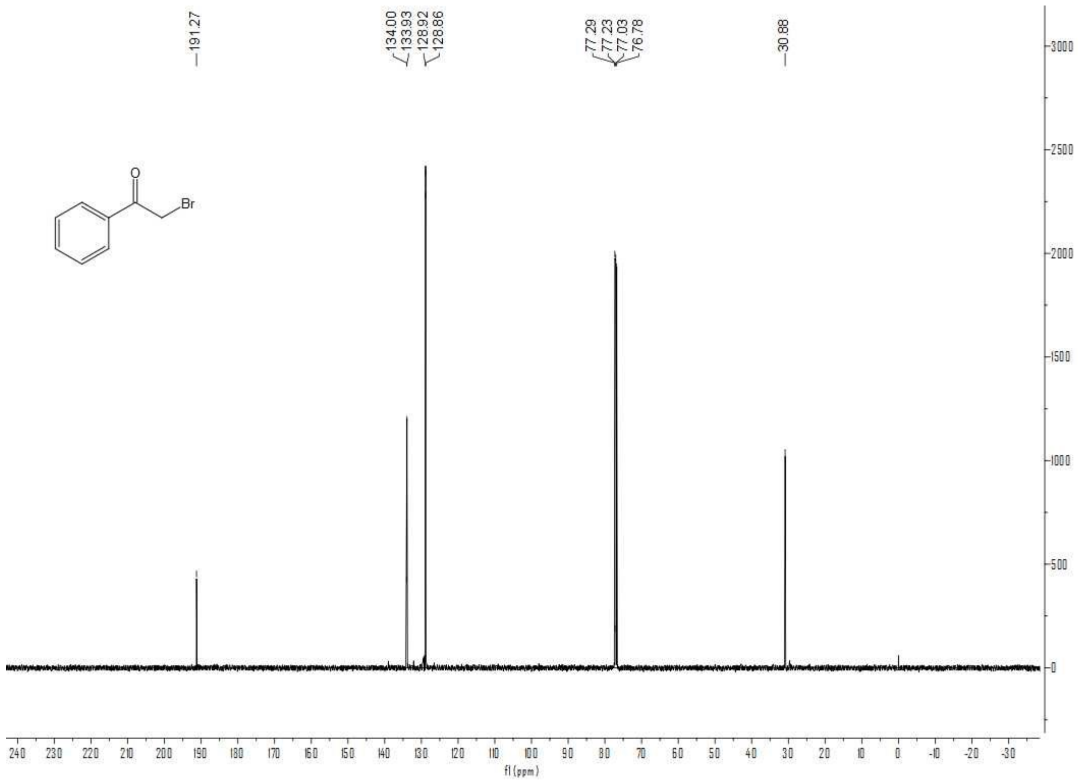
2.594
H_3C
O
Br
0.95
0.96
1.00
1.03
2.99
fl (ppm)

2l:

