A General Photoinduced Oxidative Strategy with Molecular Oxygen in Water

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Supporting Information

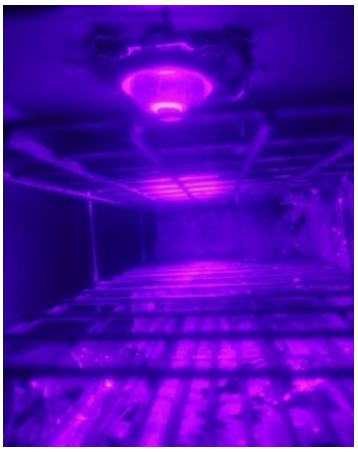
1. General Experiments

NMR spectra were recorded on an Agilent-NMR-VNMRs 400 MHz spectrometer or Bruker Advance 400 MHz spectrometer. Chemical shifts are reported in parts per million (ppm) and referenced to $CDCl_3$ (7.26 ppm) for ^1H NMR, and $CDCl_3$ (77.16 ppm) for ^{13}C NMR. Chemical shifts are reported in parts per million (ppm) and referenced to DMSO-d_6 (2.50 ppm) for ^1H NMR, and DMSO-d_6 (39.25 ppm) for ^{13}C NMR. GC-MS analyses were performed with an Agilent 8890-597BGCMSD spectrometer. UV–visible spectrum was recorded by a Shanghai Yidian 752N UV–visible spectrophotometer. The fluorescence was studied with a HORIBA FluoroMax-4 spectrophotometer. The color of the solution was taken by a cell phone Huawei Nova 6. The Column chromatography or preparative thin-layer chromatography (TLC) was performed with Qing Dao silica gel. All reagents and solvents were used directly as purchased.

2. Supplement Figures and Tables

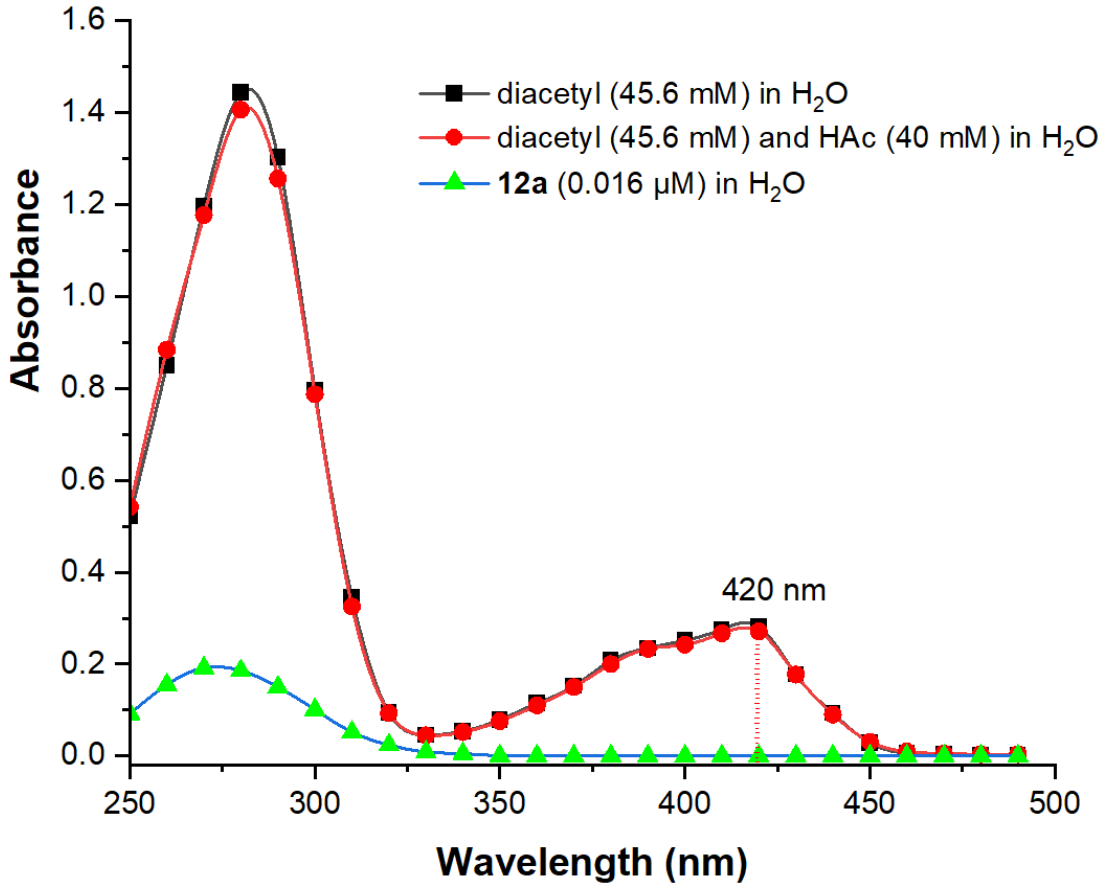
Figure S1. Supplement Figures

(a) Photo-reactors placed on the top middle of quartz tubes



(b) LED lamps (420 nm, 20 W, Taiwan Guanghong, EP-U4545k-A3)

(c) UV-visible spectra of diacetyl and **12a**



(d) Fluorescent spectra of diacetyl and **1a**

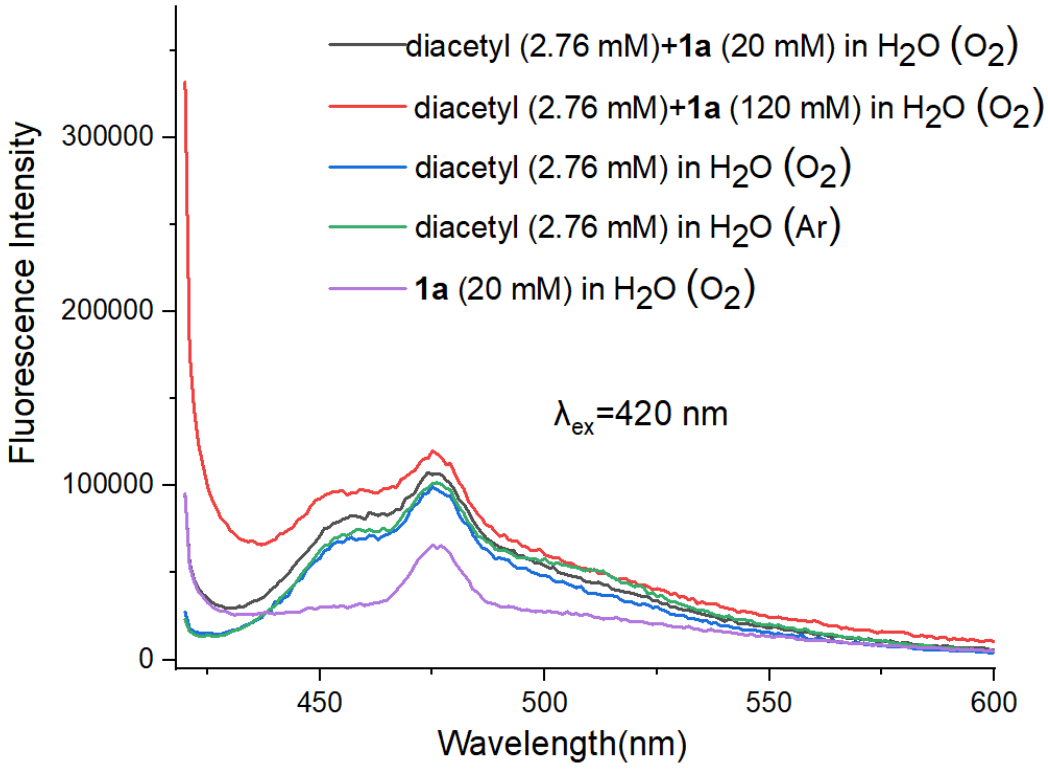
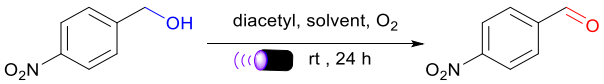


Table S1. Optimization of conditions for the oxidation of alcohol **12a**.[a]

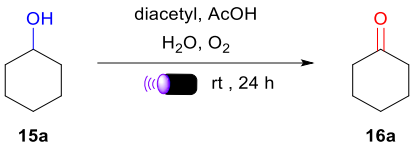


12a **13a**

entry	diacetyl/equiv	solvent	yield/%
1	-	H_2O	0
2	9	H_2O	0[b]
3	9	H_2O	0[c]
4	9	$CHCl_3$	37
5	9	MeCN	29
6	9	MeOH	37
7	9	THF	0
8	9	DMSO	43
9	9	DMF	31
10	9	acetone	53
11	1	H_2O	50
12	2	H_2O	62
13	3	H_2O	68
14	**4**	**H_2O**	**83**
15	5	H_2O	59
16	6	H_2O	57

[a] Reaction conditions: **12a** (0.15 mmol), diacetyl, solvent (0.6 mL, the reaction mixture was bubbled with O_2 for 15 min), purple LEDs (20 W), 24 h, isolated yield after column chromatography. [b] Under argon atmosphere. [c] In the dark.

Table S2. Optimization of conditions for the oxidation of alcohol **15a**.[a]



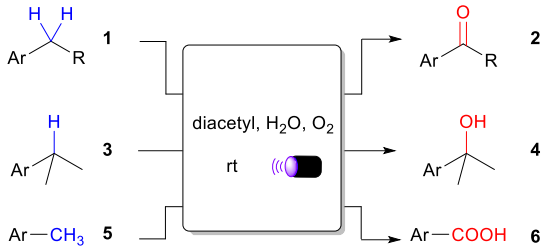
15a **16a**

entry	diacetyl/equiv	AcOH/equiv	yield/%
1	2	16.7	41
2	3	16.7	43
3	4	16.7	52
4	5	16.7	53
5	6	16.7	53
6	7	16.7	59
7	**8**	**16.7**	**96**
8	9	16.7	92
9	9	16.7	0[b]
10	9	16.7	0[c]
11	-	16.7	0
12	8	-	36
13	8	0.25	58
14	8	0.5	64
15	8	0.8	73
16	8	1	71
17	8	2	51
18	8	3	51
19	8	4	80
20	**8**	**5**	**96**
21	8	7	92
22	8	8	88
23	8	10	84
24	8	12	44

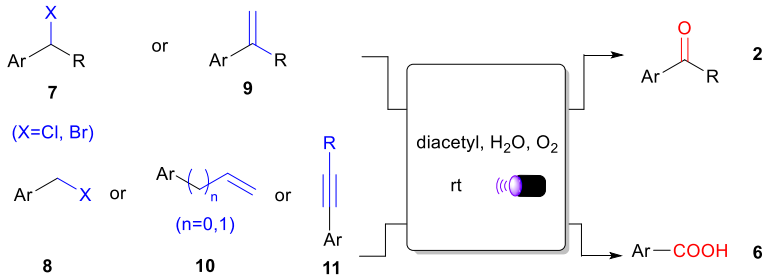
[a] Reaction conditions: **15a** (0.15 mmol), diacetyl, H_2O (0.6 mL, the reaction mixture was bubbled with O_2 for 15 min), purple LEDs (20 W), 24 h, GC-MS yield with 1,3,5-trimethylbenzene as internal standard. [b] Under argon atmosphere. [c] In the dark.

3. Experimental procedure

(1) General procedure for the oxidation of C–H bond, alkenes, alkynes, or halides



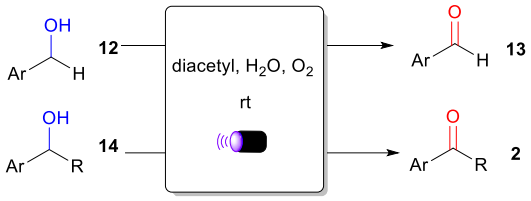
To a 10 mL quartz tube charged with a magnetic stir bar was added **1**/**3**/**5** (0.15 mmol), diacetyl (0.45 mmol, 3 equiv, 39.5 μL), and H_2O (0.6 mL). The mixture was bubbled with O_2 for 15 min and then irradiated by purple LEDs (420 nm, 20 W) with vigorous stirring at room temperature. After 24-36 h, the solution was extracted with EtOAc (3 mL×3). The combined organic layers were washed with brine (2 mL), dried with anhydrous Na_2SO_4, and purified by column chromatography with petroleum ether/EtOAc (100:0−2:1) to afford products **2**/**4**/**6** in yields as indicated in Table 2.



A similar procedure was carried out for the oxidation of **7**/**8**/**9**/**10**/**11** (0.15 mmol) to afford products **2**/**6** after 36-48 h in yields as indicated in Table 3.

A similar procedure was carried out for the scale-up oxidation of **1a** (8 mmol) to afford 0.851 g of **2a** in a yield of 53% (Scheme 2).

(2) General procedure for the oxidation of benzylic alcohols

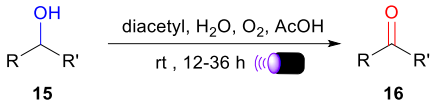


To a 10 mL quartz tube charged with a magnetic stir bar was added **12** or **14** (0.15 mmol), diacetyl (0.6 mmol, 4 equiv, 52 μL), and H_2O (0.6 mL). The mixture was bubbled with O_2 for 15 min and then irradiated

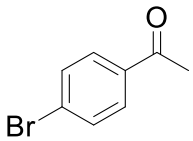
by purple LEDs (420 nm, 20 W) with vigorous stirring at room temperature. After 12-36 h, the solution was extracted with EtOAc (3 mL×3). The combined organic layers were washed with brine (2 mL), dried with anhydrous Na_2SO_4, and purified by column chromatography with petroleum ether/EtOAc (100:0−2:1) to afford products **13** or **2** in yields as indicated in Table 4.

A similar procedure was carried out for the scale-up oxidation of **14b/14d/14x** (7 mmol/6.5 mmol/5.0 mmol) to afford corresponding products in yields as indicated in Scheme 2.

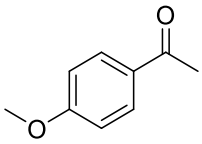
(3) General procedure for the oxidation of aliphatic alcohols



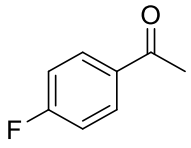
To a 10 mL quartz tube charged with a magnetic stir bar was added **15** (0.15 mmol), diacetyl (1.2 mmol, 8 equiv, 105 μL), H_2O (0.6 mL) and CH_3COOH (0.75 mmol, 5 equiv). The mixture was bubbled with O_2 for 15 min and then irradiated by purple LEDs (420 nm, 20 W) with vigorous stirring at room temperature. After 12-36 h, the solution was extracted with EtOAc (3 mL×3). The combined organic layers were washed with brine (2 mL), dried with anhydrous Na_2SO_4, and purified by column chromatography with petroleum ether/EtOAc (100:0−2:1) to afford products **16** in yields as indicated in Table 4.

1-(4-Bromophenyl)ethan-1-one (**2a**) 

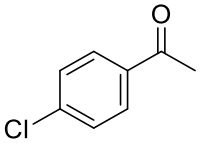
Yield 81%, 24.2 mg from **1a** in Table 1 (24 h), or yield 89%, 26.7 mg from **14a** in Table 4 (12 h), or yield 53%, 851 mg from **1a** (5 mmol) in Scheme 2 (48 h), white solid, m.p. 78.1 – 80.2℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, J = 8.6 Hz, 2H), 7.57 (d, J = 8.6 Hz, 2H), 2.56 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.1, 135.8, 131.9, 129.9, 128.4, 26.6.[1]

1-(4-Methoxyphenyl)ethan-1-one (**2b**) 

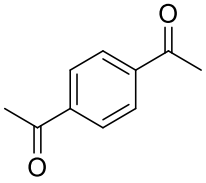
Yield 69%, 15.6 mg from **1b** in Table 2 (36 h), or yield 85%, 19.4 mg from **14b** in Table 4 (24 h), or yield 86%, 899 mg from **14b** (7 mmol) in Scheme 2 (12 h), white solid, m.p. 36.2 – 37.5 ºC. **¹**H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.9 Hz, 2H), 6.91 (d, J = 8.9 Hz, 2H), 3.84 (s, 3H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 163.5, 130.6, 130.3, 113.7, 55.5, 26.4.[1]

1-(4-Fluorophenyl)ethan-1-one (**2c**) 

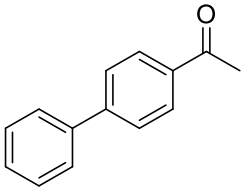
Yield 81%, 16.8 mg from **1c** in Table 2 (36 h, 2.1 mg, 11% of **1c** was recovered), or yield 69%, 14.2 mg from **9a** in Table 3 (36 h), or yield 89%, 18.5 mg from **14c** in Table 4 (24 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) 7.97 (dd, J = 8.8, 5.4 Hz, 2H), 7.13 (t, J = 8.9 Hz, 2H), 2.58 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) 196.7, 165.8 (d, J = 254.7 Hz), 133.6, 131.1 (d, J = 9.4 Hz), 115.8 (d, J = 22.0 Hz), 26.7.[1]

1-(4-Chlorophenyl)ethan-1-one (**2d**) 

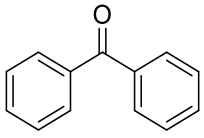
Yield 86%, 20.1 mg from **1d** in Table 2 (36 h), or yield 86%, 19.9 mg from **14d** in Table 4 (24 h), or yield 74%, 757 mg from **14d** (6.5 mmol) in Scheme 2 (24 h), white solid, m.p. 17.8 – 18.0 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (d, J = 8.8 Hz, 2H), 7.43 (d, J = 8.9 Hz, 2H), 2.59 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.0, 139.7, 135.5, 129.9, 129.0, 26.7.[2]

1,1'-(1,4-Phenylene)bis(ethan-1-one) (**2e**) 

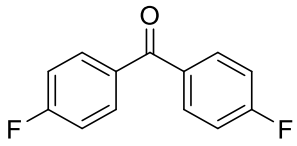
Yield 53%, 12.5 mg from **1e** in Table 2 (36 h, 7.8 mg, 35% of **1e** was recovered), white solid, m.p. 111.3 – 112.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (s, 4H), 2.65 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.7, 140.3, 128.6, 27.1.[2]

1-([1,1'-Biphenyl]-4-yl)ethan-1-one (**2f**) 

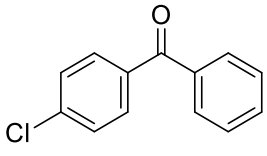
Yield 51%, 15.2 mg from **1f** in Table 2 (36 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.5 Hz, 2H), 7.63 (d, J = 6.9 Hz, 2H), 7.48 (t, J = 8.0 Hz, 2H), 7.42 (t, J = 7.0 Hz, 1H), 2.64 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.9, 145.8, 139.9, 135.9, 129.0, 129.0, 128.3, 127.4, 127.3, 26.8.[2]

Benzophenone (**2g**) 

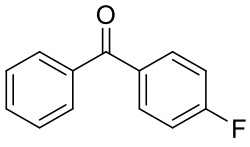
Yield 78%, 21.4 mg from **1g** in Table 2 (36 h), or yield 90%, 24.5 mg from **7a** in Table 3 (36 h), or yield 87%, 23.8 mg from **7b** in Table 3 (36 h), or yield 92%, 25.0 mg from **9b** in Table 3 (36 h), or yield 83%, 22.7 mg from **14g** in Table 4 (24 h), white solid, m.p. 47.8 – 48.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) 7.81 (d, J = 7.0 Hz, 4H), 7.64 – 7.56 (m, 2H), 7.49 (t, J = 7.5 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.9, 137.7, 132.6, 130.2, 128.4.[1]

Bis(4-fluorophenyl)methanone (**2h**) 

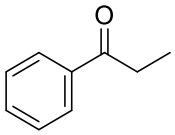
Yield 89%, 29 mg from **1h** in Table 2 (36 h), or yield 93%, 30.6 mg from **7d** in Table 3 (36 h), or yield 91%, 29.9 mg from **14h** in Table 4 (24 h), white solid, m.p. 104.1 – 104.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.79 (m, 4H), 7.17 (t, J = 8.6 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.0, 165.5 (d, J = 254.4 Hz), 133.8 (d, J = 3.1 Hz), 132.6 (d, J = 9.2 Hz), 115.7 (d, J = 21.9 Hz).[1]

(4-Chlorophenyl)(phenyl)methanone (**2i**) 

Yield 90%, 29.4 mg from **1i** in Table 2 (36 h), or yield 93%, 30.2 mg from **7c** in Table 3 (36 h), white solid, m.p. 80.0 – 82.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.71 (m, 4H), 7.59 (t, J = 7.4 Hz, 1H), 7.52 – 7.42 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.6, 138.9, 137.3, 135.9, 132.7, 131.6, 130.0, 128.7, 128.5.[1]

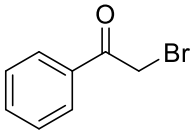
(4-Fluorophenyl)(phenyl)methanone (**2j**) 

Yield 94%, 28.4 mg from **1j** in Table 2 (36 h), white solid, m.p. 46.2 – 48.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.71 (m, 4H), 7.62 – 7.56 (m, 1H), 7.52 – 7.41 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.4, 165.5 (d, J = 254.1 Hz), 137.6, 133.9 (d, J = 3.0 Hz), 132.8 (d, J = 9.2 Hz), 132.6, 130.0, 128.5, 115.6 (d, J = 21.8 Hz).[1]

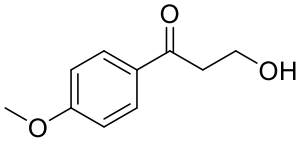
Propiophenone (**2k**) 

Yield 78%, 16.3 mg from **1k** in Table 2 (36 h), or yield 90%, 18.1 mg from **14k** in Table 4 (24 h), yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, J = 7.1 Hz, 2H), 7.54 (d, J = 7.3 Hz, 1H), 7.46 (t, J = 7.5 Hz,

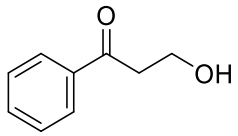
2H), 3.01 (q, *J* = 7.2 Hz, 2H), 1.23 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.0, 137.0, 133.0, 128.7, 128.1, 31.9, 8.4.[1]

2-Bromo-1-phenylethan-1-one (**2l**) 

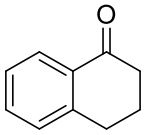
Yield 61%, 18.1 mg from **1l** in Table 2 (36 h), white solid, m.p. 49.6 – 50.7 °C. **1**H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.0 Hz, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 4.46 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.3, 134.0, 133.9, 129.0, 128.9, 31.2.[2]

3-*H*ydroxy-1-(4-methoxyphenyl) propan-1-one (**2m**) 

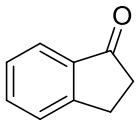
Yield 57%, 15.3 mg from **1m** in Table 2 (36 h using 0.60 mmol diacetyl), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) 7.94 (d, *J* = 8.9 Hz, 2H), 6.94 (d, *J* = 8.9 Hz, 2H), 4.01 (t, *J* = 5.3 Hz, 2H), 3.87 (s, 3H), 3.18 (t, *J* = 5.3 Hz, 2H), 2.51 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.3, 163.9, 130.5, 129.8, 113.9, 58.4, 55.6, 40.0.[3]

3-*H*ydroxy-1-phenylpropan-1-one (**2n**) 

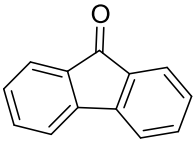
Yield 32%, 7.1 mg from **1n** in Table 2 (36 h), colorless liquid (11.3 mg **1n** was recovered). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 6.9 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 2H), 4.04 (t, *J* = 5.3 Hz, 2H), 3.24 (t, *J* = 5.3 Hz, 2H), 2.40 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.7, 136.7, 133.7, 128.8, 128.2, 58.2, 40.5.[2]

3,4-Dihydronaphthalen-1(2H)-one (**2o**) 

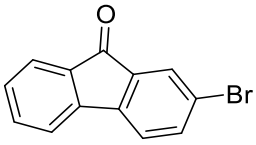
Yield 87%, 19 mg from **1o** in Table 2 (36 h), or yield 79%, 17.4 mg from **14o** in Table 4 (24 h), yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.8 Hz, 1H), 7.47 (t, *J* = 7.5 Hz, 1H), 7.29 (d, *J* = 7.0 Hz, 1H), 7.25 (d, *J* = 7.0 Hz, 1H), 2.97 (t, *J* = 6.1 Hz, 2H), 2.69 – 2.61 (m, 2H), 2.17 – 2.11 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.6, 144.6, 133.5, 132.7, 128.9, 127.3, 126.8, 39.3, 29.8, 23.4.[1]

2,3-Dihydro-1*H*-inden-1-one (**2p**) 

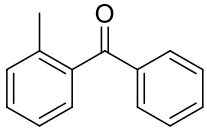
Yield 80%, 15.8 mg from **1p** in Table 2 (36 h), or yield 73%, 14.4 mg from **14p** in Table 4 (24 h), yellowish-brown solid, m.p. 39.0 – 40.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, J = 7.6 Hz, 1H), 7.61 – 7.55 (m, 1H), 7.48 (d, J = 7.7 Hz, 1H), 7.39 – 7.34 (m, 1H), 3.17 – 3.12 (m, 2H), 2.72 – 2.66 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 207.3, 155.3, 137.2, 134.7, 127.4, 126.8, 123.8, 36.3, 25.9.[4]

9*H*-Fluoren-9-one (**2q**) 

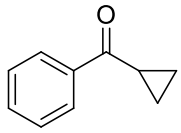
Yield 42%, 11.3 mg from **1q** in Table 2 (36 h in the solvent of CH_3CN, 11.3 mg, 42% of **1q** was recovered), or yield 44%, 11.9 mg from **7f** in Table 3 (36 h, 13.8 mg, 51% of **7f** was recovered), or yield 67%, 18.0 mg from **14q** in Table 4 (36 h), yellow solid, m.p. 83.0 – 83.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, J = 7.0 Hz, 2H), 7.50 – 7.44 (m, 4H), 7.31 – 7.25 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.9, 144.4, 134.7, 134.1, 129.1, 124.3, 120.3.[1]

2-Bromo-9*H*-fluoren-9-one (**2r**) 

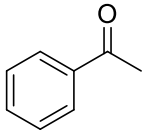
Yield 44%, 17.2 mg from **1r** in Table 2 (36 h in the solvent of CH_3CN), yellow solid, m.p. 147.1 – 148.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$) 7.69 (d, J = 1.9 Hz, 1H), 7.63 – 7.52 (m, 2H), 7.49 – 7.43 (m, 2H), 7.35 – 7.27 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 143.8, 143.1, 137.2, 135.8, 135.2, 133.8, 129.6, 127.7, 124.8, 123.0, 121.9, 120.6.[5]

1-Benzyl-2-methylbenzene (**2s**) 

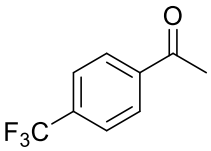
Yield 65%, 19.2 mg from **7e** in Table 3 (36 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.79 (m, 2H), 7.60 – 7.55 (m, 1H), 7.47 – 7.43 (m, 2H), 7.41 – 7.37(m, 1H), 7.33 – 7.22 (m, 3H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.7, 138.6, 137.8, 136.8, 133.2, 131.1, 130.3, 130.2, 128.6, 128.5, 125.3, 20.1.[4]

Cyclopropyl phenyl ketone (**2t**) 

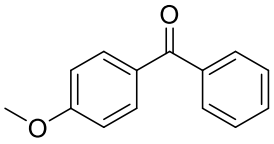
Yield 63%, 13.8 mg from **9c** in Table 3 (36 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 – 7.95 (m, 2H), 7.57 – 7.49 (m, 1H), 7.43 (t, J = 8.1 Hz, 2H), 2.67 – 2.61 (m, 1H), 1.23 – 1.20 (m, 2H), 1.04 – 0.98 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.4, 137.8, 132.6, 128.4, 127.9, 17.0, 11.5.[6]

Acetophenone (**2v**) 

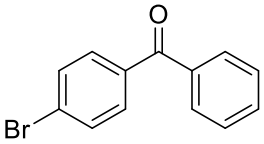
Yield 81%, 14.5 mg from **14v** in Table 4 (24 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J = 7.0 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.5 Hz, 2H), 2.61 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.4, 137.2, 133.3, 128.7, 128.4, 26.8.[1]

1-(4-(Trifluoromethyl)phenyl)ethan-1-one (**2w**) 

Yield 82%, 23.1 mg from **14w** in Table 4 (36 h), white solid, m.p. 30.5 – 31.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 8.7 Hz, 2H), 7.72 (d, J = 8.1 Hz, 2H), 2.64 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.2, 139.7, 134.5 (q, J = 32.8 Hz), 128.8, 125.7 (q, J = 3.7 Hz), 122.4 (q, J = 272.6 Hz), 26.9.[1]

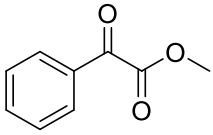
(4-Methoxyphenyl)(phenyl)methanone (**2x**) 

Yield 92%, 29.1 mg from **14x** in Table 4 (24 h), or yield 88%, 932.8 mg from **14x** (5 mmol) in Scheme 2 (24 h), white solid, m.p. 60.7 – 61.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 8.8 Hz, 2H), 7.76 (d, J = 6.9 Hz, 2H), 7.56 (d, J = 7.4 Hz, 1H), 7.47 (t, J = 7.5 Hz, 2H), 6.96 (d, J = 8.8 Hz, 2H), 3.89 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.7, 163.3, 138.4, 132.7, 132.0, 130.2, 129.9, 128.3, 113.6, 55.6.[1]

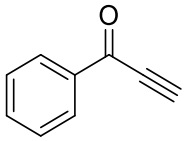
4-Bromobenzophenone (**2y**) 

Yield 81%, 31.8 mg from **14y** in Table 4 (24 h), white solid, m.p. 79.6 – 79.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, J = 6.9 Hz, 2H), 7.68 (d, J = 8.6 Hz, 2H), 7.64 (s, 2H), 7.60 (d, J = 7.5 Hz, 1H), 7.49 (t, J

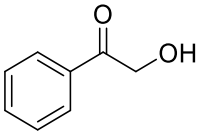
= 7.6 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.8, 137.2, 136.4, 132.8, 131.8, 131.7, 130.1, 128.6, 127.7.[7]

Methyl-2-oxo-2-phenylacetate (**2z**) 

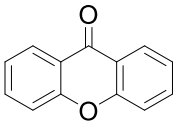
Yield 80%, 19.7 mg from **14z** in Table 4 (24 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, J = 6.6 Hz, 2H), 7.67 (t, J = 7.4 Hz, 1H), 7.53 (d, J = 8.2 Hz, 2H), 3.98 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.2, 164.2, 135.2, 132.5, 130.2, 129.0, 53.0.[8]

1-Phenyl-2-propyn-1-one (**2aa**) 

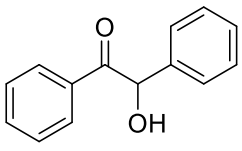
Yield 66%, 12.8 mg from **14aa** in Table 4 (30 h), yellow solid, m.p. 50.1 – 51.3 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.09 (dd, J = 8.4, 1.4 Hz, 2H), 7.80 – 7.71 (m, 1H), 7.62 (d, J = 8.1 Hz, 2H), 5.12 (s, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 177.1, 135.7, 135.0, 129.3, 129.2, 85.6, 80.3.[9]

2-Hydroxyacetophenone (**2ab**) 

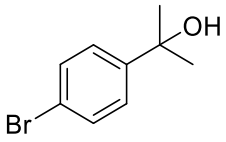
Yield 91%, 18.3 mg from **14ab** in Table 4 (36 h), white solid, m.p. 87.2 – 88.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 6.6 Hz, 2H), 7.63 (q, J = 7.6 Hz, 1H), 7.55 – 7.45 (m, 2H), 4.90 (s, 2H), 3.55 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.5, 134.47, 133.4, 129.1, 127.8, 65.6.[10]

9*H*-Xanthen-9-one (**2ac**) 

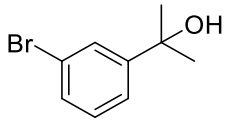
Yield 90%, 26.6 mg from **14ac** in Table 4 (36 h), white solid, m.p. 172.5 – 173.8 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (d, J = 8.0 Hz, 2H), 7.76 – 7.69 (m, 2H), 7.49 (d, J = 8.5 Hz, 2H), 7.38 (t, J = 7.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.4, 156.3, 135.0, 126.8, 124.0, 121.9, 118.1.[1]

2-Hydroxy-1,2-diphenylethan-1-one (**2ad**) 

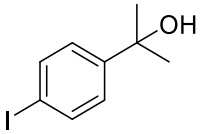
Yield 80%, 25.1 mg from **14ad** in Table 4 (36 h), white solid, m.p. 136.2 – 137.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 7.3 Hz, 2H), 7.52 (t, J = 7.4 Hz, 1H), 77.44 – 7.26 (m, 7H), 5.96 (s, 1H), 4.59 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.0, 139.1, 134.1, 133.5, 129.3, 129.3, 128.8, 128.7, 127.9, 76.3.[11]

2-(4-Bromophenyl)propan-2-ol (**4a**) 

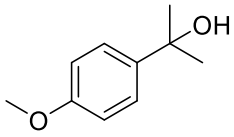
Yield 71%, 23 mg from **3a** in Table 2 (36 h), white solid, m.p. 45.3 – 46.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 8.6 Hz, 2H), 7.36 (d, J = 8.7 Hz, 2H), 1.81 (s, 1H), 1.56 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.2, 131.4, 126.5, 120.7, 72.4, 31.8.[12]

2-(3-Bromophenyl)propan-2-ol (**4b**) 

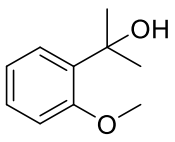
Yield 63%, 20.4 mg from **3b** in Table 2 (36 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.43 – 7.35 (m, 2H), 7.21 (t, J = 8.0 Hz, 1H), 1.74 (s, 1H), 1.57 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.6, 130.0, 129.9, 127.9, 123.2, 122.6, 72.4, 31.8.[13]

2-(4-Iodophenyl)propan-2-ol (**4c**) 

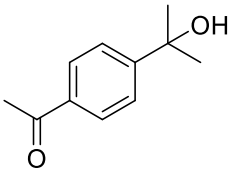
Yield 47%, 18.6 mg from **3c** in Table 2 (36 h), yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, J = 8.6 Hz, 2H), 7.24 (d, J = 8.6 Hz, 2H), 1.80 (s, 1H), 1.55 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.9, 137.4, 126.7, 92.3, 72.5, 31.8.[12]

2-(4-Methoxyphenyl)propan-2-ol (**4d**) 

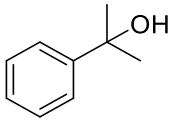
Yield 66%, 16.4 mg from **3d** in Table 2 (36 h), yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 8.3 Hz, 2H), 6.87 (d, J = 8.6 Hz, 2H), 3.80 (s, 3H), 1.73 (s, 1H), 1.57 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.4, 141.4, 125.7, 113.6, 72.4, 55.5, 31.9.[14]

2-(2-Metoxyphenyl)-2-propanol (**4e**) 

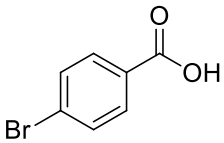
Yield 41%, 10.1 mg from **3e** in Table 2 (36 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (d, J = 7.7 Hz, 1H), 7.25 – 7.22 (m, 1H), 6.98 – 6.91 (m, 2H), 3.92 (s, 3H), 1.61 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.0, 135.8, 128.3, 125.9, 121.1, 111.4, 72.7, 55.4, 29.8.[15]

1-(4-(2-Hydroxypropan-2-yl)phenyl)ethan-1-one (**4f**) 

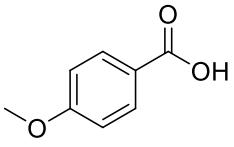
Yield 50%, 13.4 mg from **3f** in Table 2 (36 h), white solid, m.p. 85.1 – 85.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 8.4 Hz, 2H), 7.58 (d, J = 7.0 Hz, 2H), 2.60 (s, 3H), 1.85 (s, 1H), 1.60 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.0, 154.6, 135.7, 128.5, 124.9, 72.7, 31.8, 26.9.[12]

2-Phenylpropan-2-ol (**4g**) 

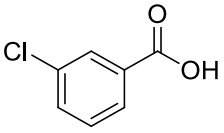
Yield 50%, 10.1 mg from **3g** in Table 2 (36 h), white solid, m.p. 30.5 – 31.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, J = 8.6 Hz, 2H), 7.36 (t, J = 7.6 Hz, 2H), 7.26 (t, J = 7.3 Hz, 1H), 2.11 (s, 1H), 1.59 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.2, 128.4, 126.8, 124.5, 72.7, 31.9.[13]

4-Bromobenzoic acid (**6a**) 

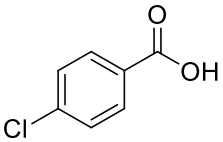
Yield 35%, 10.5 mg from **5a** in Table 2 (36 h, 11.0 mg, 43% of **5a** was recovered), white solid, m.p. 253.2 – 254.3 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 13.16 (s, 1H), 7.86 (d, J = 8.5 Hz, 2H), 7.71 (d, J = 8.5 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.4, 131.5, 131.1, 129.8, 126.7.[2]

4-Methoxybenzoic acid (**6b**) 

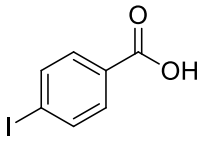
Yield 89%, 20.3 mg from **5b** in Table 2 (36 h), or yield 44%, 9.9 mg from **10b** in Table 3 (36 h), or yield 61%, 13.7 mg from **11a** in Table 3 (36 h), white solid, m.p. 183.1 – 184.4 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.64 (s, 1H), 7.89 (d, J = 6.9 Hz, 2H), 7.01 (d, J = 9.0 Hz, 2H), 3.81 (s, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 167.1, 162.9, 131.4, 123.0, 113.9, 55.5.[2]

3-Chlorobenzoic acid (**6c**) 

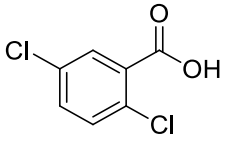
Yield 63%, 14.8 mg from **5c** in Table 2 (36 h), white solid, m.p. 156.2 – 157.0 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.90 – 7.88 (m, 2H), 7.69 (d, J = 8.7 Hz, 1H), 7.54 (t, J = 8.1 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 165.9, 133.1, 133.0, 132.4, 130.4, 128.6, 127.7.[2]

4-Chlorobenzoic acid (**6d**) 

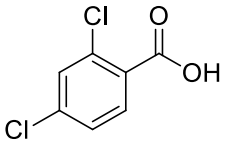
Yield 49%, 11.5 mg from **5d** in Table 2 (36 h), white solid, m.p. 241.3 – 242.0 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 13.22 (s, 1H), 7.94 (d, J = 8.5 Hz, 2H), 7.57 (d, J = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.6, 137.8, 131.2, 129.7, 128.8.[2]

4-Iodobenzoic acid (**6e**) 

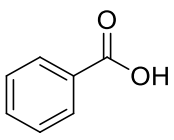
Yield 67%, 25.1 mg from **5e** in Table 2 (36 h), white solid, m.p. 272.0 – 273.3 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.88 (d, J = 8.4 Hz, 2H), 7.69 (d, J = 8.3 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.8, 137.3, 130.8, 130.2, 100.9.[16]

2,5-Dichlorobenzoic acid (**6f**) 

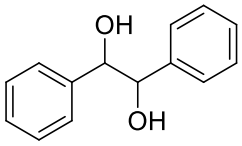
Yield 51%, 14.5 mg from **5f** in Table 2 (36 h), white solid, m.p. 151.1 – 152.3 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 13.70 (s, 1H), 7.90 – 7.88 (m, 1H), 7.56 – 7.50 (m, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 165.6, 133.2, 132.45, 132.36, 131.9, 130.5, 130.4.[17]

2,4-Dichlorobenzoic acid (**6g**) 

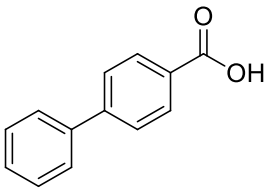
Yield 31%, 11.7 mg from **5g** in Table 2 (36 h, 11.7 mg, 48% of **5g** was recovered), white solid, m.p. 158.3 – 159.7 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.80 (d, J = 8.4 Hz, 1H), 7.72 (d, J = 2.1 Hz, 1H), 7.52 (dd, J = 8.4, 2.1 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.0, 136.4, 133.0, 132.3, 130.6, 130.2, 127.5.[17]

Benzoic acid (**6h**) 

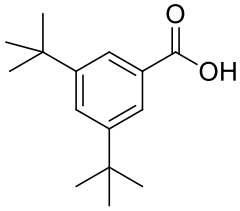
Yield 86%, 9.9 mg from **8a** in Table 3 (36 h), or yield 61%, 13.7 mg from **10a** in Table 3 (24 h), or yield 65%, 9.9 mg from **11d** in Table 3 (36 h), or yield 24%, 20.3 mg from **11e** in Table 3 (48 h in the solvent of CH_3CN and H_2O), white solid, m.p. 183.1 – 184.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.59 (s, 1H), 8.15 (d, J = 7.3 Hz, 2H), 7.63 (t, J = 7.7 Hz, 1H), 7.49 (t, J = 7.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.8, 134.0, 130.4, 129.4, 128.6.[1]

1,2-Diphenylethane-1,2-diol (**6h'**) 

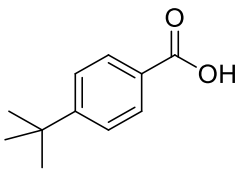
Yield 49%, 15.8 mg from **11e** in Table 3 (36 h), white solid, m.p. 132.7 – 135.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.29 (m, 6H), 7.27 – 7.23 (m, 4H), 4.83 (s, 2H), 2.00 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.8, 128.4, 128.3, 127.2, 78.2.[18]

[1,1'-Biphenyl]-4-carboxylic acid (**6i**) 

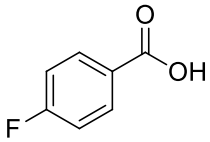
Yield 24%, 9.5 mg from **8b** in Table 3 (48 h in the solvent of CH_3CN, 21.1 mg, 57% of **8b** was recovered), or yield 68%, 20.3 mg from **11c** in Table 3 (48 h in the solvent of CH_3CN), white solid, m.p. 224.3 – 225.2 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.03 (d, J = 7.8 Hz, 2H), 7.75 (dd, J = 26.9, 7.8 Hz, 4H), 7.48 (t, J = 7.6 Hz, 2H), 7.40 (t, J = 7.4 Hz, 1H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 167.0, 144.1, 138.8, 129.8, 129.4, 128.9, 128.1, 126.7, 126.6.[2]

3,5-Di-tert-butylbenzoic acid (**6j**) 

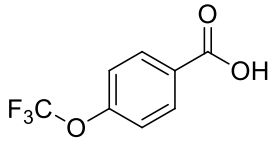
Yield 38%, 13.5 mg from **8c** in Table 3 (36 h, 20.0 mg, 47% of **8c** was recovered), white solid, m.p. 174.2 – 175.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (s, 2H), 7.70 (s, 1H), 1.37 (s, 18H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 151.3, 128.8, 128.2, 124.6, 35.1, 31.5.[19]

4-(tert-Butyl)benzoic acid (**6k**) 

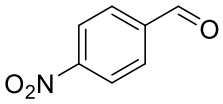
Yield 82%, 21.9 mg from **8d** in Table 3 (36 h, 4.4 mg, 13% of **8d** was recovered), white solid, m.p. 163.4 – 164.8 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.87 (d, J = 8.5 Hz, 2H), 7.51 (d, J = 8.5 Hz, 2H), 1.29 (s, 9H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 167.4, 155.8, 129.3, 128.1, 125.4, 34.8, 30.9.[19]

4-Fluorobenzoic acid (**6l**) 

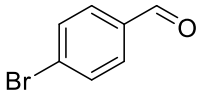
Yield 88%, 18.5 mg from **8e** in Table 3 (36 h), or yield 55%, 11.5 mg from **10c** in Table 3 (36 h), or yield 35%, 7.3 mg from **11b** in Table 3 (36 h), white solid, m.p. 182.3 – 183.9 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 13.05 (s, 1H), 7.99 (dd, J = 8.6, 5.7 Hz, 2H), 7.28 (t, J = 8.9 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.4 (d, J = 24.3 Hz), 163.8, 132.2 (d, J = 13.8 Hz), 127.5 (d, J = 2.8 Hz), 132.2 (d, J = 13.8 Hz).[1]

4-(Trifluoromethoxy)benzoic acid (**6m**) 

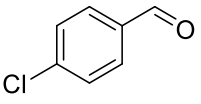
Yield 68%, 20.9 mg from **8f** in Table 3 (36 h, 10.1 mg, 26% of **8f** was recovered), white solid, m.p. 153.1 – 153.8 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.06 (d, J = 8.8 Hz, 2H), 7.48 (d, J = 8.3 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 166.3, 151.5, 131.8, 129.9, 121.3, 119.8 (q, J = 211.2 Hz).[17]

4-Nitrobenzaldehyde (**13a**) 

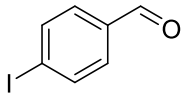
Yield 83%, 18.9 mg from **12a** in Table 4 (24 h), white solid, m.p. 104.7 – 105.2 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.16 (s, 1H), 8.40 (d, J = 8.7 Hz, 2H), 8.08 (d, J = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.5, 151.2, 140.1, 130.6, 124.4.[8]

4-Bromobenzaldehyde (**13b**) 

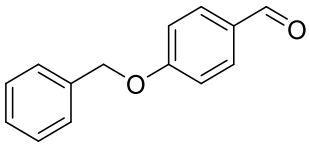
Yield 83%, 23.2 mg from **12b** in Table 4 (36 h, 4.3 mg, 15% of 4-bromobenzoic acid was isolated as a byproduct), white solid, m.p. 56.5 – 57.4 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.97 (s, 1H), 7.74 (d, J = 8.5 Hz, 2H), 7.68 (d, J = 8.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.3, 135.1, 132.6, 131.1, 129.9.[8]

4-Chlorobenzaldehyde (**13c**) 

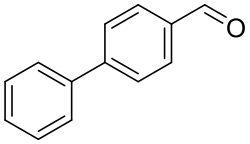
Yield 65%, 13.7 mg from **12c** in Table 4 (36 h, 3.7 mg, 16% of 4-chlorobenzoic acid was isolated as a byproduct), white solid, m.p. 44.4 – 47.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.82 (d, J = 8.4 Hz, 2H), 7.51 (d, J = 8.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.1, 141.1, 134.8, 131.0, 129.6.[8]

4-Iodobenzaldehyde (**13d**) 

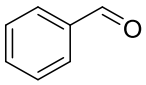
Yield 73%, 25.3 mg from **12d** in Table 4 (36 h), white solid, m.p. 81.3 – 81.9 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.95 (s, 1H), 7.91 (d, J = 8.3 Hz, 2H), 7.59 (d, J = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.6, 138.6, 135.7, 131.0, 103.0.[8]

4-(Benzyloxy)benzaldehyde (**13e**) 

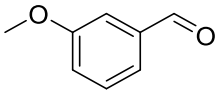
Yield 61%, 19.3 mg from **12e** in Table 4 (36 h, 10.3 mg, 30% of 4-(benzyloxy)benzoic acid was isolated as a byproduct), white solid, m.p. 72.0 – 73.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.89 (s, 1H), 7.84 (d, J = 8.4 Hz, 2H), 7.45 – 7.34 (m, 5H), 7.08 (d, J = 8.4 Hz, 2H), 5.15 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.0, 163.8, 136.0, 132.1, 130.2, 128.9, 128.5, 127.6, 115.2, 70.4.[8]

[1,1'-Biphenyl]-4-carbaldehyde (**13f**) 

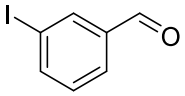
Yield 50%, 23.6 mg from **12f** in Table 3 (36 h, 9.8 mg, 33% of [1,1'-biphenyl]-4-carboxylic acid was isolated as a byproduct), white solid, m.p. 53.2 – 54.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.96 (d, J = 8.4 Hz, 2H), 7.75 (d, J = 7.9 Hz, 2H), 7.64 (d, J = 8.0 Hz, 2H), 7.49 (t, J = 7.2 Hz, 2H), 7.43 (t, J = 7.1 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.1, 147.2, 139.8, 135.2, 130.4, 129.1, 128.6, 127.8, 127.4.[8]

Benzaldehyde (**13g**) 

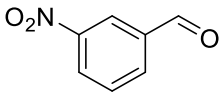
Yield 56%, 10.0 mg from $BnCH_2OH$ (**12g**) in Table 4 (36 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.02 (s, 1H), 7.88 (d, J = 7.9 Hz, 2H), 7.63 (t, J = 7.4 Hz, 1H), 7.57 – 7.50 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.6, 136.4, 134.6, 129.8, 129.1.[8]

3-Methoxybenzaldehyde (**13h**) 

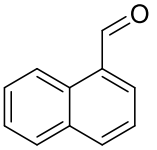
Yield 72%, 14.3 mg from **12h** in Table 4 (36 h, 4.3 mg, 19% of 3-methoxybenzoic acid was isolated as a byproduct), colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.97 (s, 1H), 7.49 – 7.42 (m, 2H), 7.39 (d, J = 1.4 Hz, 1H), 7.21 – 7.15(m, 1H), 3.86 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.4, 160.2, 137.9, 130.2, 123.8, 121.7, 112.0, 55.6.[8]

3-Iodobenzaldehyde (**13i**) 

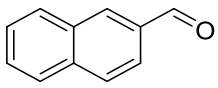
Yield 50%, 18.5 mg from **12i** in Table 4 (36 h, 1.0 mg, 3% of 3-iodobenzoic acid was isolated as a byproduct), yellow solid, m.p. 58.3 – 59.7 °C. **1**H NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 8.21 (s, 1H), 7.96 (d, J = 8.6 Hz, 1H), 7.84 (d, J = 7.7 Hz, 1H), 7.33 – 7.24 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.9, 143.3, 138.6, 138.1, 130.9, 129.0, 94.8.[20]

3-Nitrobenzaldehyde (**13j**) 

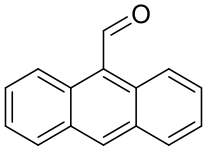
Yield 59%, 13.4 mg from **12j** in Table 4 (36 h, 1.7 mg, 7% of 3-nitrobenzoic acid was isolated as a byproduct), pale yellow powder, m.p. 124.1 – 125.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.13 (s, 1H), 8.72 (s, 1H), 8.50 (d, J = 8.2 Hz, 1H), 8.24 (d, J = 7.7 Hz, 1H), 7.77 (t, J = 7.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.0, 148.9, 137.5, 134.8, 130.5, 128.8, 124.7.[21]

1-Naphthaldehyde (**13k**) 

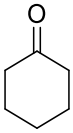
Yield 78%, 18.3 mg from **12k** in Table 4 (36 h, 5.6 mg, 21% of 1-naphthoic acid was isolated as a byproduct), yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.40 (s, 1H), 9.26 (d, J = 8.3 Hz, 1H), 8.11 (d, J = 8.2 Hz, 1H), 8.00 (dd, J = 7.1, 1.3 Hz, 1H), 7.93 (d, J = 8.2 Hz, 1H), 7.70 (t, J = 7.7 Hz, 1H), 7.66 – 7.57 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.8, 136.9, 135.5, 133.8, 131.5, 130.6, 129.2, 128.6, 127.1, 124.6.[8]

2-Naphthaldehyde (**13l**) 

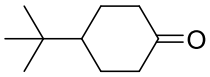
Yield 58%, 13.6 mg from **12l** in Table 4 (36 h), white solid, m.p. 60.6 – 61.0 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.17 (s, 1H), 8.35 (s, 1H), 8.05 – 7.85 (m, 4H), 7.68 – 7.63 (m, 1H), 7.63 – 7.57 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.5, 136.6, 134.8, 134.2, 132.7, 129.7, 129.3, 129.2, 128.2, 127.2, 122.8.[8]

Anthracene-9-carbaldehyde (**13m**) 

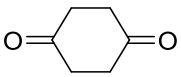
Yield 41%, 12.7 mg from **12m** in Table 4 (36 h), yellow solid, m.p. 100.8 – 102.5 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.50 (s, 1H), 8.96 (d, J = 9.0 Hz, 2H), 8.65 (s, 1H), 8.04 (d, J = 8.5 Hz, 2H), 7.77 – 7.60 (m, 2H), 7.59 – 7.49 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.1, 135.4, 132.2, 131.1, 129.4, 129.2, 125.8, 124.7, 123.6.[21]

Cyclohexanone (**16a**) 

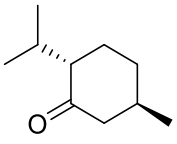
Yield 96% (GC-MS yield with 1,3,5-trimethylbenzene as internal standard) from **15a** in Table 4 (12 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.32 (t, J = 6.7 Hz, 4H), 1.89 – 1.80 (m, 4H), 1.75 – 1.66 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 212.4, 42.1, 27.1, 25.1.[1]

4-(tert-Butyl)cyclohexanone (**16b**) 

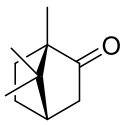
Yield 71%, 16.7 mg from **15b** in Table 4 (36 h), white solid, m.p. 45.2 – 46.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.42 – 2.34 (m, 2H), 2.34 – 2.24 (m, 2H), 2.11 – 2.02 (m, 2H), 1.50 – 1.36 (m, 3H), 0.90 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 212.9, 46.8, 41.4, 32.6, 27.7.[22]

Cyclohexan-1,4-dione (**16c**) 

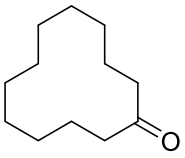
Yield 53%, 8.9 mg from **15c** in Table 4 (24 h), yellow solid, m.p. 75.9 – 77.9 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.69 (s, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.6, 36.7.[22]

(2S, 5R)-Menthone (**16d**) 

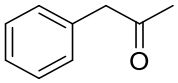
Yield 59%, 14.6 mg from **15d** in Table 4 (36 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.34 (ddd, J = 12.9, 3.9, 2.2 Hz, 1H), 2.18 – 1.94 (m, 4H), 1.93 – 1.79 (m, 2H), 1.45 – 1.26 (m, 2H), 1.00 (d, J = 6.3 Hz, 3H), 0.90 (d, J = 6.8 Hz, 3H), 0.84 (d, J = 6.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 212.7, 56.0, 51.0, 35.6, 34.0, 28.0, 26.0, 22.4, 21.4, 18.8.[23]

(1S,4S)-1,7,7-Trimethylbicyclo[2.2.1]heptan-2-one (**16e**) 

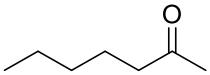
Yield 64%, 14.6 mg from **15e** in Table 4 (36 h), white solid, m.p. 173.3 – 176.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.34 – 2.28 (m, 1H), 2.05 (t, J = 4.6 Hz, 1H), 1.97 – 1.86 (m, 1H), 1.80 (d, J = 18.2 Hz, 1H), 1.68 – 1.61 (m, 1H), 1.41 – 1.24 (m, 2H), 0.92 (s, 3H), 0.87 (s, 3H), 0.79 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 219.8, 57.8, 46.8, 43.4, 43.1, 30.0, 27.1, 19.9, 19.2, 9.3.[24]

Cyclododecanone (**16f**) 

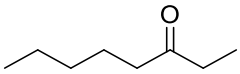
Yield 72%, 19.5 mg from **15f** in Table 4 (36 h), white solid, m.p. 57.1 – 58.7 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.46 – 2.39 (m, 4H),1.64 – 1.58 (m, 4H), 1.29 – 1.12 (m, 14H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 211.9, 39.7, 24.2, 24.2, 23.8, 22.0, 22.0.[1]

1-Phenylpropan-2-one (**16g**) 

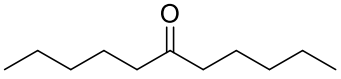
Yield 71%, 14.3 mg from **14g** in Table 4 (36 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (t, J = 7.2 Hz, 2H), 7.30 – 7.26 (m, 1H), 7.21 (d, J = 8.0 Hz, 2H), 3.70 (s, 2H), 2.16 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.7, 134.3, 129.5, 128.9, 127.2, 51.2, 29.4.[25]

Heptan-2-one (**16h**) 

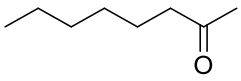
Yield 60%, 10.3 mg from **15h** in Table 4 (24 h), colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.41 (t, J = 7.5 Hz, 2H), 2.12 (s, 3H), 1.61 – 1.51 (m, 2H), 1.35 – 1.19 (m, 4H), 0.88 (t, J = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.6, 43.9, 31.5, 30.0, 23.7, 22.6, 14.0.[1]

Octan-3-one (**16i**) 

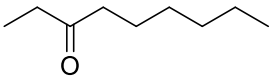
Yield 73%, 14.0 mg from **15i** in Table 4 (36 h), yellow liquid. ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.44 – 2.36 (m, 4H), 1.48 – 1.41 (m, 2H), 1.28 – 1.17 (m, 4H), 0.90 (t, J = 7.3 Hz, 3H), 0.85 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 211.0, 41.5, 35.0, 30.9, 23.0, 22.0, 13.9, 7.7.[1]

6-Undecanone (**16j**) 

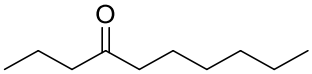
Yield 64%, 16.4 mg from **15j** in Table 4 (36 h), yellow liquid. ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.38 (t, J = 7.3 Hz, 4H), 1.48 – 1.40 (m, 4H), 1.28 – 1.16 (m, 8H), 0.84 (t, J = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 210.6, 41.8, 30.9, 23.0, 22.0, 13.9.[26]

2-Octanone (**16k**) 

Yield 78%, 15 mg from **15k** in Table 4 (36 h), yellow liquid. ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.39 (t, J = 7.3 Hz, 2H), 2.05 (s, 3H), 1.47 – 1.39 (m, 2H), 1.29 – 1.17 (m, 6H), 0.85 (t, J = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 208.3, 42.7, 31.2, 29.6, 28.3, 23.2, 22.0, 13.9.[27]

Nonan-3-one (**16l**) 

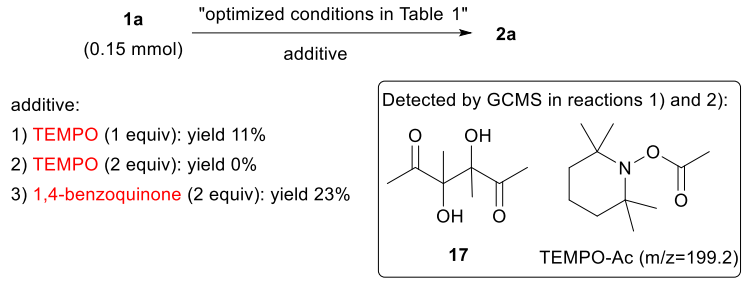
Yield 60%, 12.8 mg from **15l** in Table 4 (36 h), yellow liquid. ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.44 – 2.36 (m, 4H), 1.47 – 1.40 (m, 2H), 1.27 – 1.20 (m, 6H), 0.90 (t, J = 7.3 Hz, 3H), 0.85 (t, J = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 211.0, 41.5, 35.0, 31.1, 28.3, 23.3, 22.0, 13.9, 7.7.[28]

4-Decanone (**16m**) 

Yield 52%, 12.2 mg from **15m** in Table 4 (36 h), colorless liquid. ^{1}H NMR (400 MHz, DMSO-d_6) δ 2.40 – 2.33 (m, 4H), 1.50 – 1.40 (m, 4H), 1.22 (s, 6H), 0.86 – 0.80 (m, 6H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 210.5, 43.8, 41.9, 31.2, 28.4, 23.2, 22.1, 16.7, 14.0, 13.6.[27]

4. Mechanistic Studies

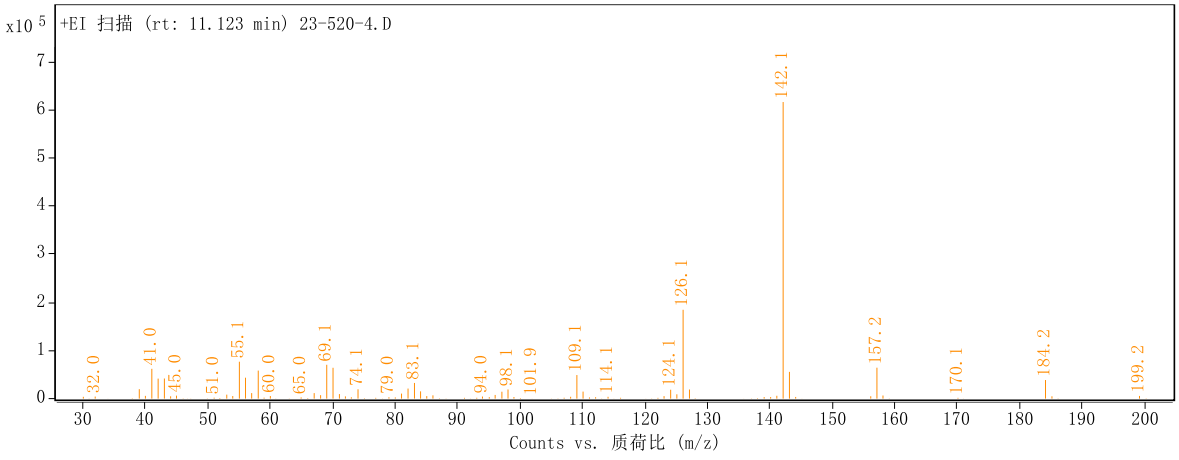
(1) Radical trapping and superoxide scavenger experiments



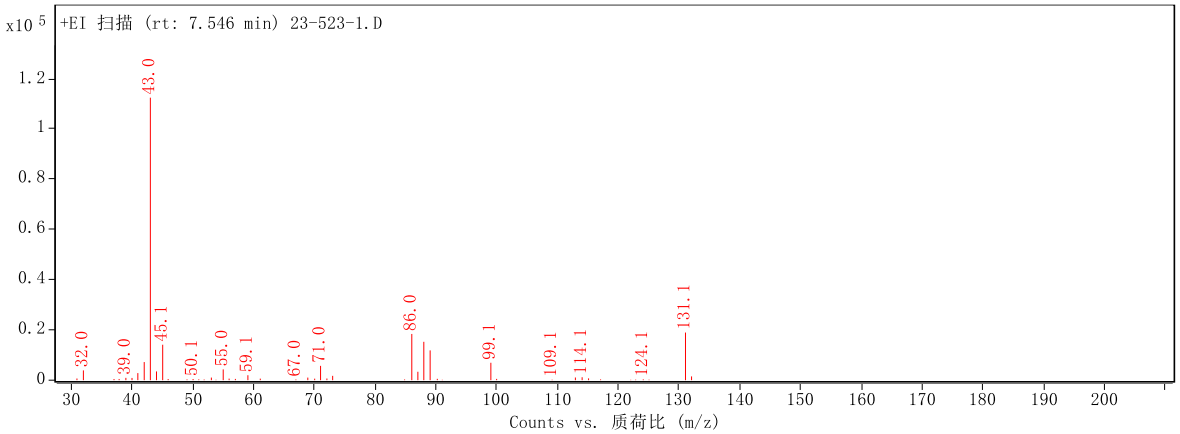
The procedures were the same as the above-optimized procedure for the oxidation of **1a** (27.8 mg, 0.15 mmol), with the addition of TEMPO (23.4 mg, 0.15 mmol, or 46.9 mg, 0.30 mmol, 1 or 2 equiv) or 1,4-benzoquinone (32.3 mg, 0.30 mmol, 2 equiv), which afforded 3.4 mg, 0 mg, or 6.8 mg of **2a** after purified by column chromatography in a yield of 11%, 0%, or 23%, respectively. In reactions 1) or reaction 2), TEMPO-Ac was detected by GC-MS in a molecular weight of 199.2 at 11.123 min (Figure S2a).[29] A pair of diastereoisomers of **17** was detected by GC-MS in a molecular weight of 131.1 ([M-Ac]$^+$) at 7.546 min and 7.778 min (Figure S2b).

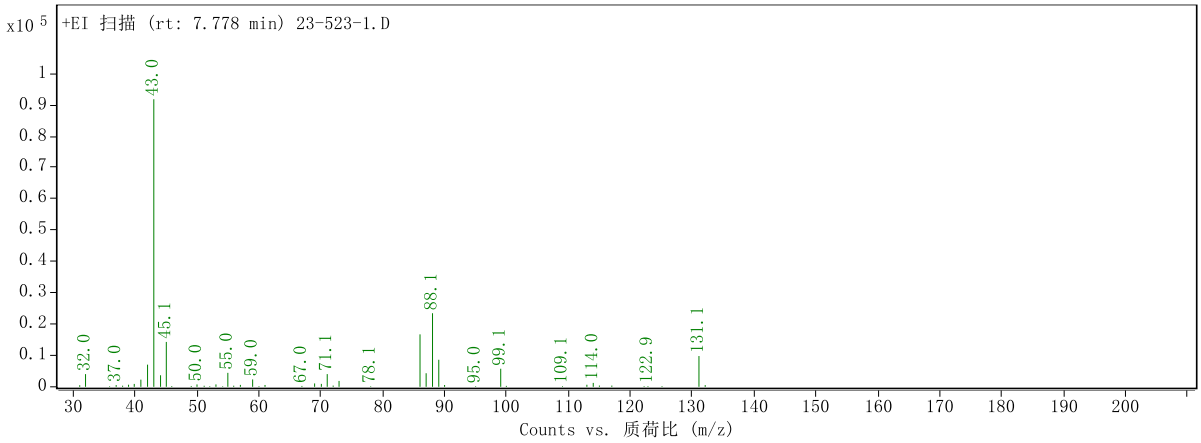
Figure S2. Spectrum of TEMPO-Ac and **17**

(a) GC-MS of compound TEMPO-Ac (t_R=11.123 min) in Scheme 3a

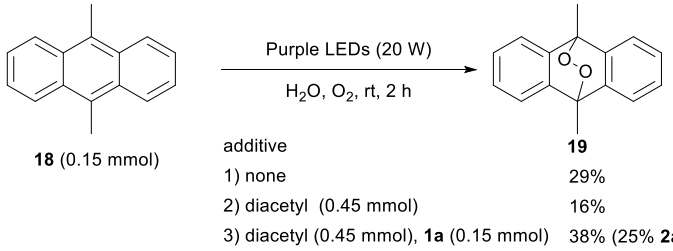


(b) GC-MS of **17** (t_R=7.546 min and 7.778 min) in Scheme 3a





(2) Trapping $^1O_2$ by 9,10-Dimethylanthracene



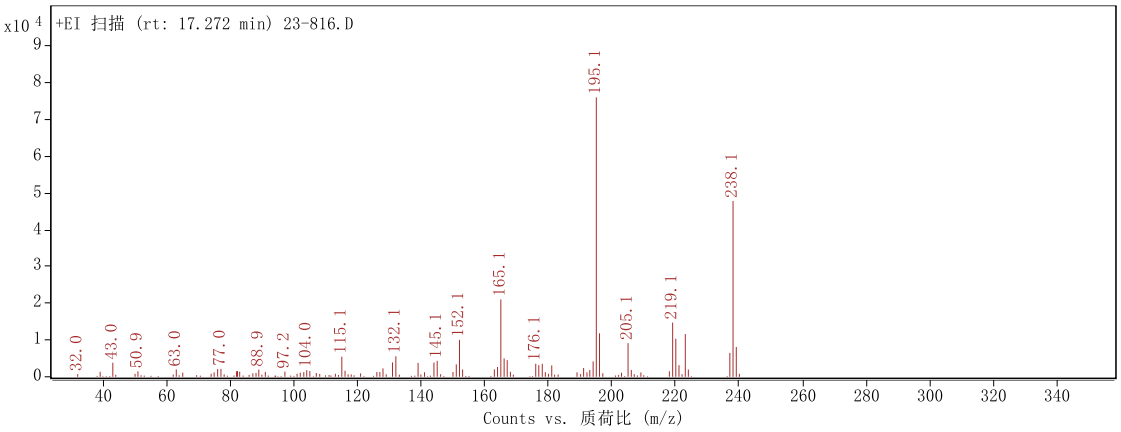
additive	**19**
1) none	29%
2) diacetyl (0.45 mmol)	16%
3) diacetyl (0.45 mmol), **1a** (0.15 mmol)	38% (25% **2a**)

To three 10 mL quartz tubes charged with a magnetic stir bar were added 9,10-dimethylanthracene **18** (30.9 mg, 0.15 mmol), H_2O (0.6 mL), diacetyl (0 mmol, 0.45 mmol, or 0.45 mmol), and **1a** (0 mmol, 0 mmol, or 0.45 mmol), respectively. The mixture was bubbled with O_2 for 15 min and then irradiated by purple LEDs (420 nm, 20 W) with vigorous stirring at room temperature. After 24 h, the solution was extracted with EtOAc (3 mL×3). The combined organic layers were washed with brine (2 mL), dried with anhydrous Na_2SO_4, and purified by column chromatography with petroleum ether/EtOAc (10:1) to afford 10.4 mg, 5.8 mg, or 13.6 mg of **19** in a yield of 29%, 16% or 38%, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.37 (m, 4H), 7.32 – 7.26 (m, 4H), 2.16 (s, 6H). ^{13}C NMR (400 MHz, $CDCl_3$) δ 140.8, 127.5, 120.8, 79.7,

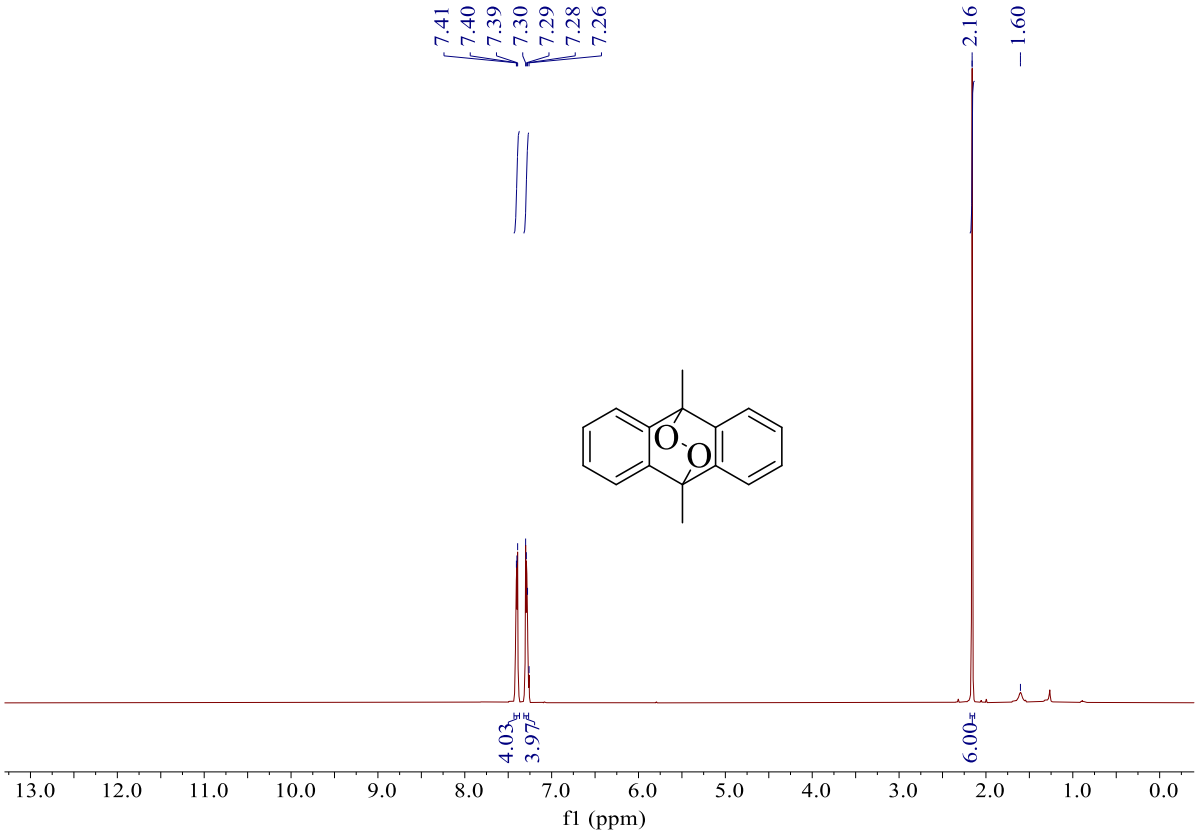
13.9. GC-MS (EI): m/z 238.1, 219.1, 195.1 (Figure S3).[30] 7.8 mg of **2a** was obtained as well for the third reaction in a yield of 25%.

Figure S3. GC-MS and NMR spectra of **19**

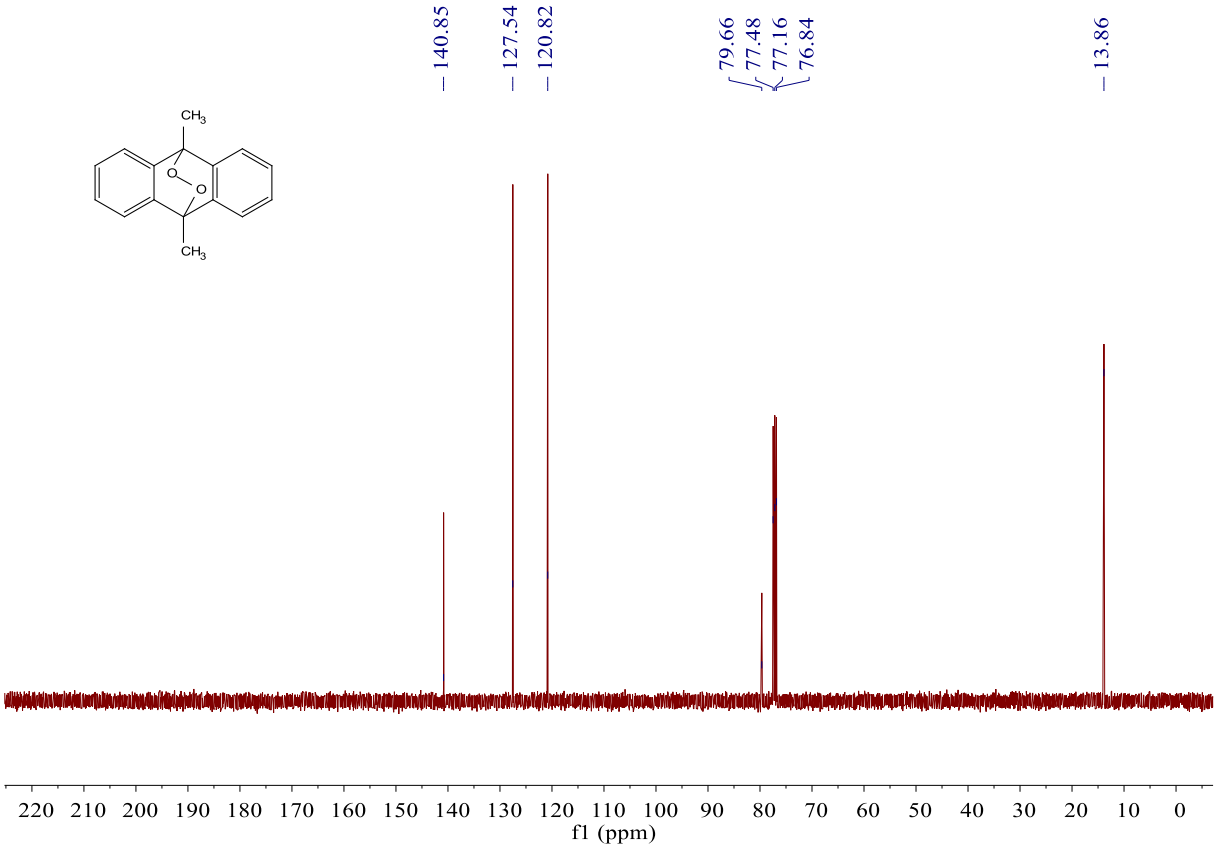
(a) GC-MS report of product **19** (t_R=17.272 min)



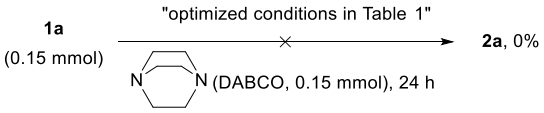
(b) ^{1}H NMR of **19**



(c) ^{13}C NMR of **19**

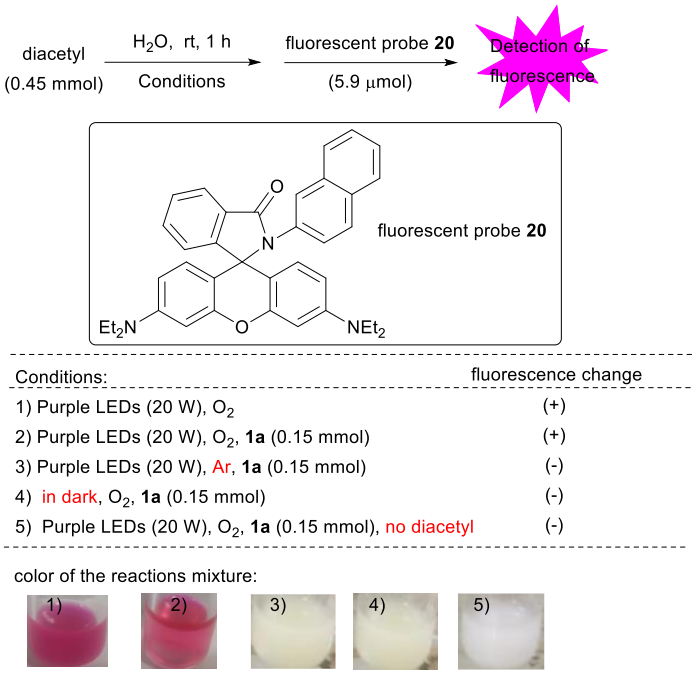


(3) DABCO as a $^1O_2$ quencher



To a 10 mL quartz tube charged with a magnetic stir bar was added **1a** (27.8 mg, 0.15 mmol), H_2O (0.6 mL), diacetyl (0.45 mmol, 3 equiv, 39.5 μL) and 1,4-diazabicyclo[2.2.2]octane (DABCO, 16.7 mg, 0.15 mmol). The mixture was bubbled with O_2 for 15 min and then irradiated by purple LEDs (420 nm, 20 W) with vigorous stirring at room temperature. After 24 h, the solution was extracted with EtOAc (3 mL×3) and the combined organic phase was detected by GC-MS, which indicated no formation of **2a**.

(4) Fluorescent detection of radical intermediates



Reaction 1): To a 10 mL quartz tube charged with a magnetic stir bar was added diacetyl (0.45 mmol, 39.5 μL) and H_2O (0.6 mL). The mixture was bubbled with O_2 for 15 min and then irradiated by purple LEDs (420 nm, 20 W) with vigorous stirring at room temperature for 1 h. The fluorescent probe **20** (5.9 μmol, 3.3 mg) in CH_3CN (0.4 mL) was added to the above reaction mixture. The color of the mixture was taken by a cell phone, and then the solution was analyzed by a fluorescence spectrophotometer (Figure S4).

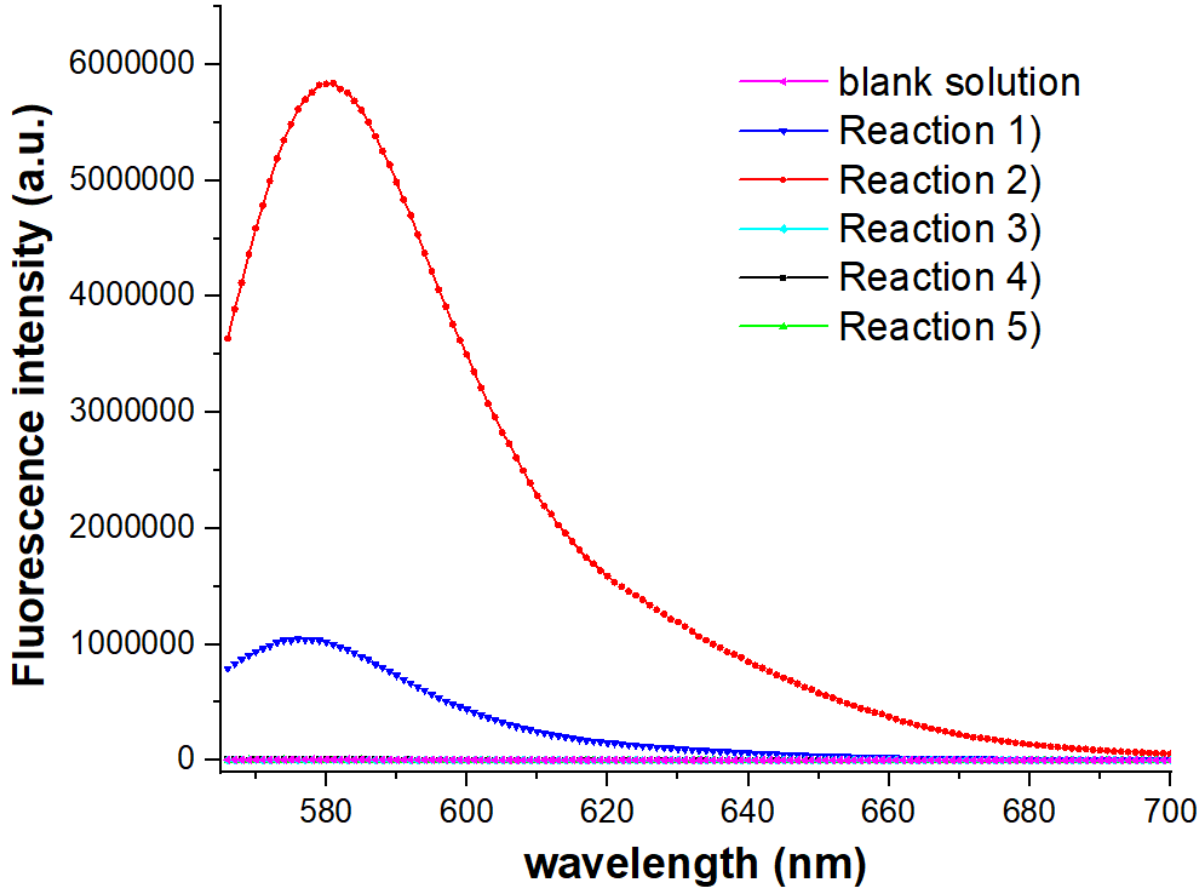
Reaction 2): A similar procedure was carried out except with an addition of **1a** (27.8 mg, 0.15 mmol).

Reaction 3): A similar procedure was carried out except with an addition of **1a** (27.8 mg, 0.15 mmol), and the mixture was bubbled with argon for 15 min.

Reaction 4): A similar procedure was carried out except with an addition of **1a** (27.8 mg, 0.15 mmol), and the mixture was stirred in the dark.

Reaction 5): A similar procedure was carried out except with an addition of **1a** (27.8 mg, 0.15 mmol), and no diacetyl was added.

Figure S4. Fluorescent spectra of radical intermediates with the addition of probe 20.



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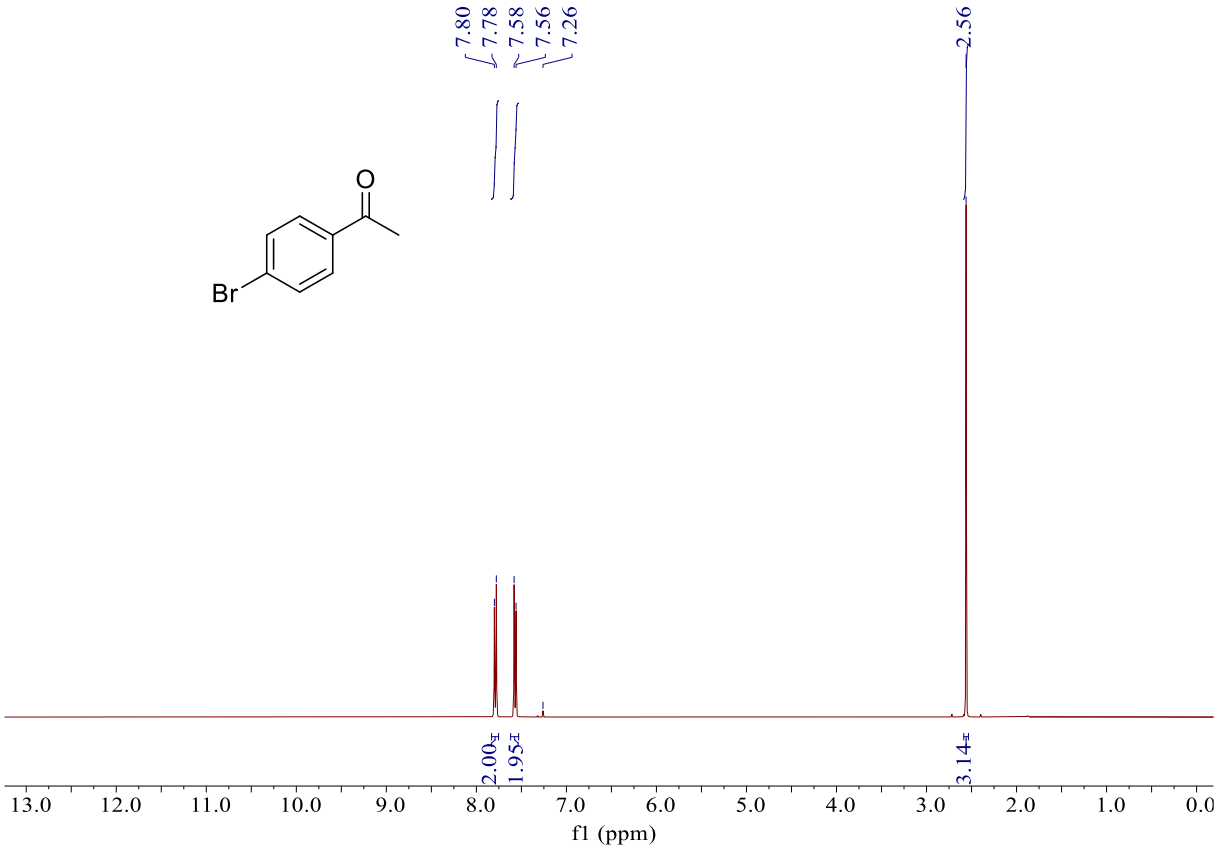
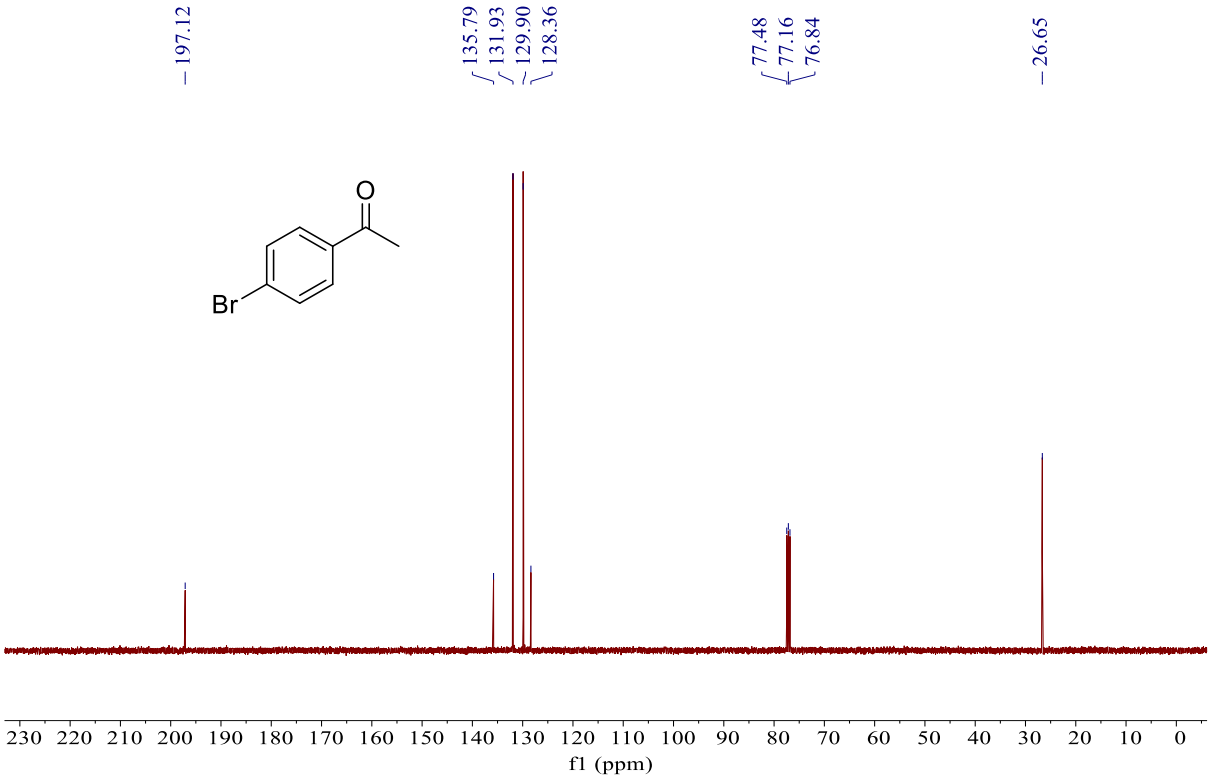
6. NMR Spectra

^{1}H NMR of compound 2a:



^{13}C NMR of compound 2a:



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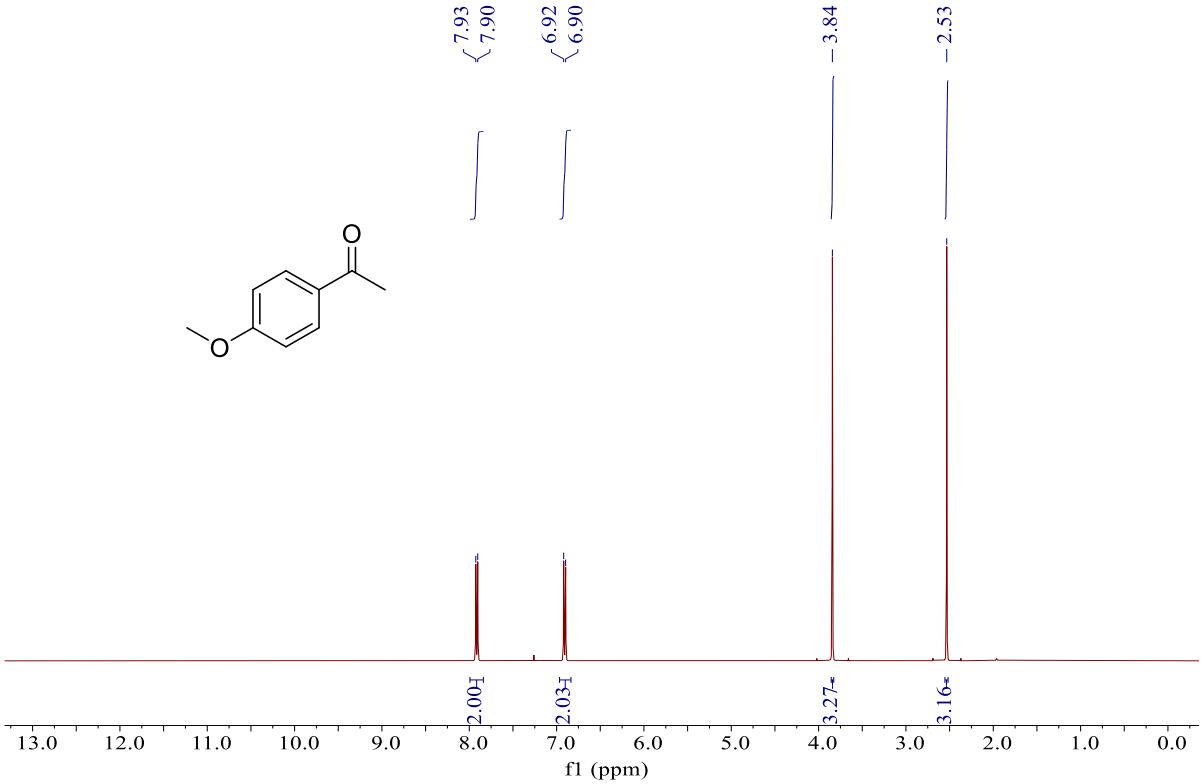

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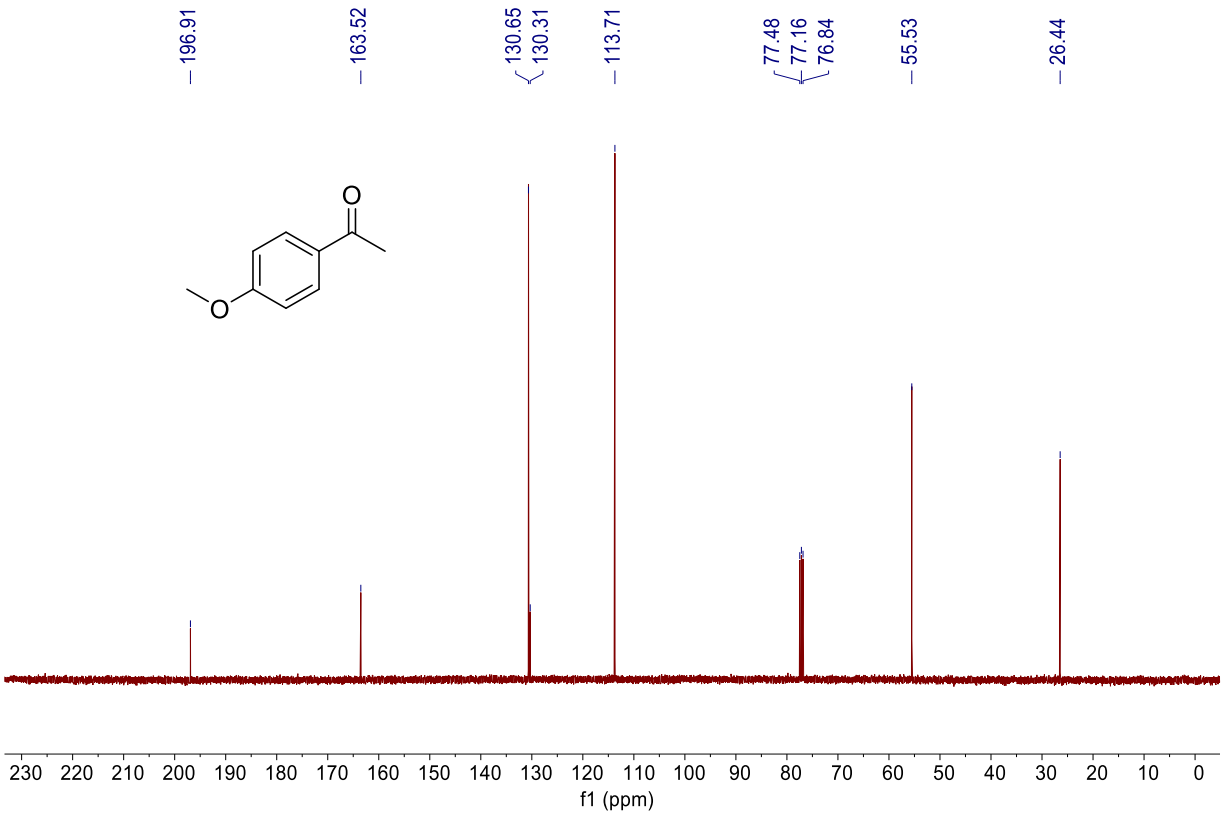
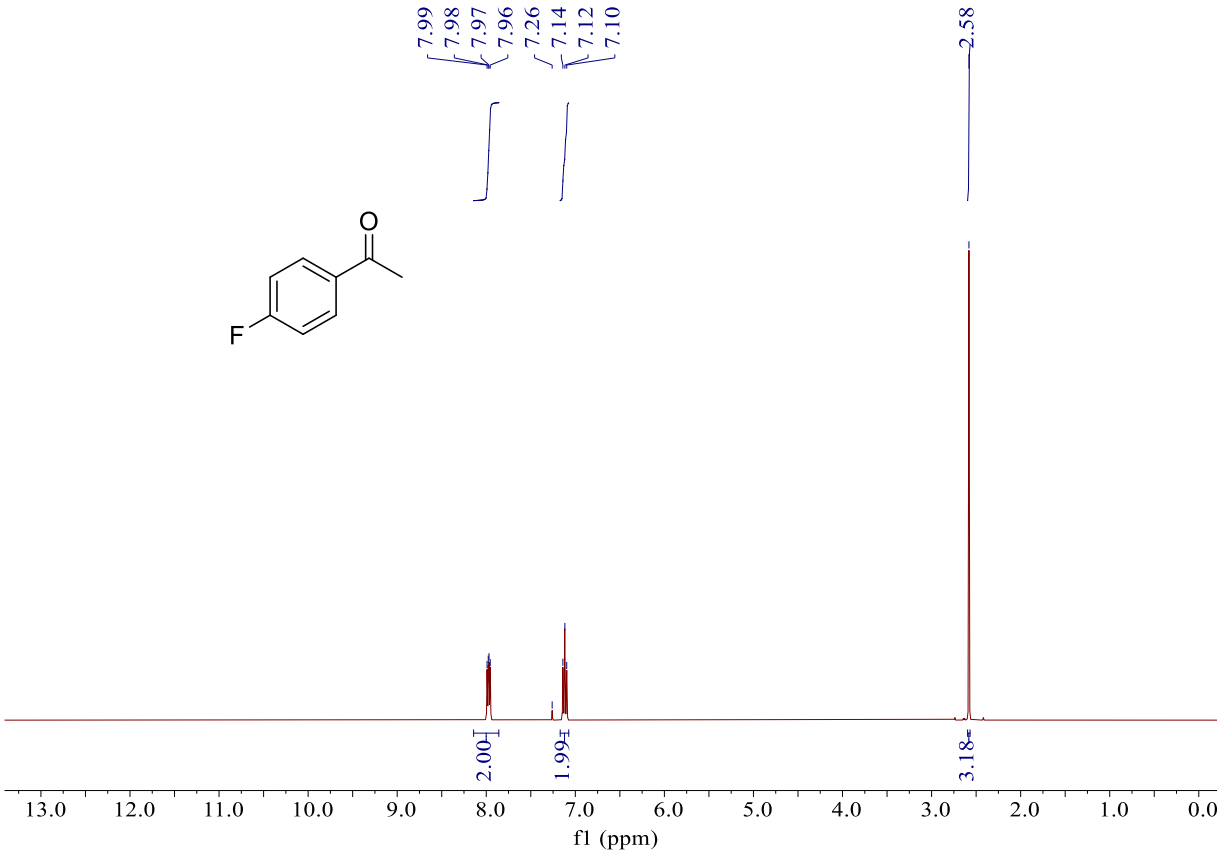


^{1}H NMR of compound 2c:



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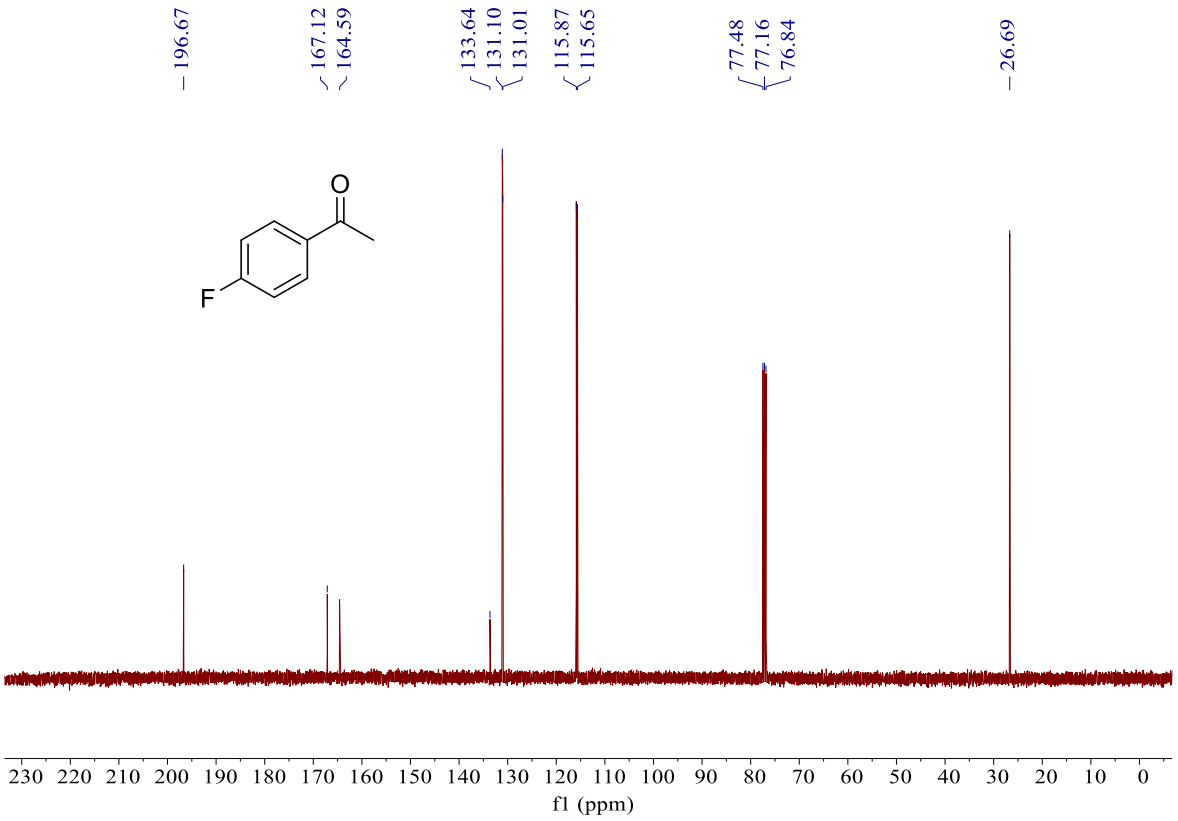