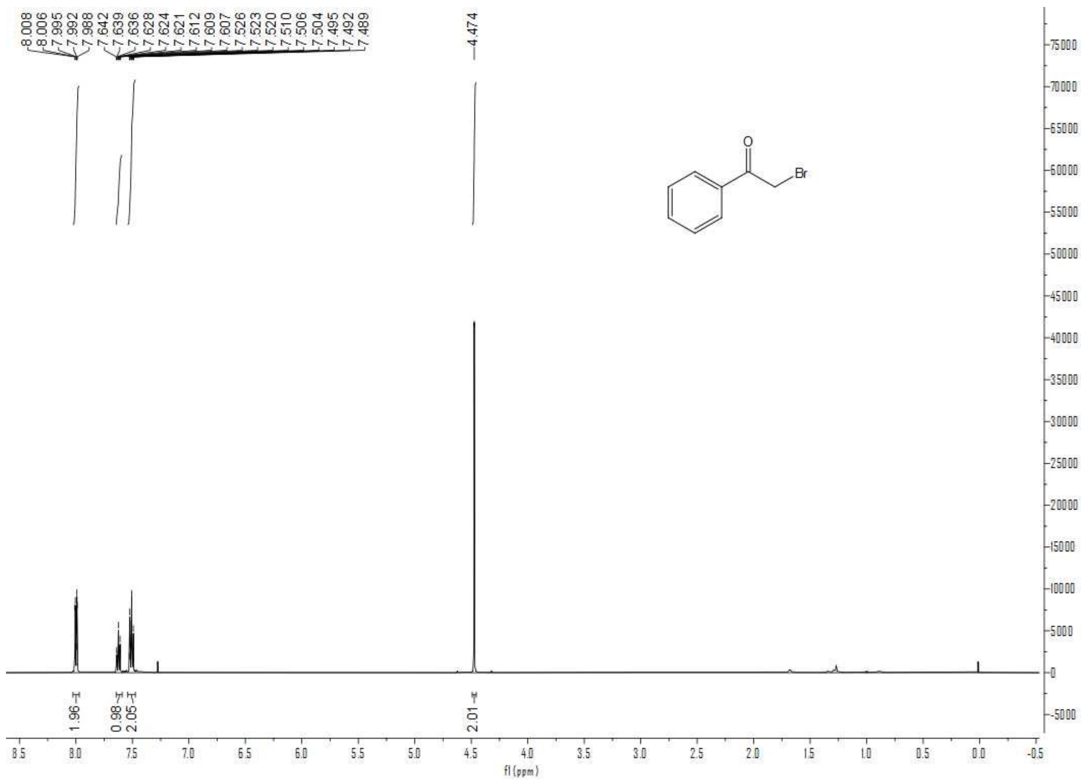

196.973
135.864
131.899
129.839
128.295
26.516
H3C
O
Br
f1 (ppm)


7.835
7.818
7.622
7.605
2.593
H3C
O
Br
1.90
1.89
3.00
f1 (ppm)

2m:

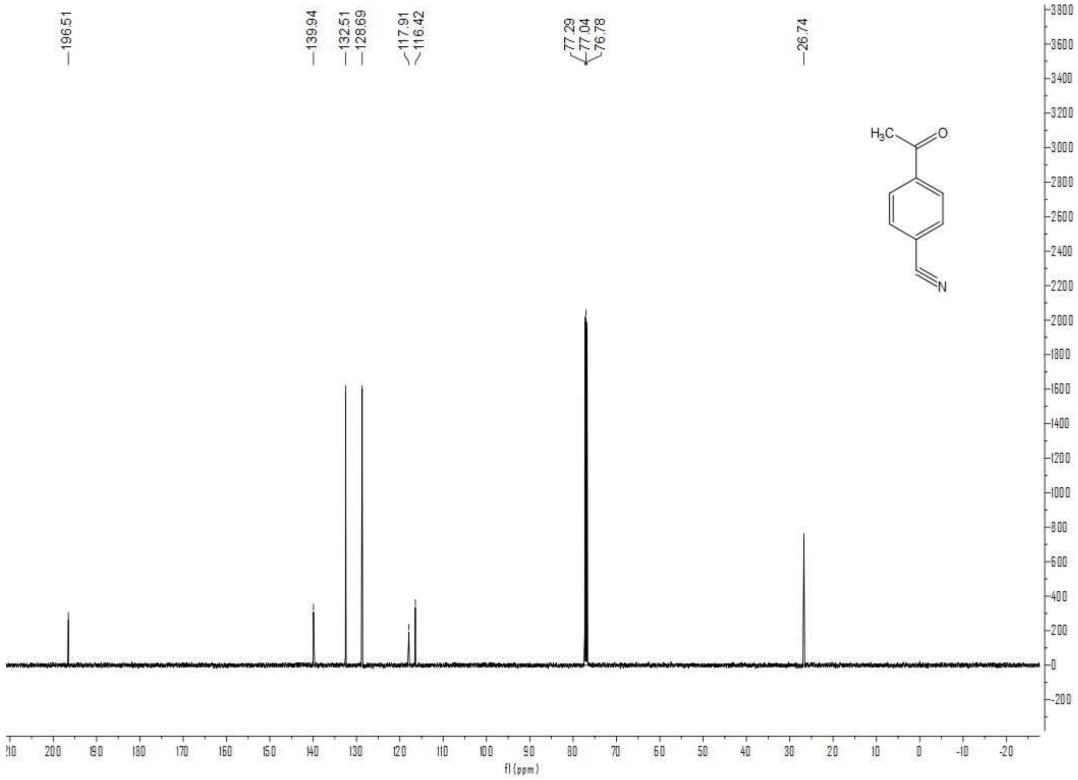


191.27
134.00
133.93
128.92
128.86
77.29
77.23
77.03
76.78
30.88
Br
O
f1 (ppm)