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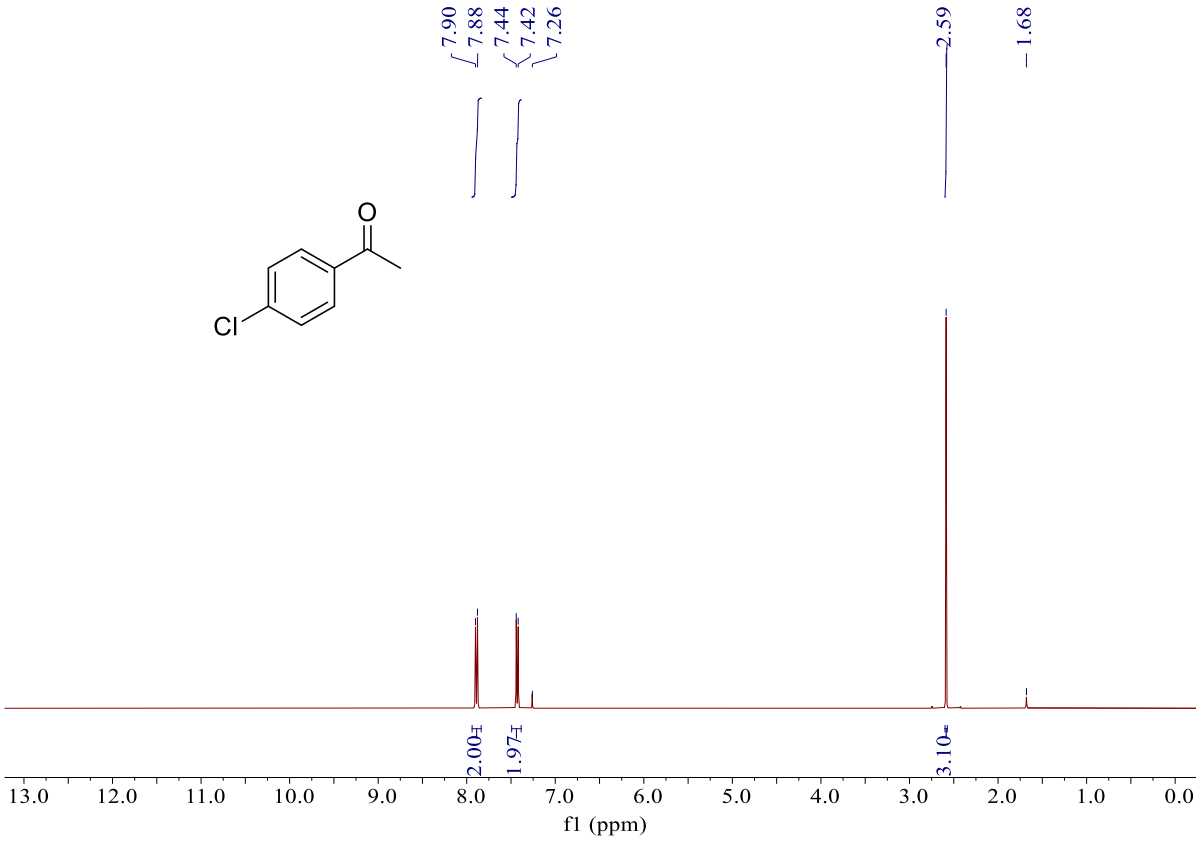

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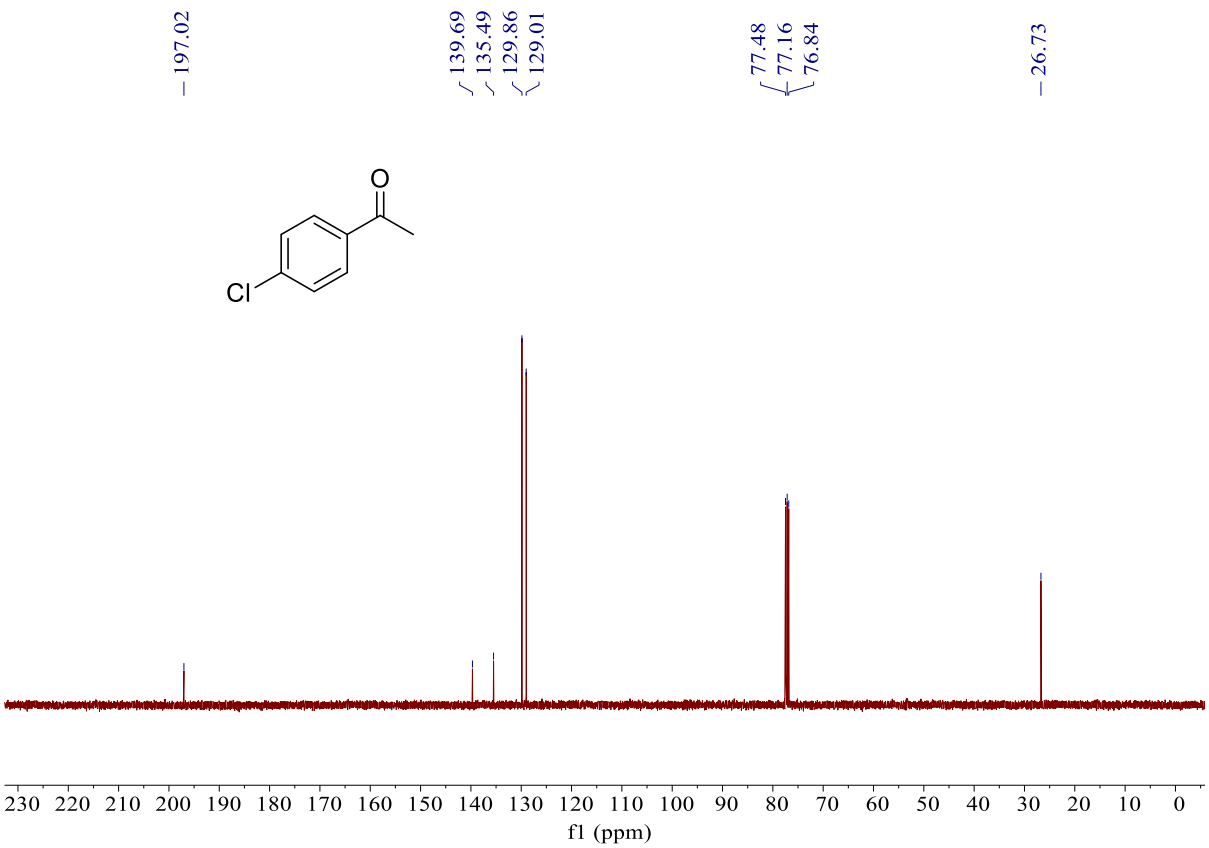

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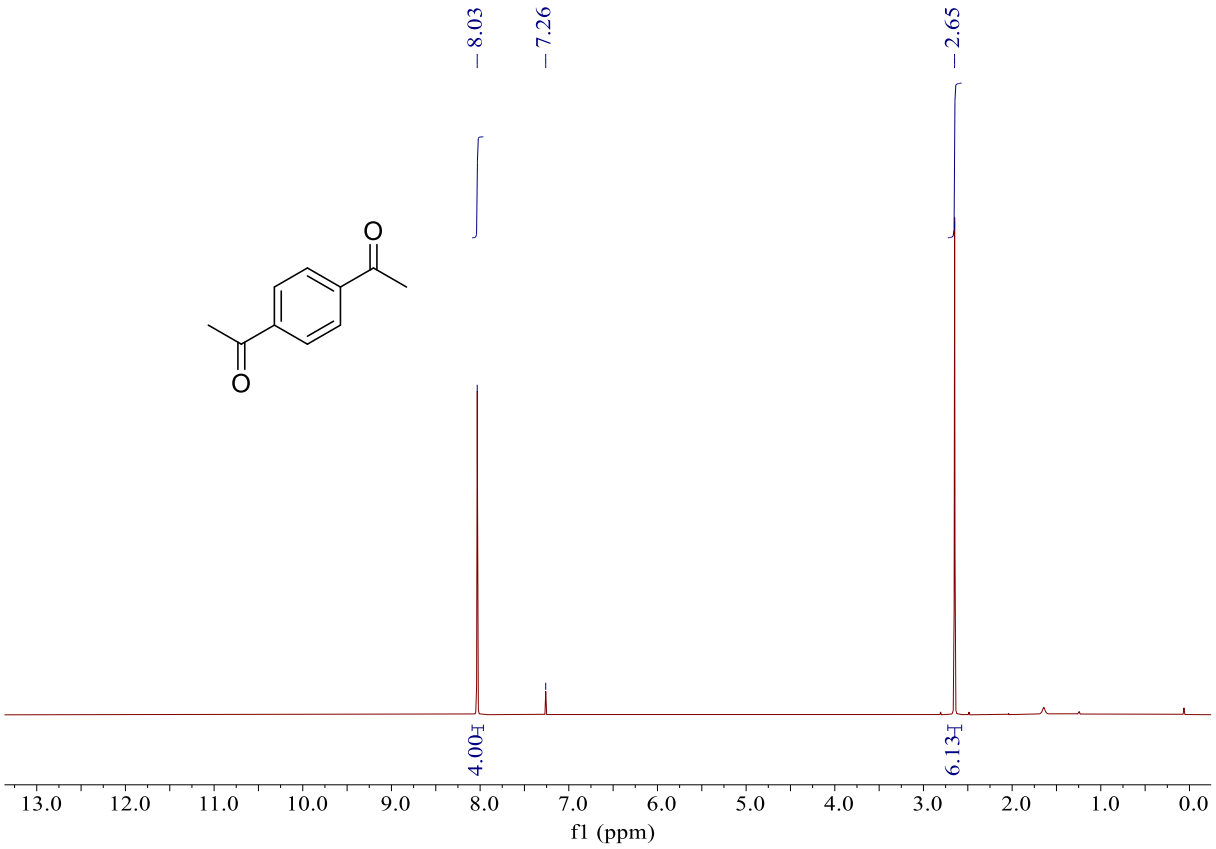

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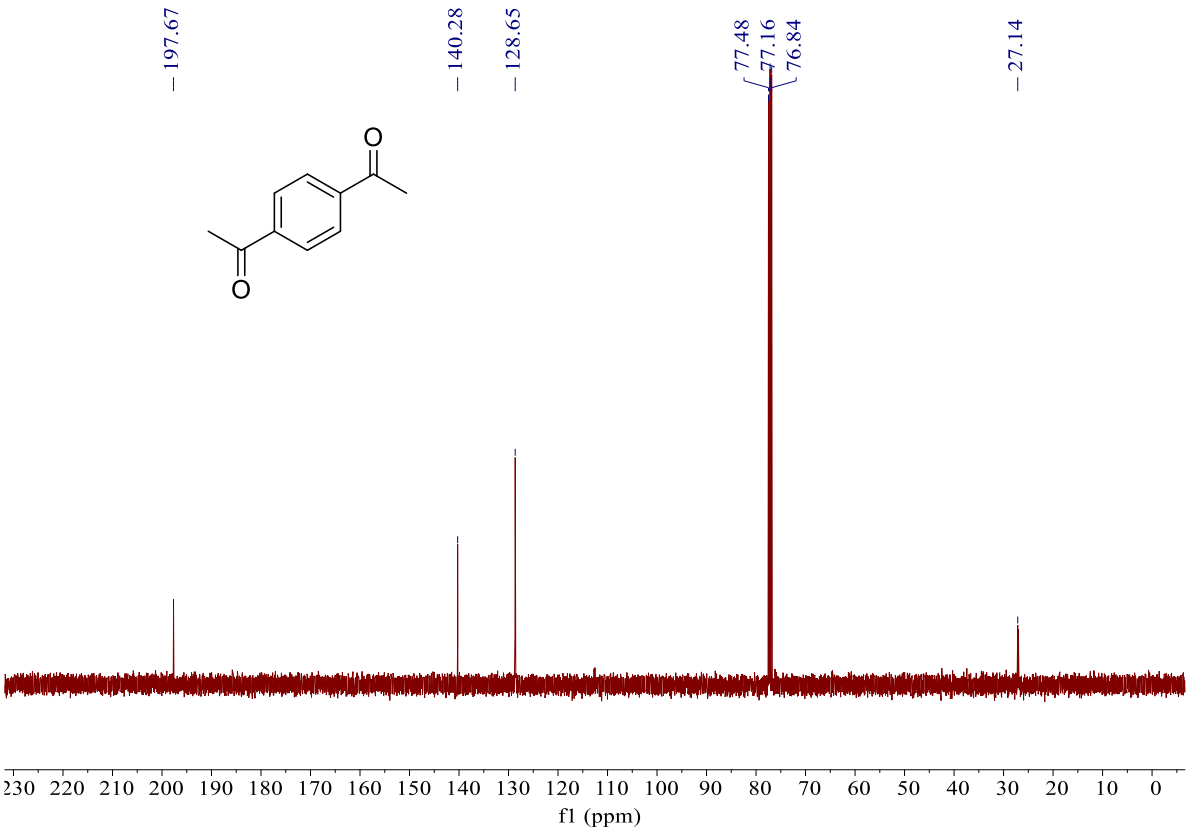

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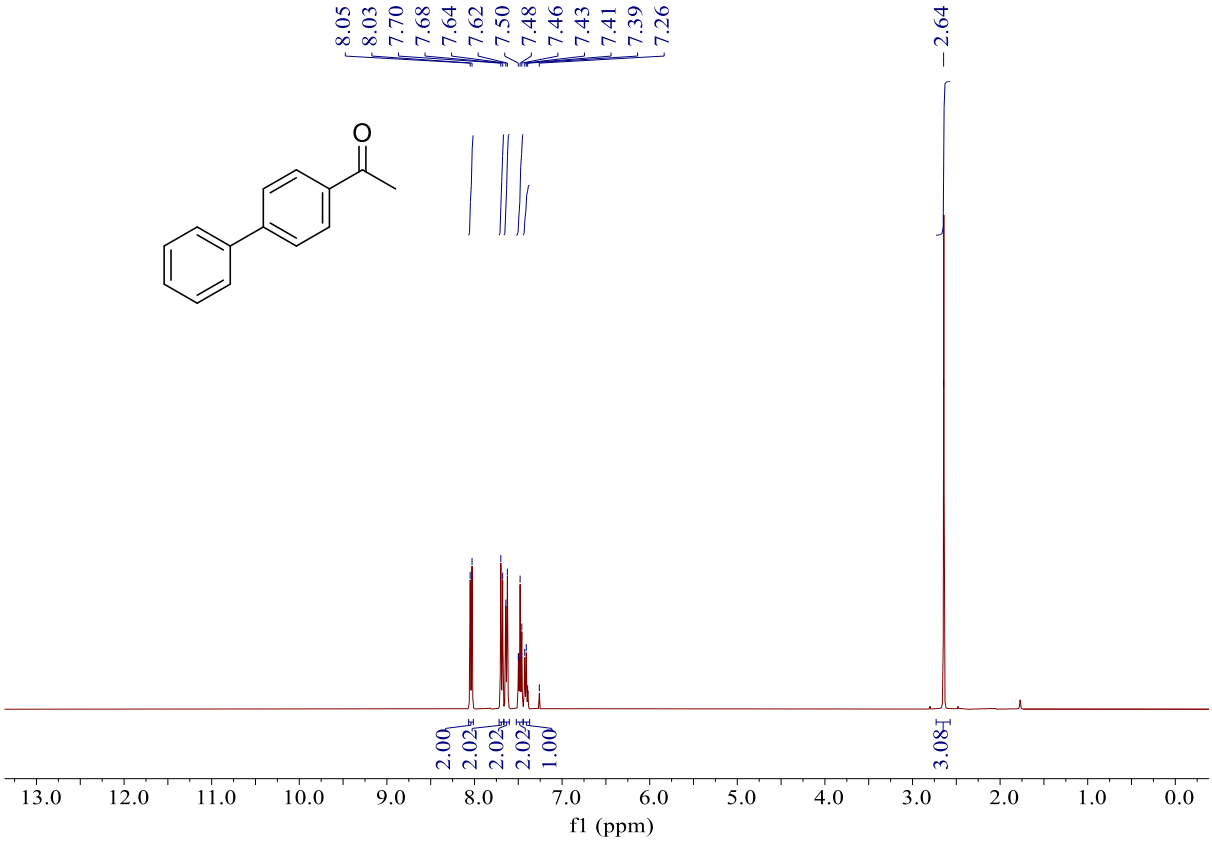

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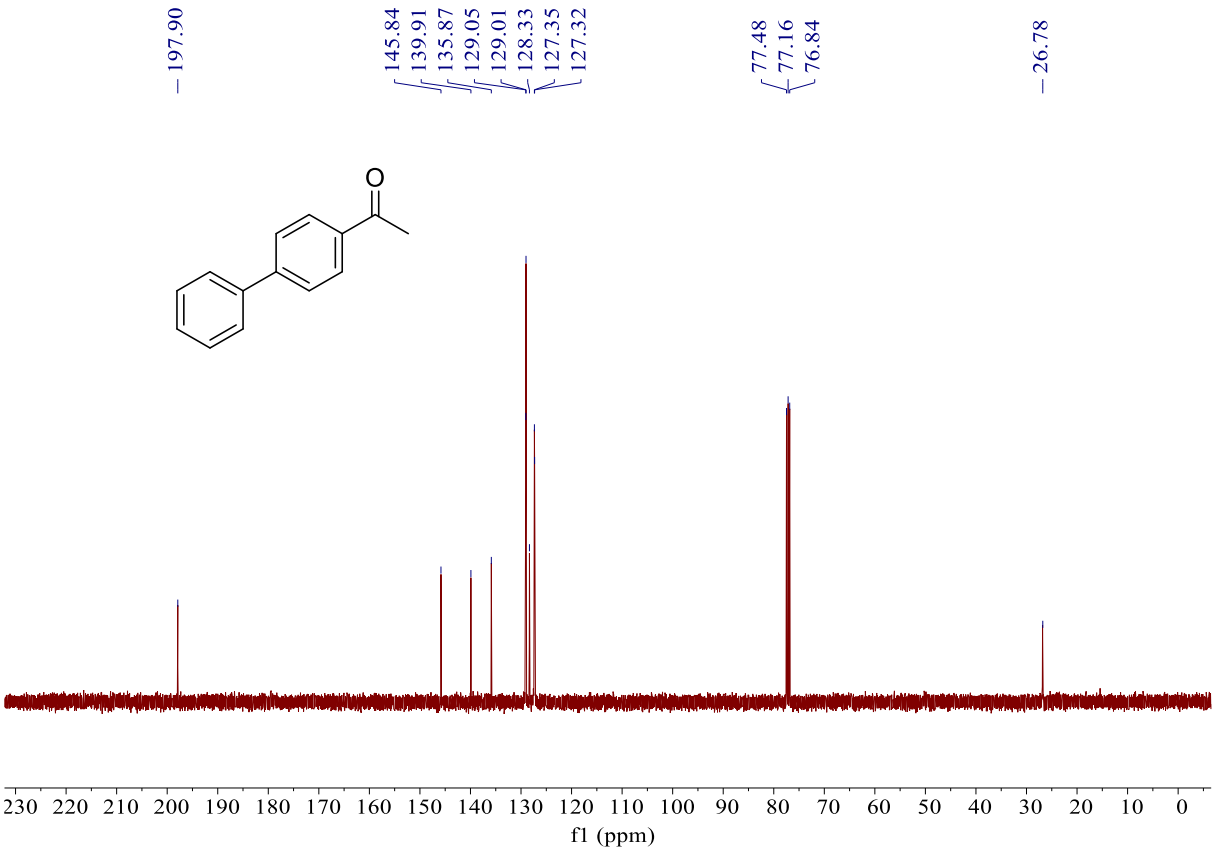

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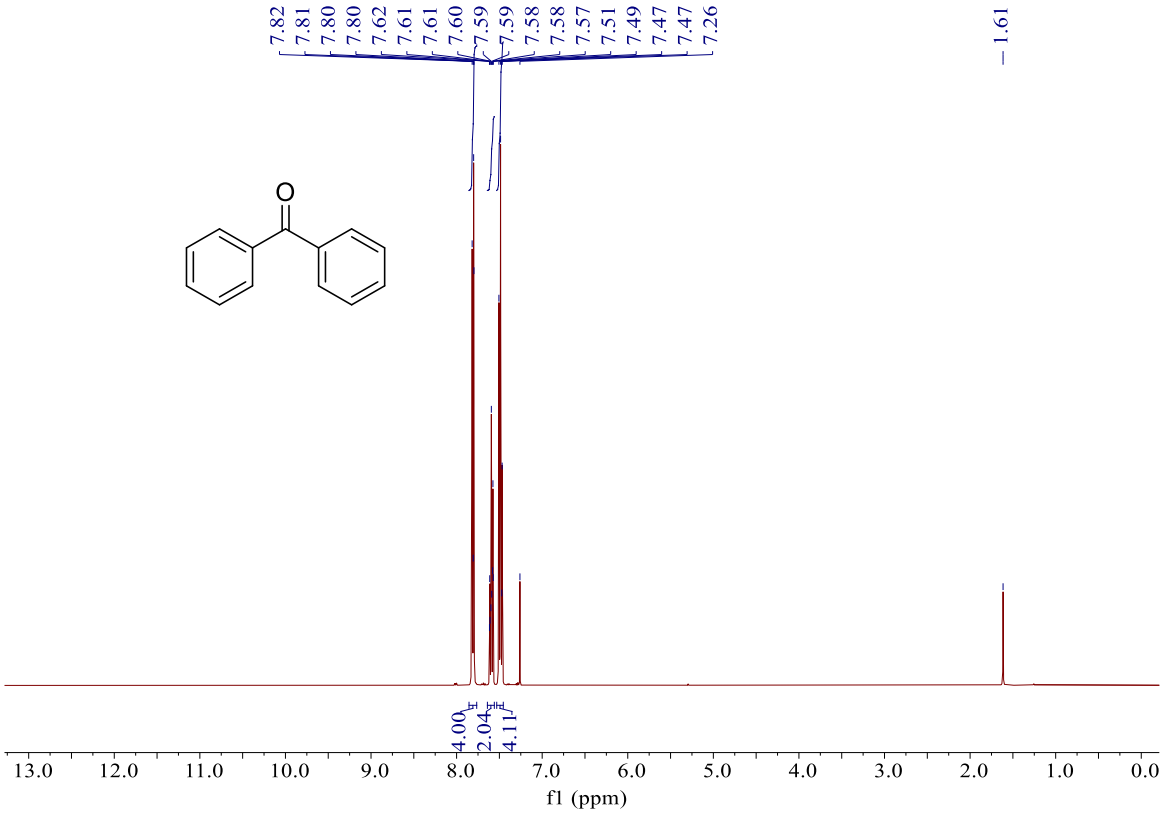

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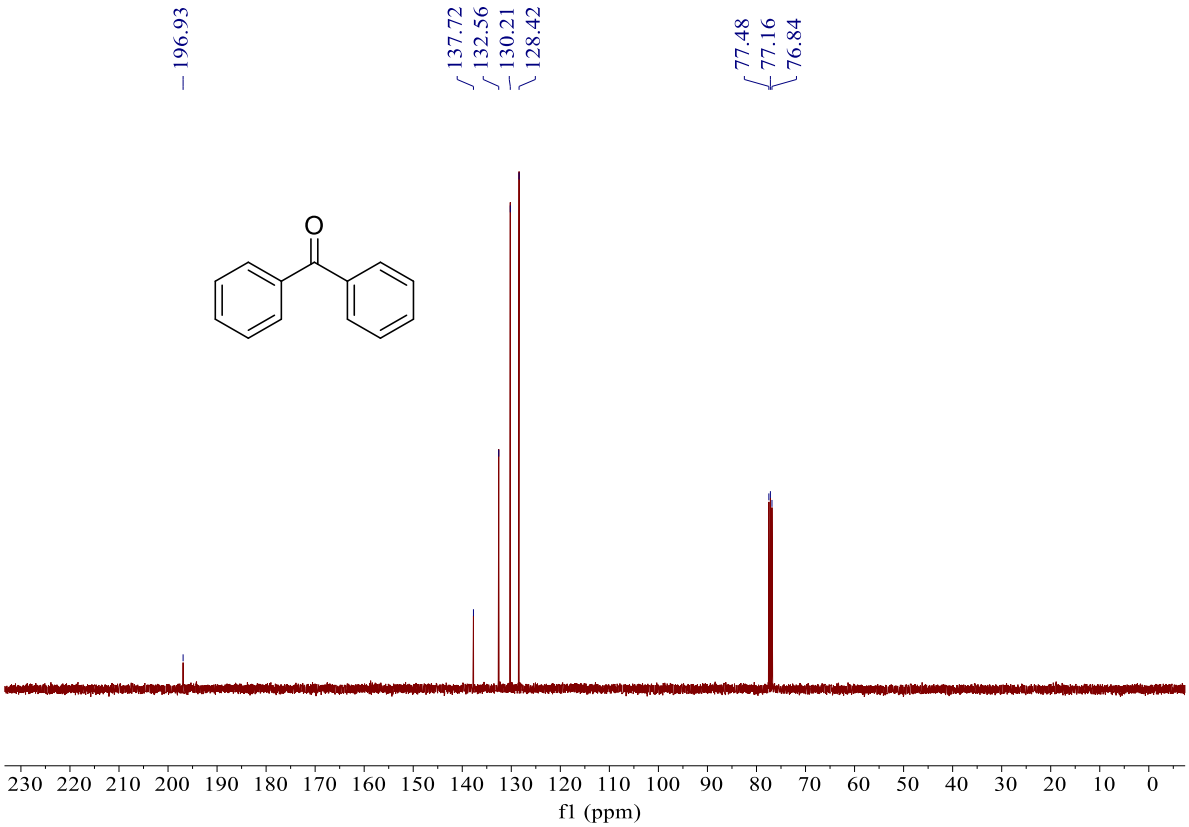

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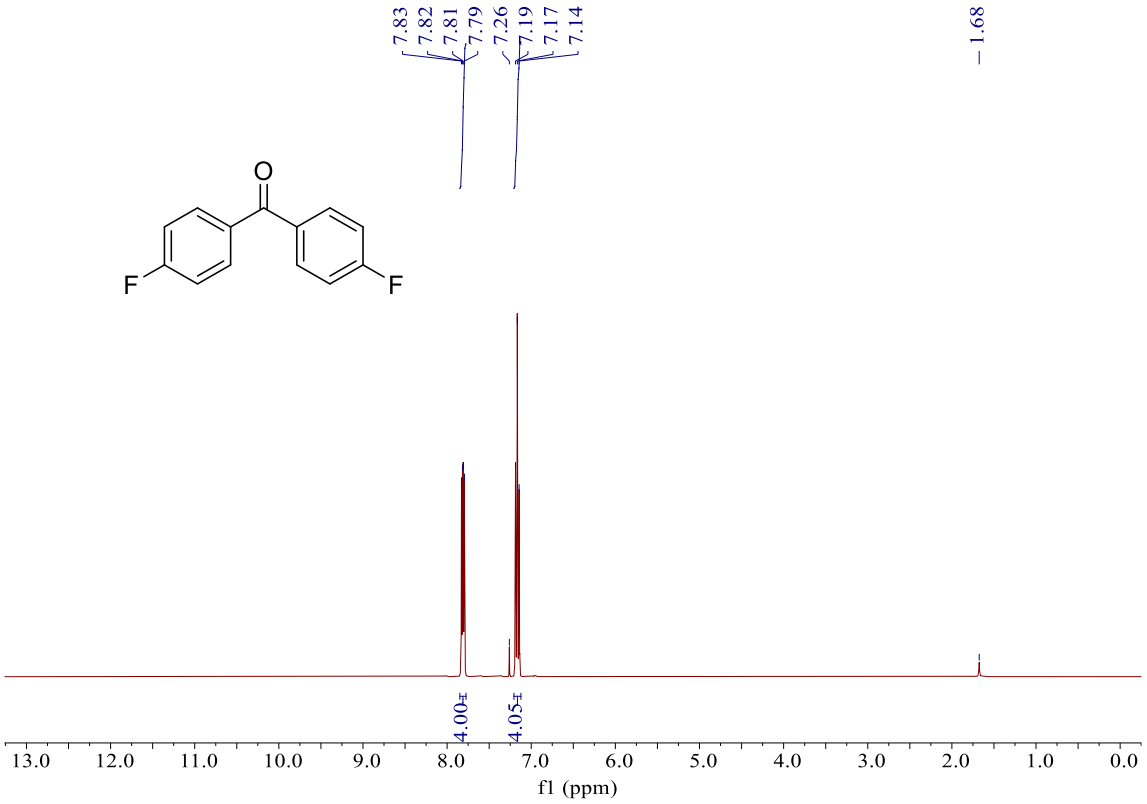

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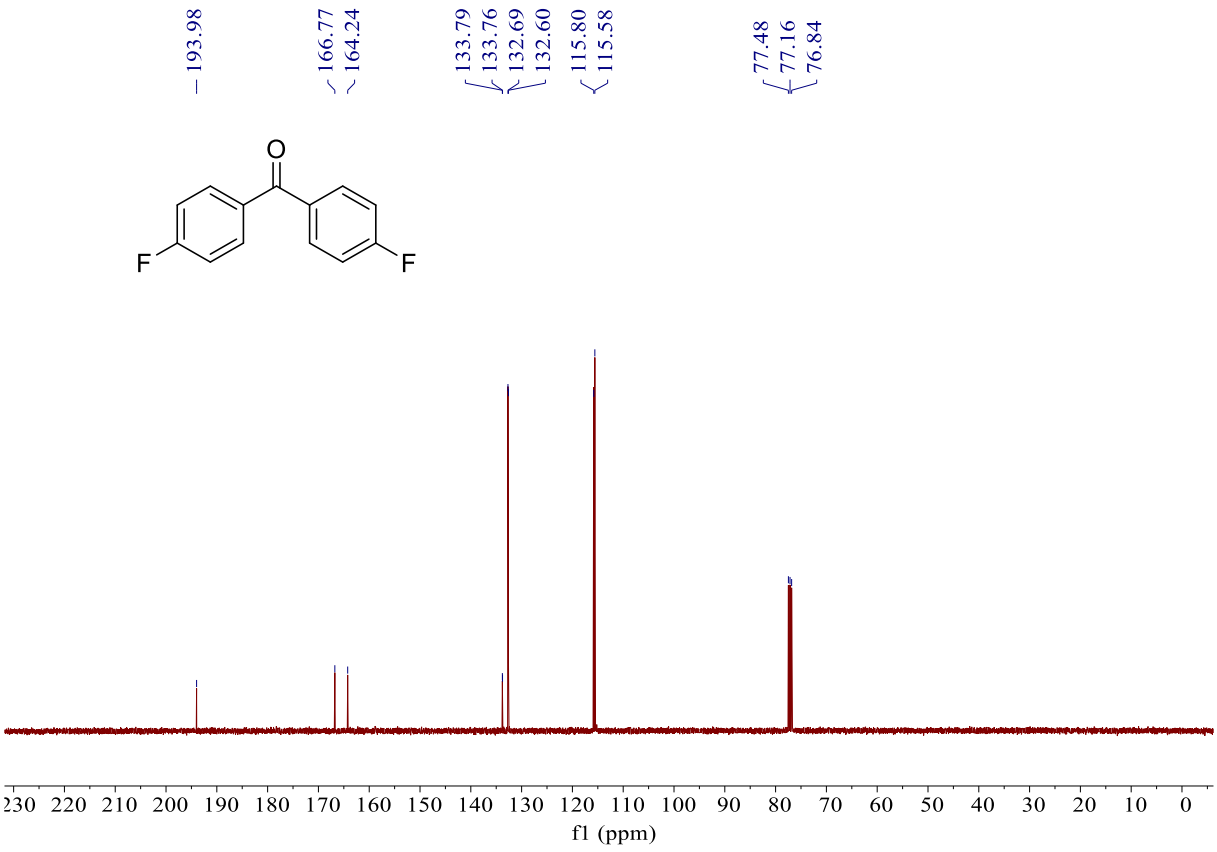

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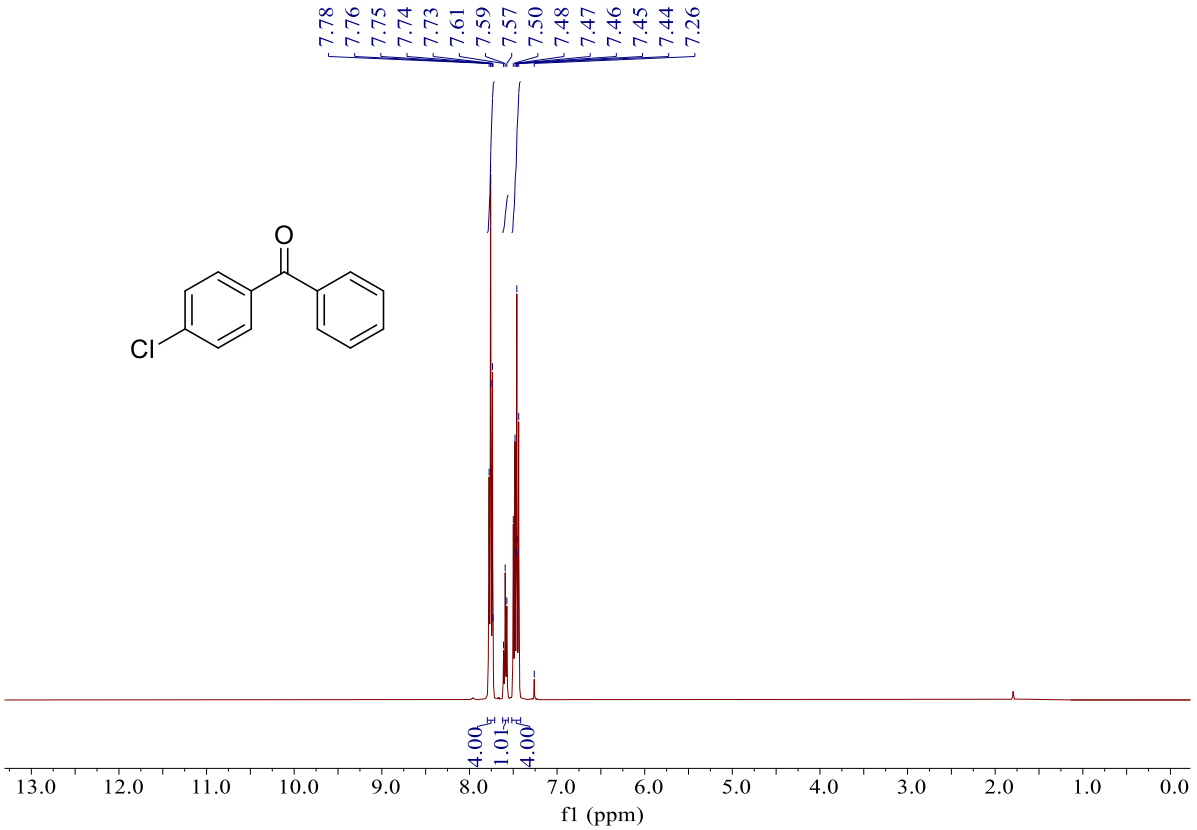

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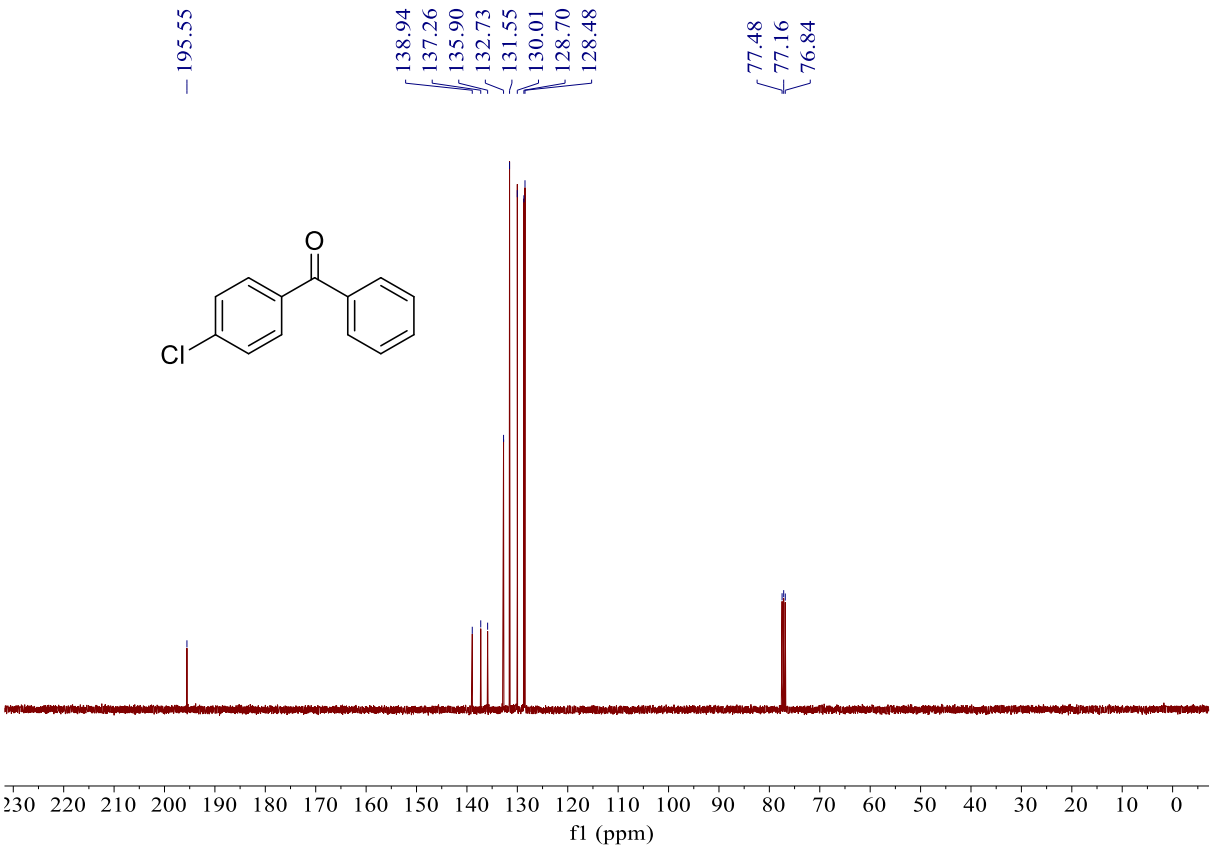

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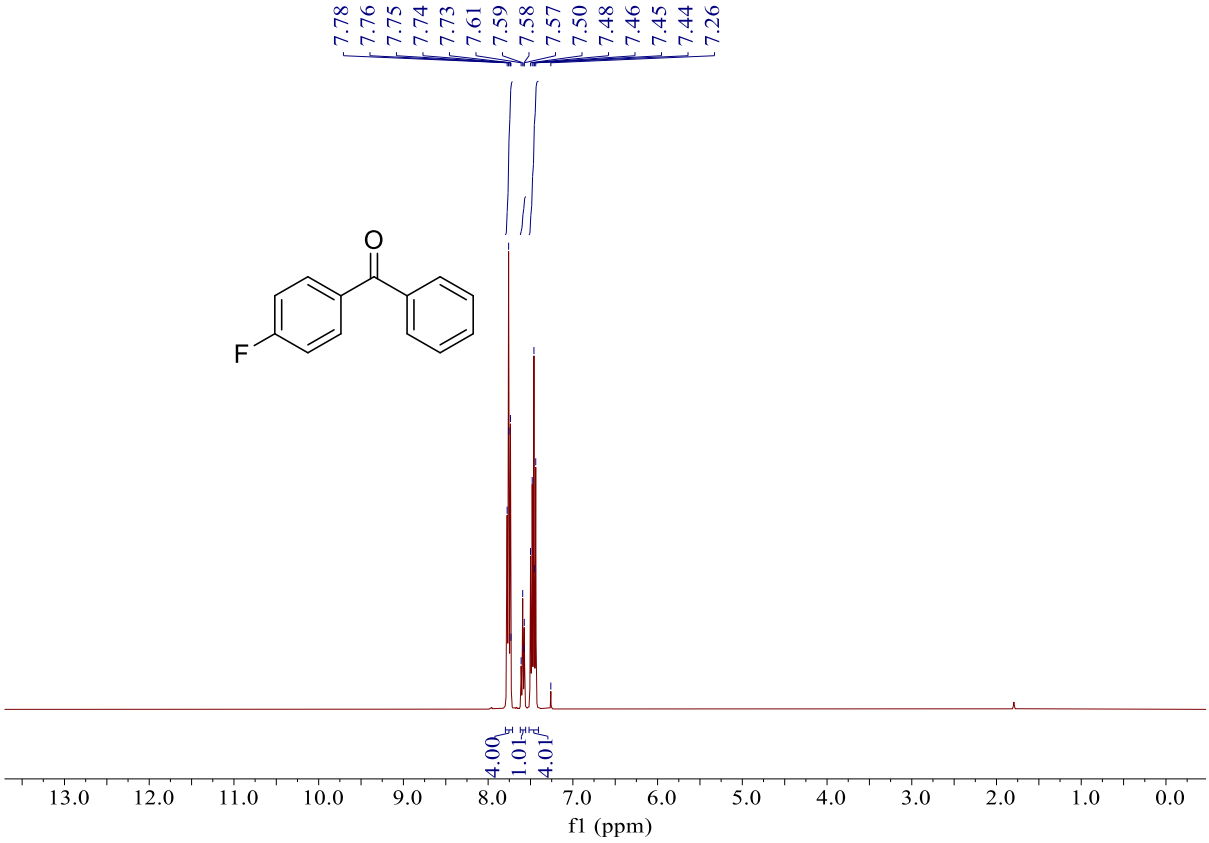

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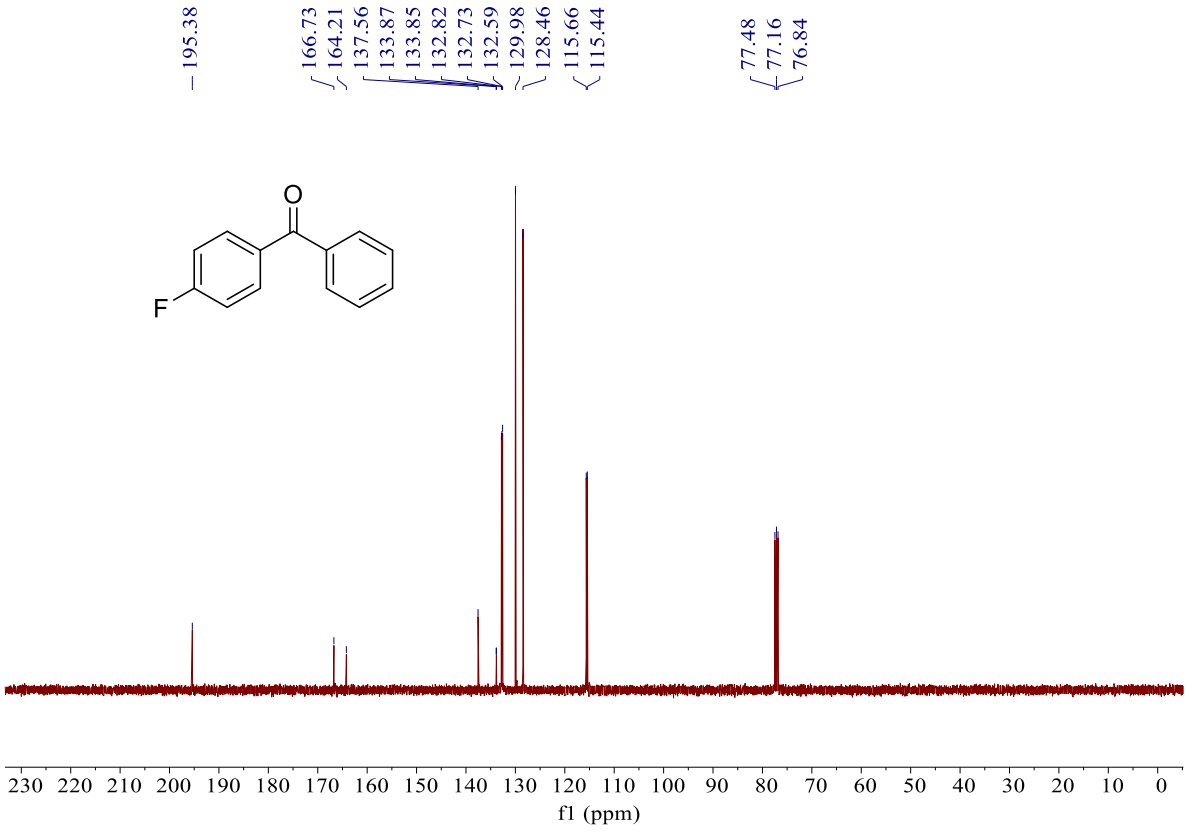

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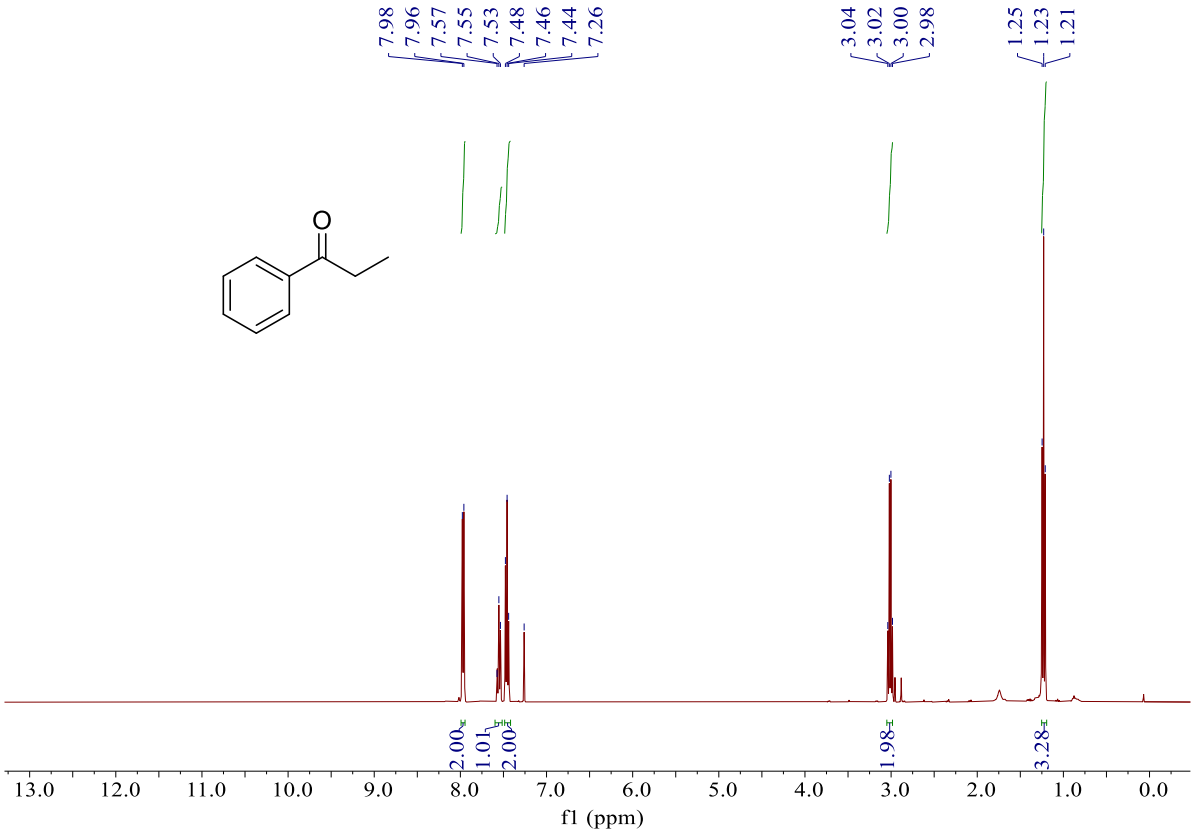

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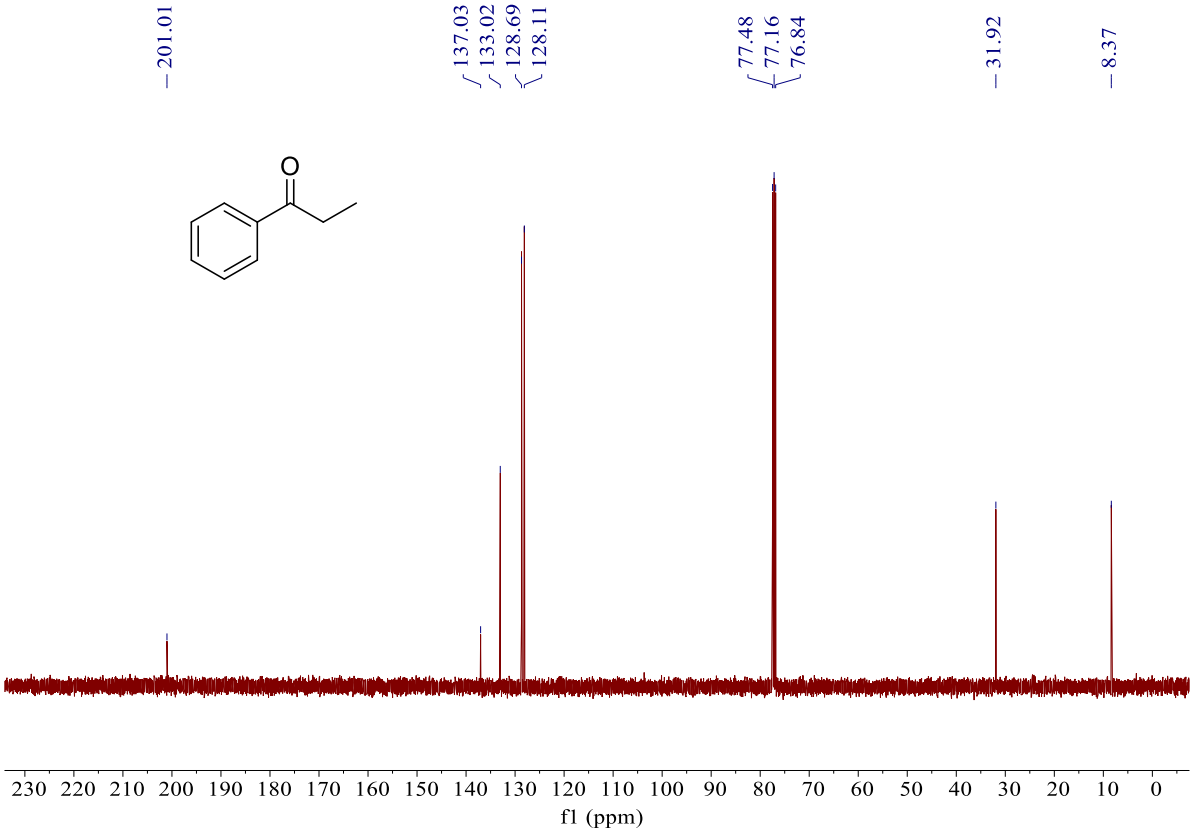

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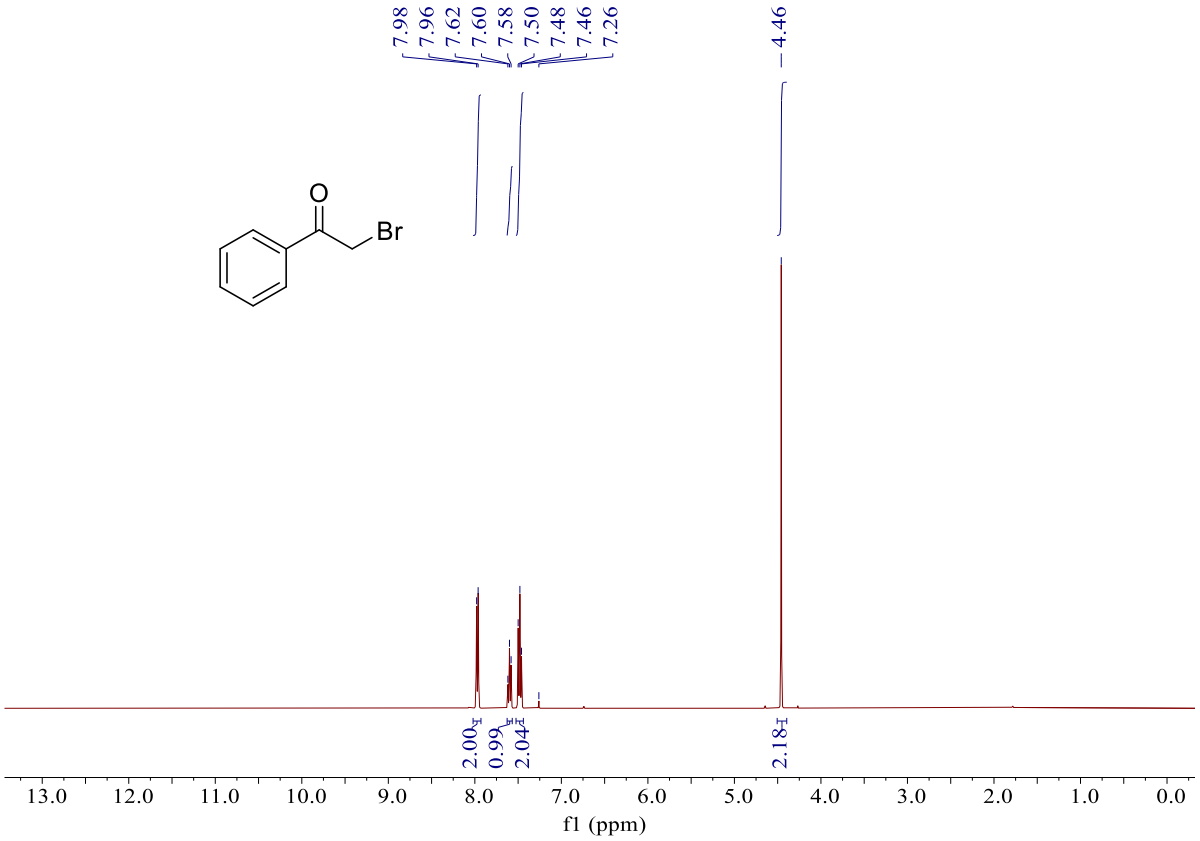

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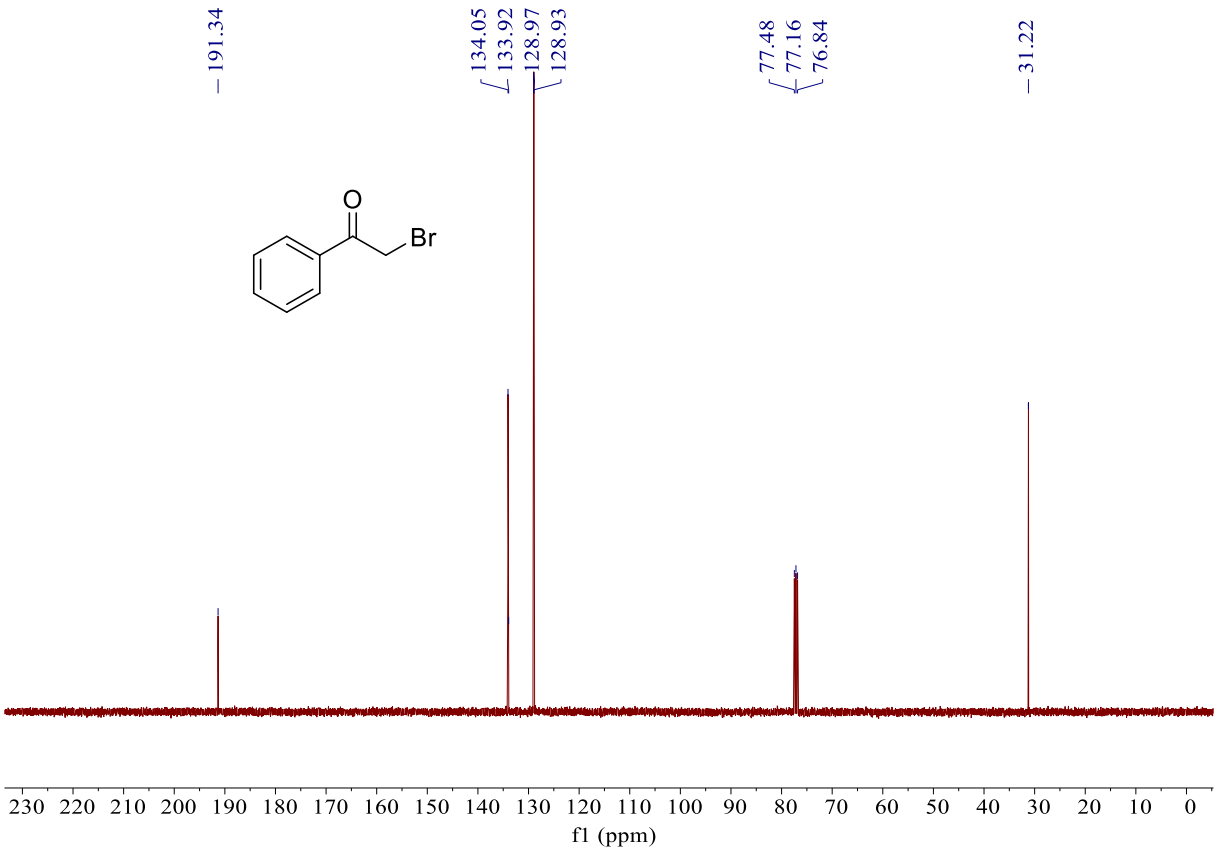

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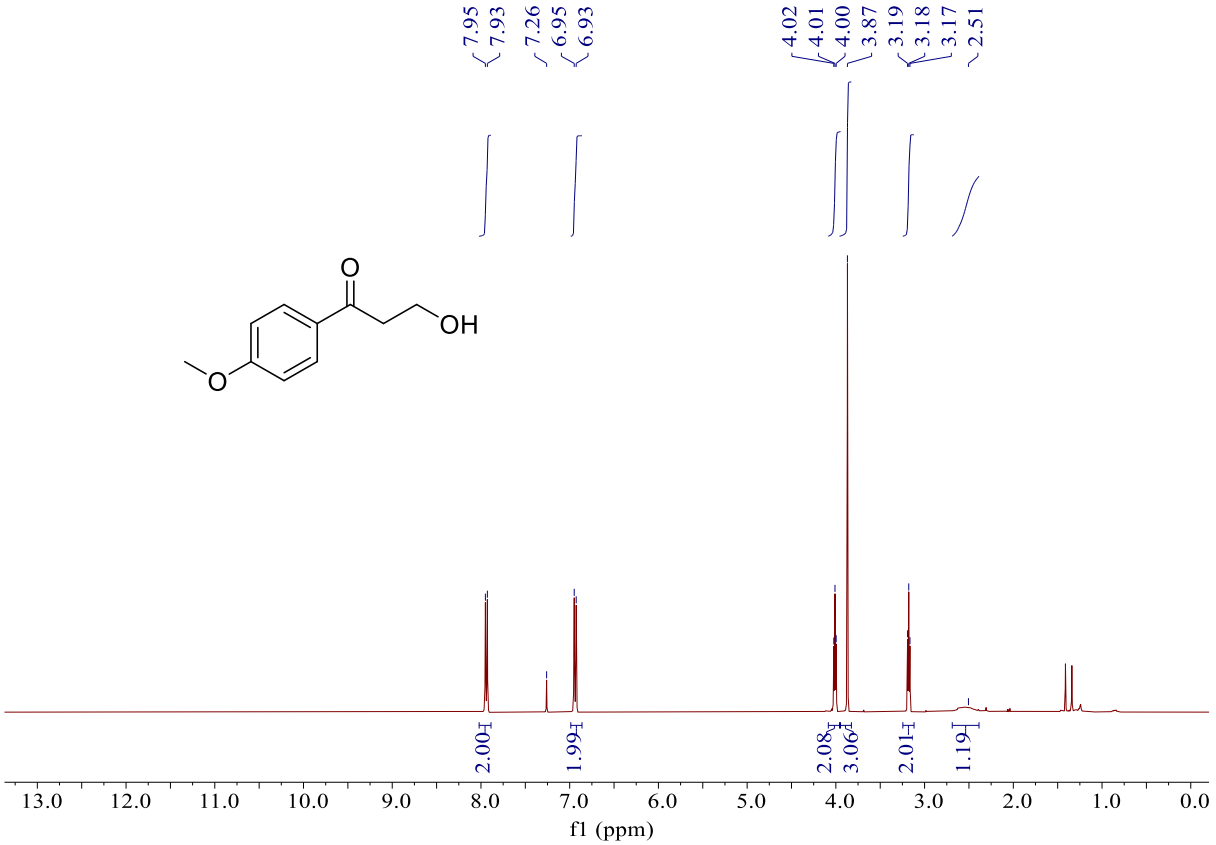

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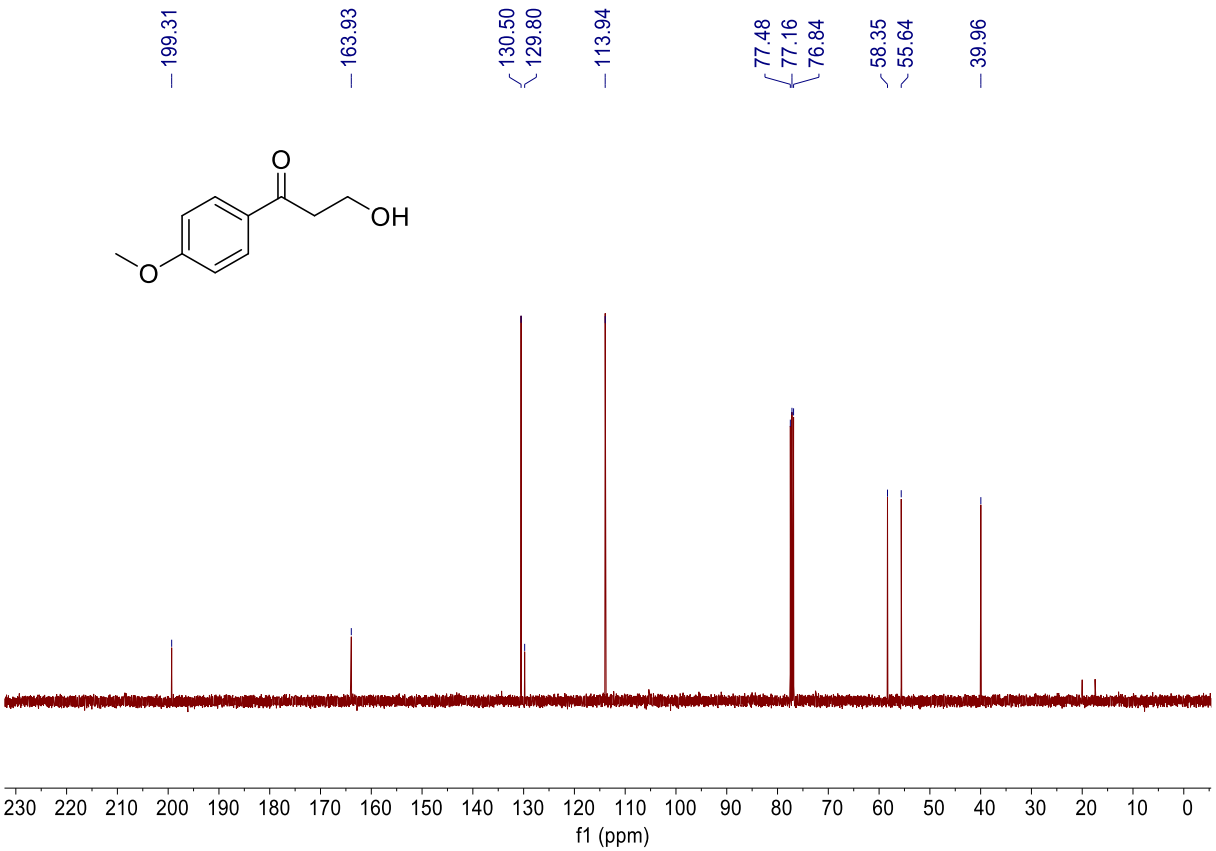

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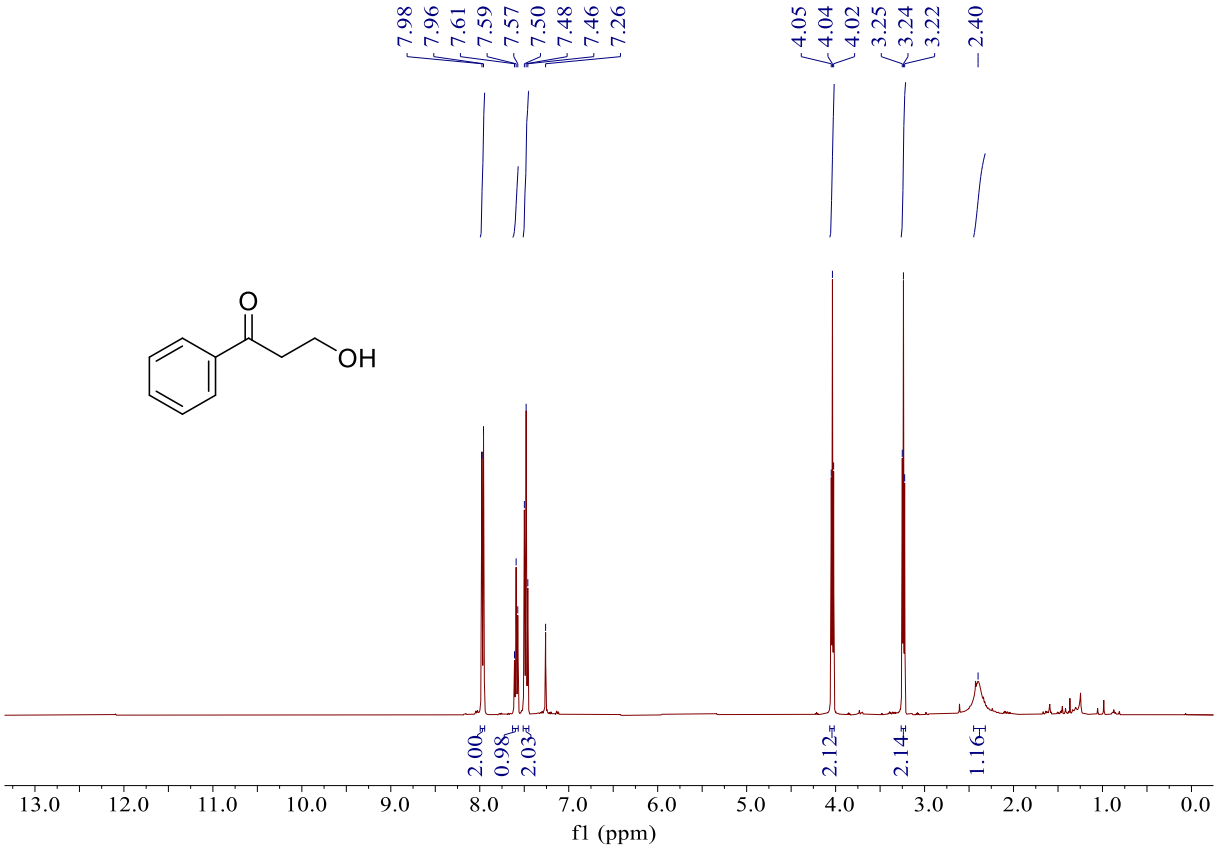

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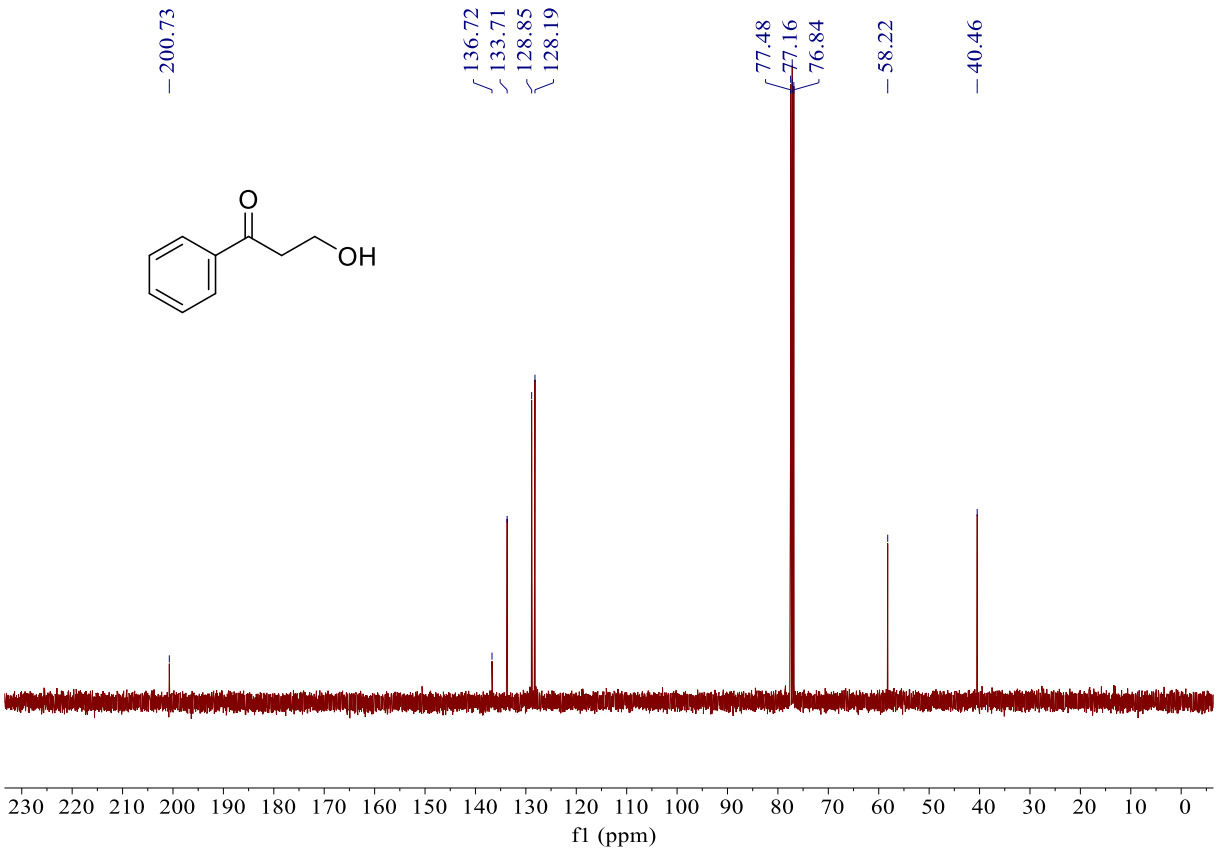

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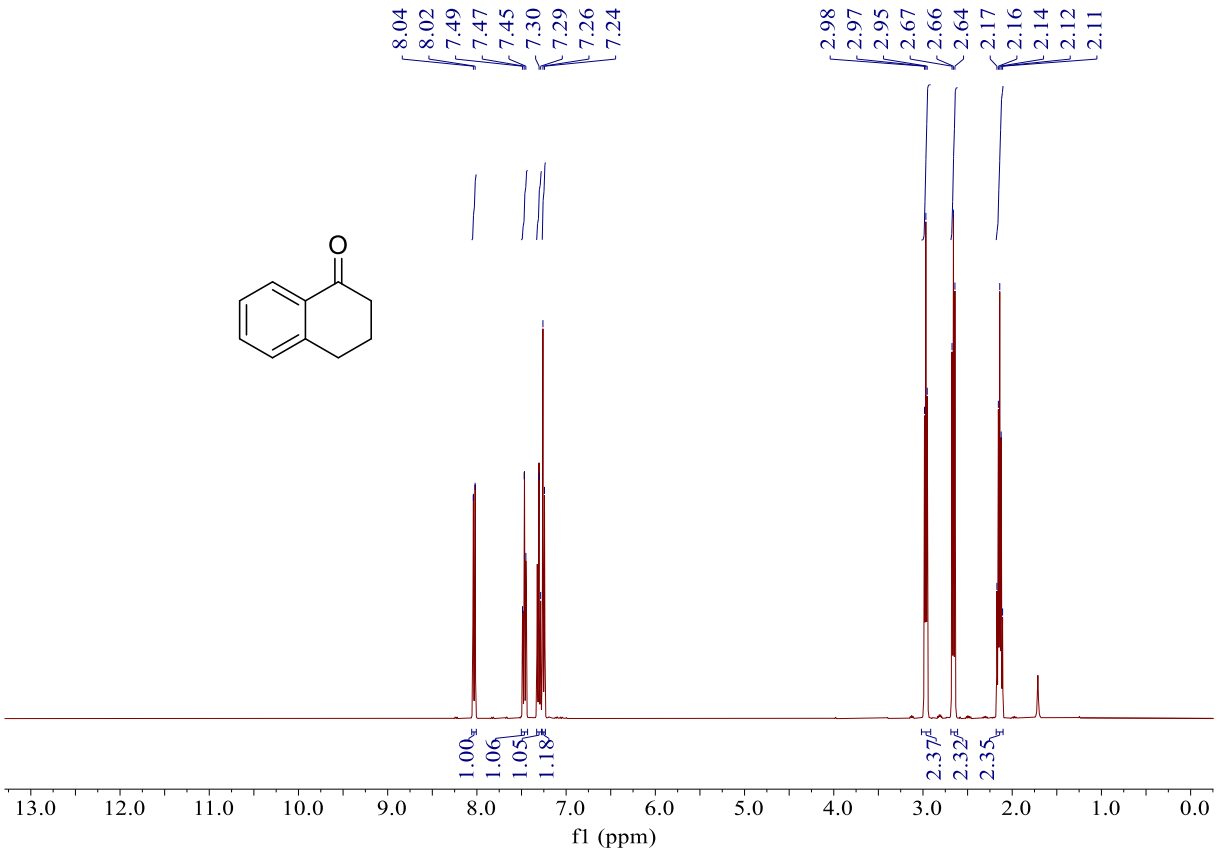

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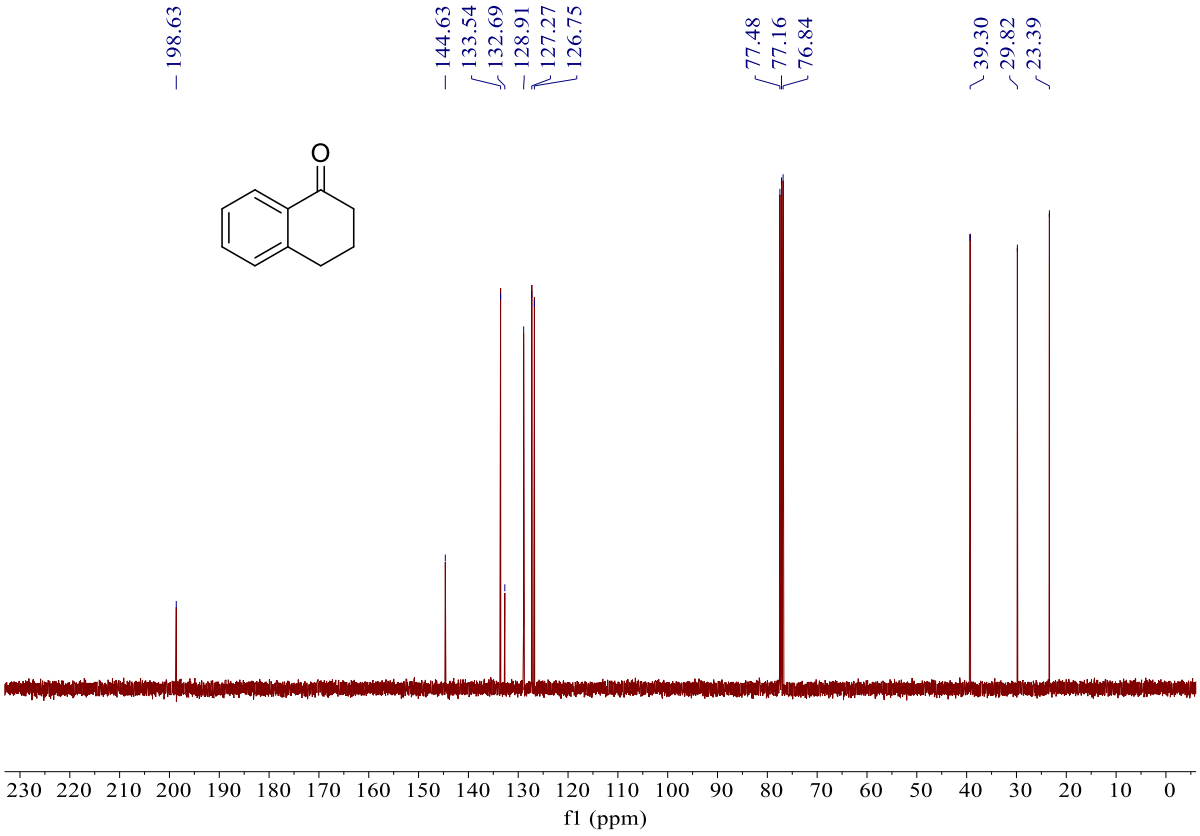

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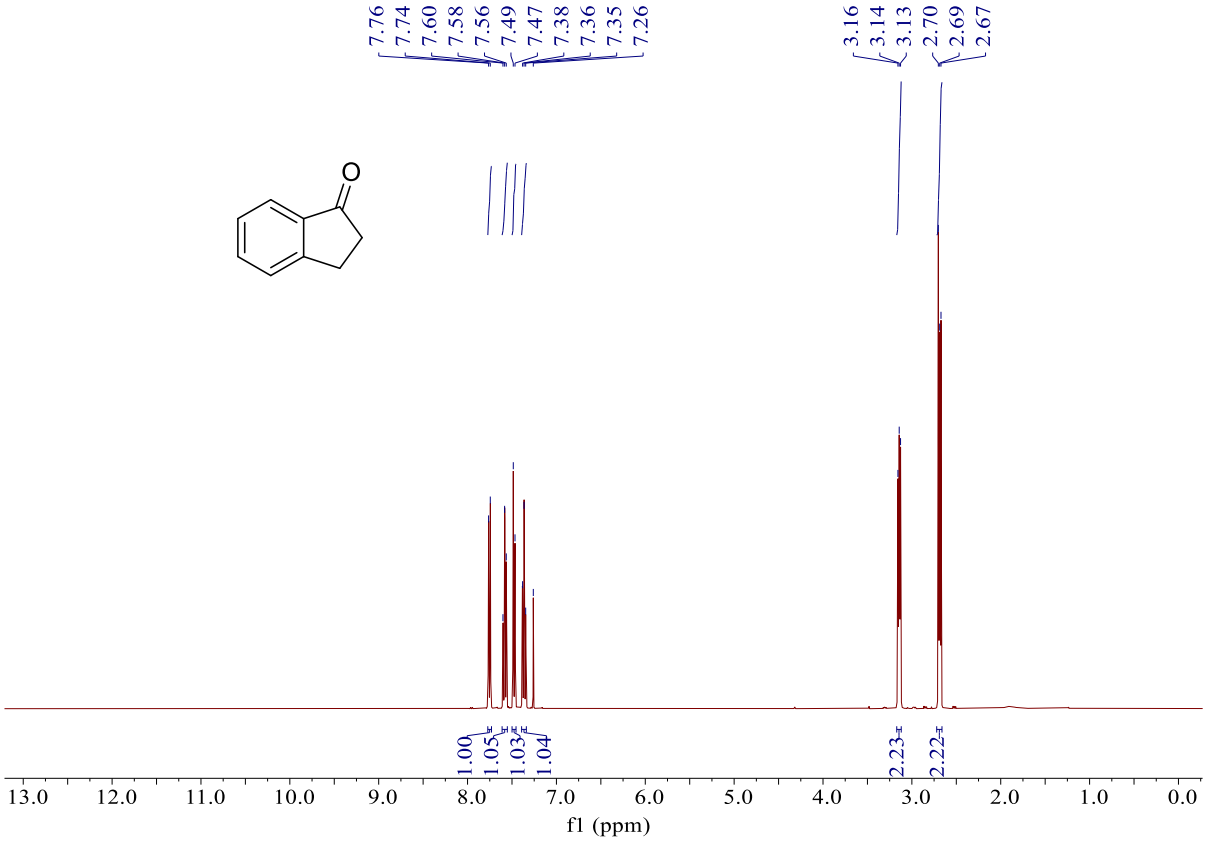

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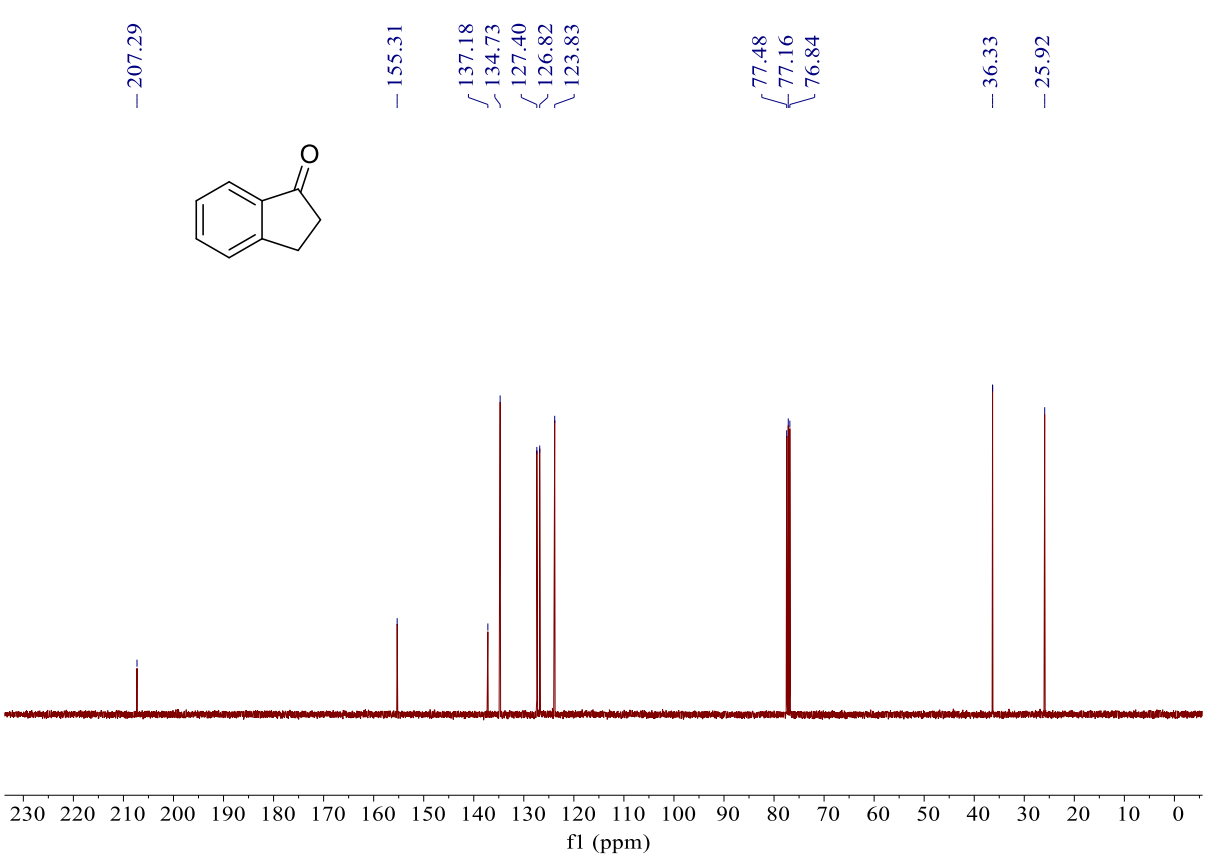

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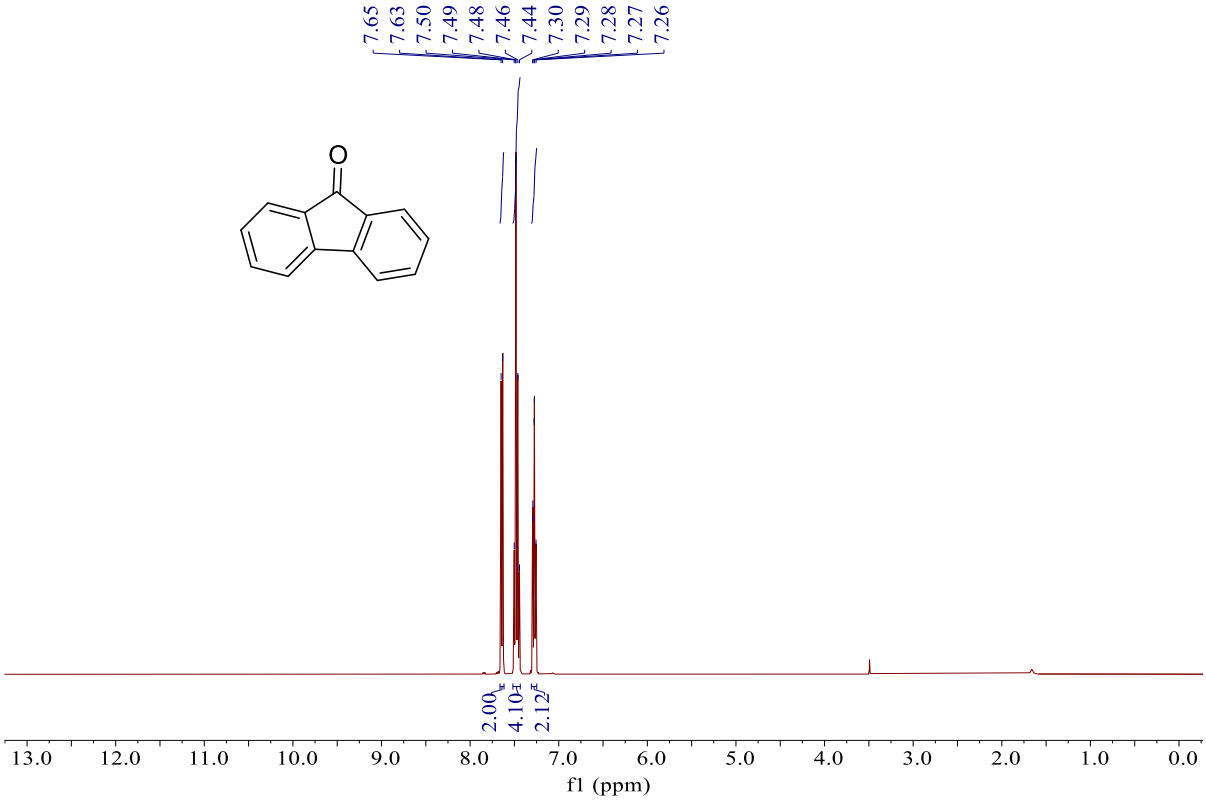

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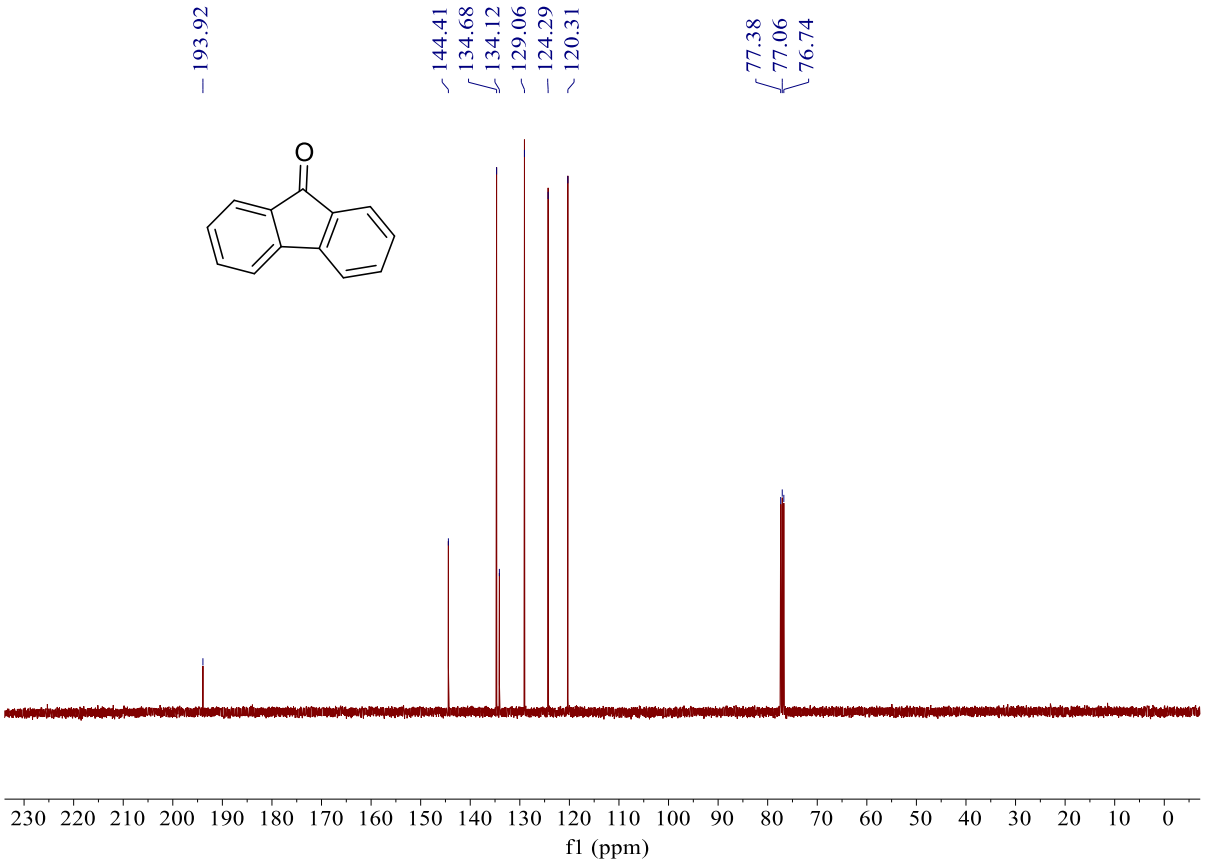

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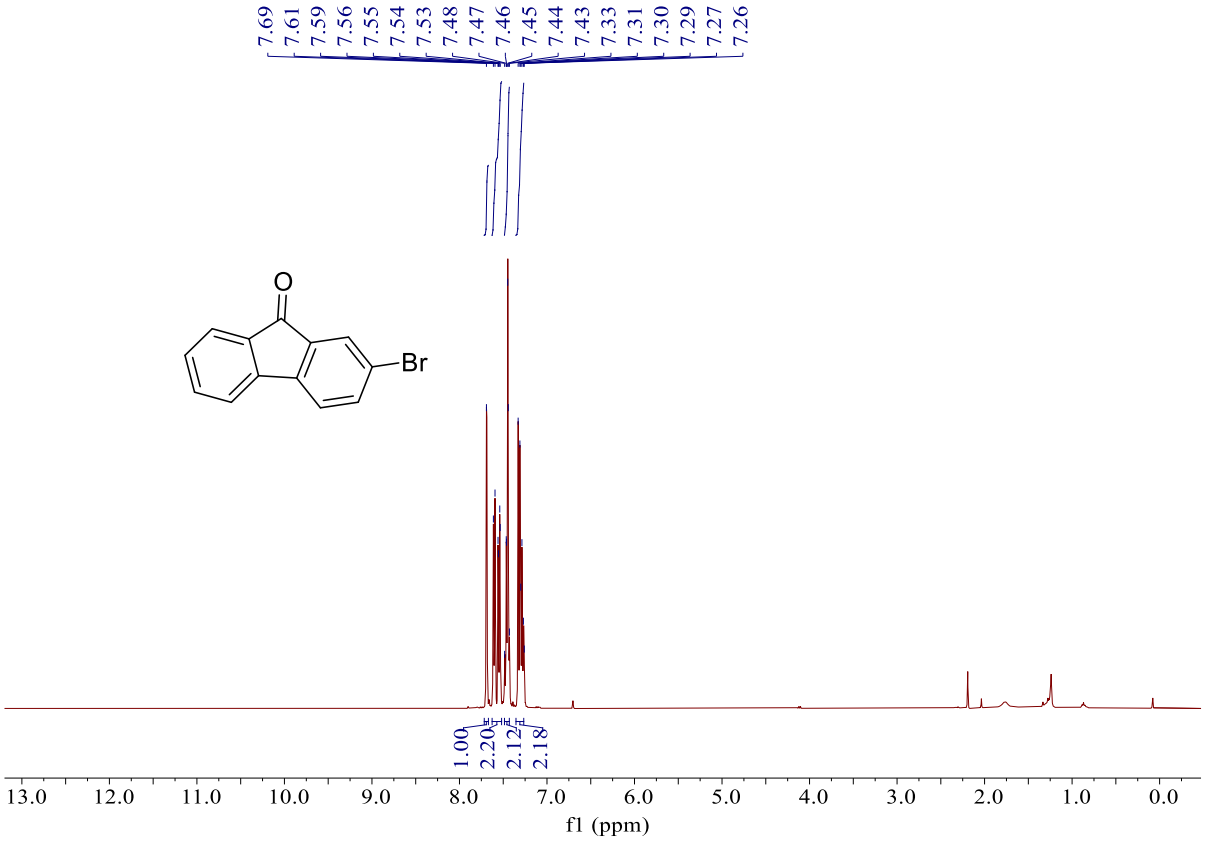

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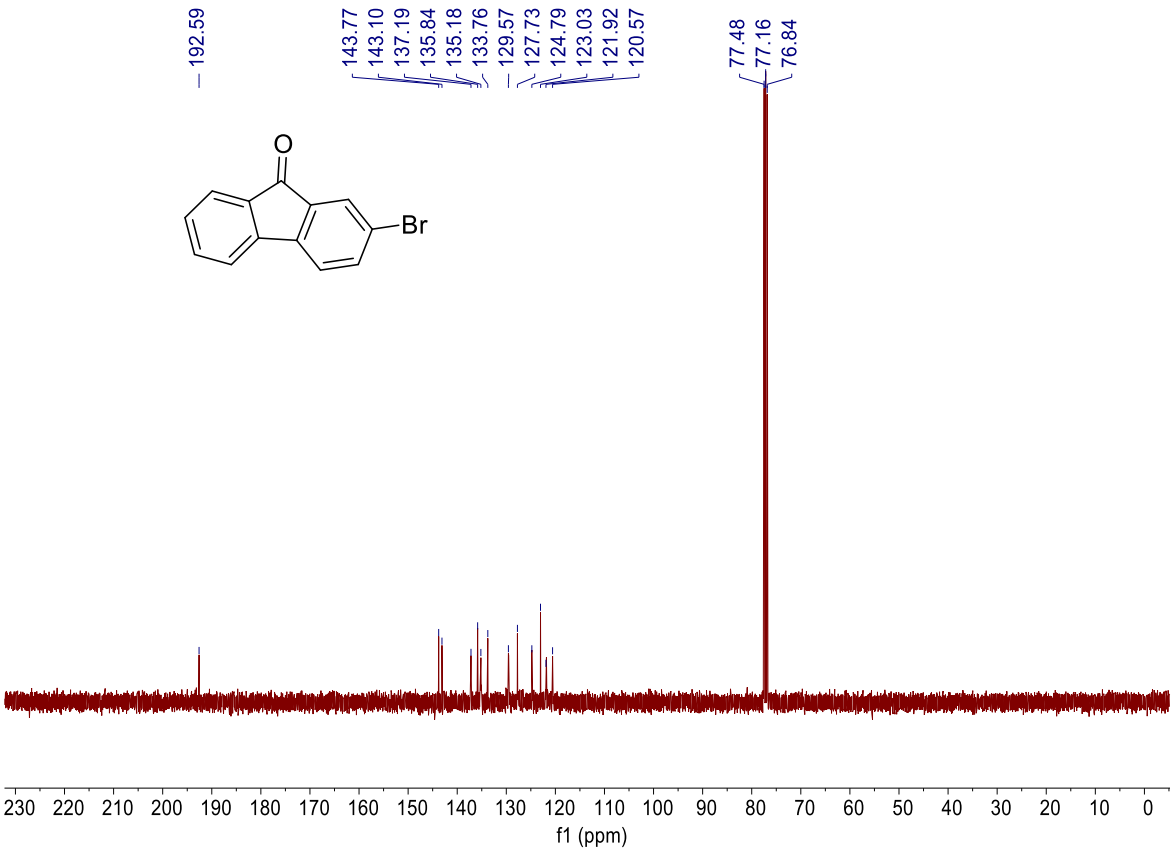

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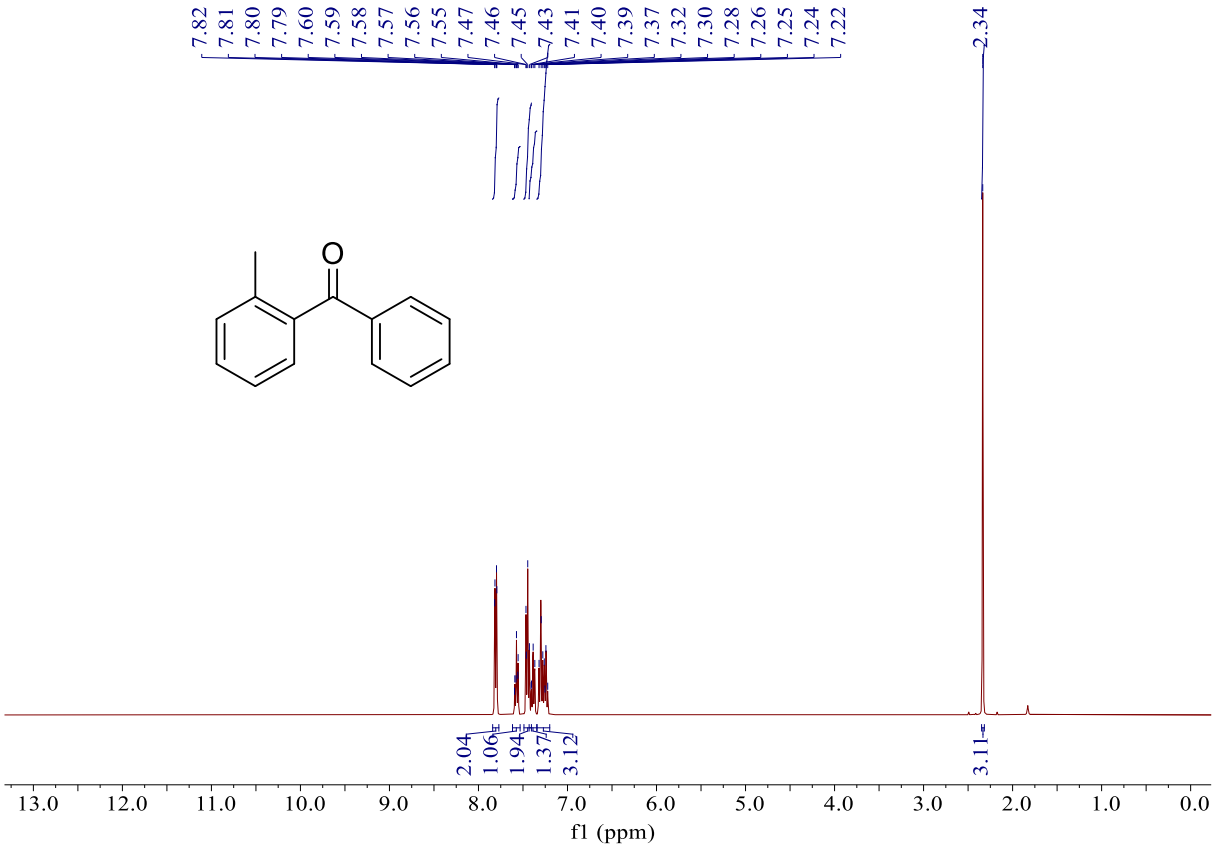