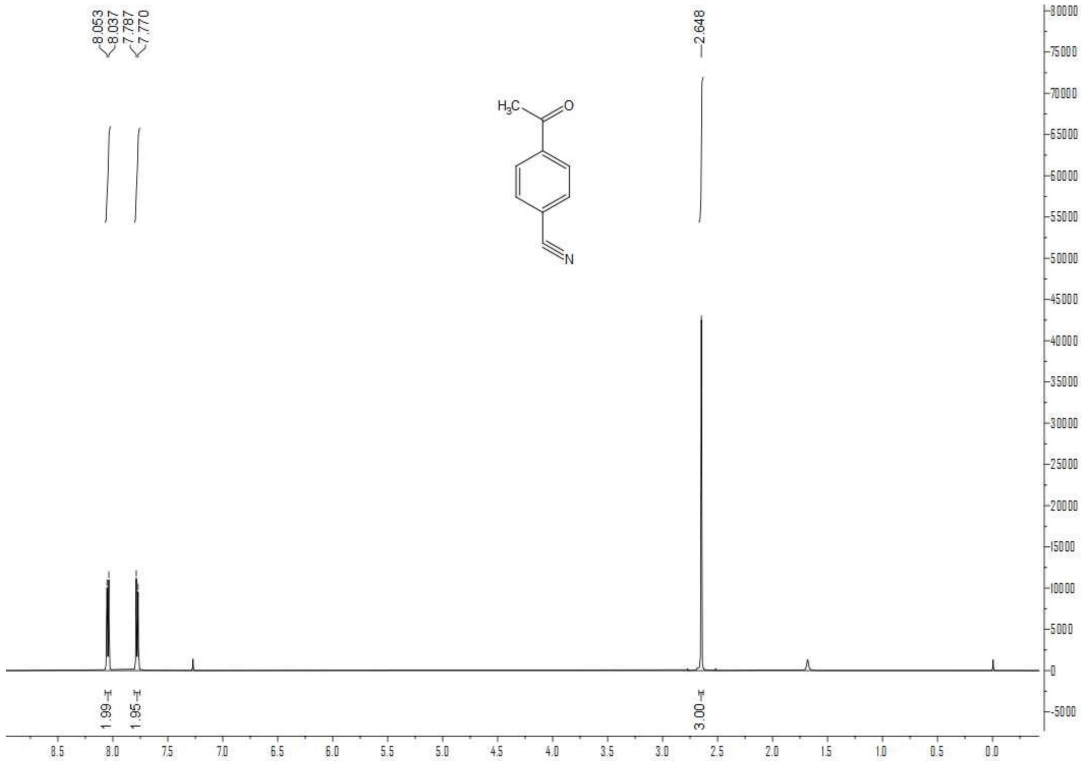


8.008
8.006
7.995
7.992
7.988
7.642
7.639
7.636
7.628
7.624
7.621
7.612
7.609
7.607
7.526
7.523
7.520
7.510
7.506
7.504
7.495
7.492
7.489
4.474
O
Br
1.96
0.98
2.05
2.01
f1 (ppm)

2n:

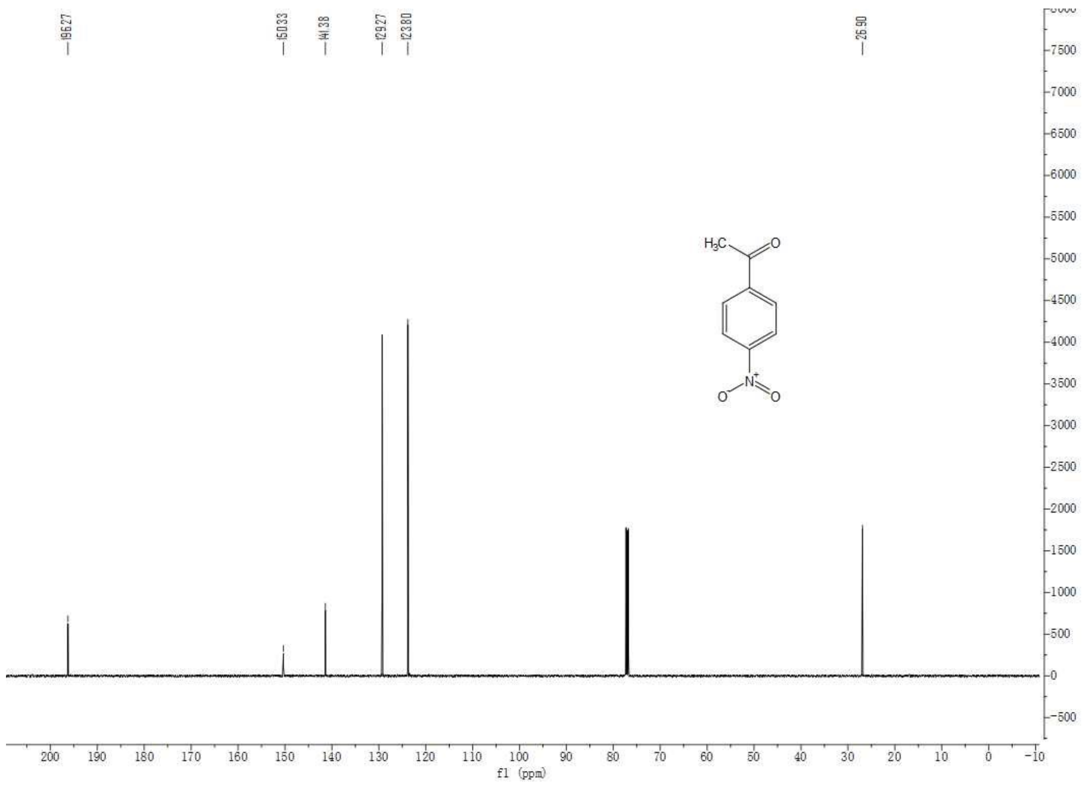


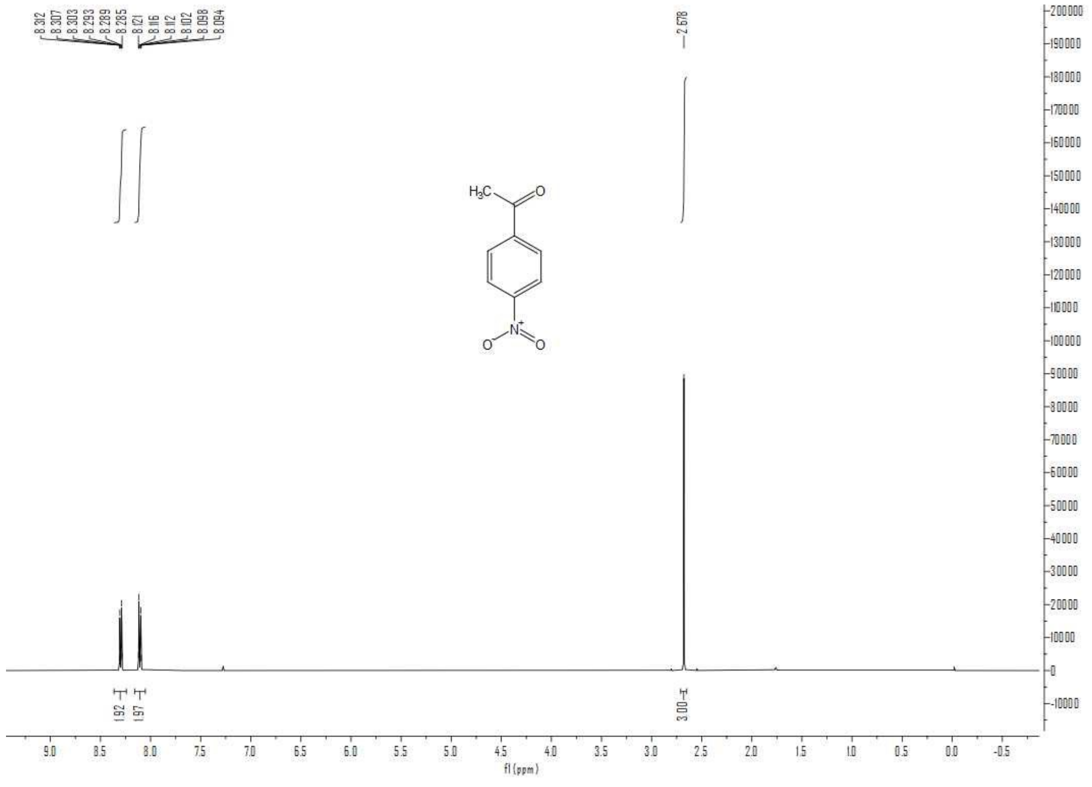
196.51
139.94
132.51
128.69
117.91
116.42
77.29
77.04
76.78
26.74
H3C
O
N
f1 (ppm)