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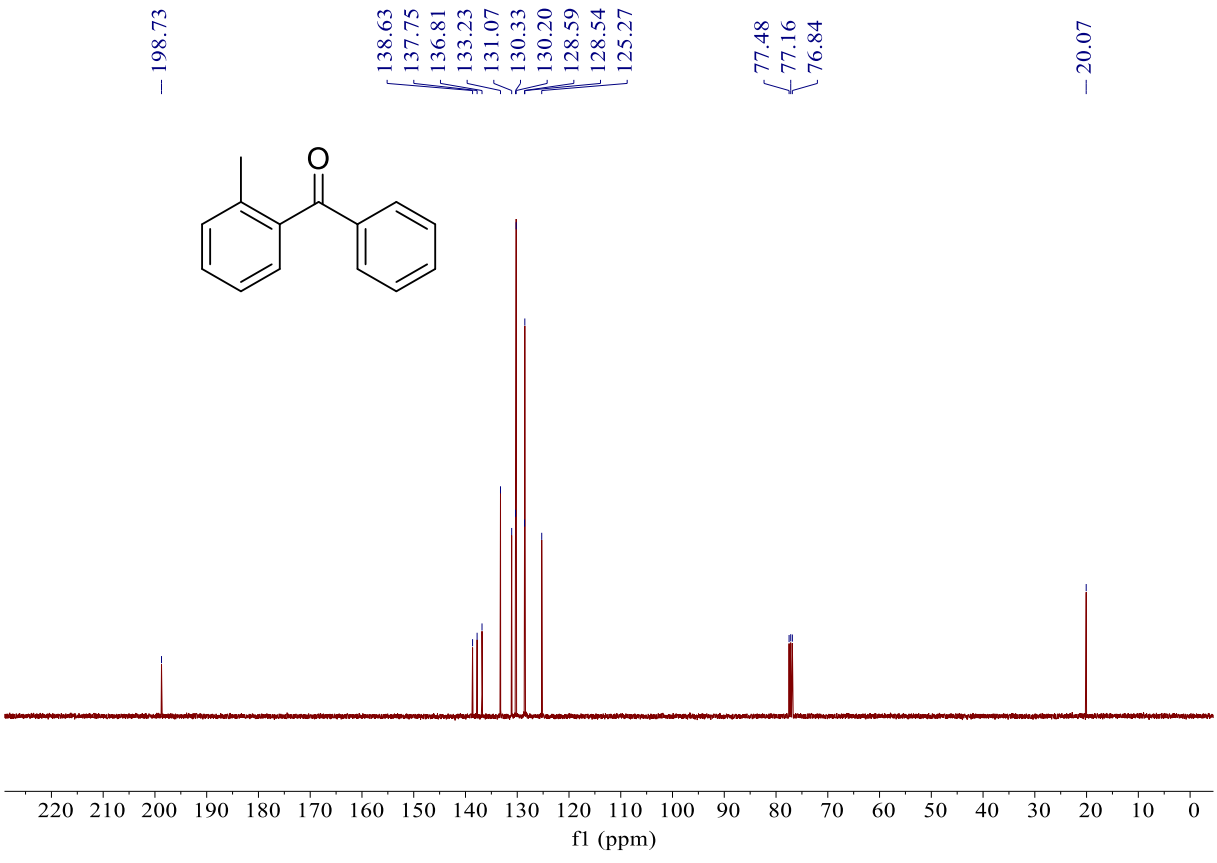

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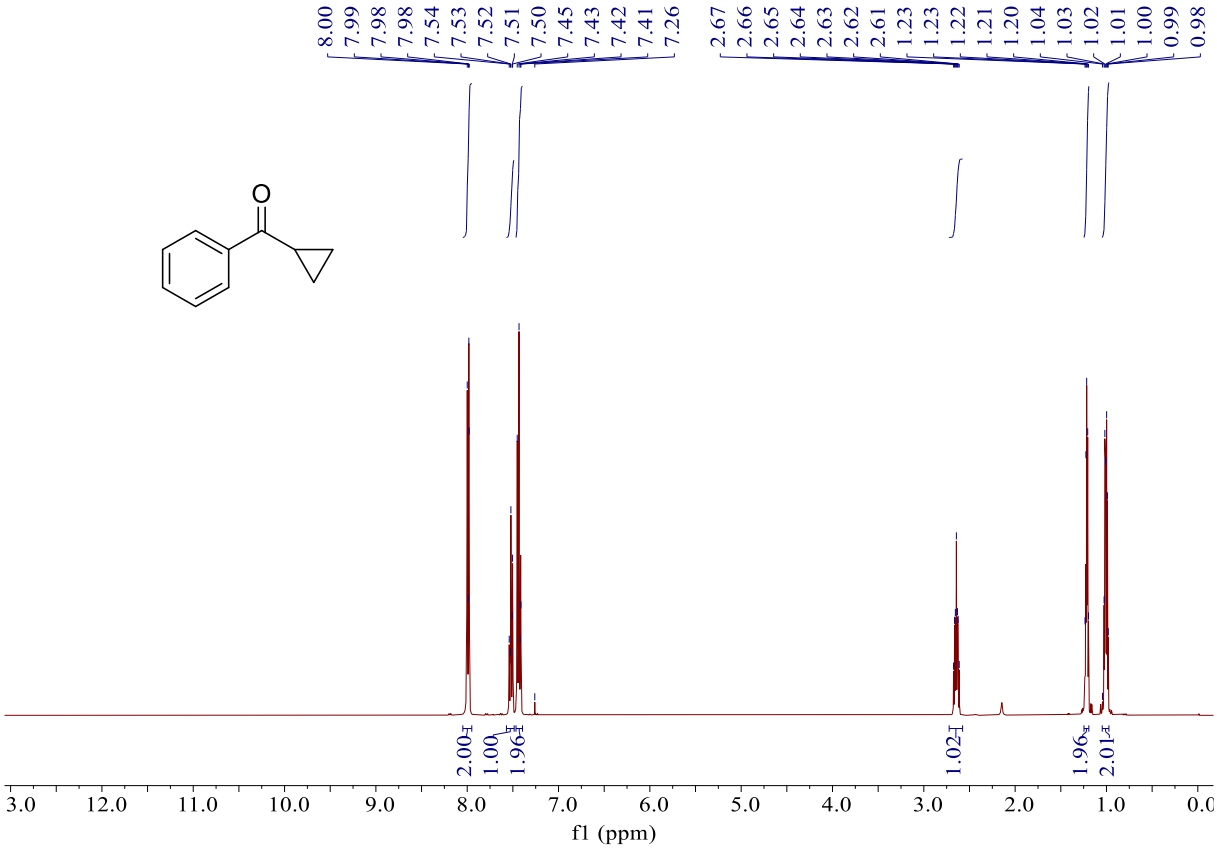

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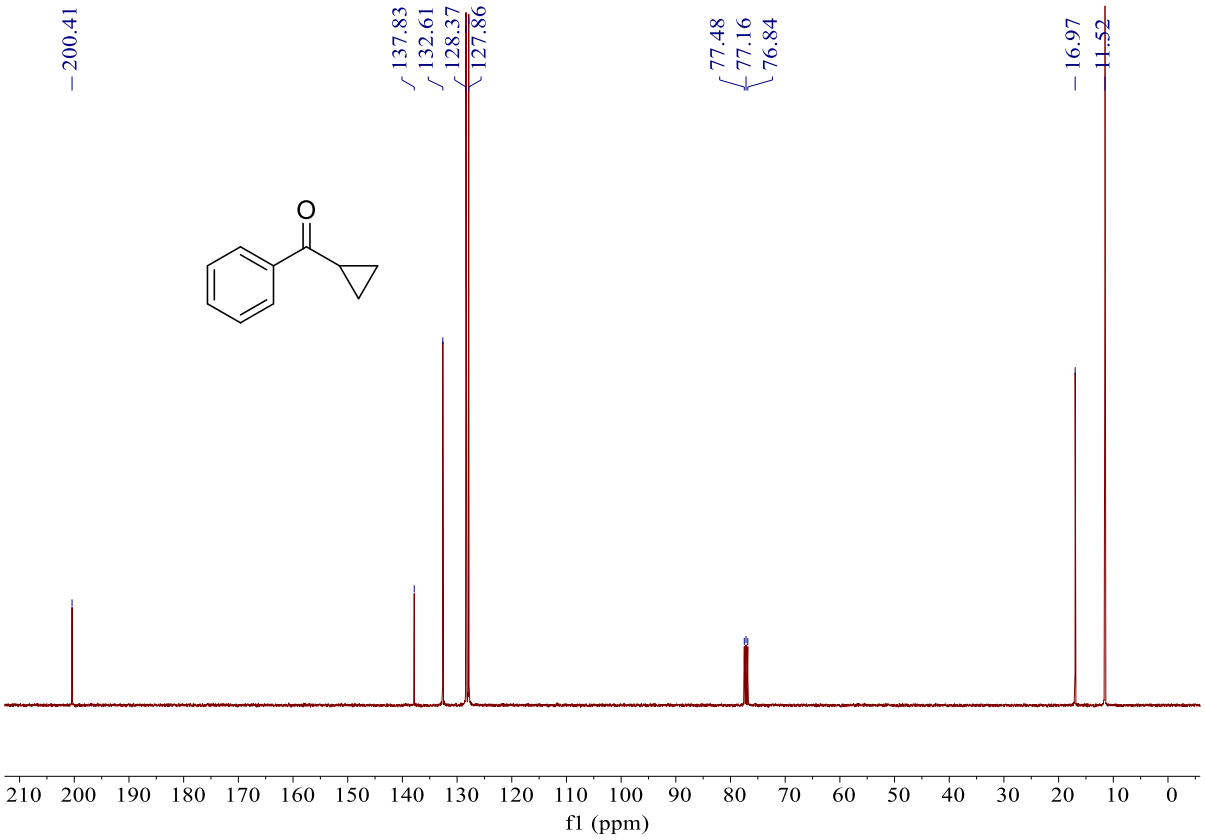

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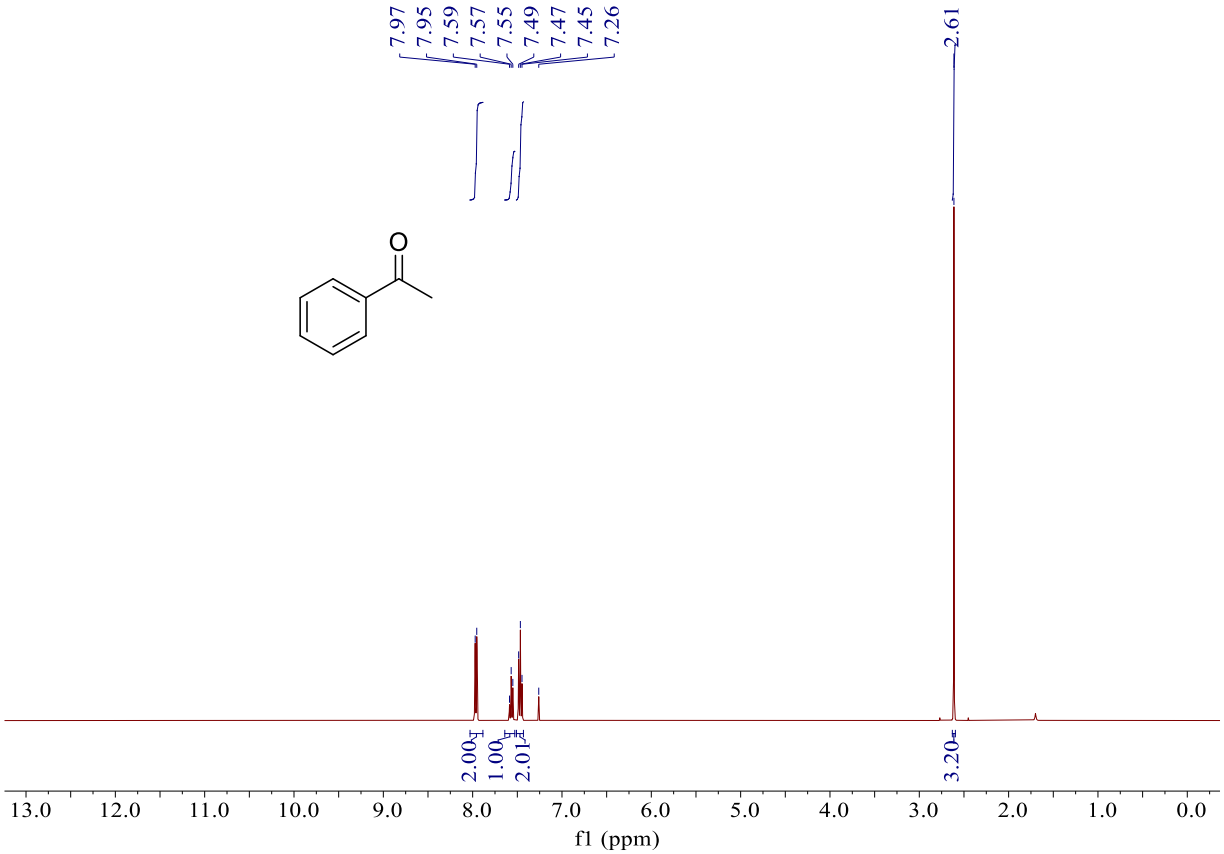

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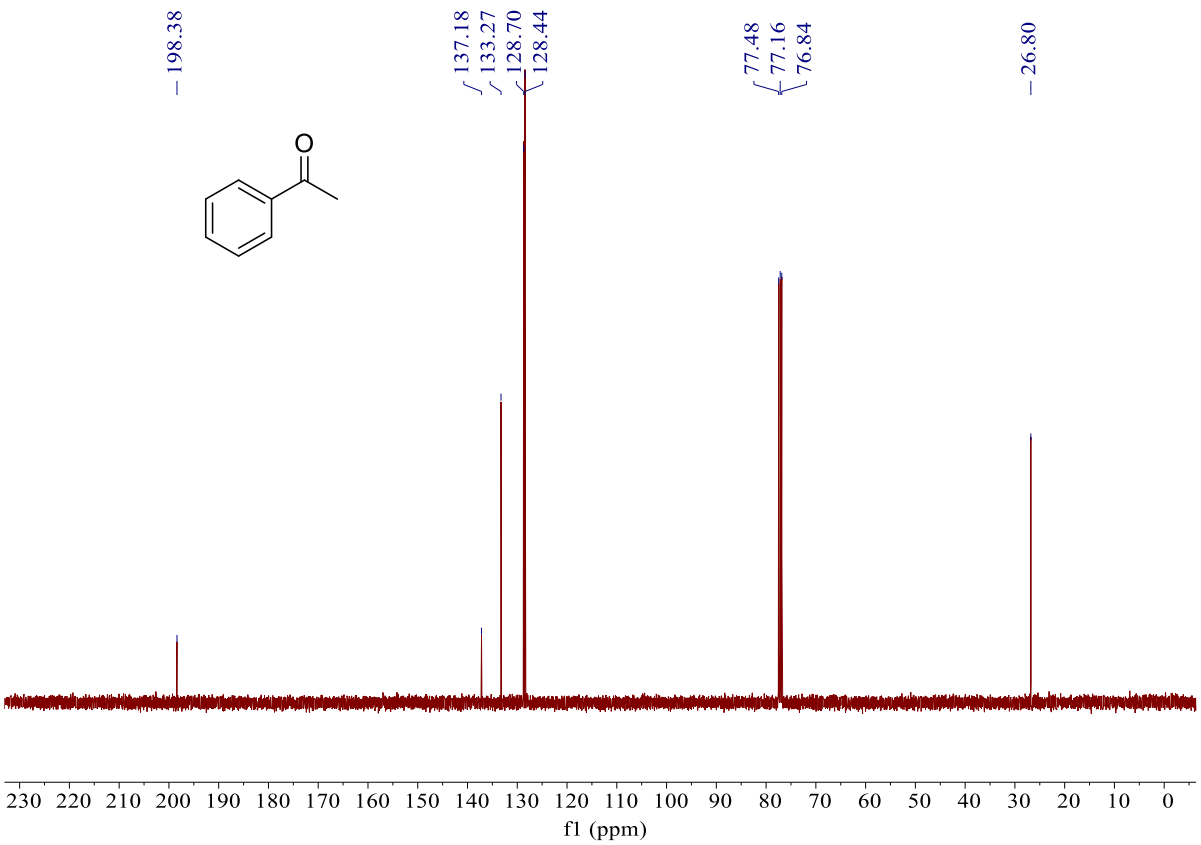

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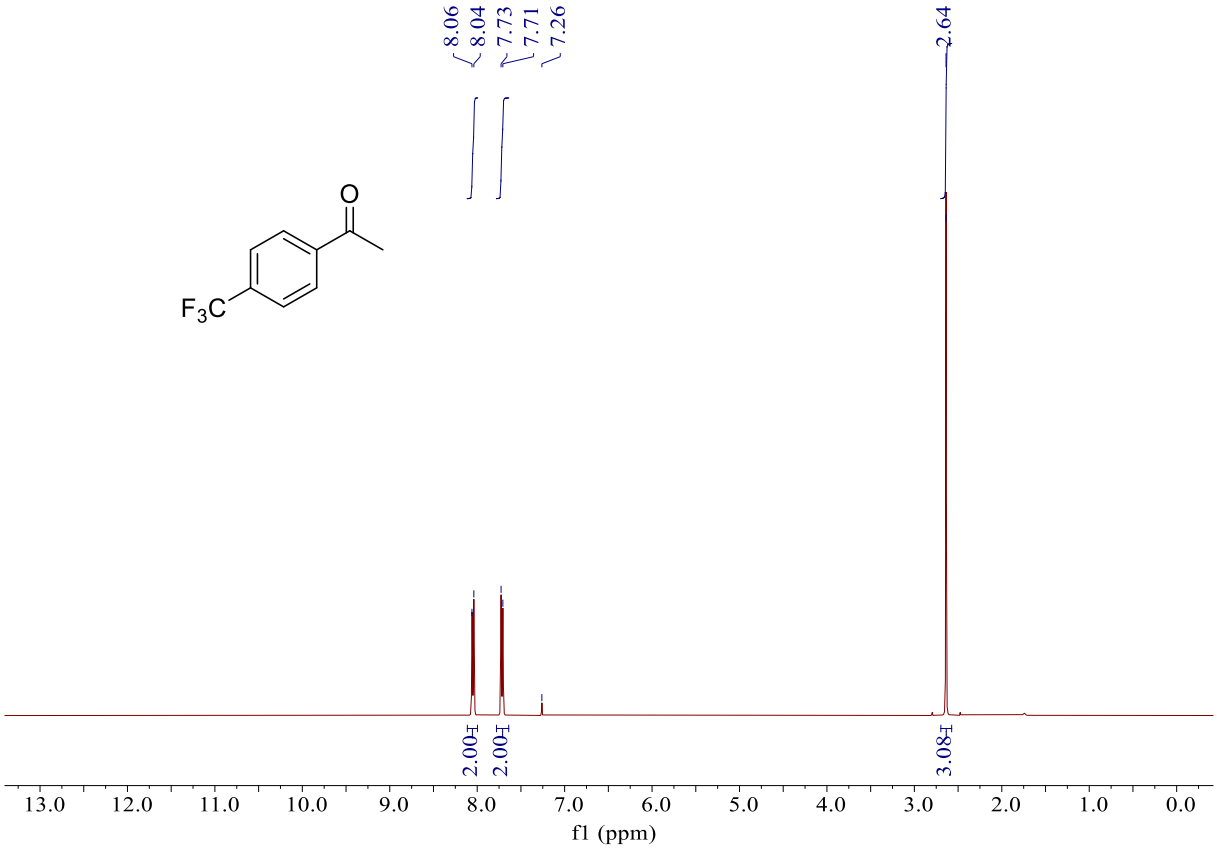

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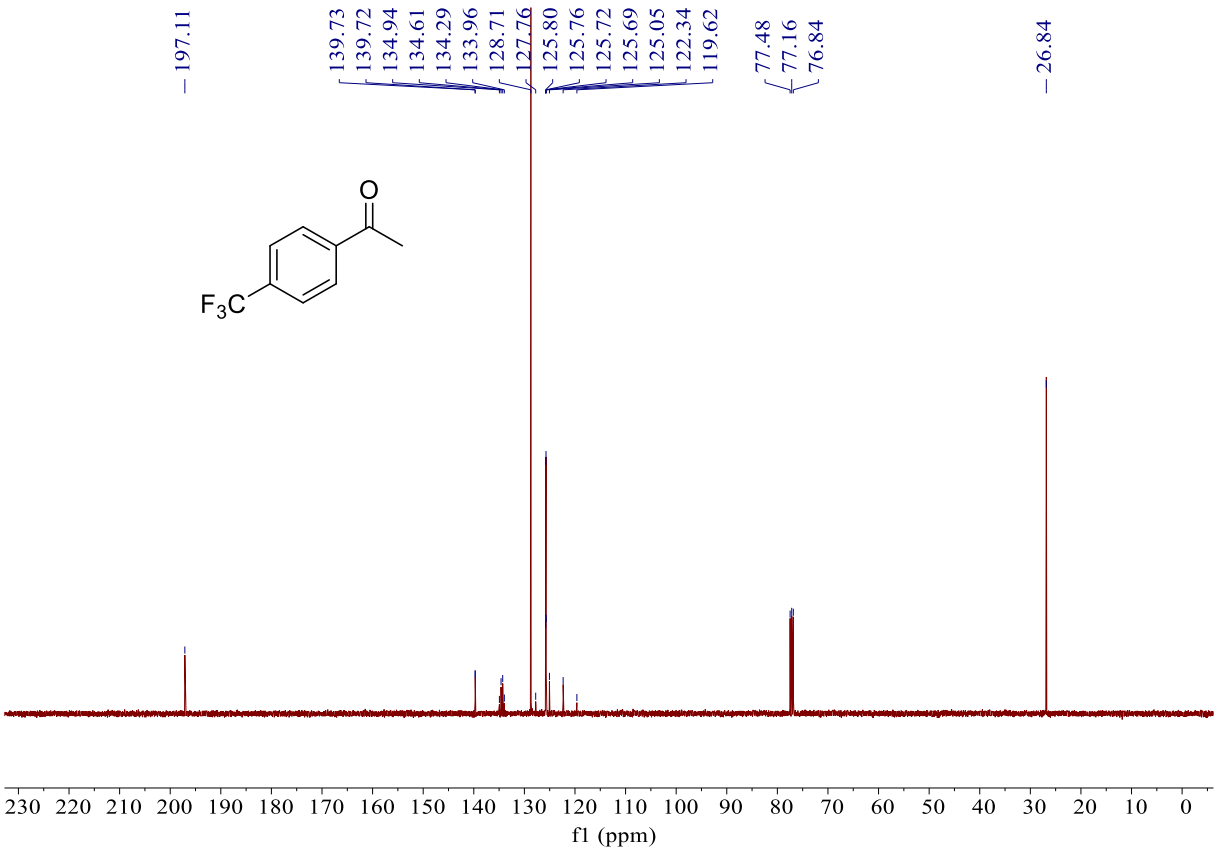

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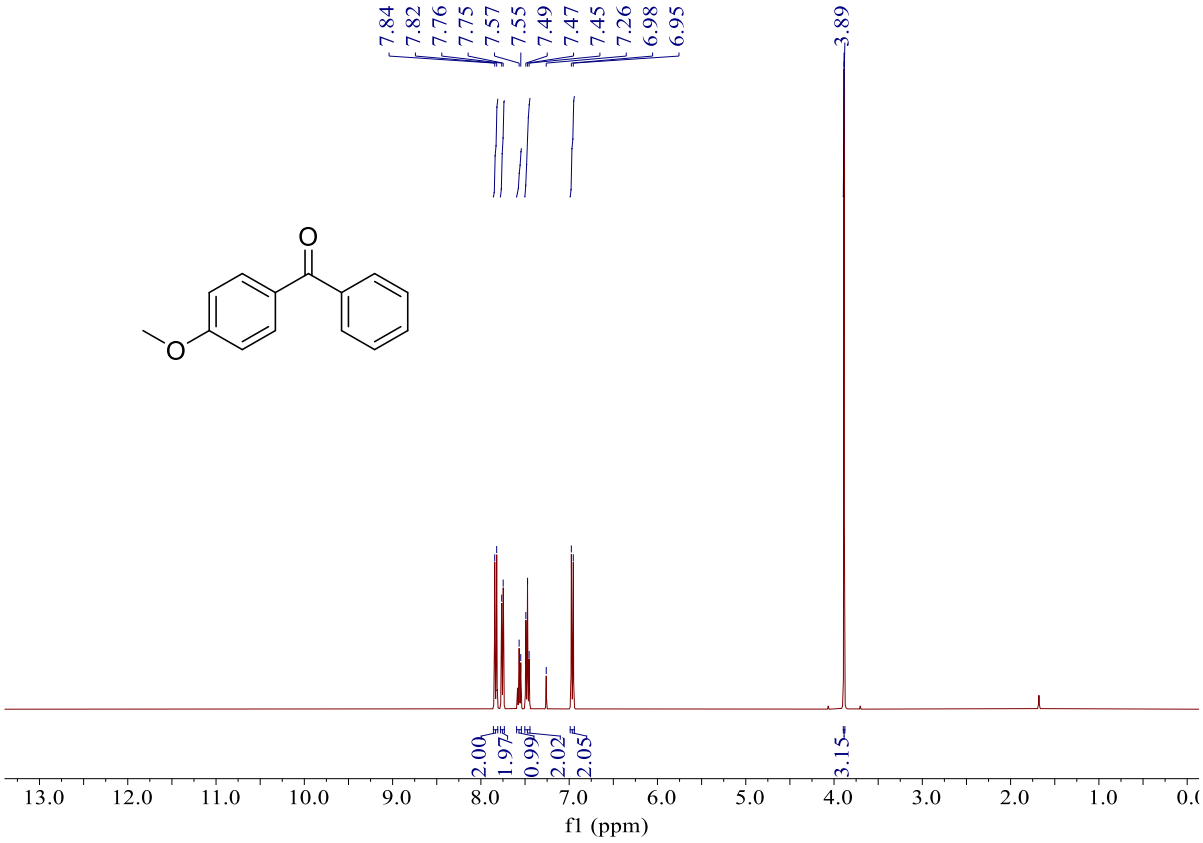

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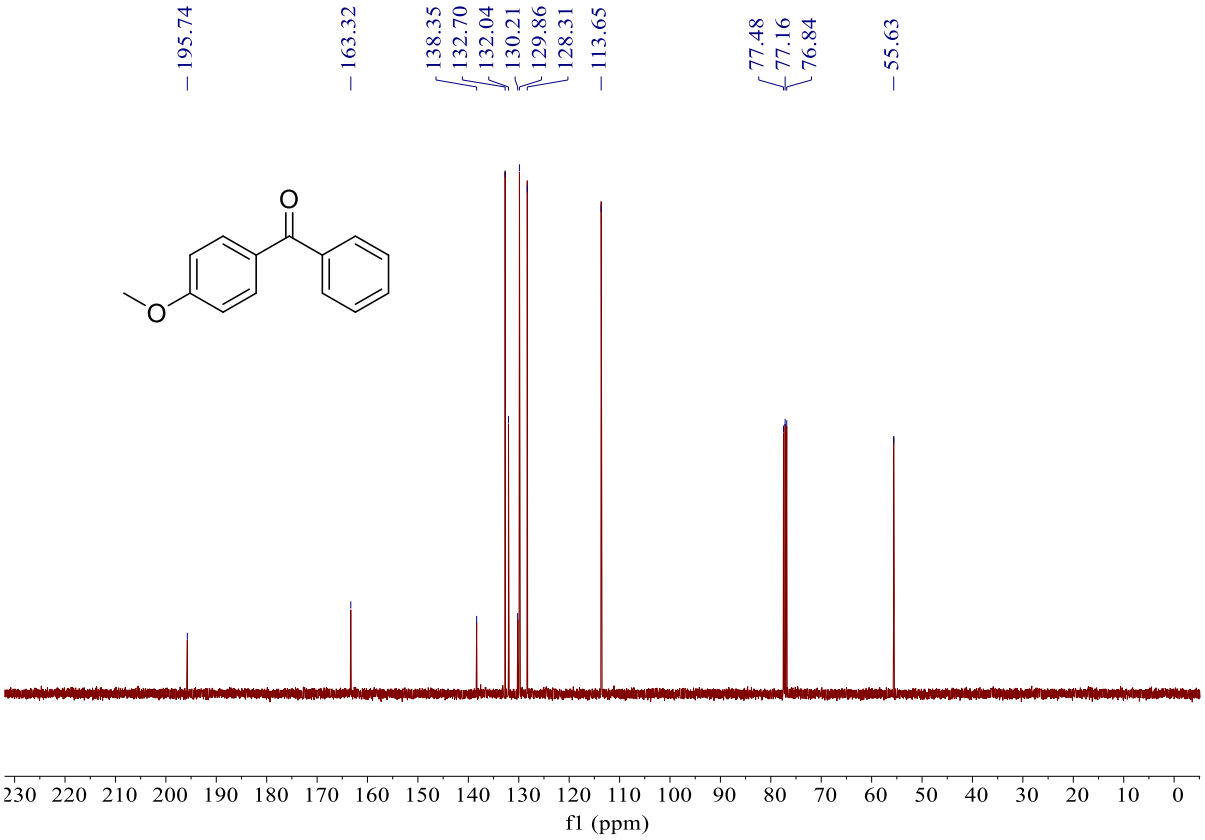

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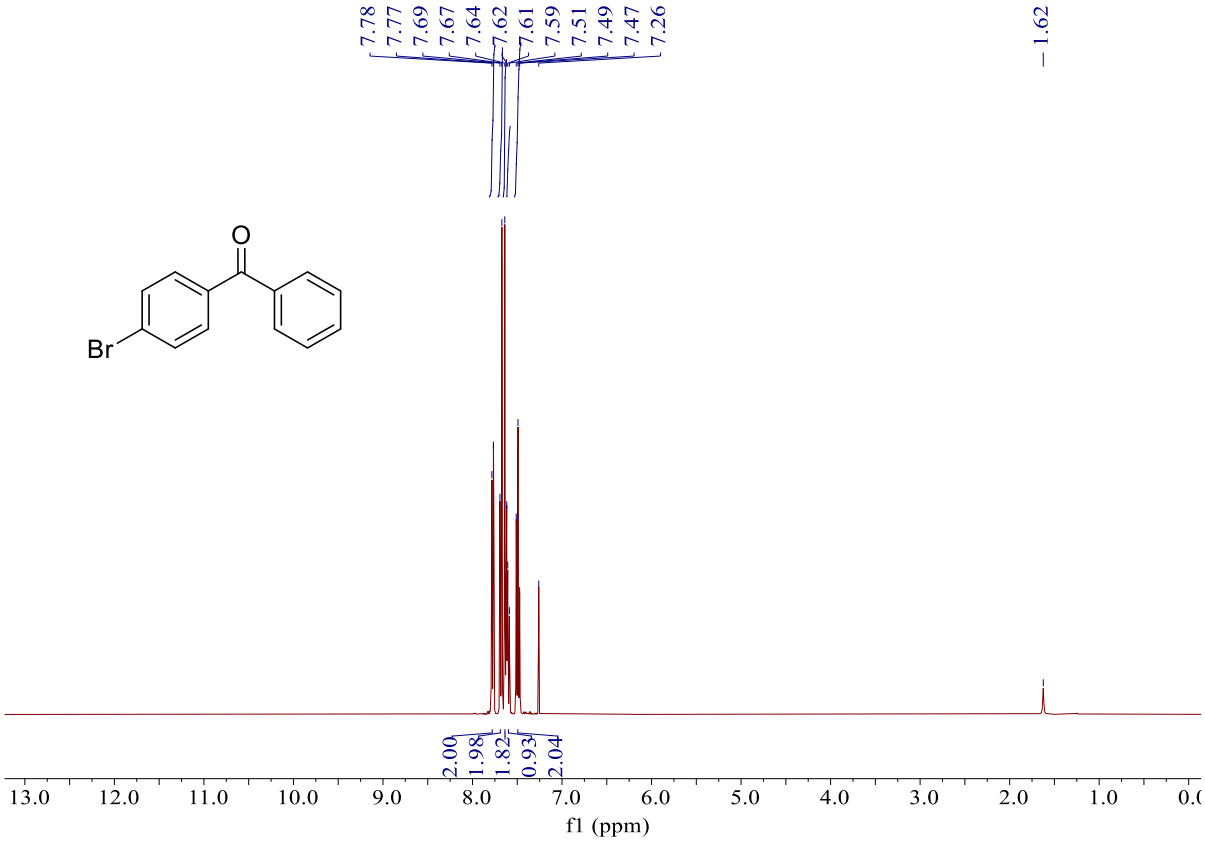

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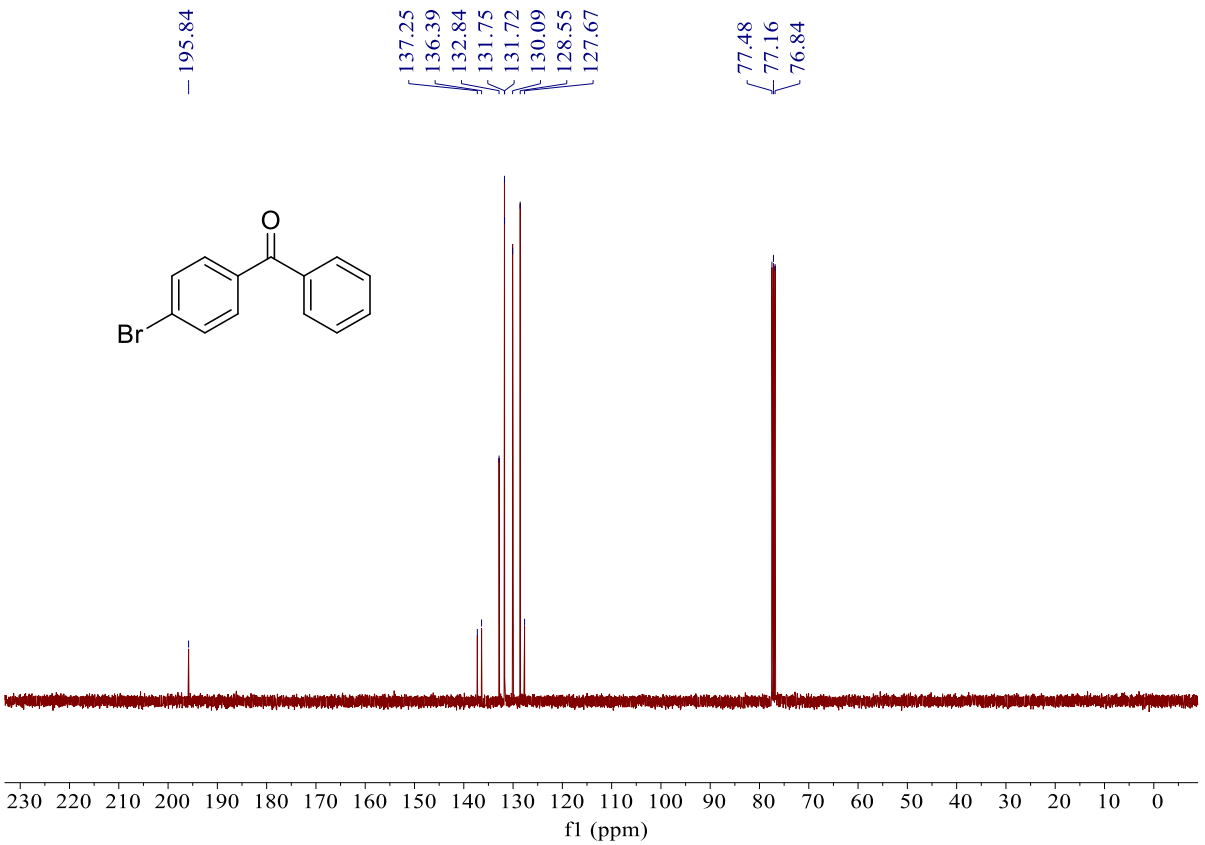

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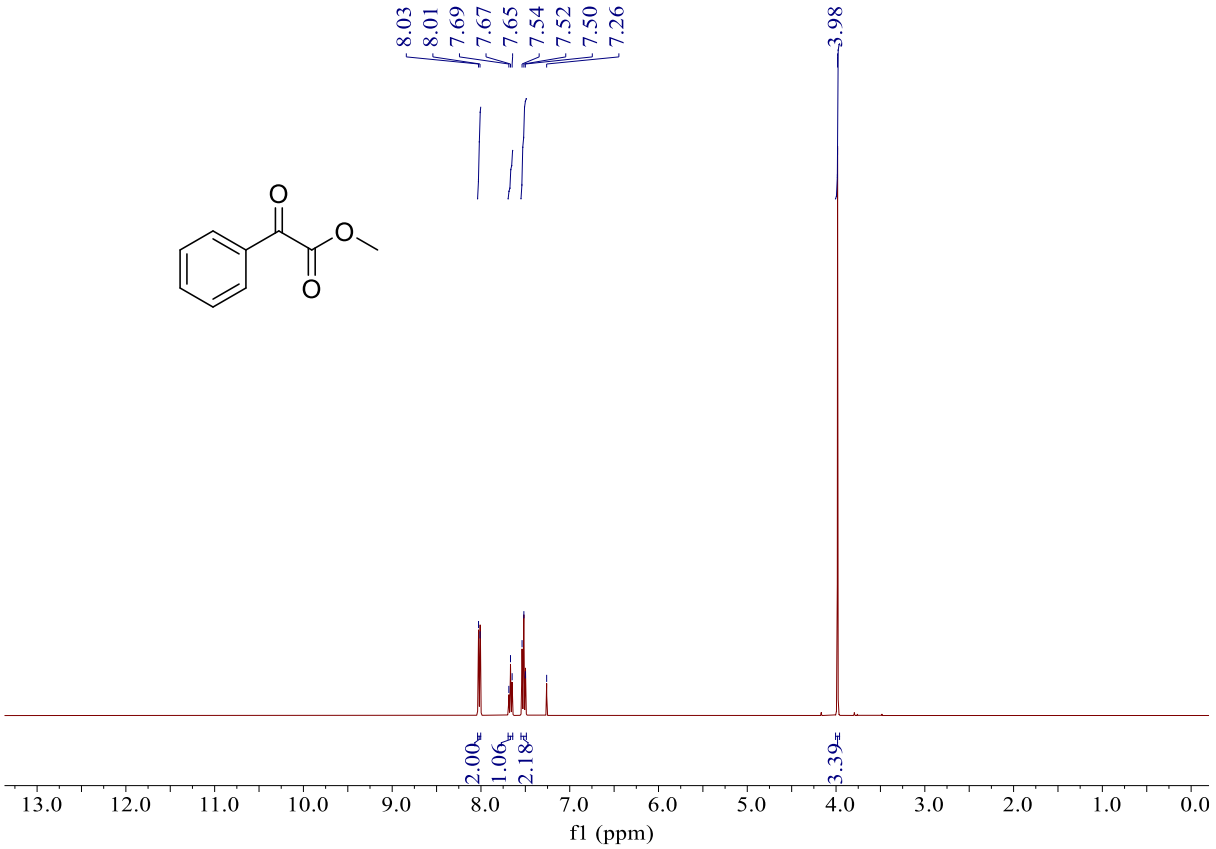

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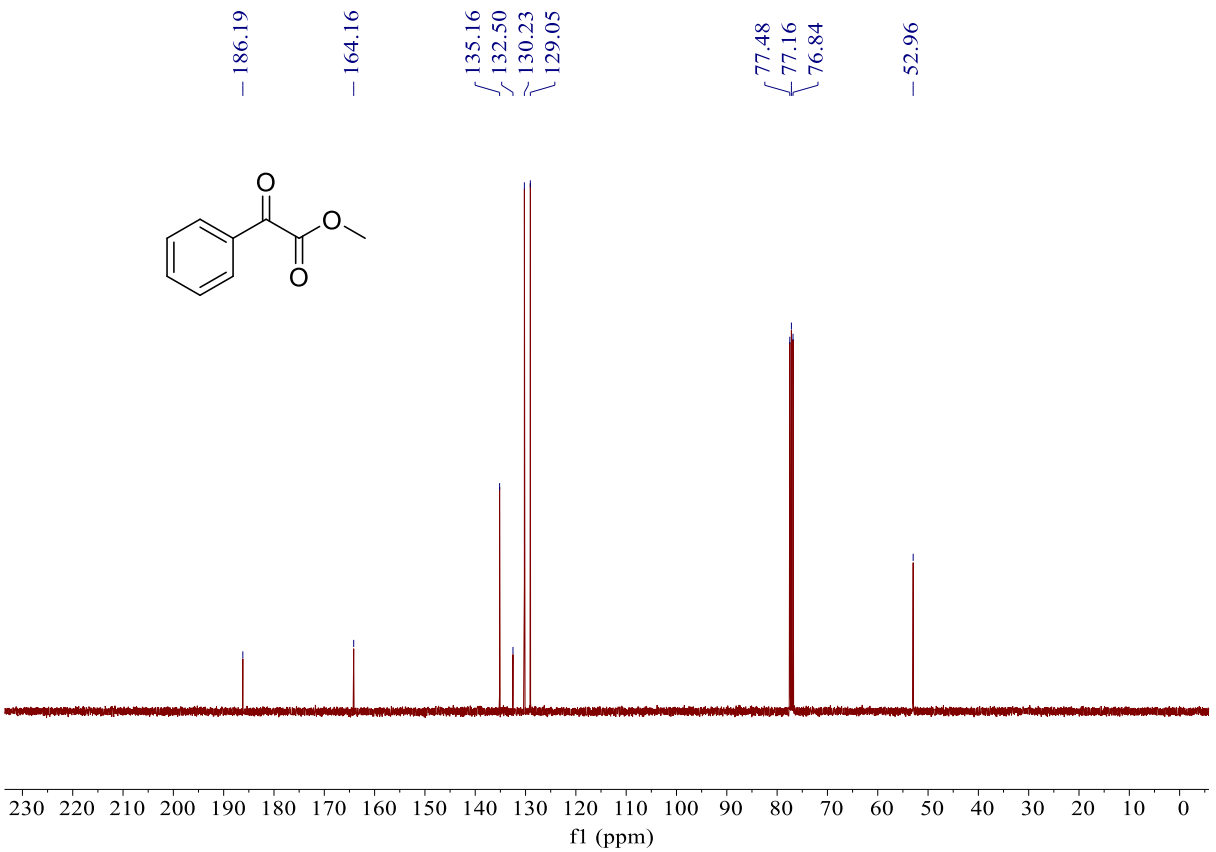

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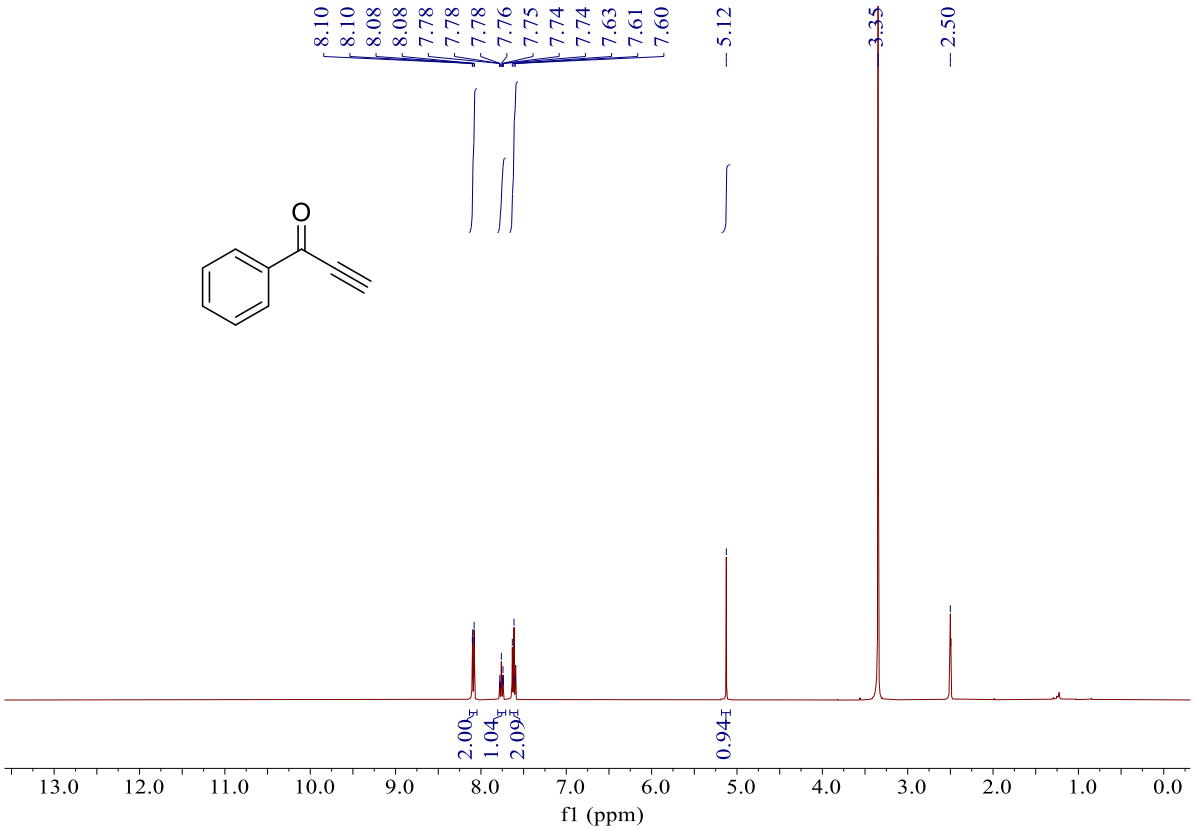

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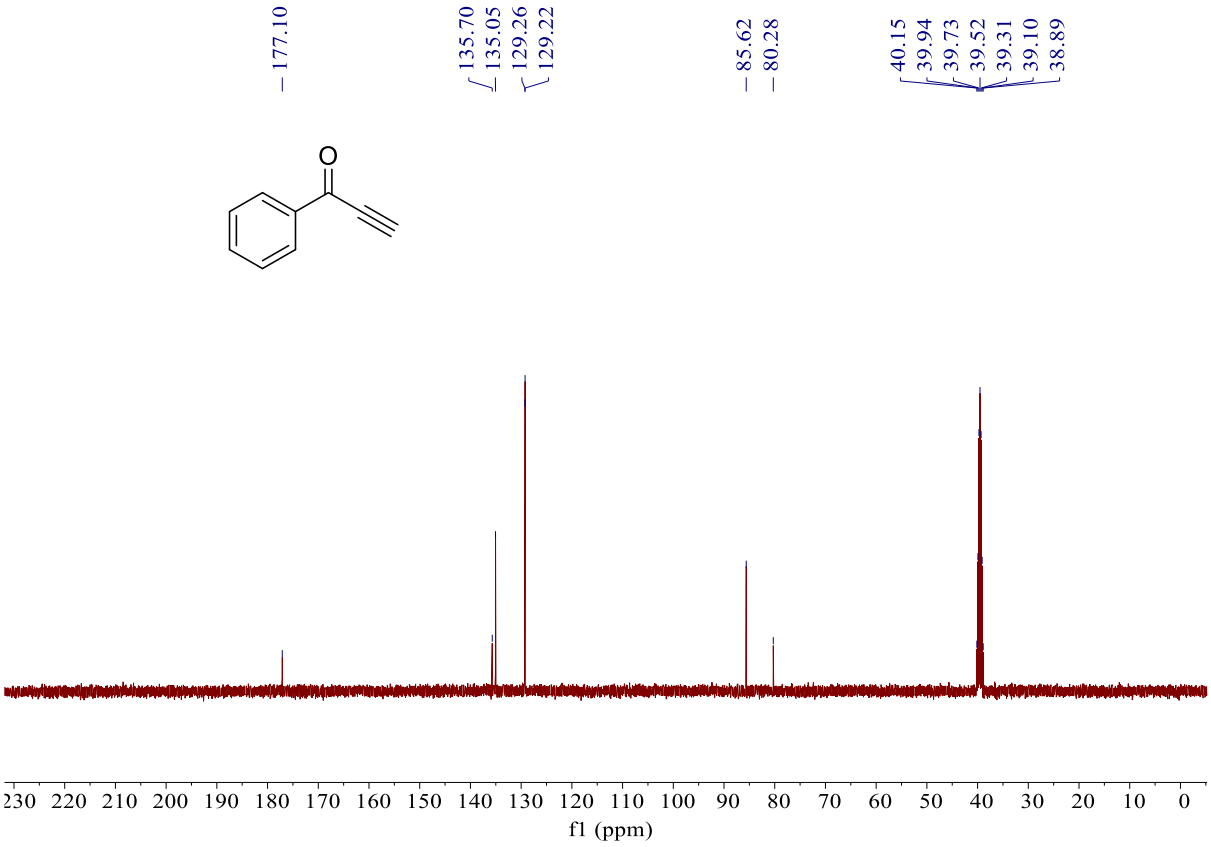

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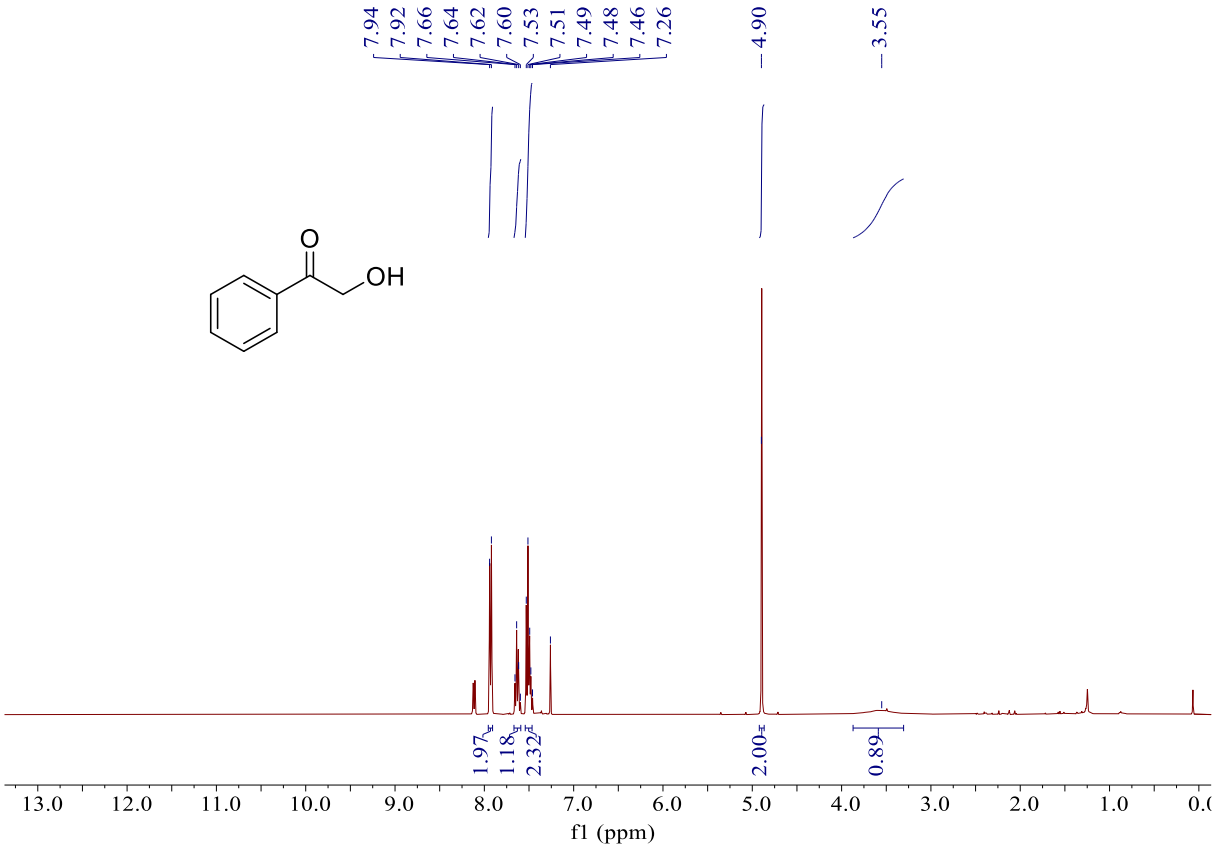

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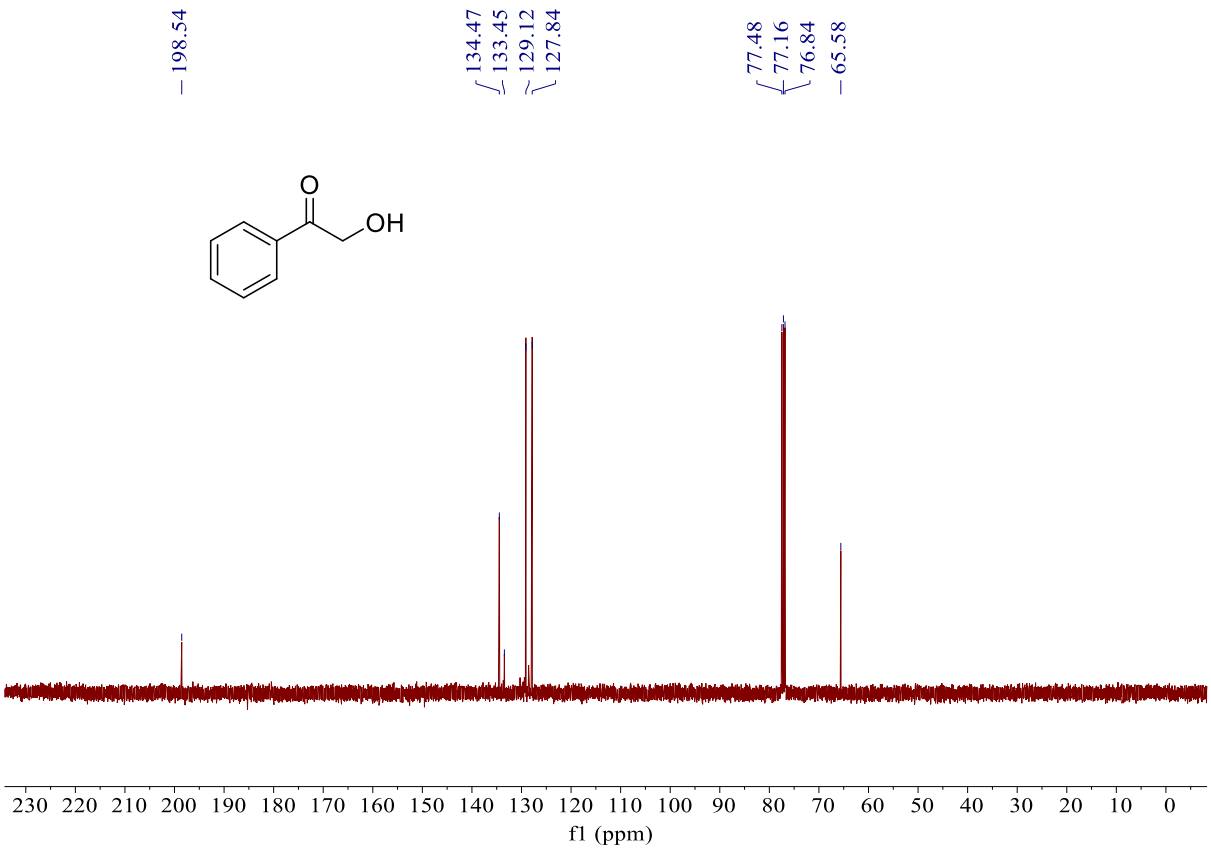
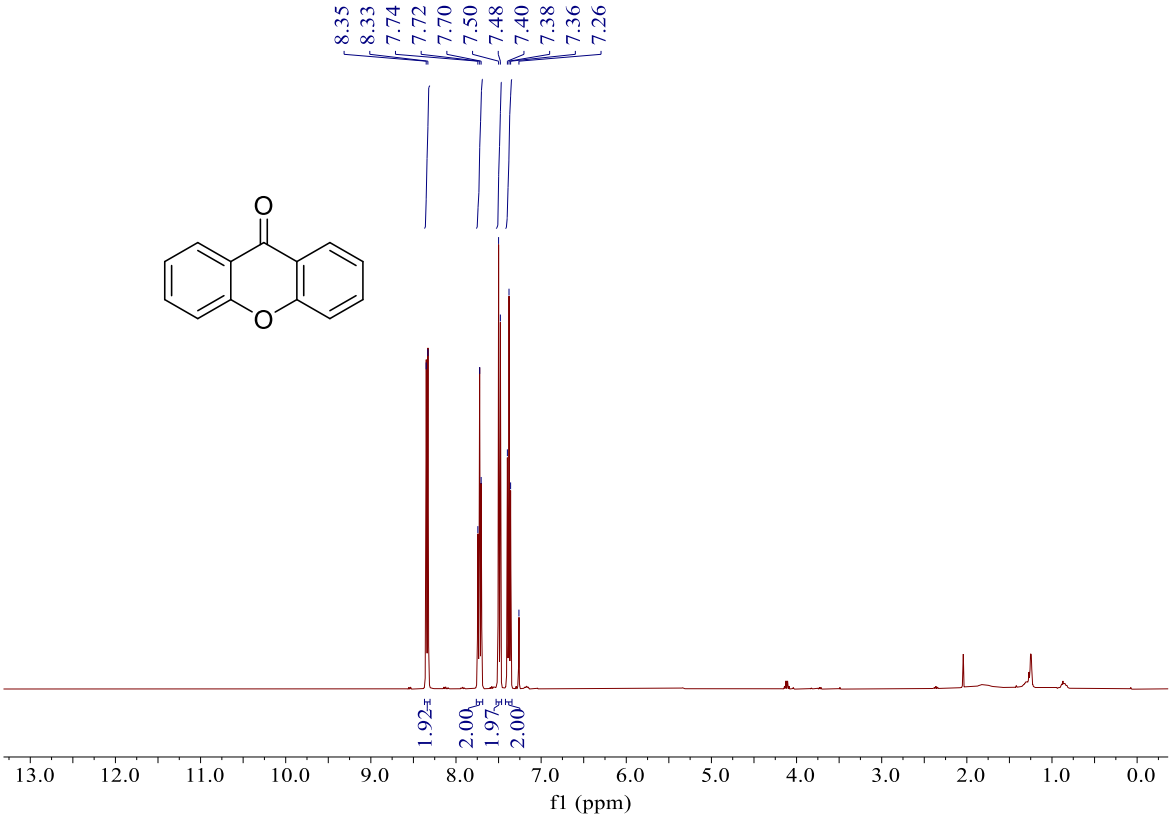
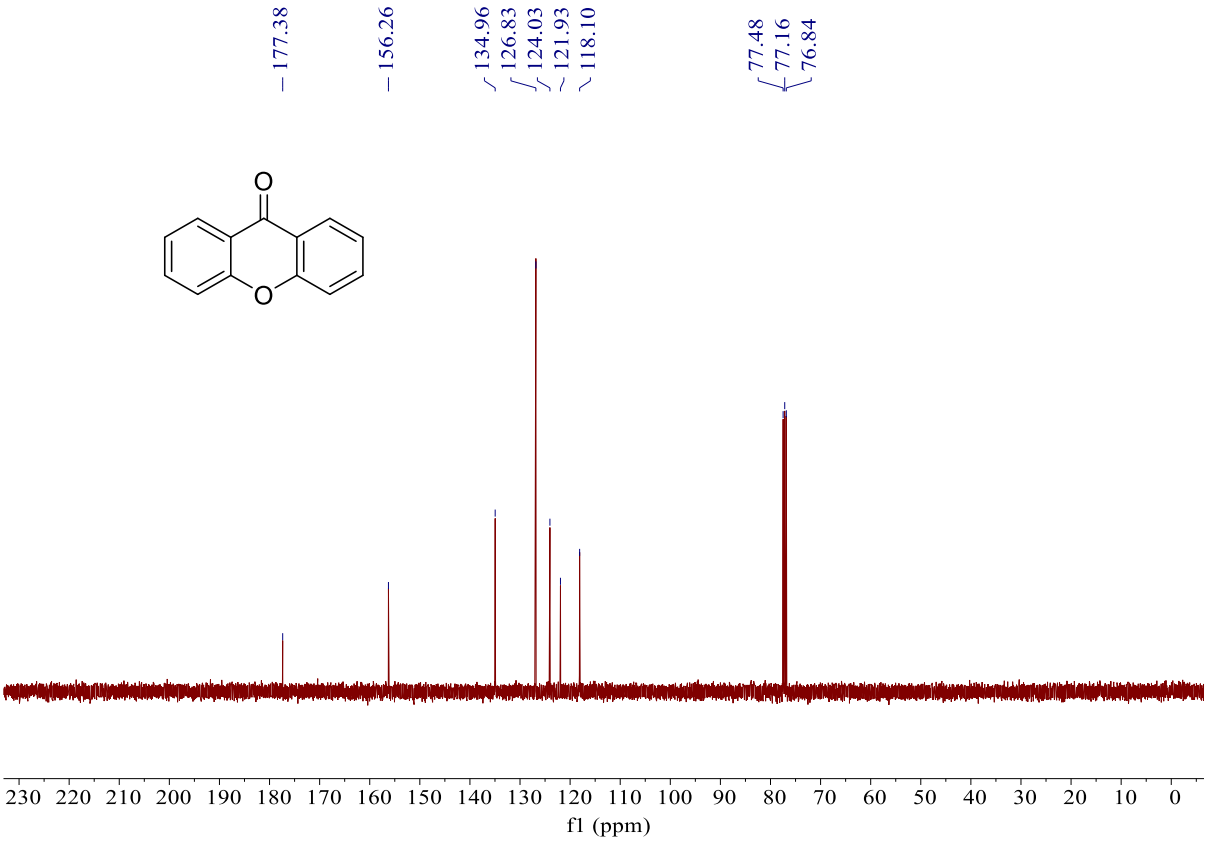


^{1}H NMR of compound 2ac:



^{13}C NMR of compound 2ac:



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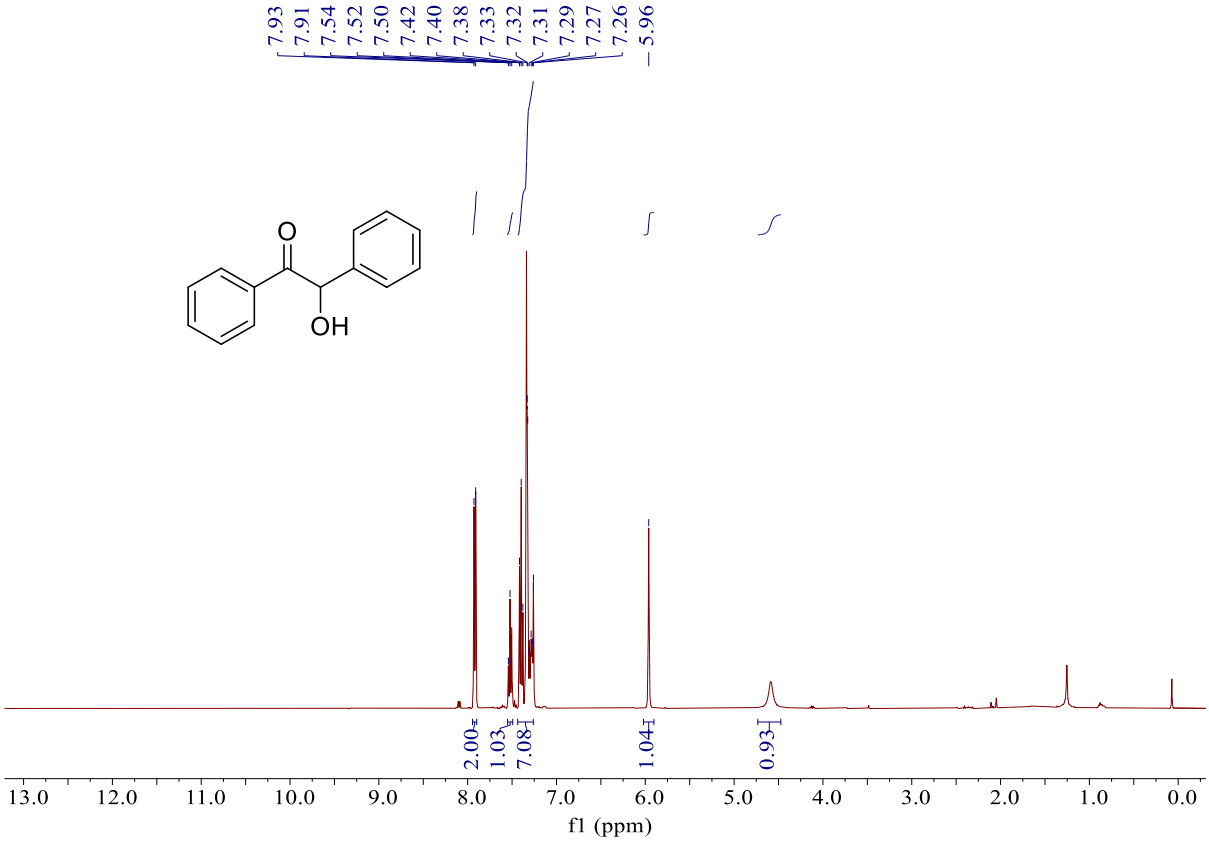

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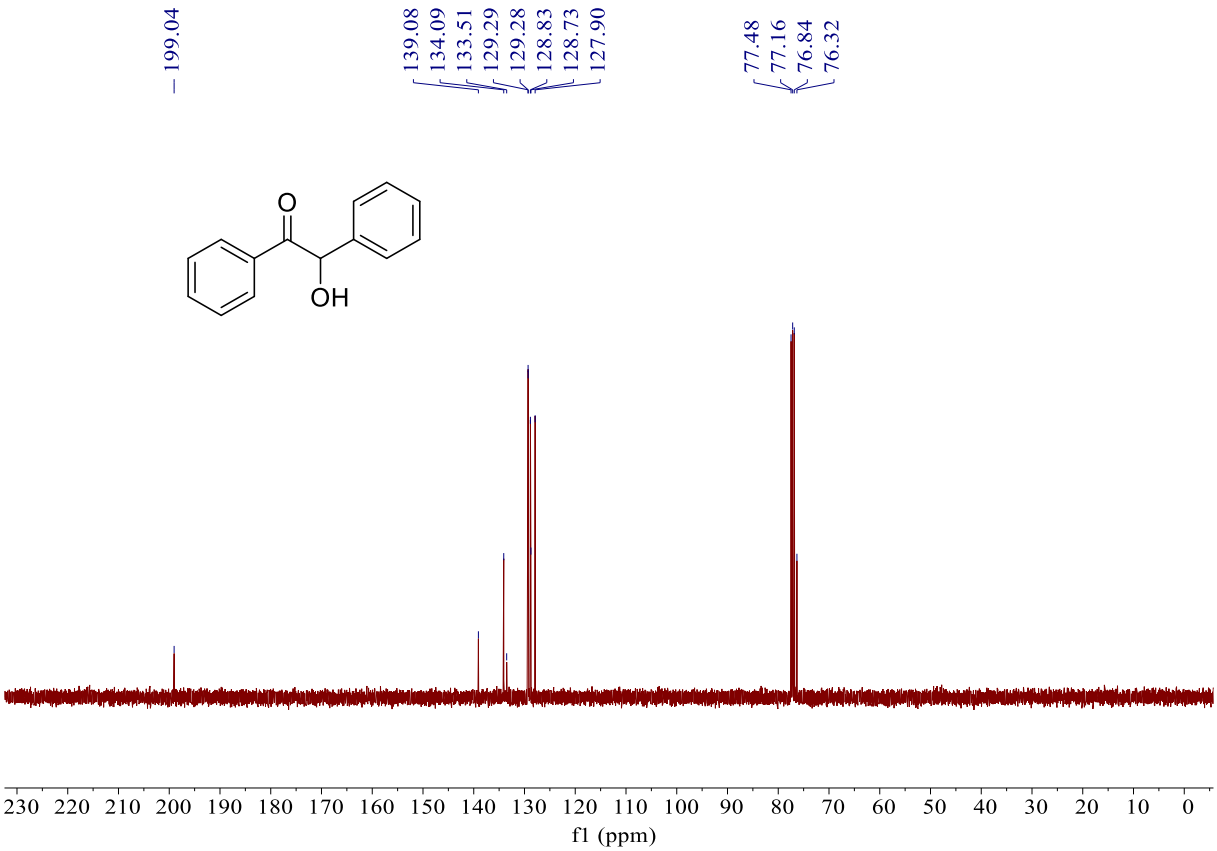
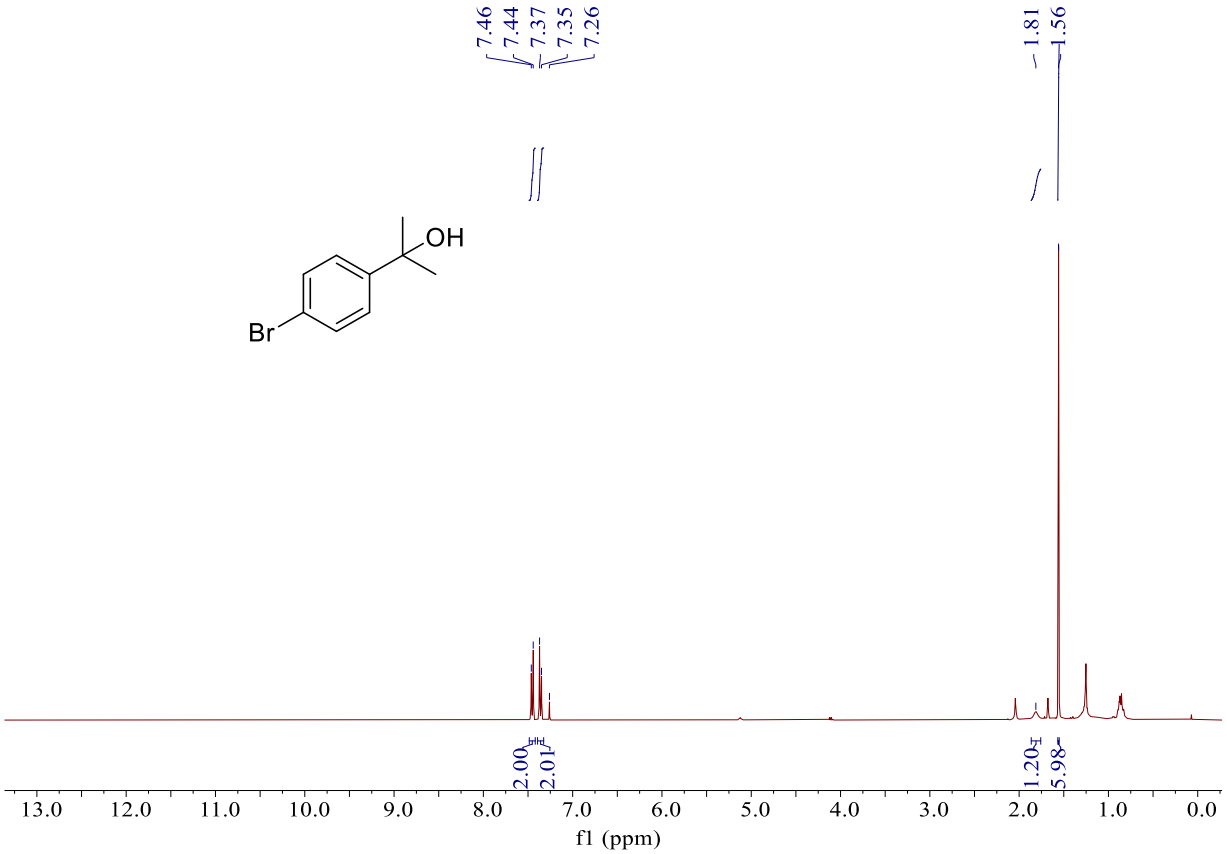


^{1}H NMR of compound 4a:



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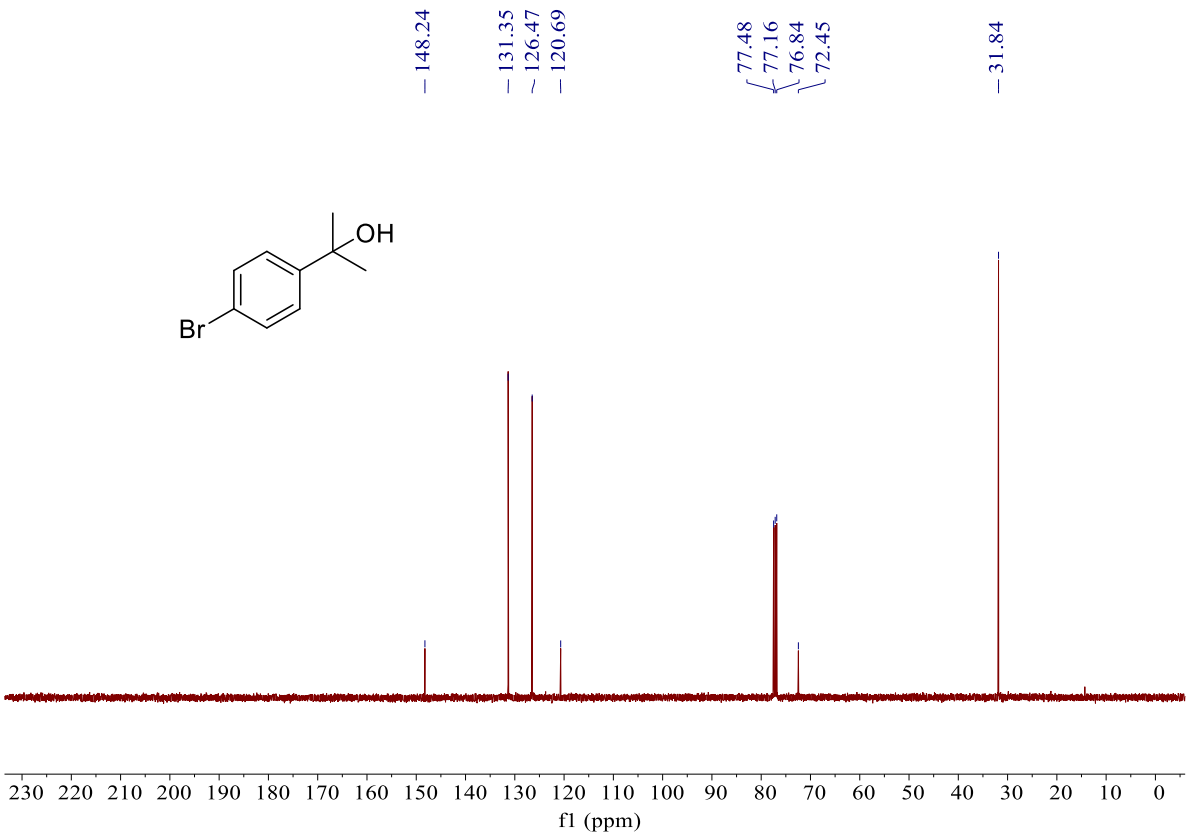

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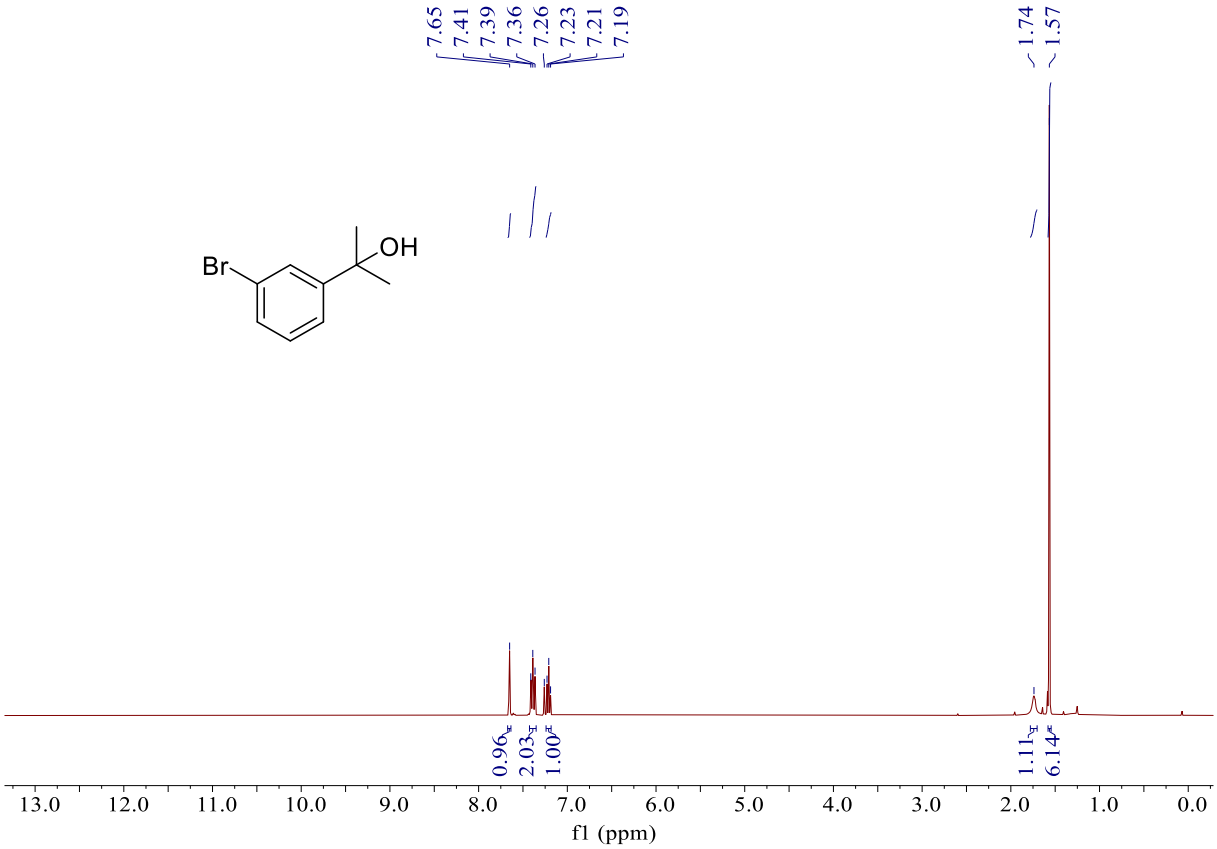
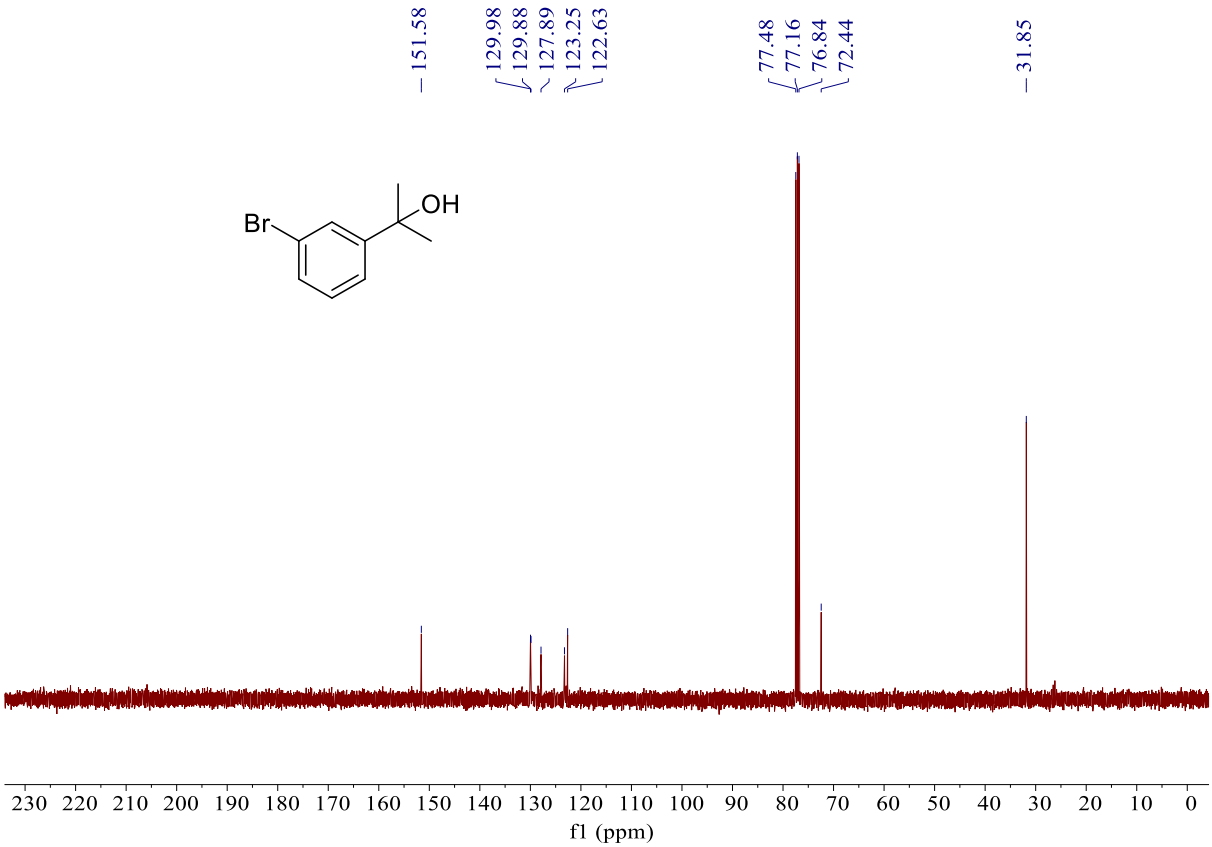


^{13}C NMR of compound 4b:



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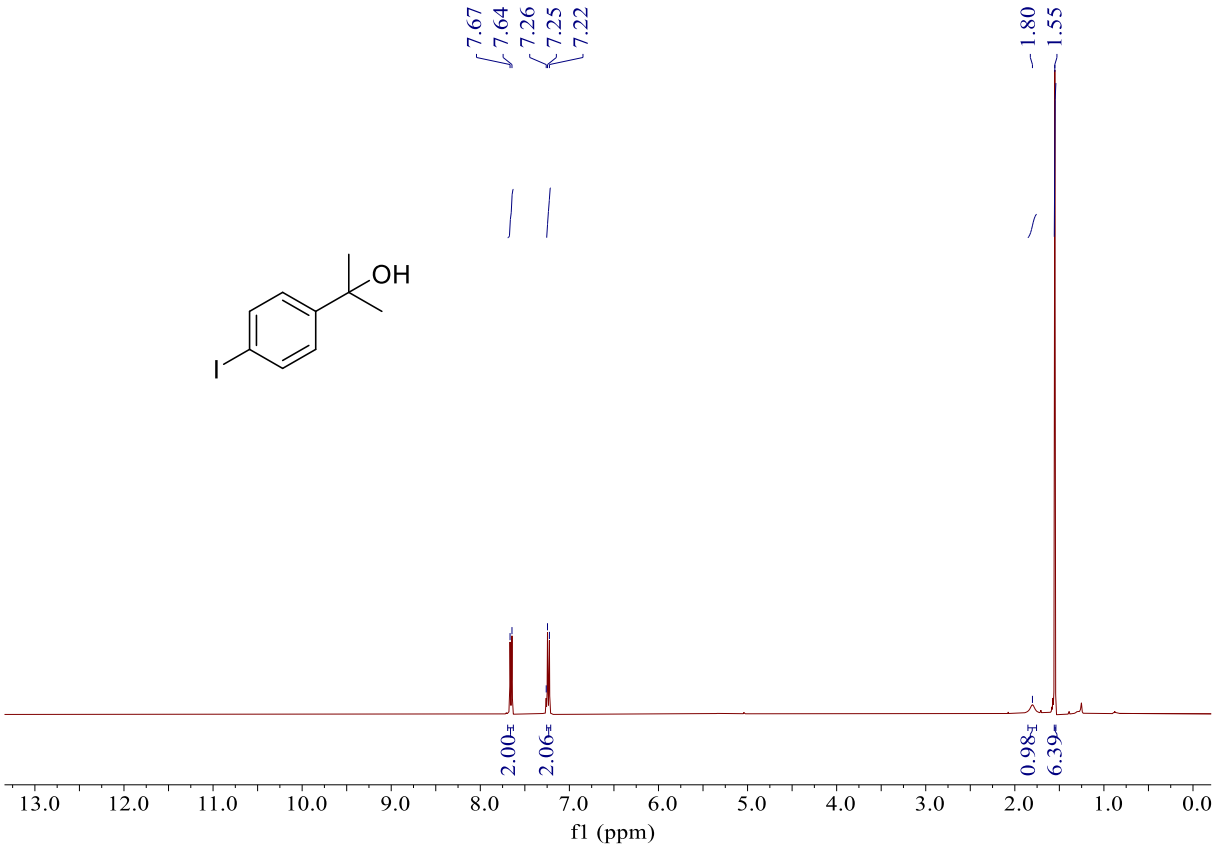

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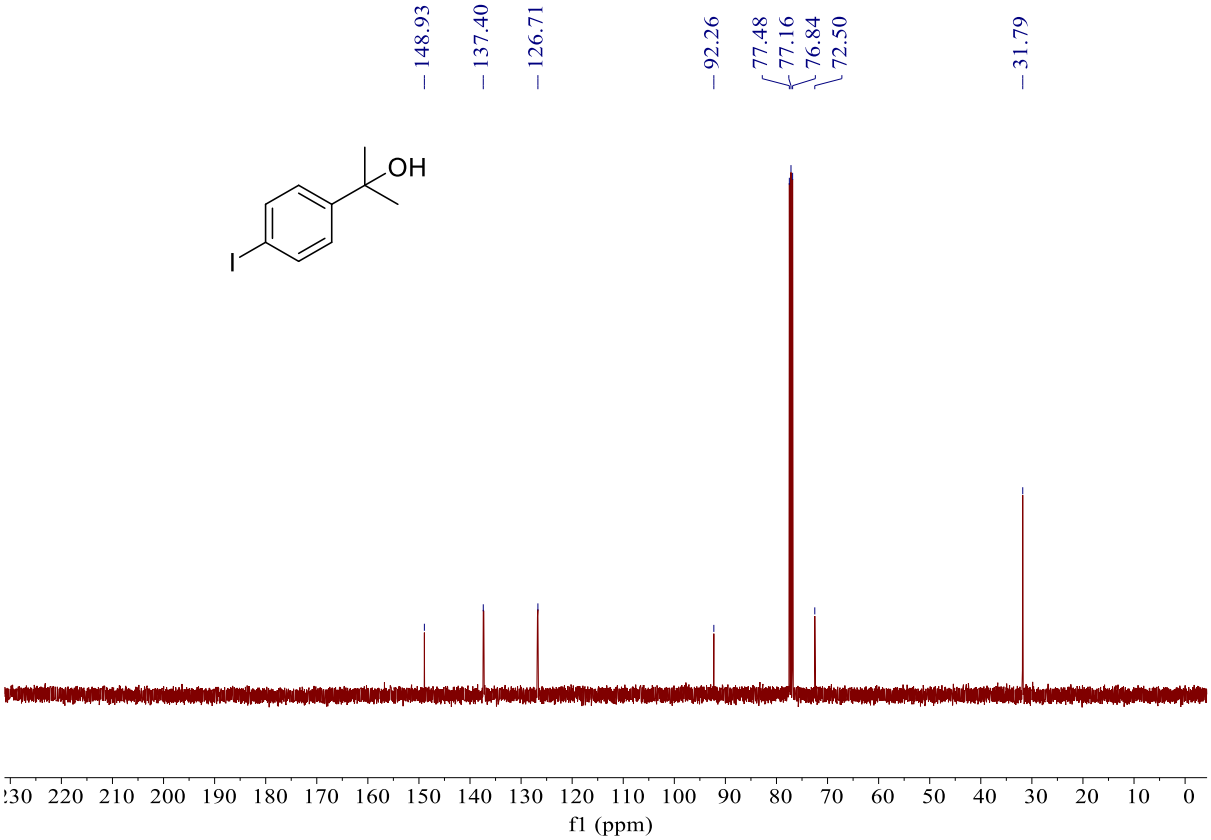
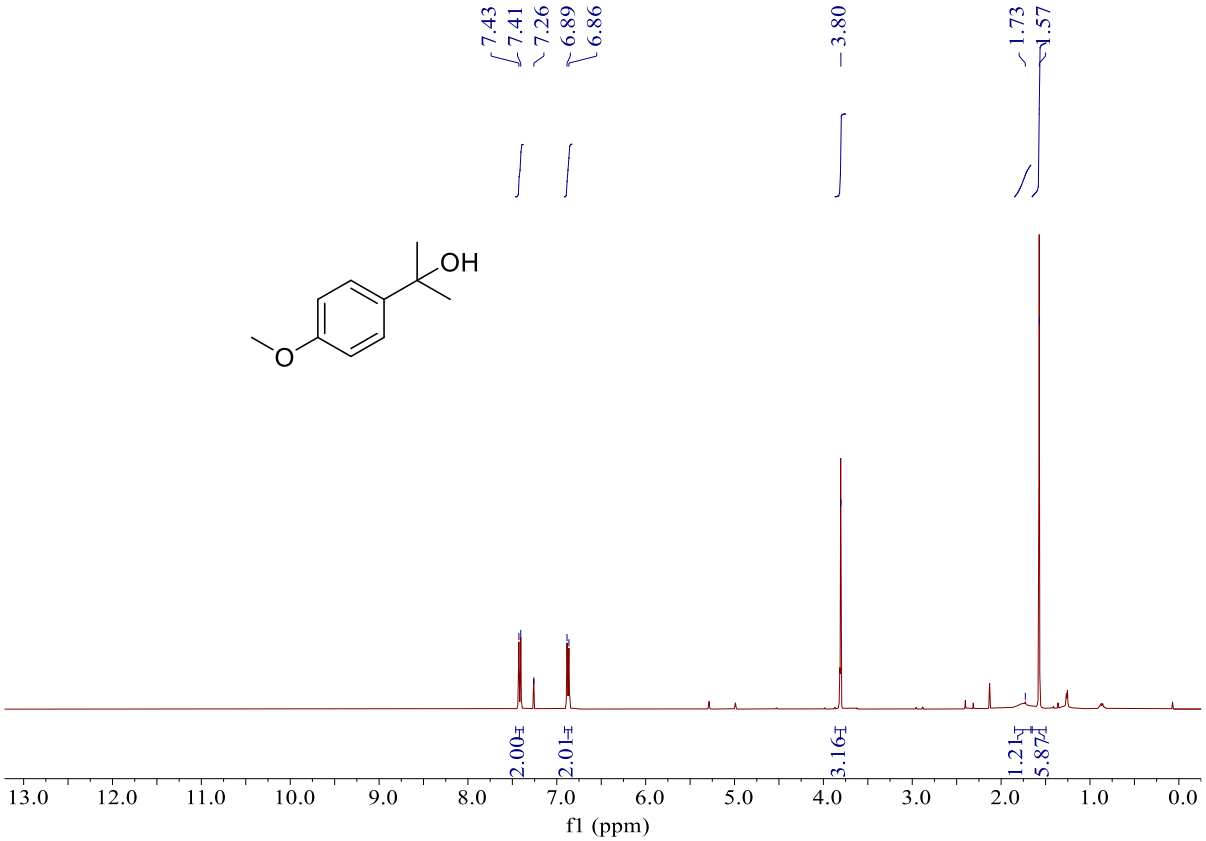


^{1}H NMR of compound 4d:



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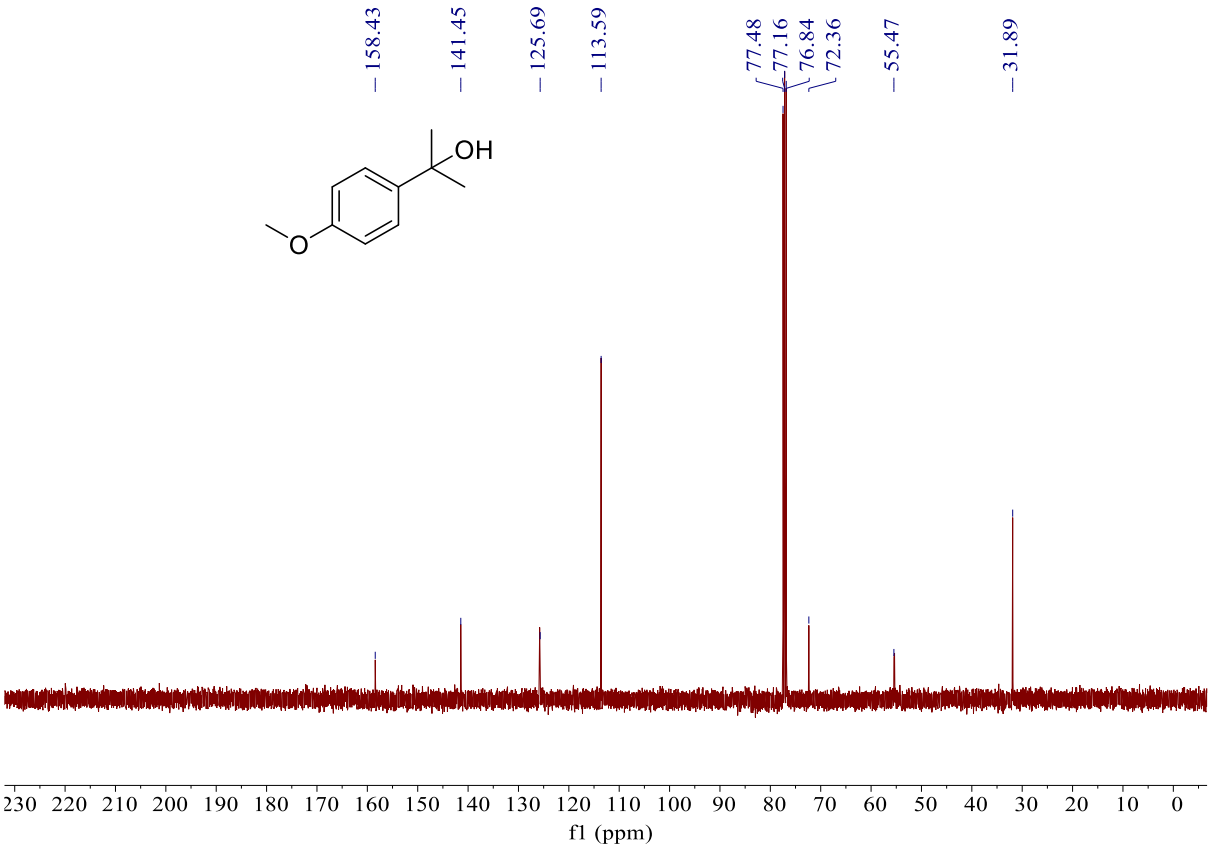

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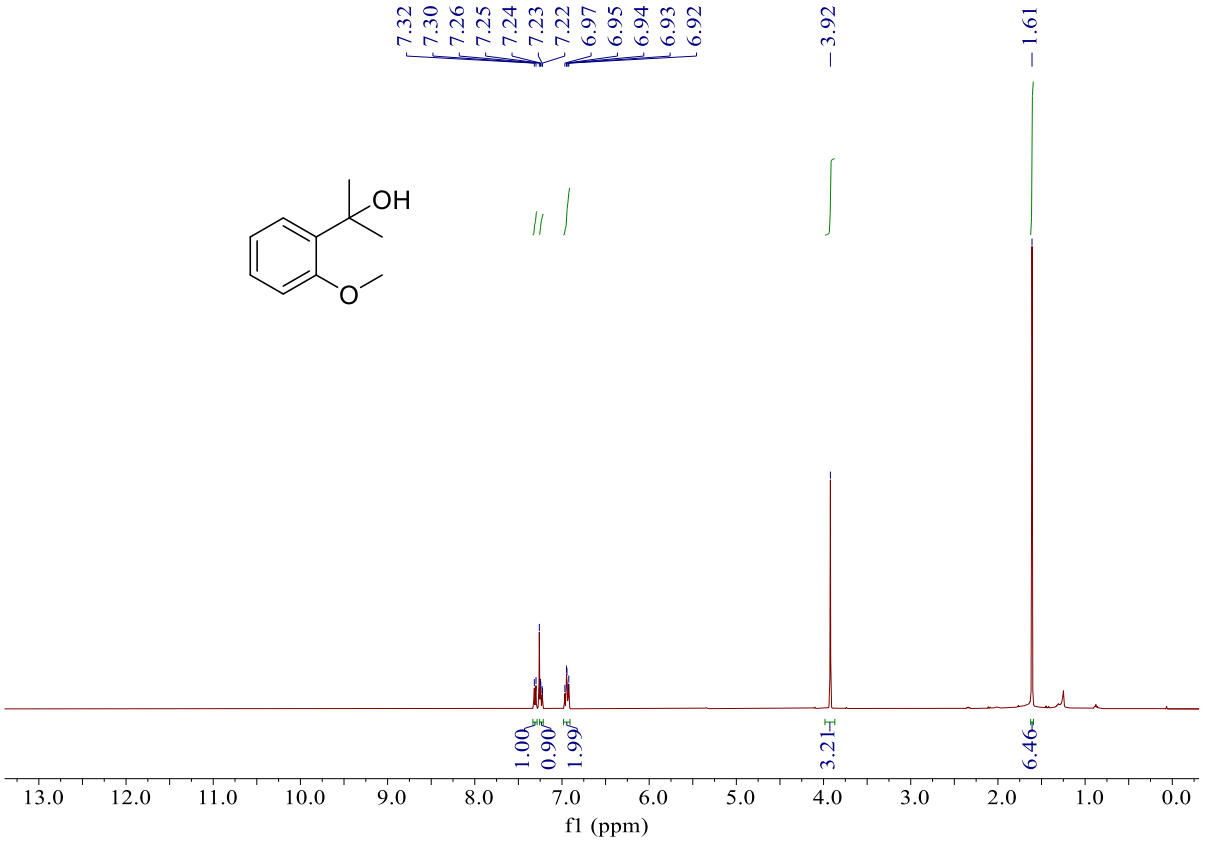

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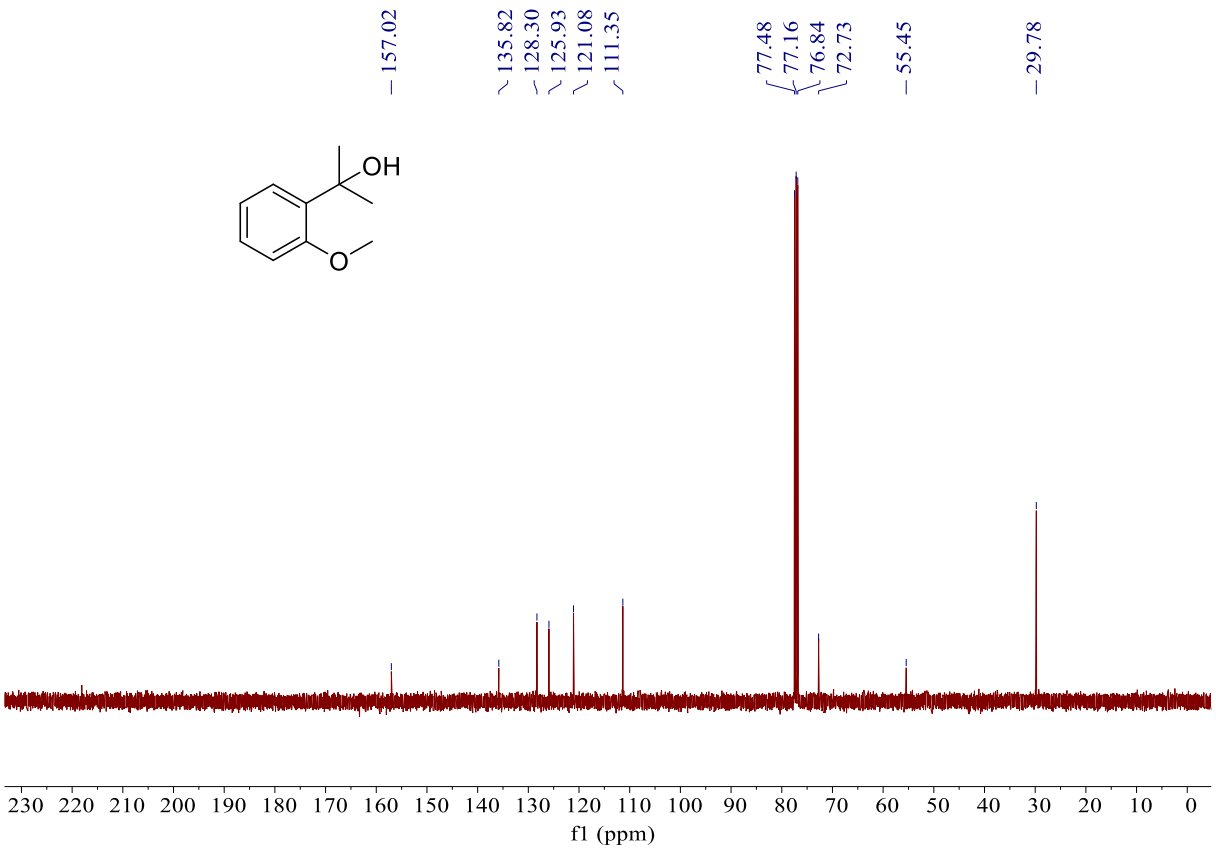
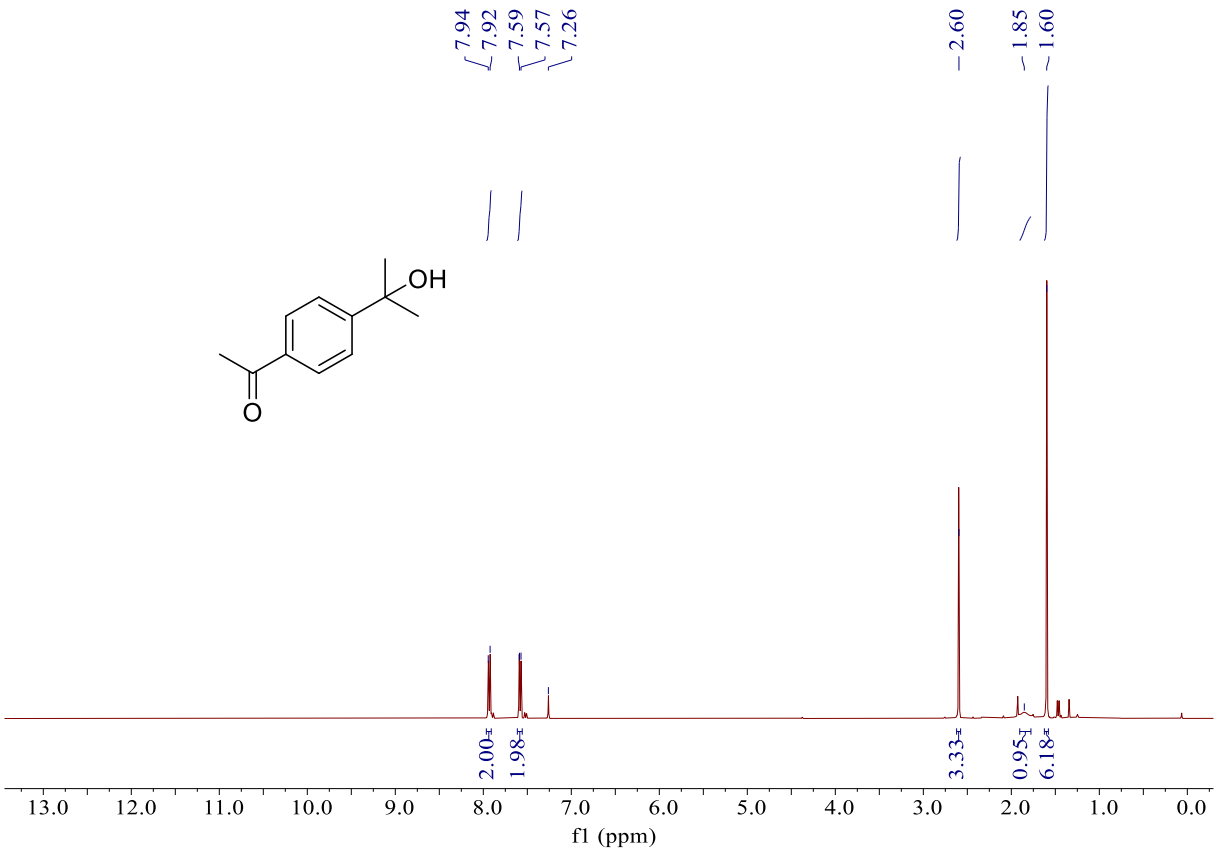