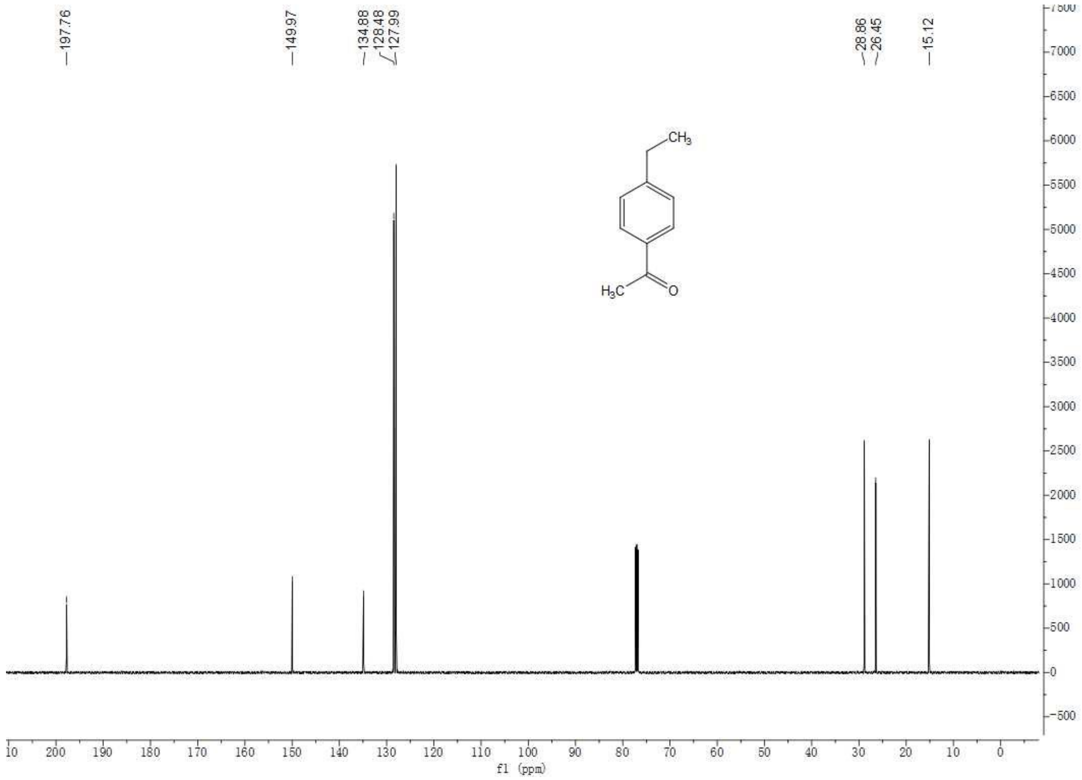
8.053
8.037
7.787
7.770
2.648
H3C
O
N
1.99
1.95
3.00
f1 (ppm)

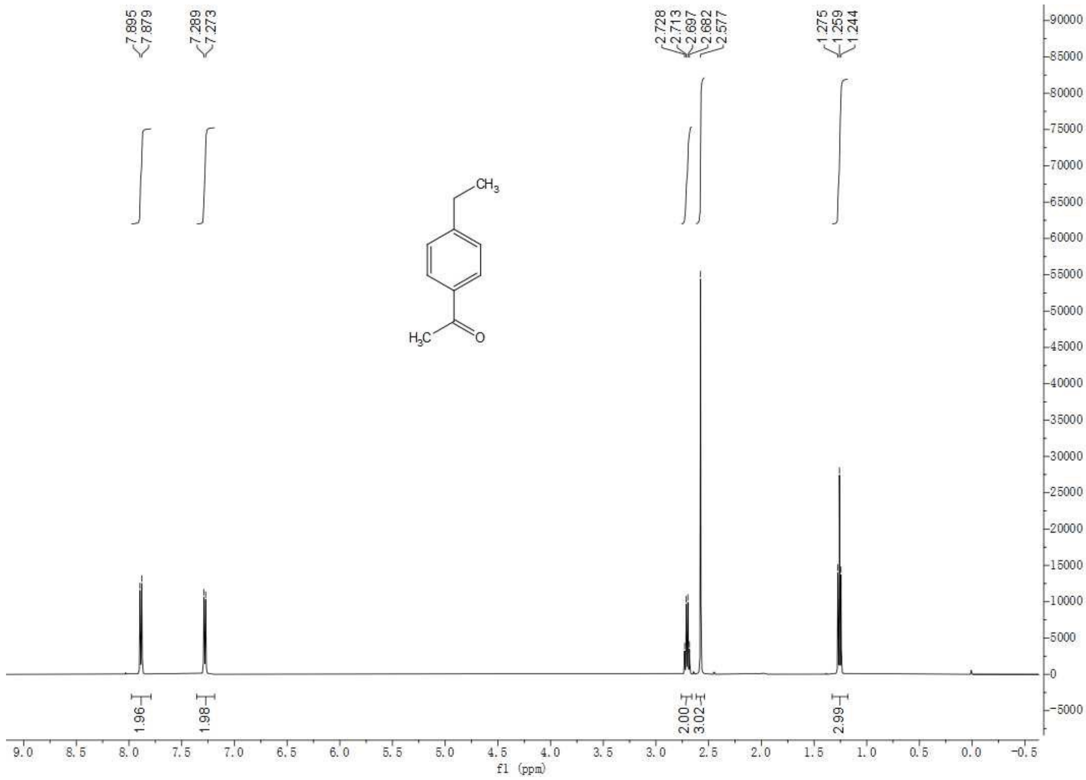
2o:



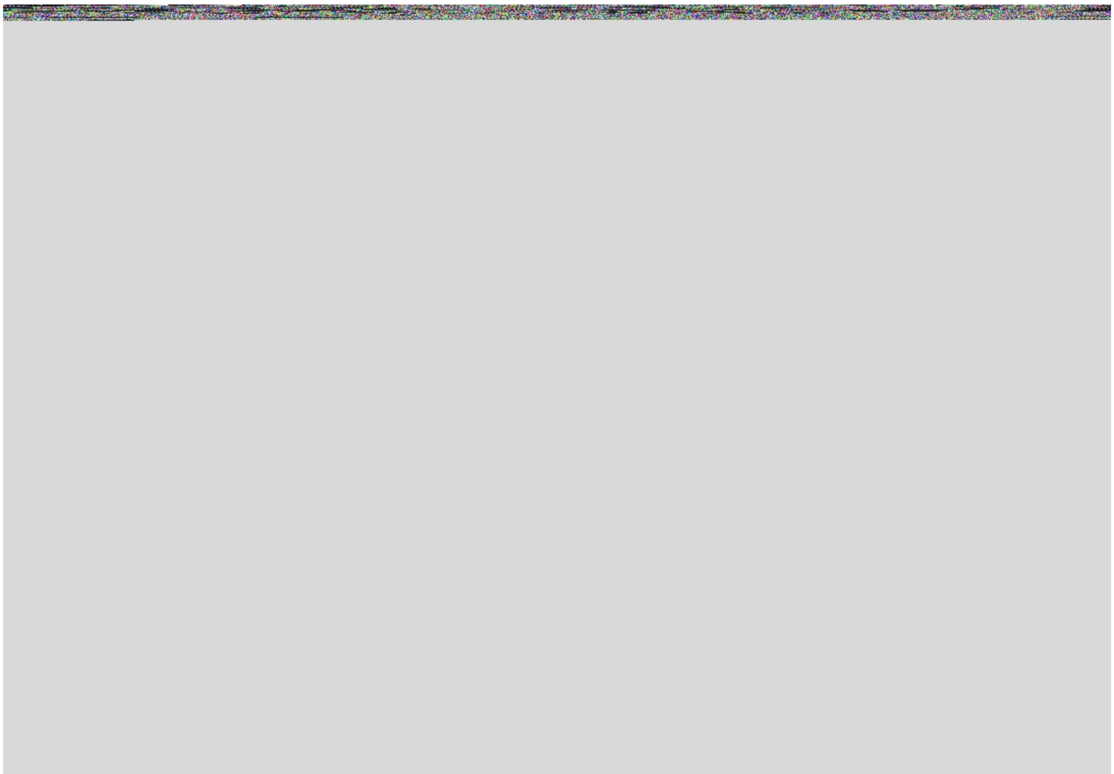


2p:

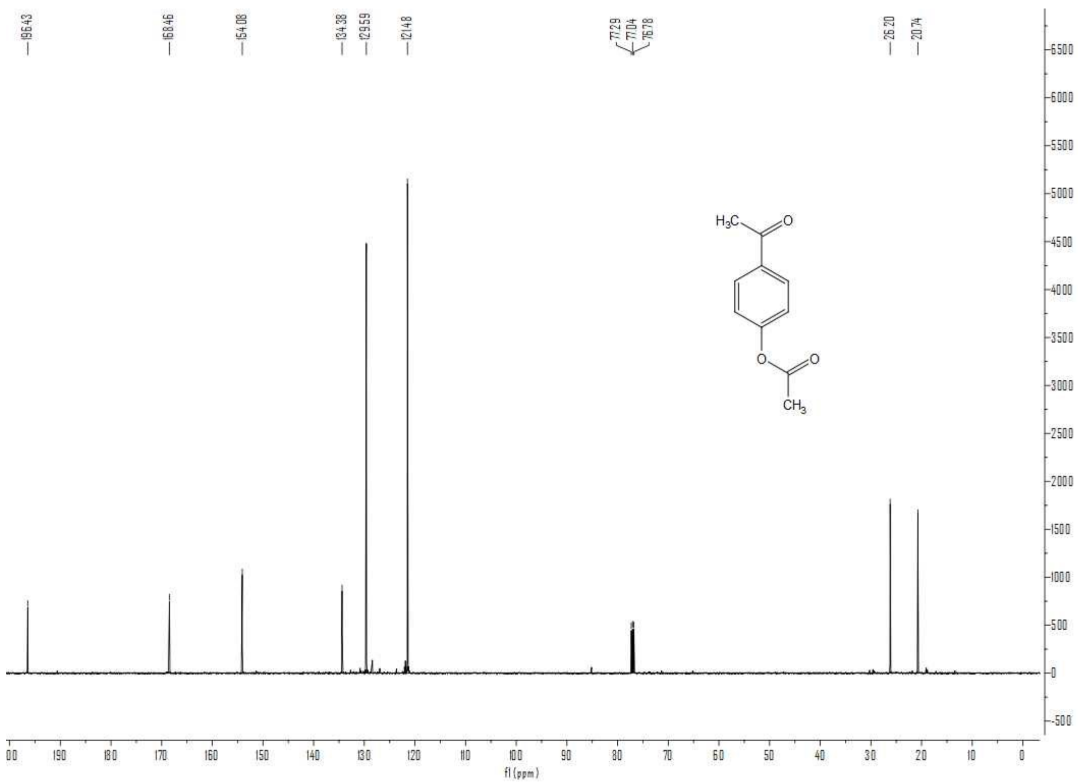

197.76
149.97
134.88
128.48
127.99
28.86
26.45
15.12
CH3
H3C
O
f1 (ppm)

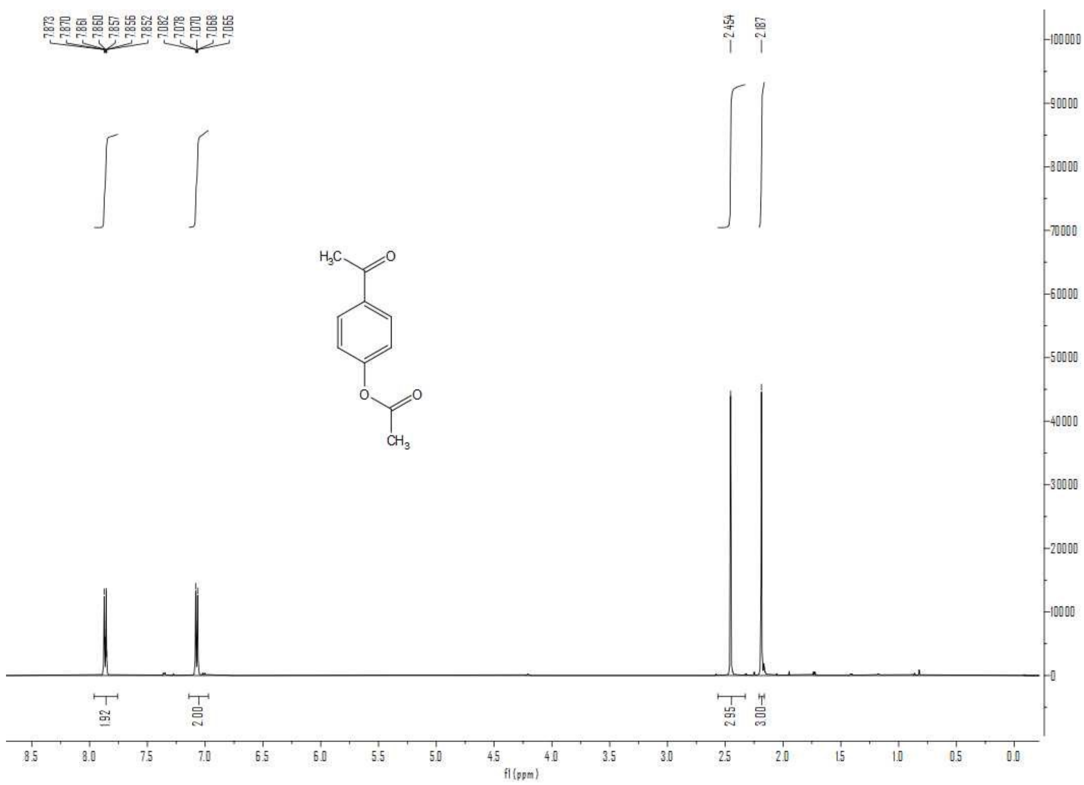

7.895
7.879
7.289
7.273
2.728
2.713
2.697
2.682
2.577
1.275
1.259
1.244
CH3
H3C
O
1.96
1.98
2.00
3.02
2.99
f1 (ppm)

2q:

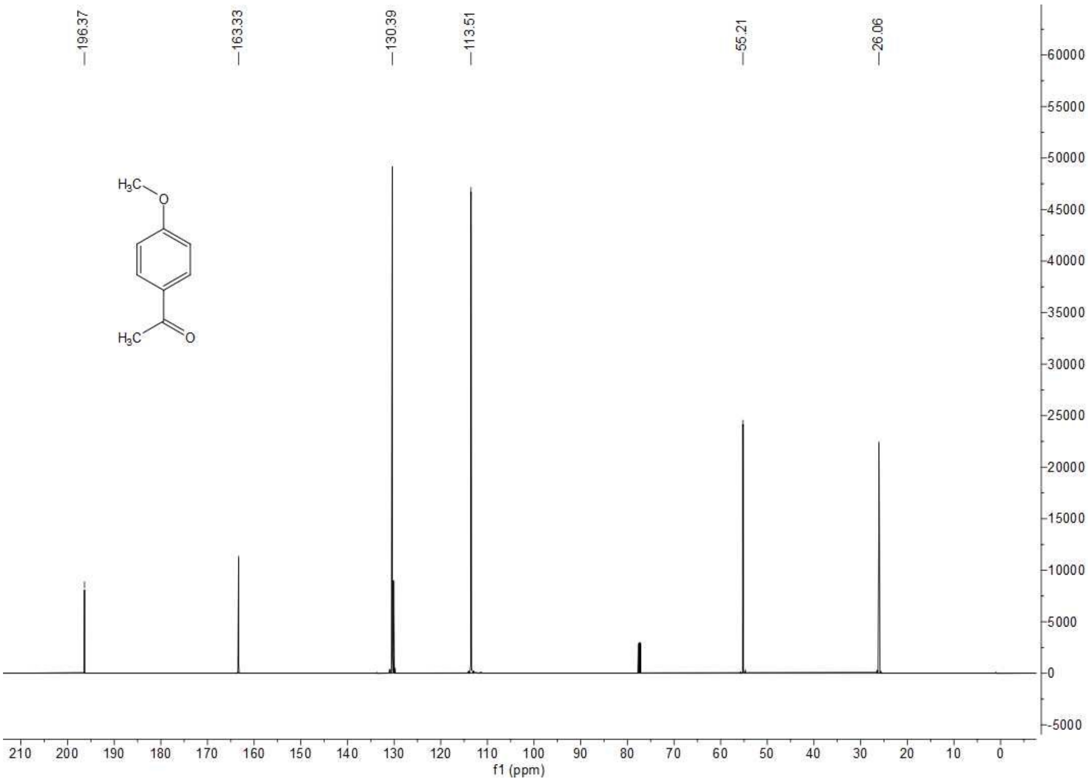


2r:





2s:



196.37
163.33
130.39
113.51
55.21
26.06
f1 (ppm)

7.752 7.747 7.744 7.740 7.736 7.732 7.728 7.725 7.721
6.748 6.743 6.739 6.736 6.732 6.728 6.724 6.721 6.717
3.657 3.653 3.650 3.645
2.353 2.349 2.347 2.342
2.00
2.03
3.08
3.12
f1 (ppm)

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