^{1}H NMR of compound 4f:



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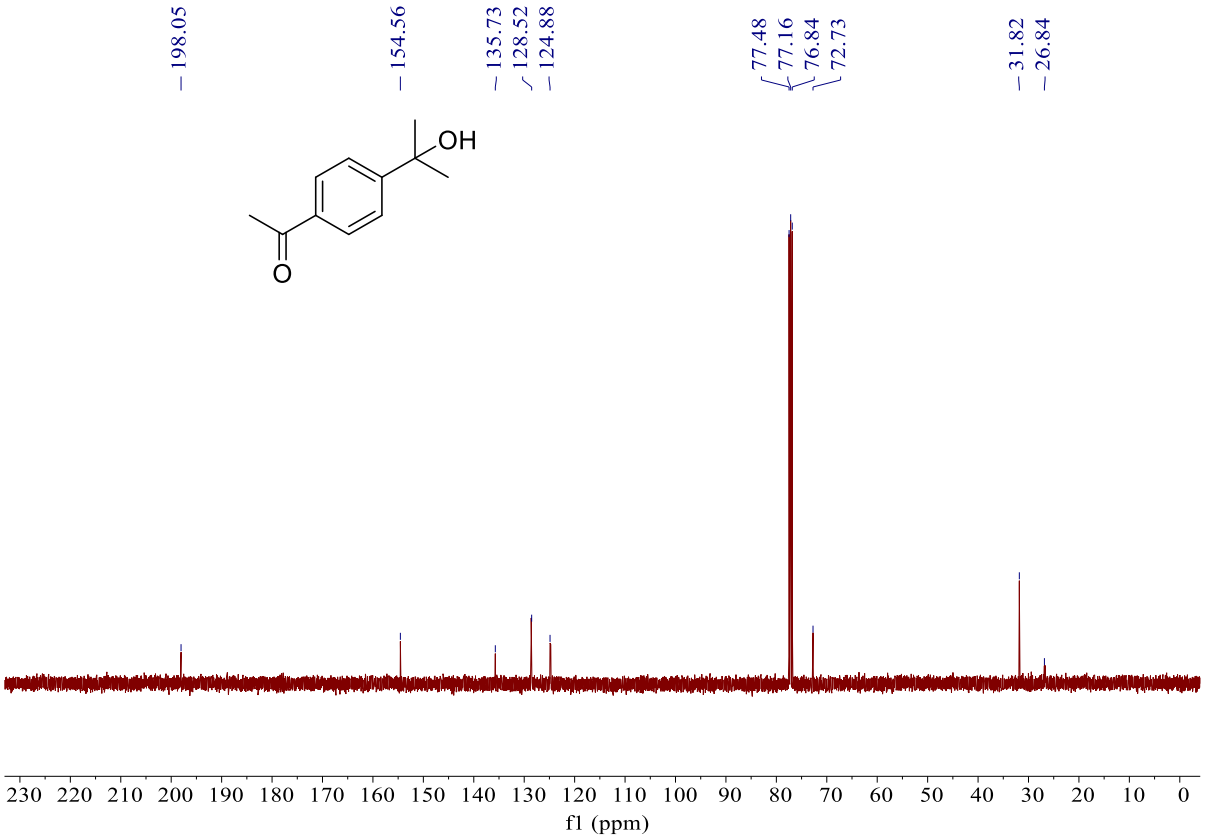
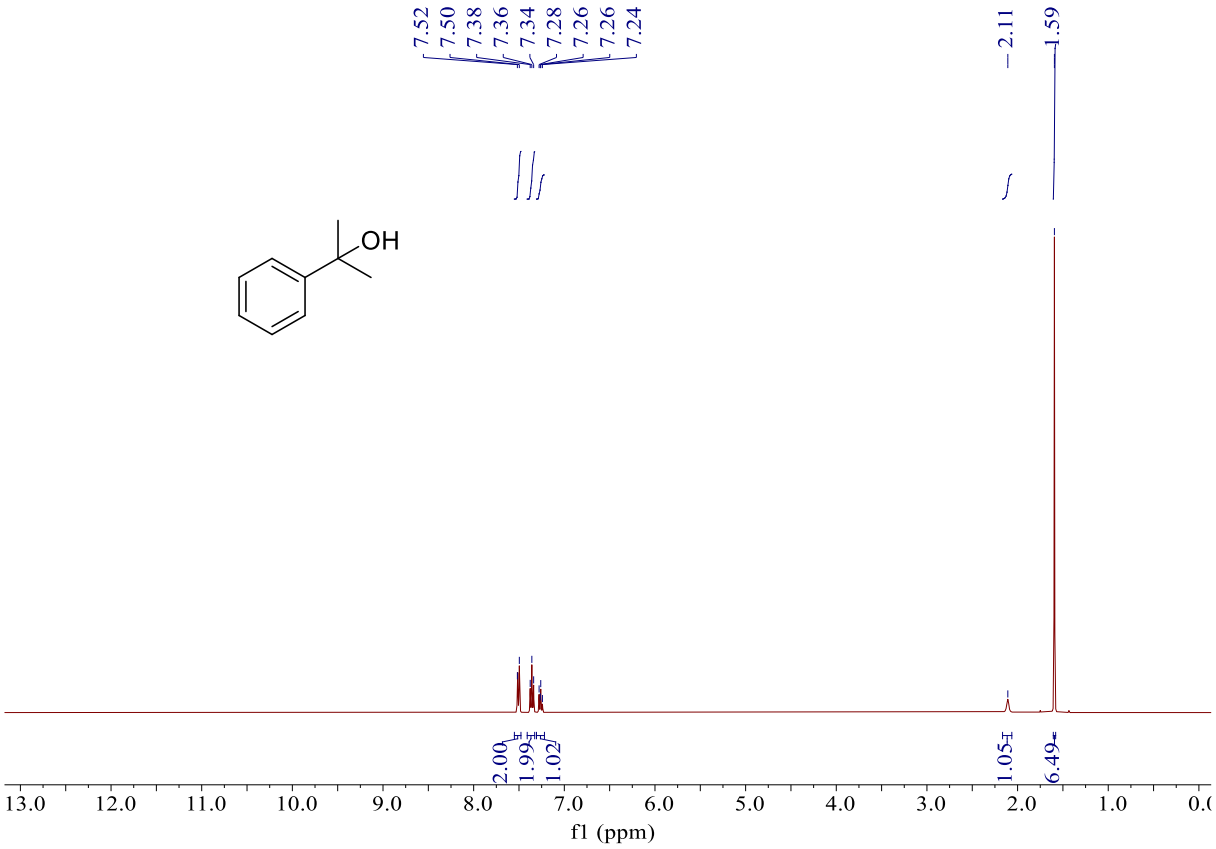
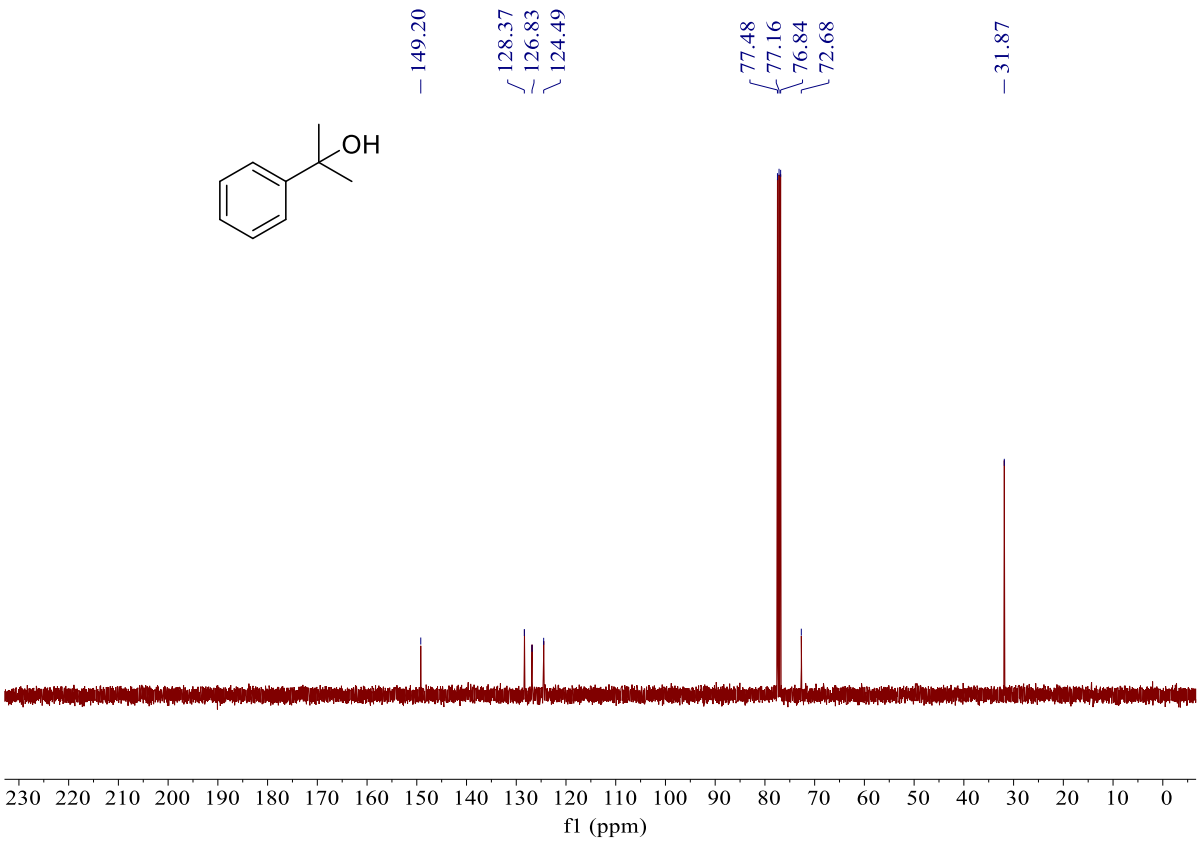


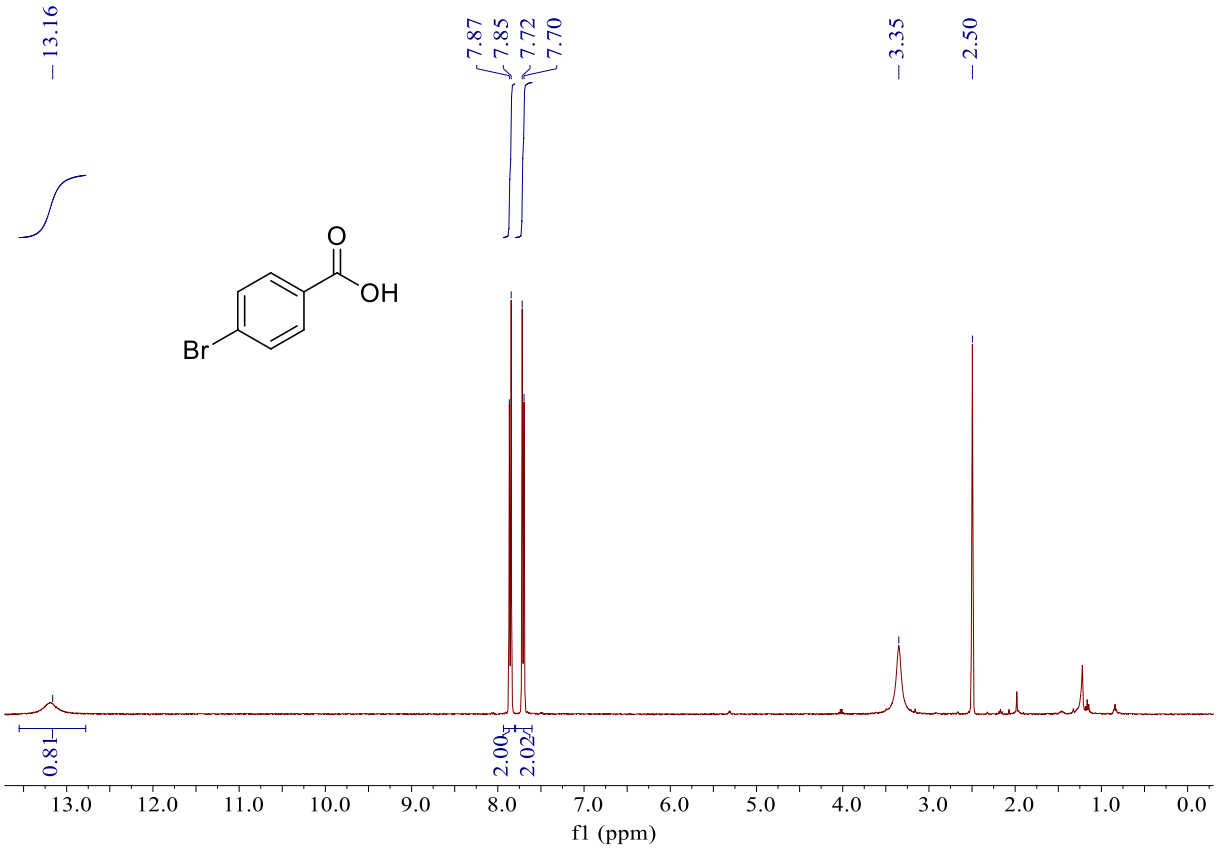
^{1}H NMR of compound 4g:



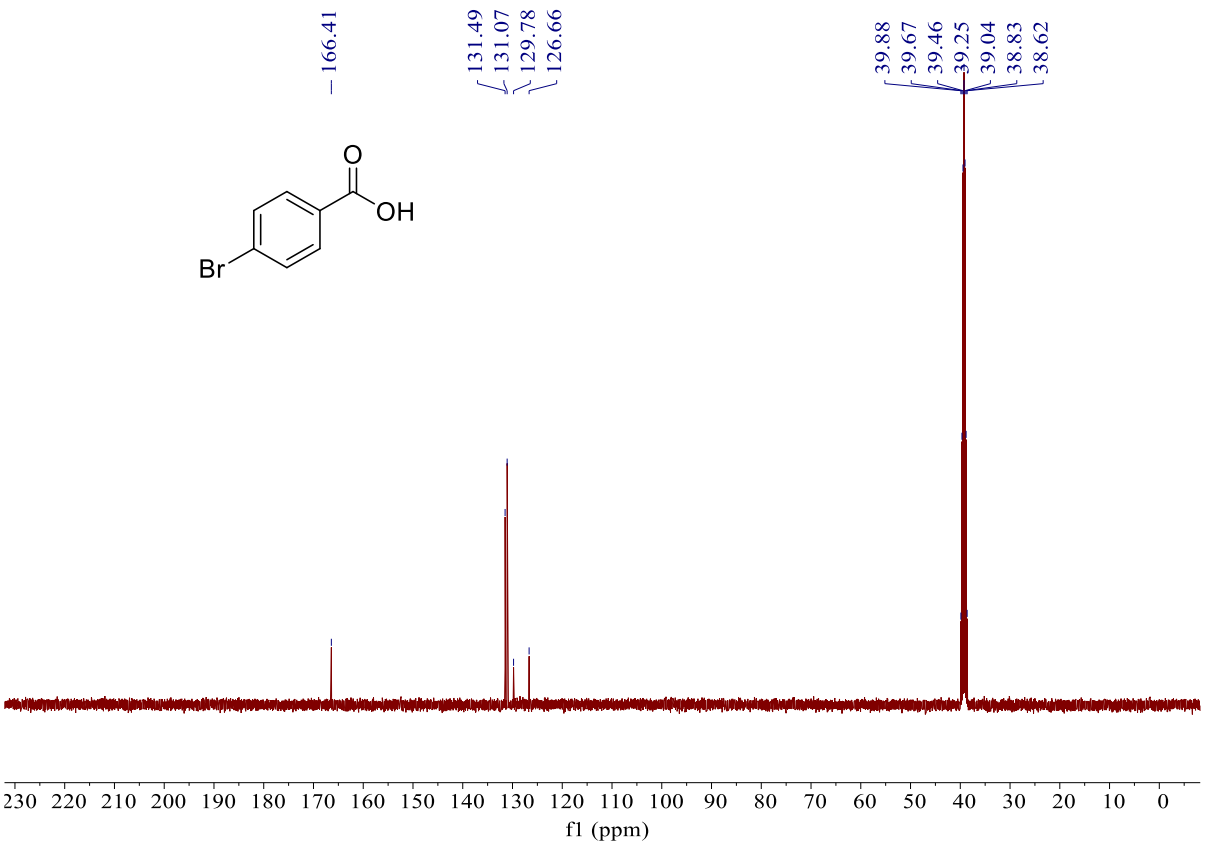
^{13}C NMR of compound 4g:



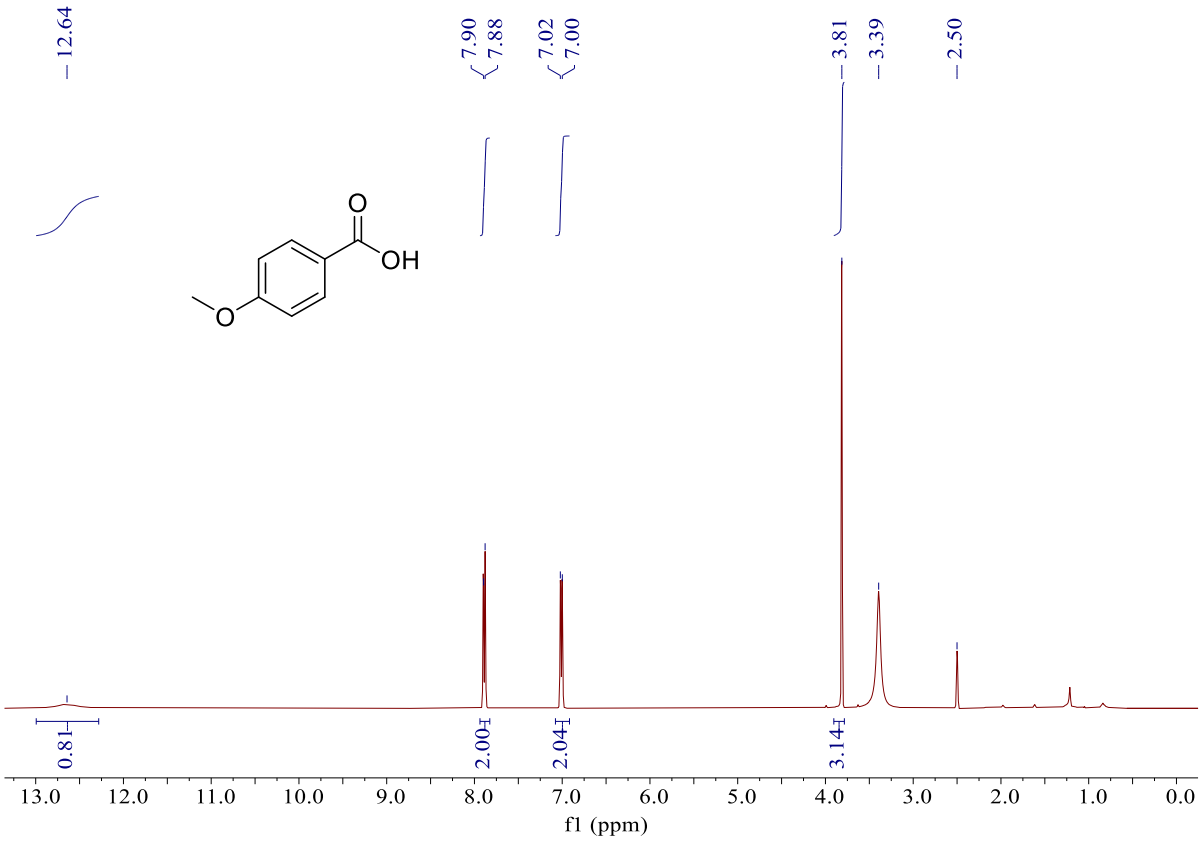
^{1}H NMR of compound 6a:



^{13}C NMR of compound 6a:



^{1}H NMR of compound 6b:



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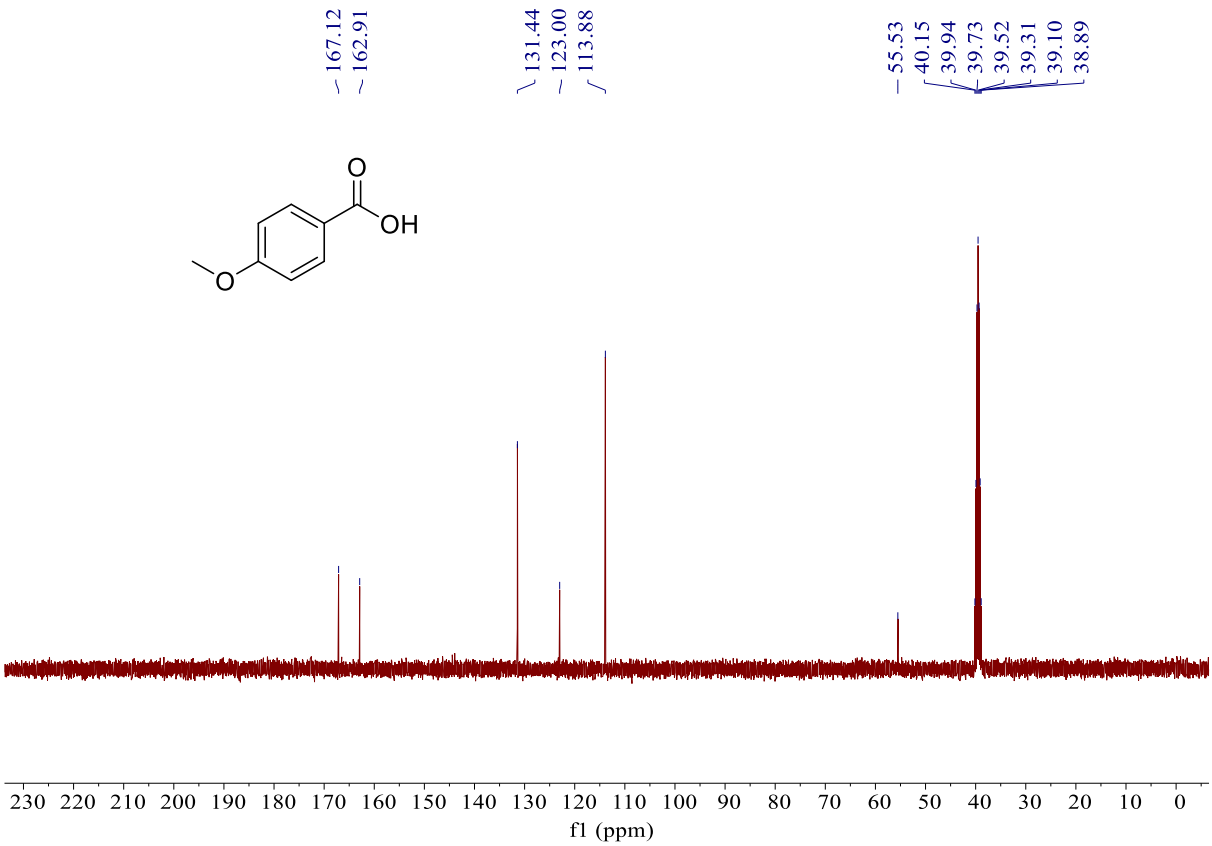

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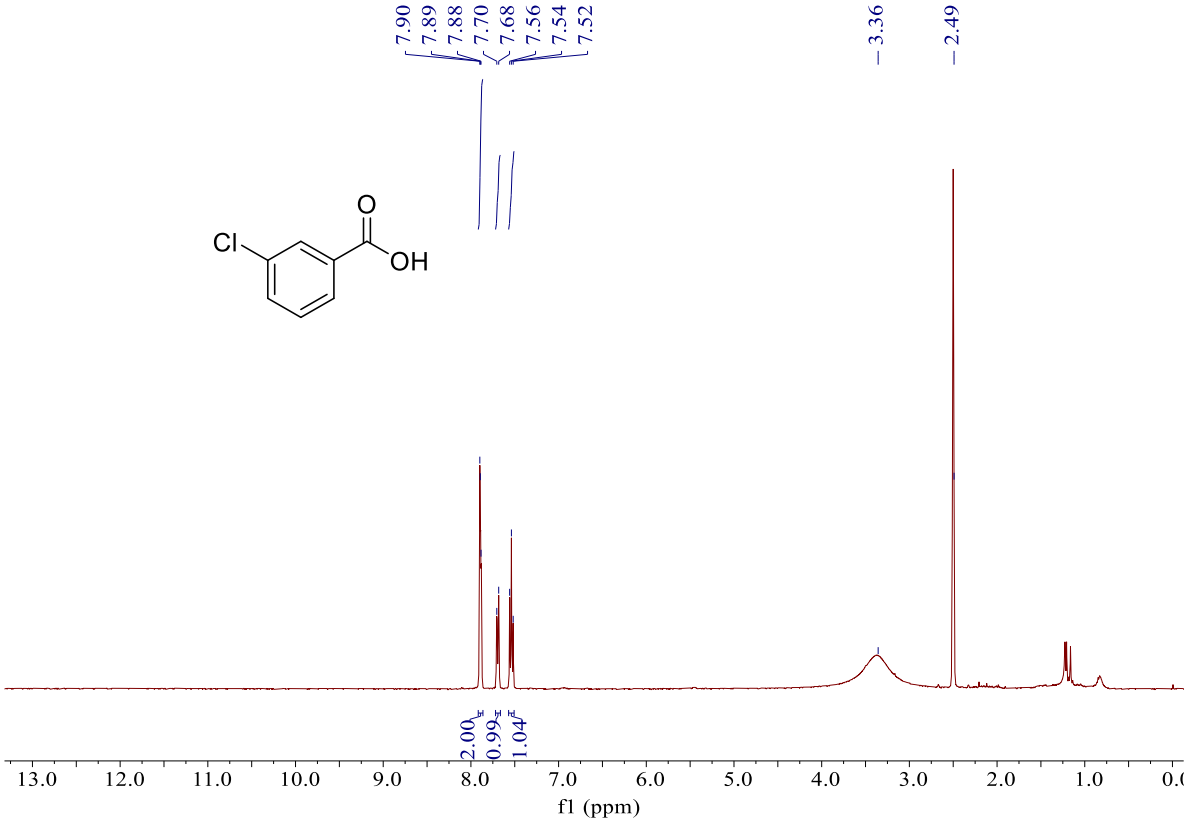

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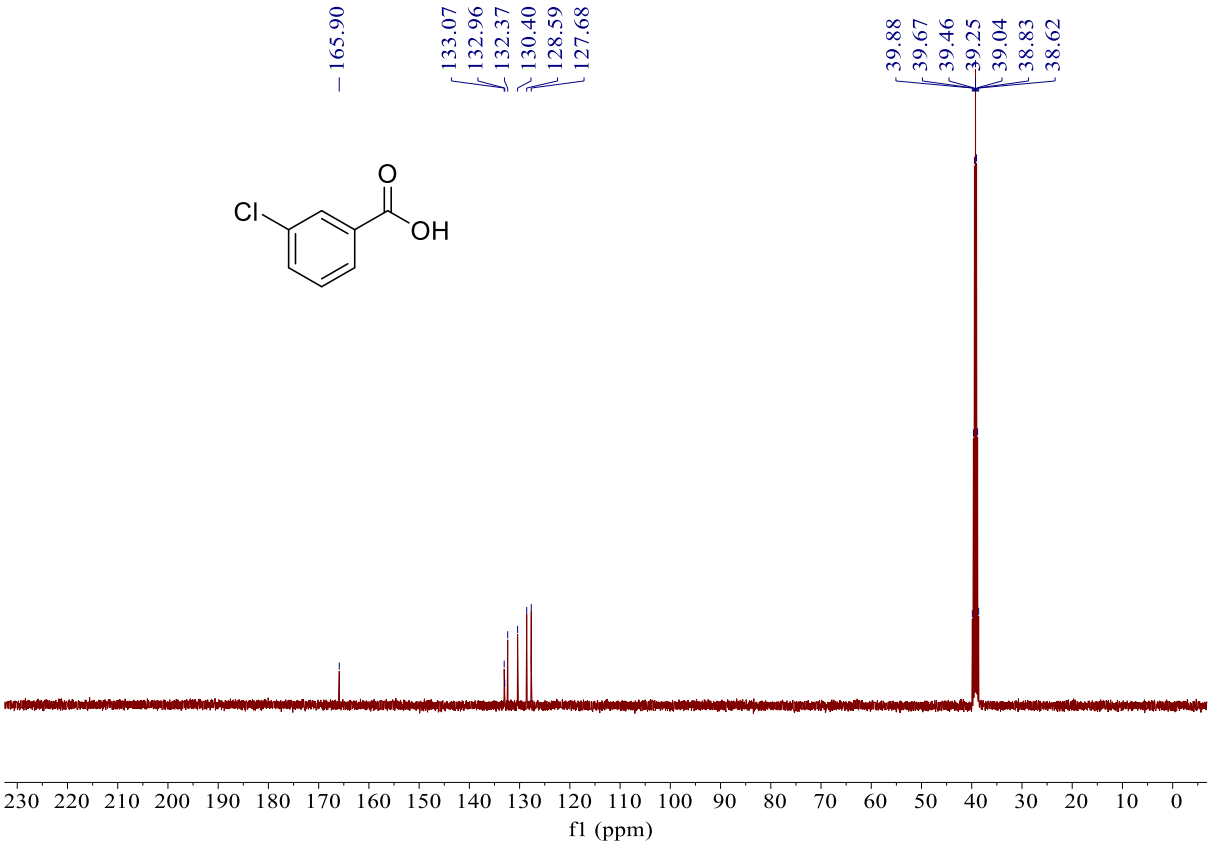

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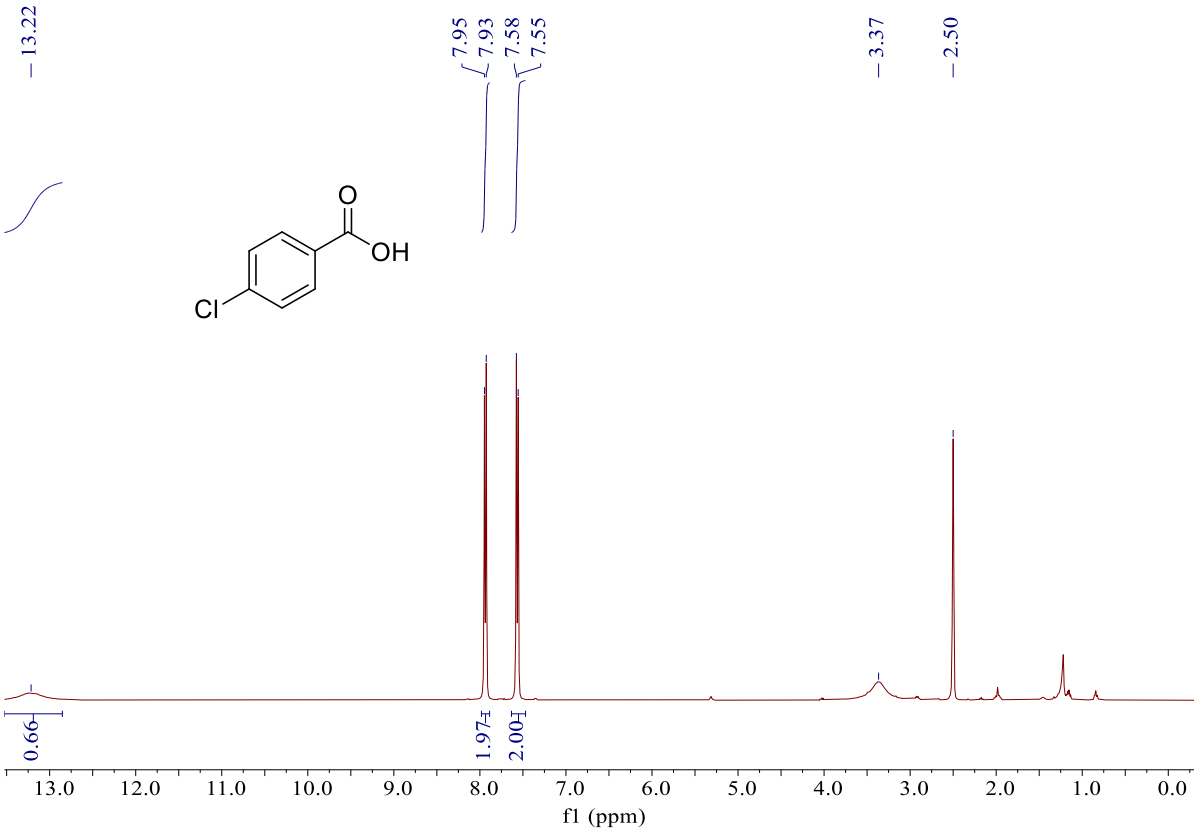

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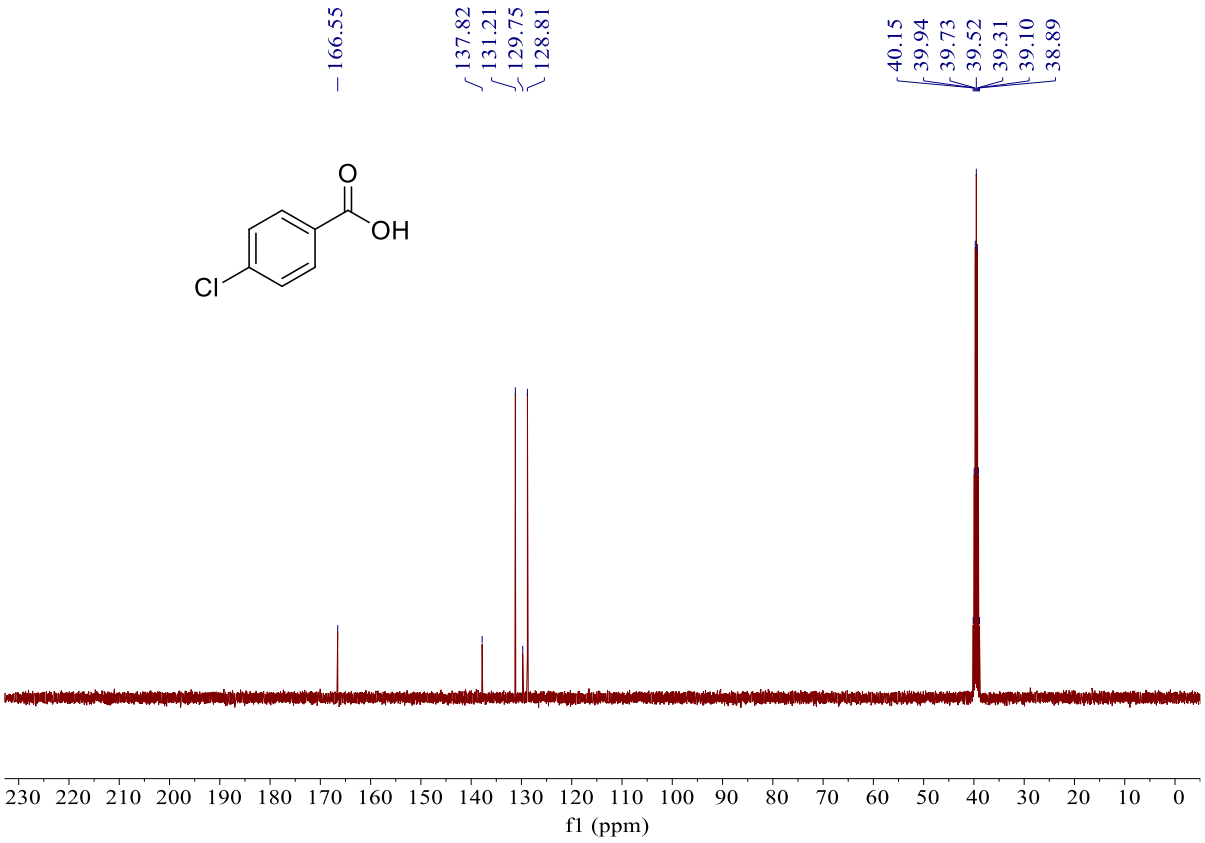

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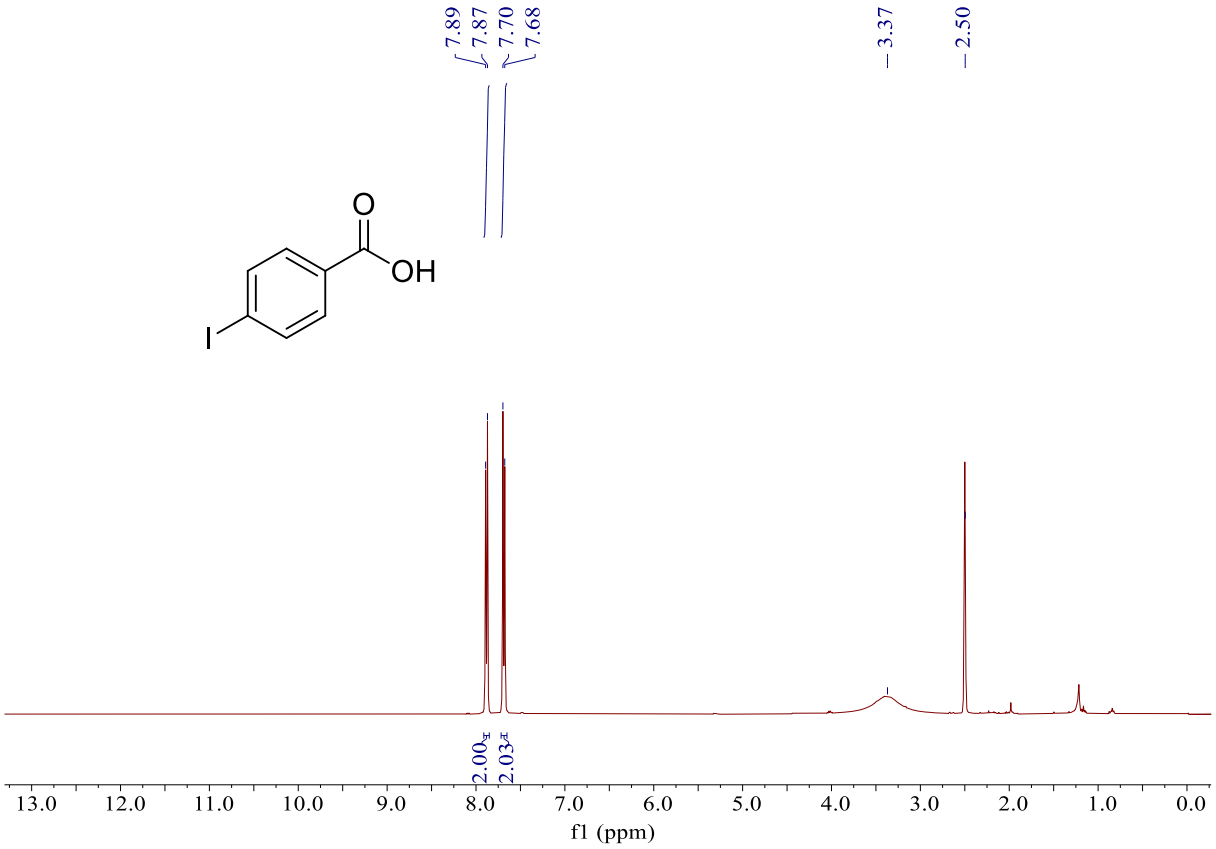

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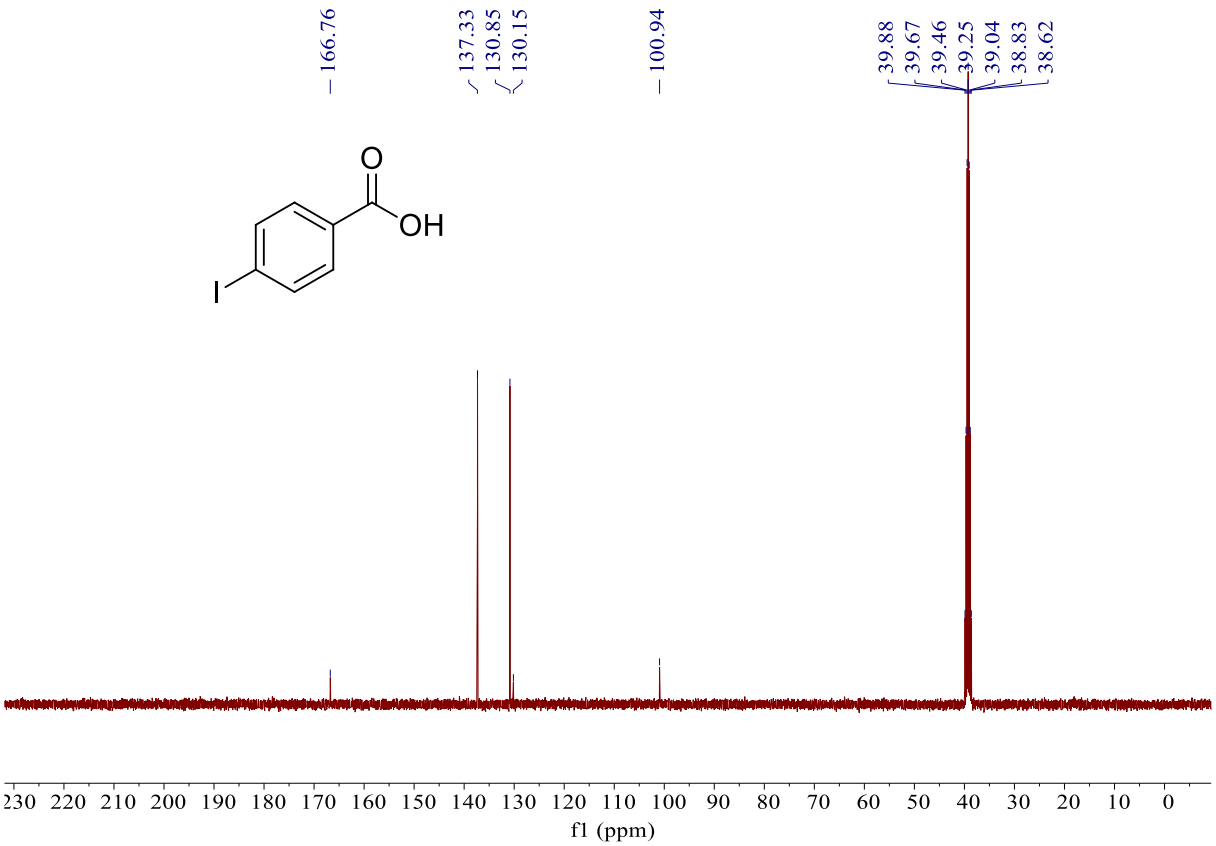
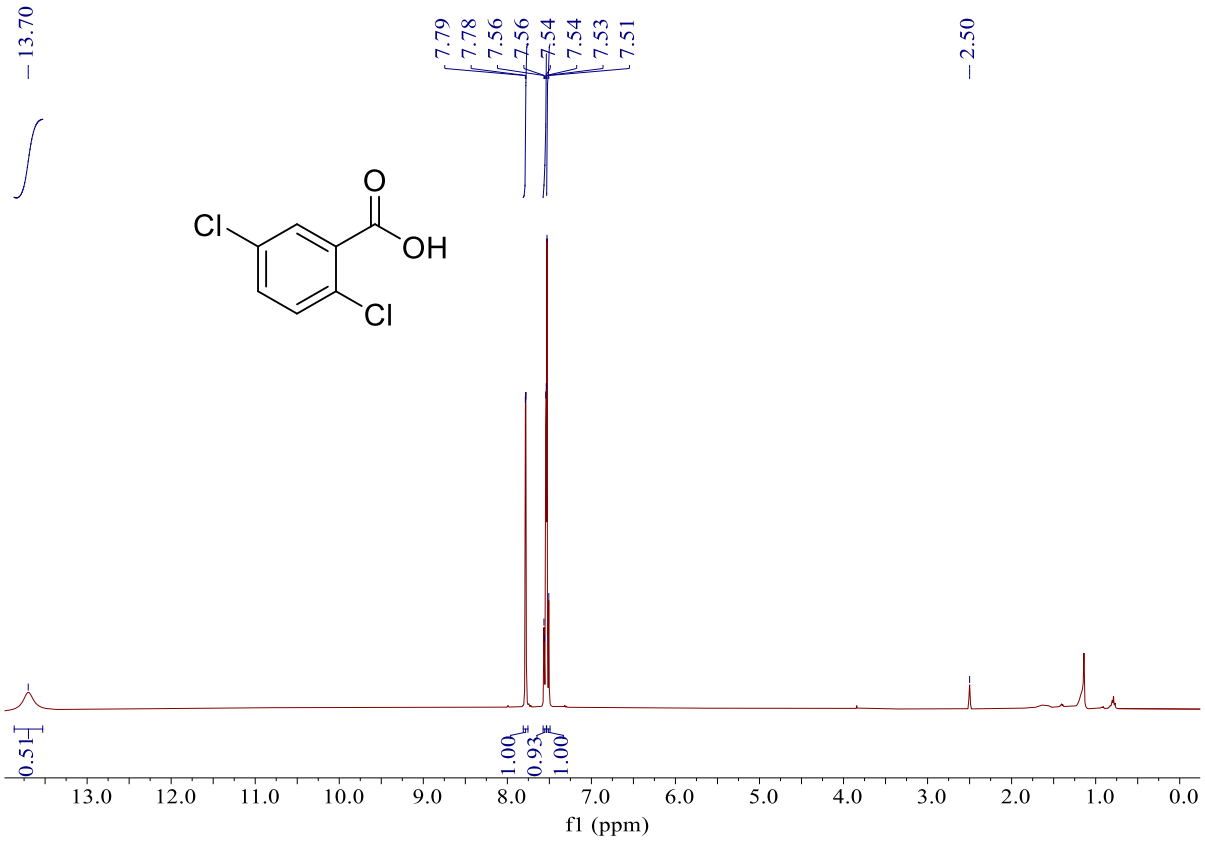


^{1}H NMR of compound 6f:



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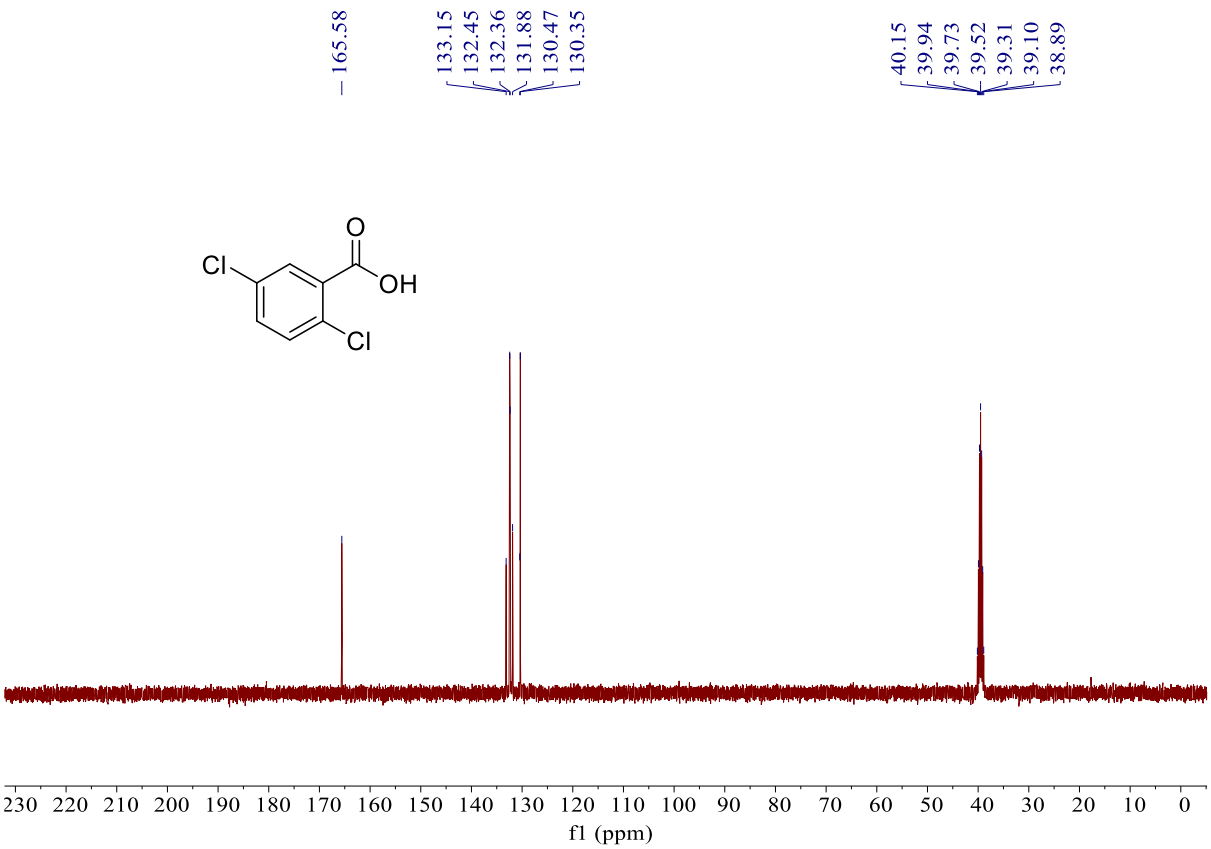

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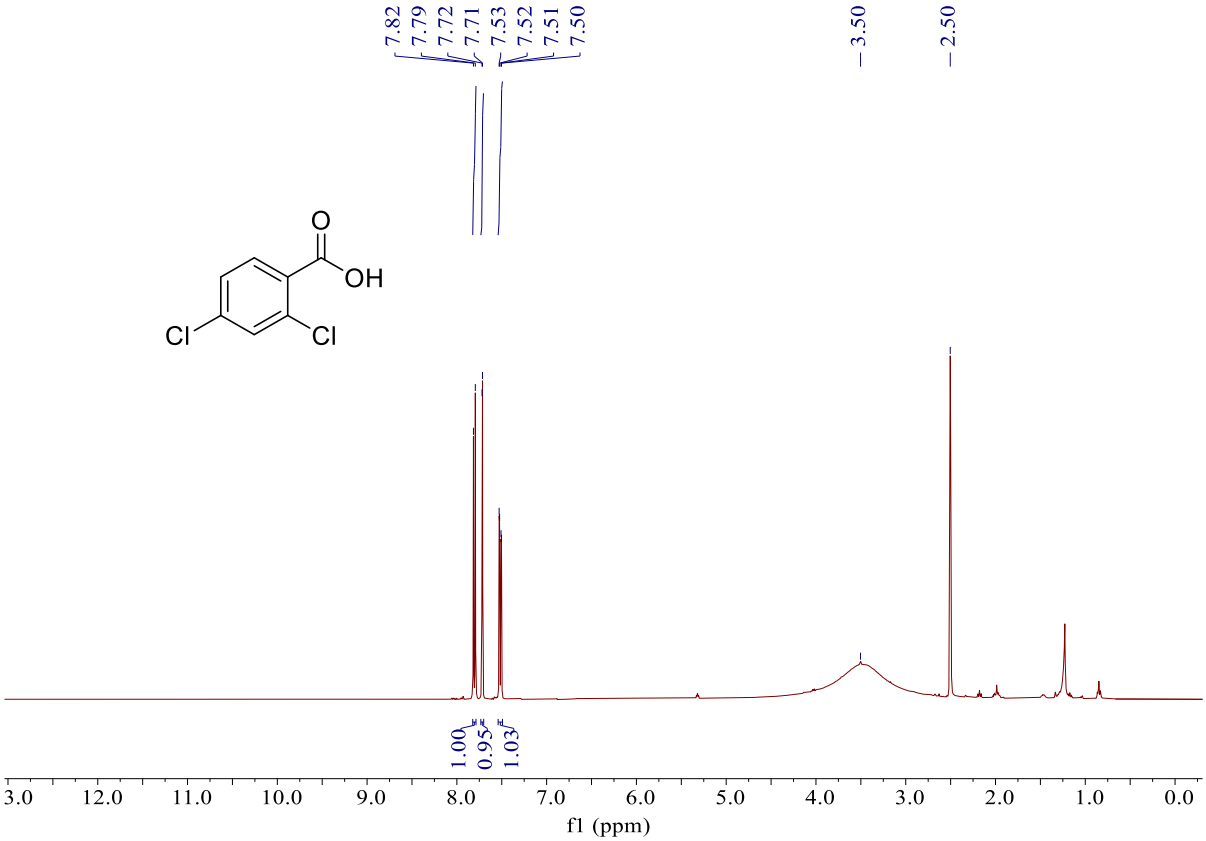

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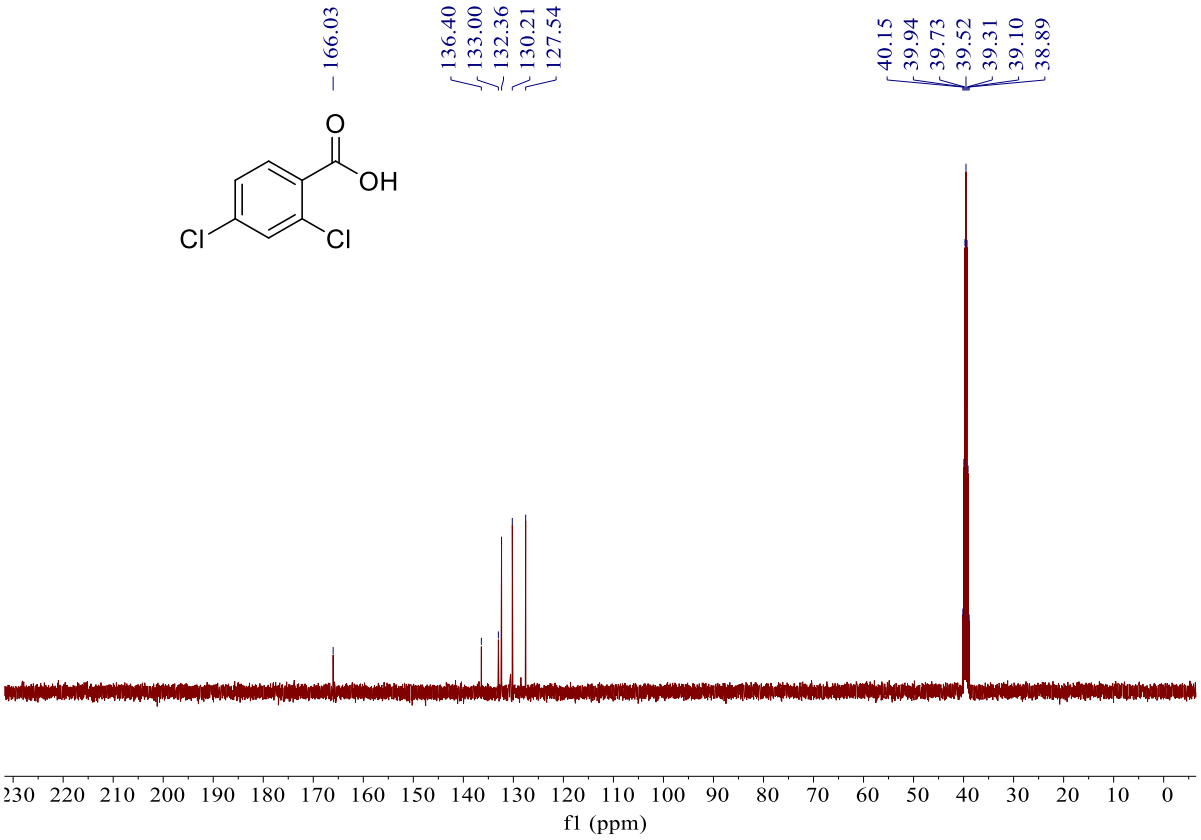
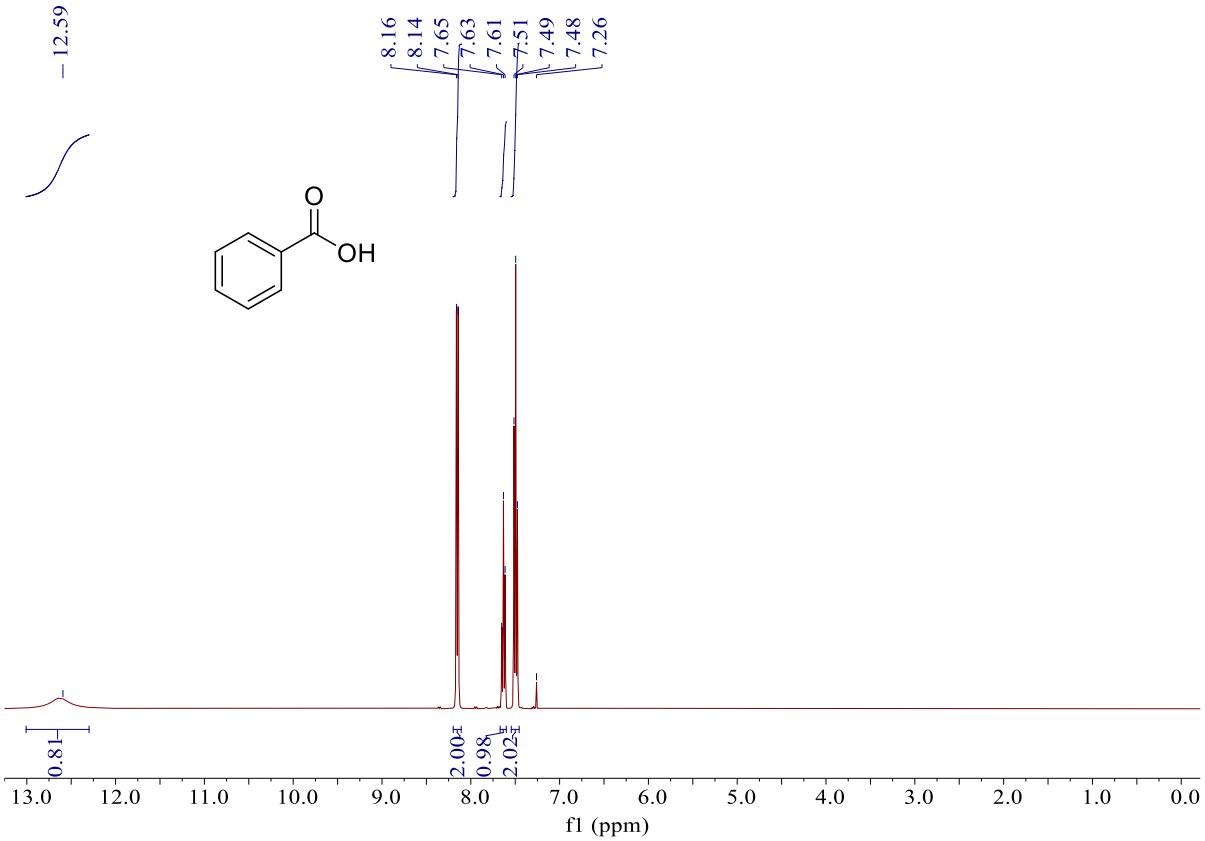


^{1}H NMR of compound 6h:



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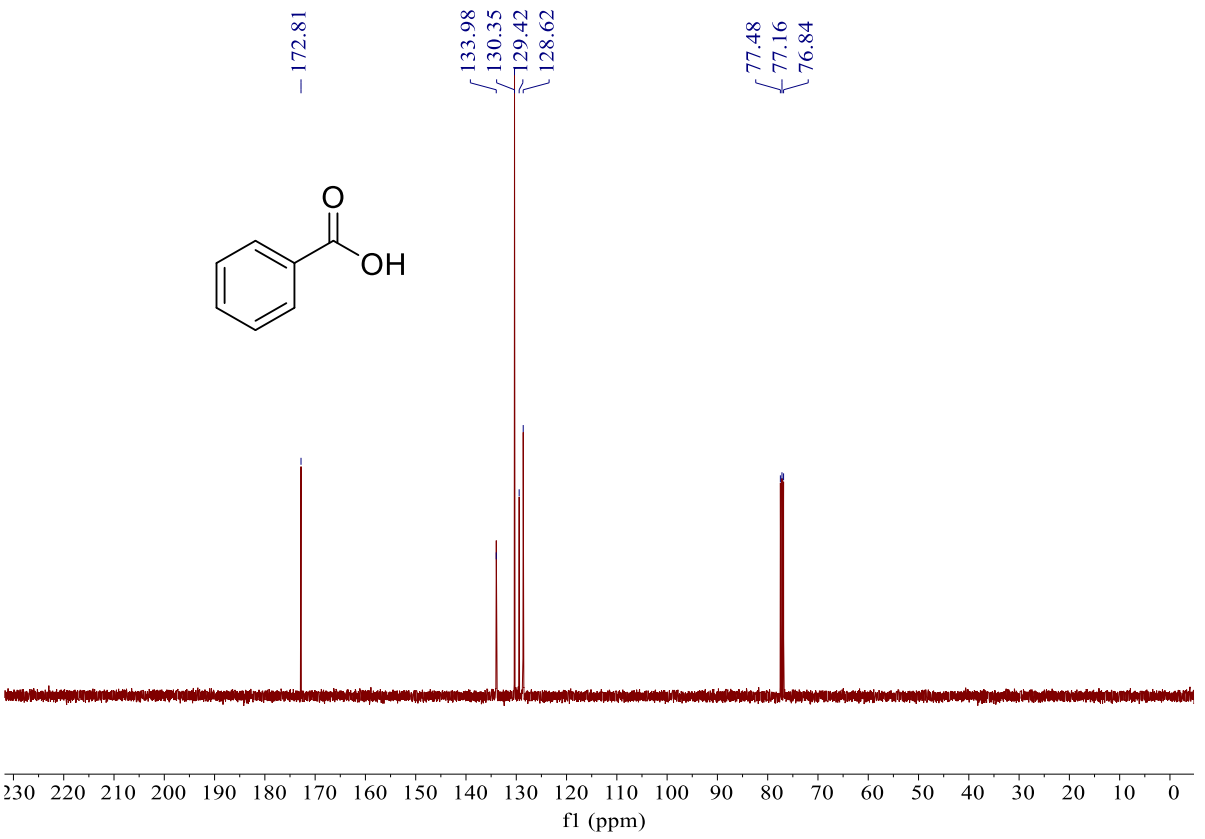

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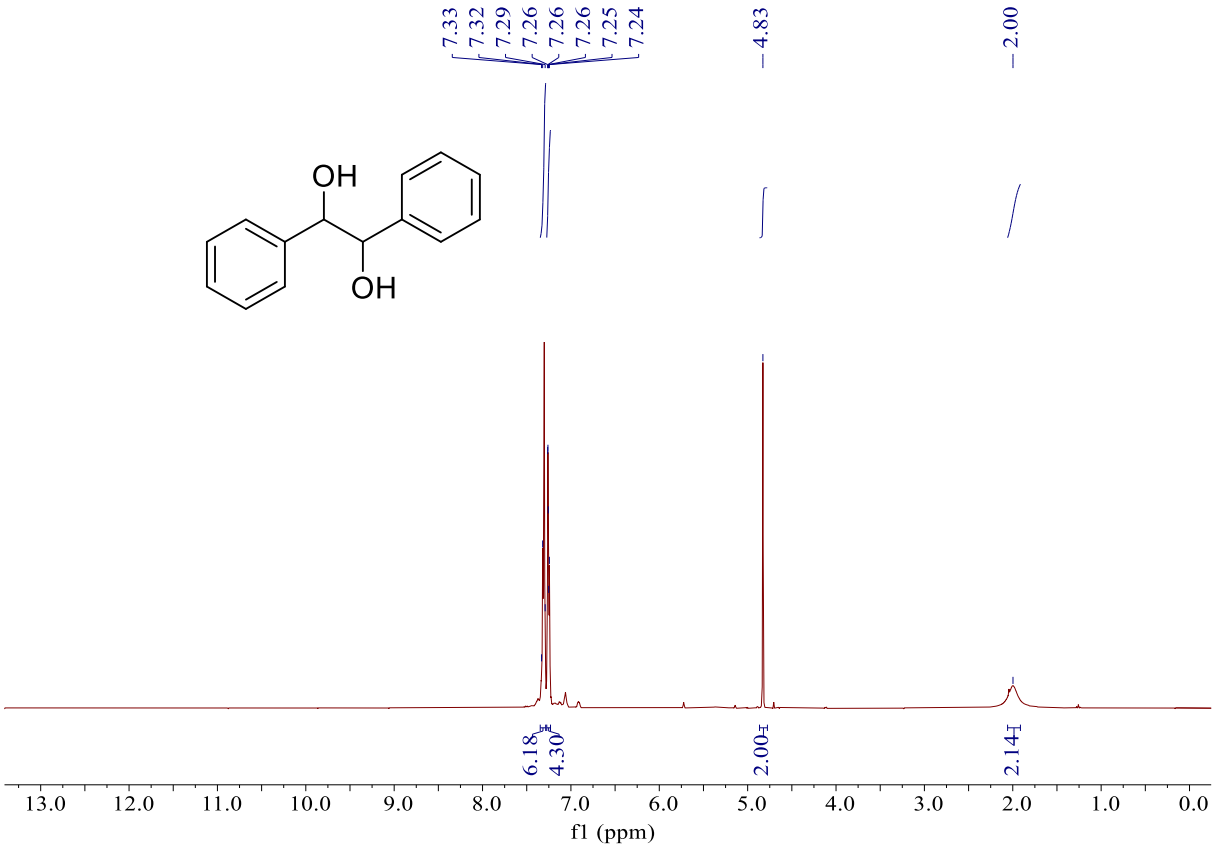

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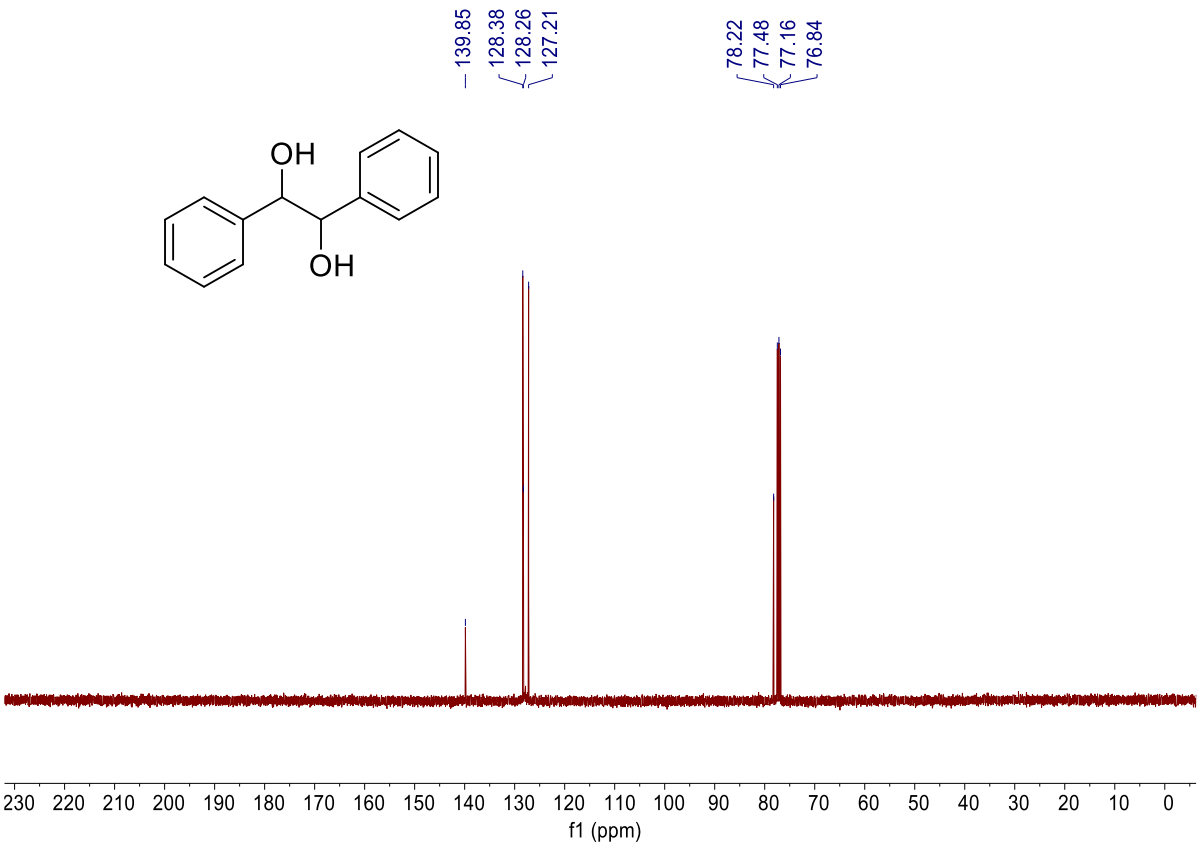

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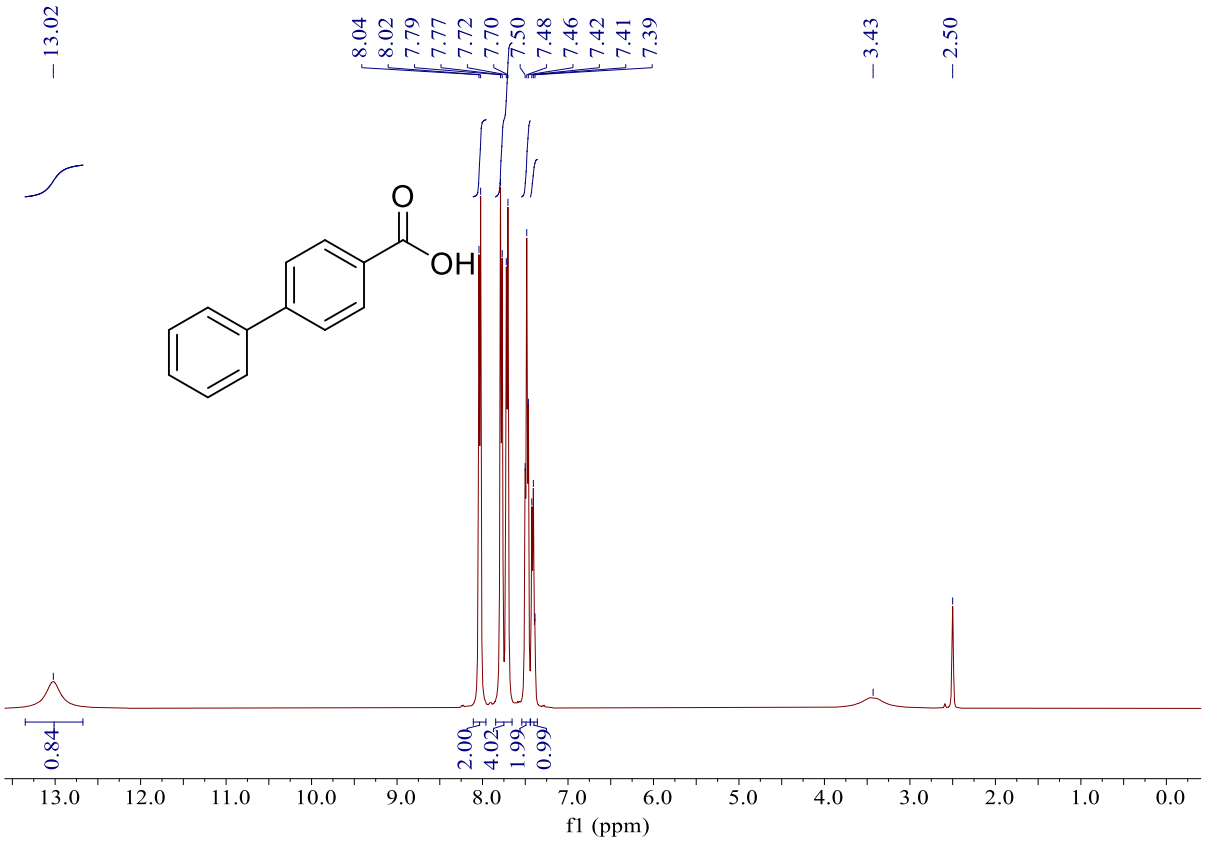

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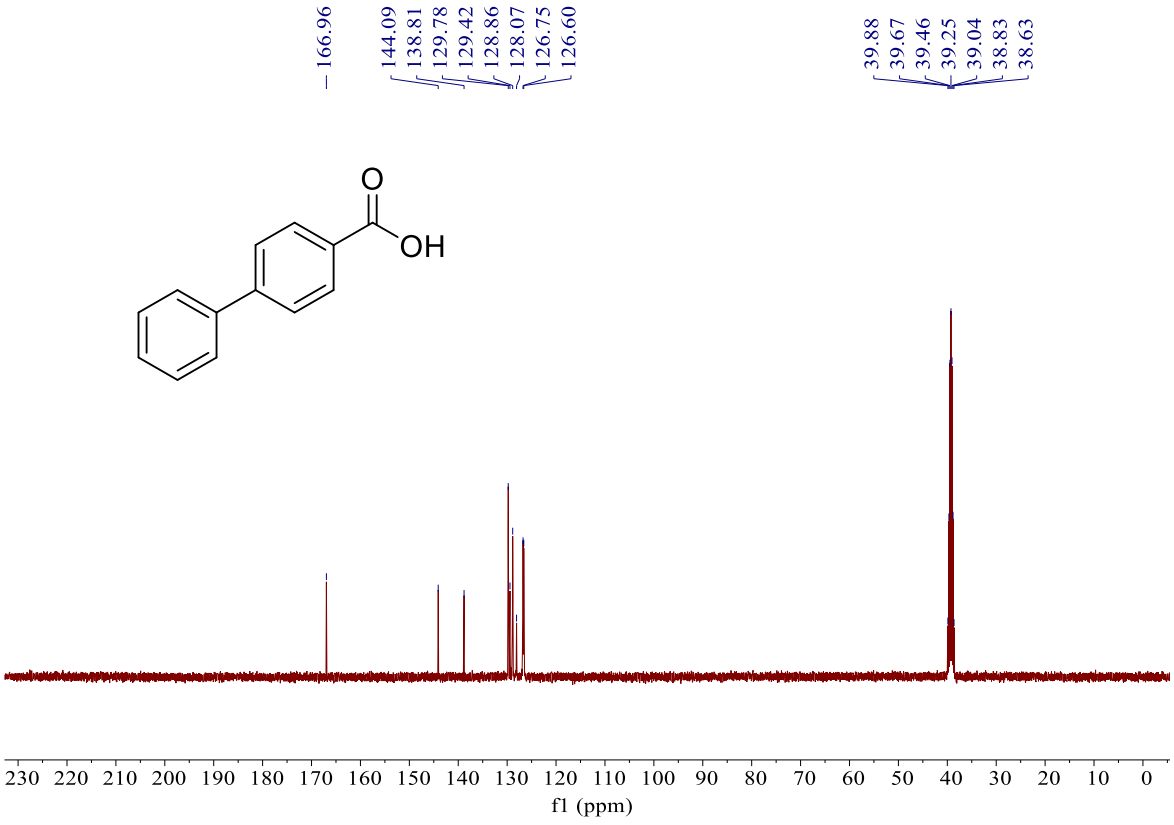

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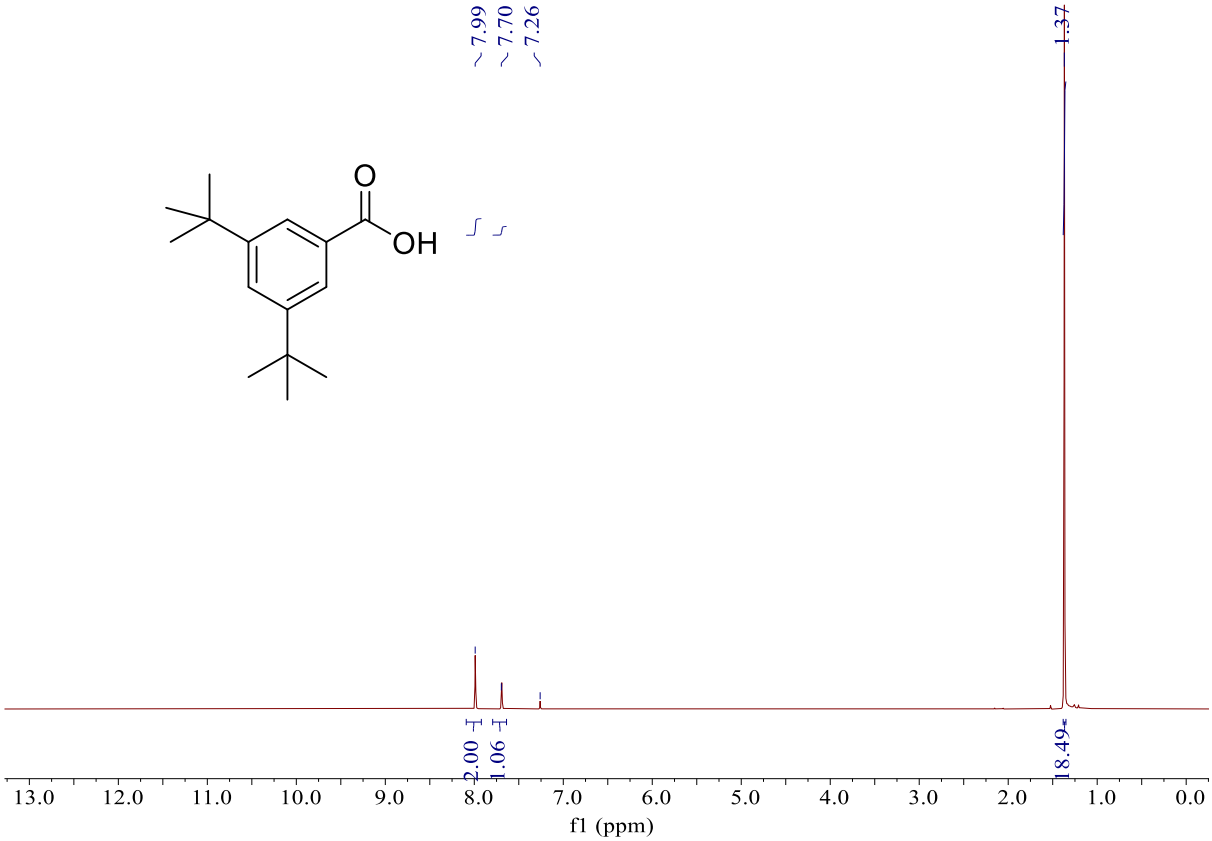

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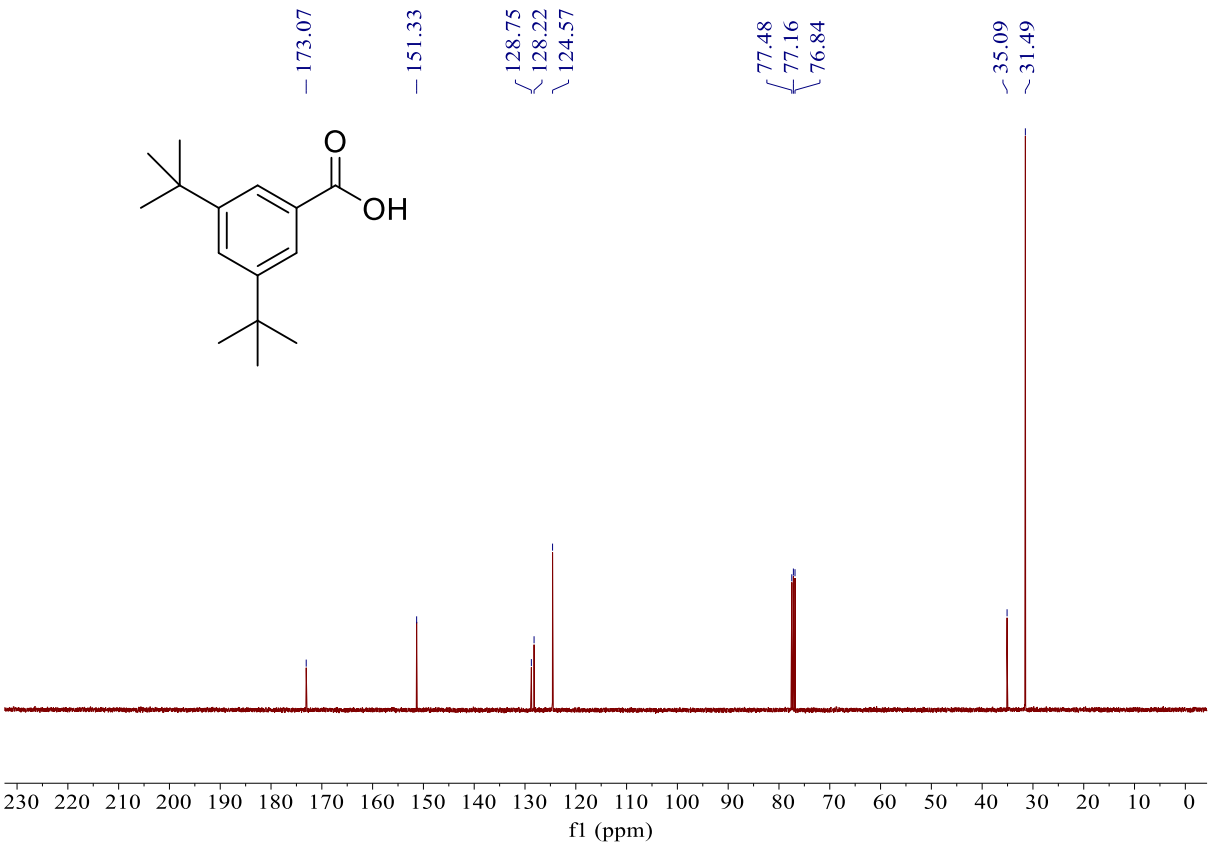
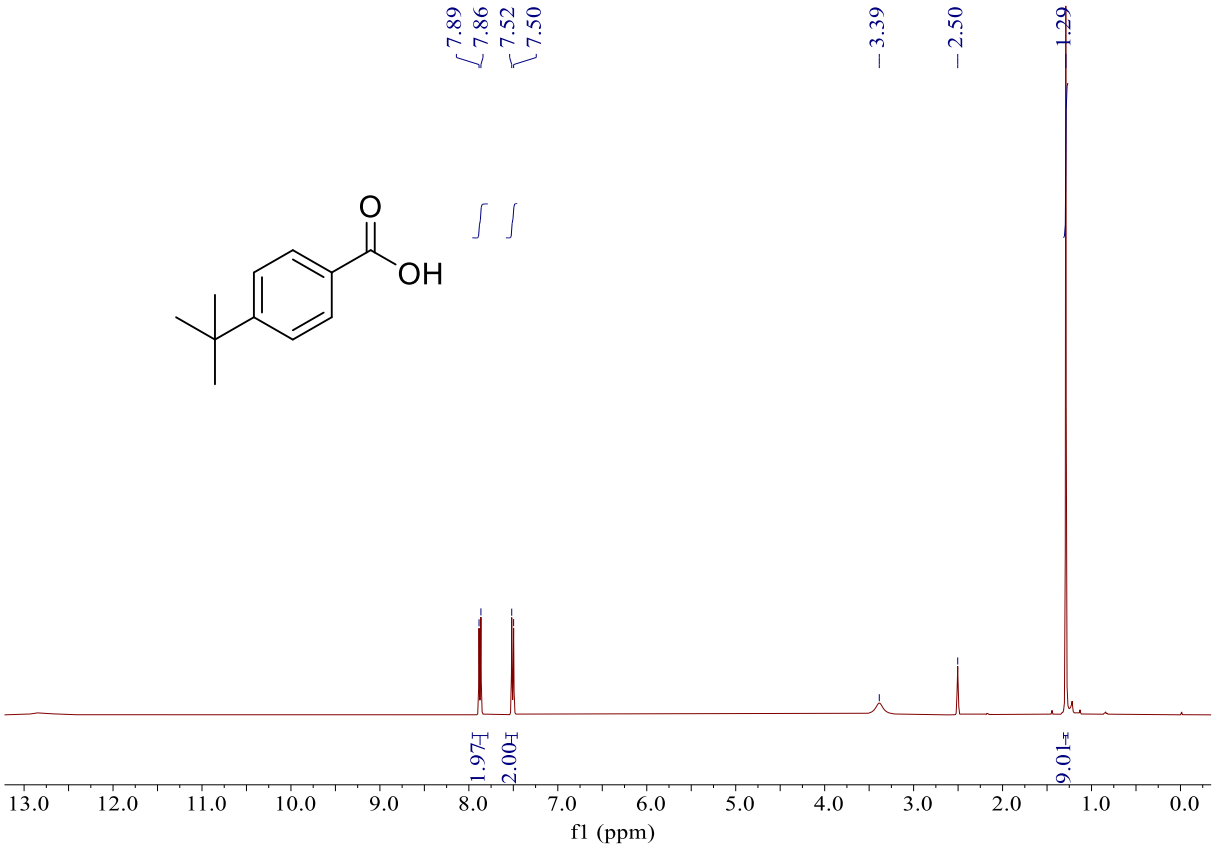


^{1}H NMR of compound 6k:



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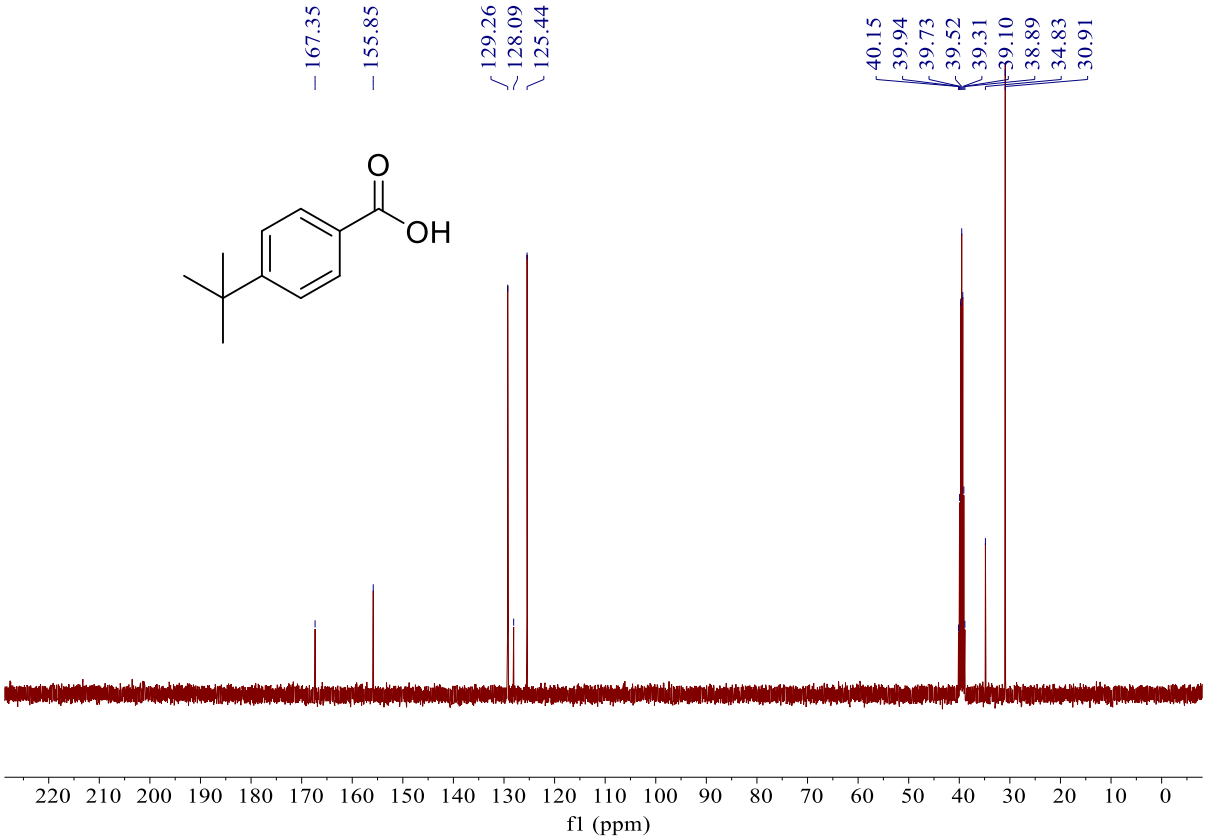

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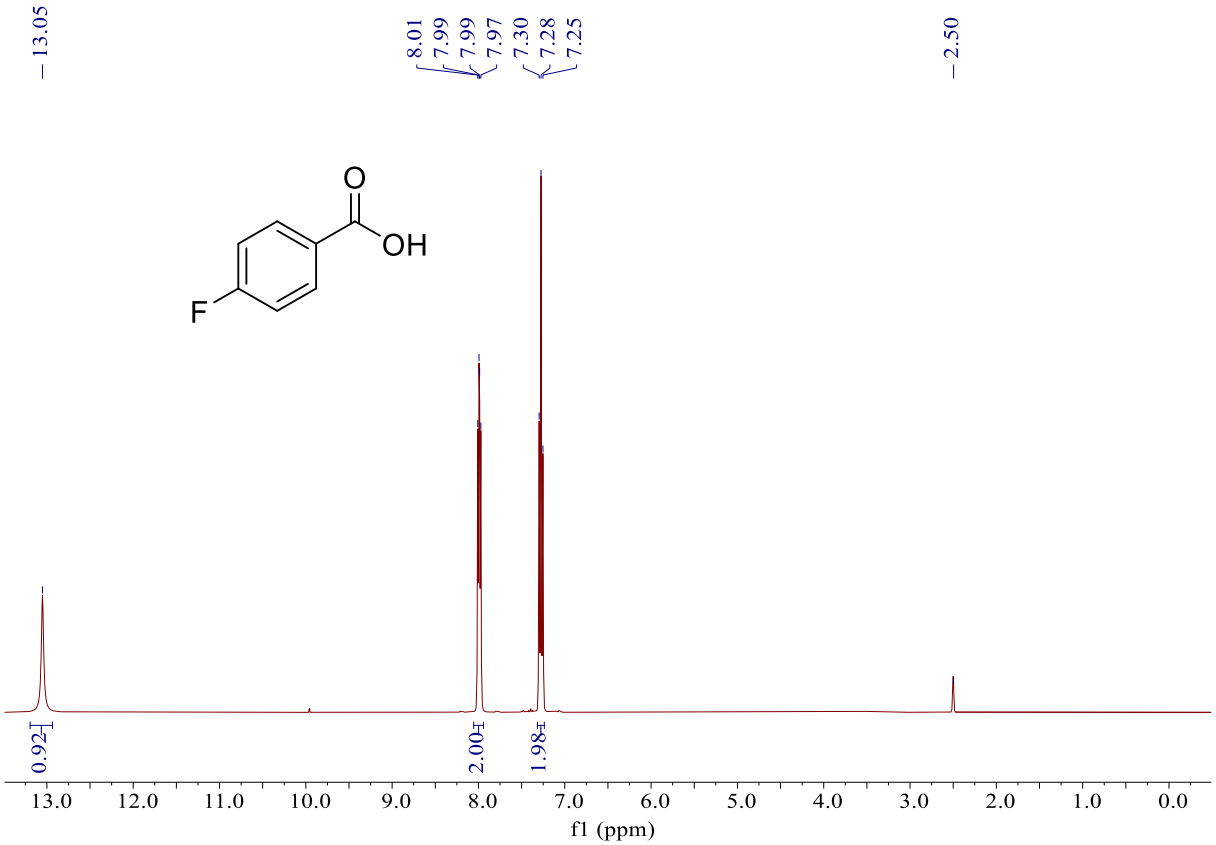

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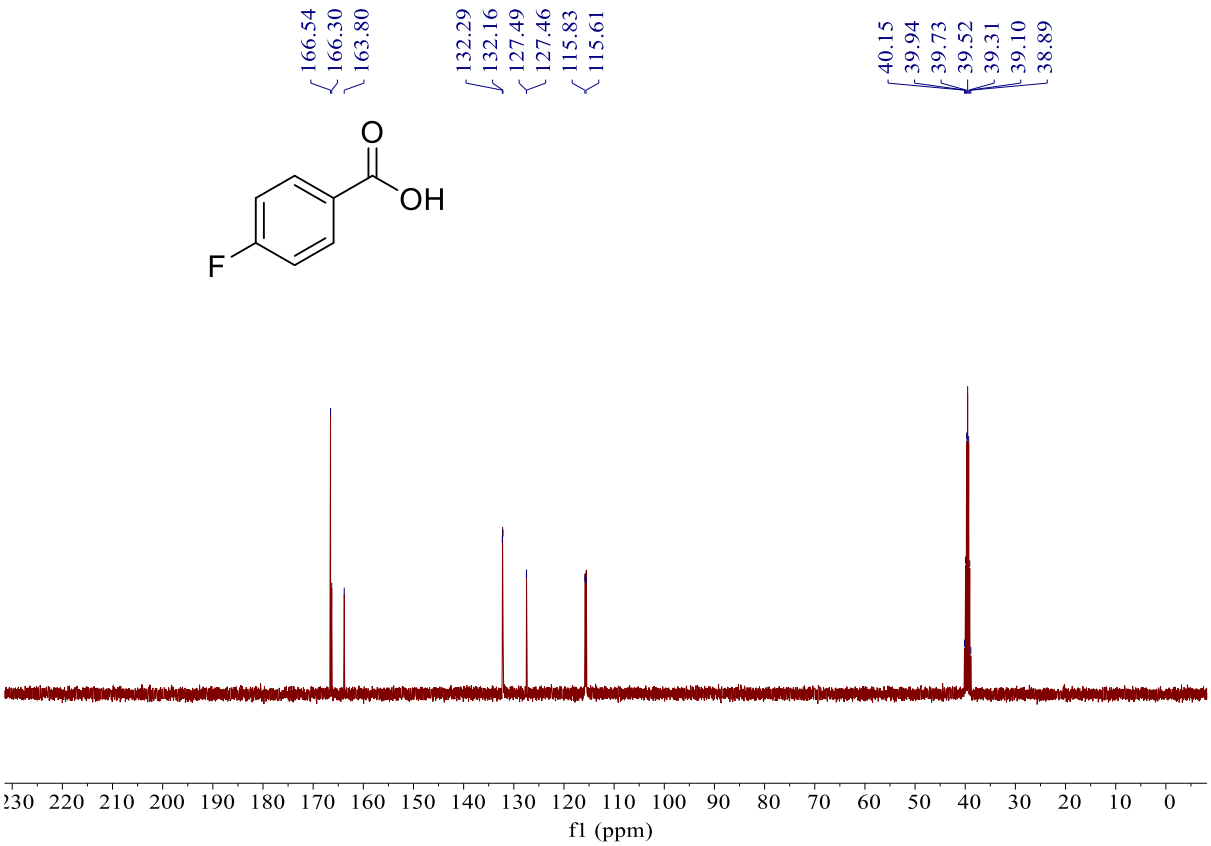
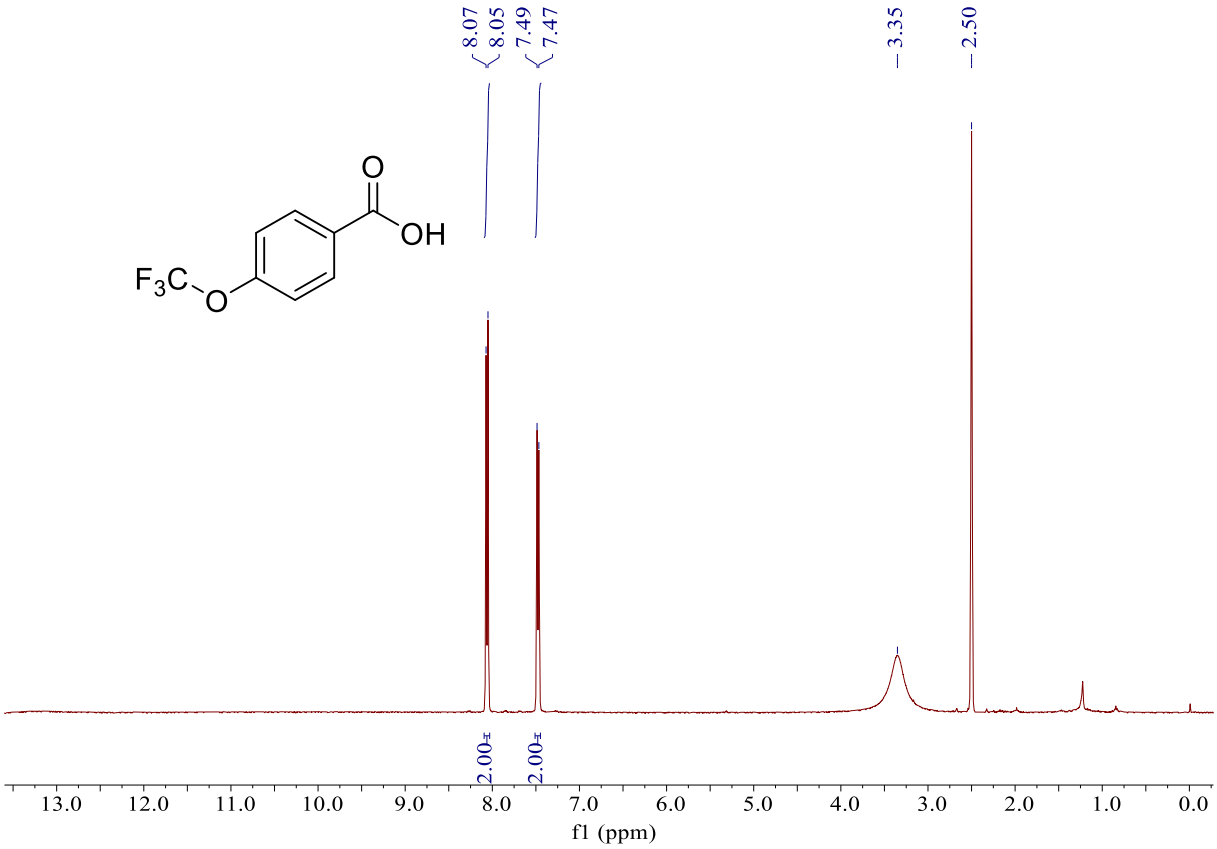


^{1}H NMR of compound 6m:



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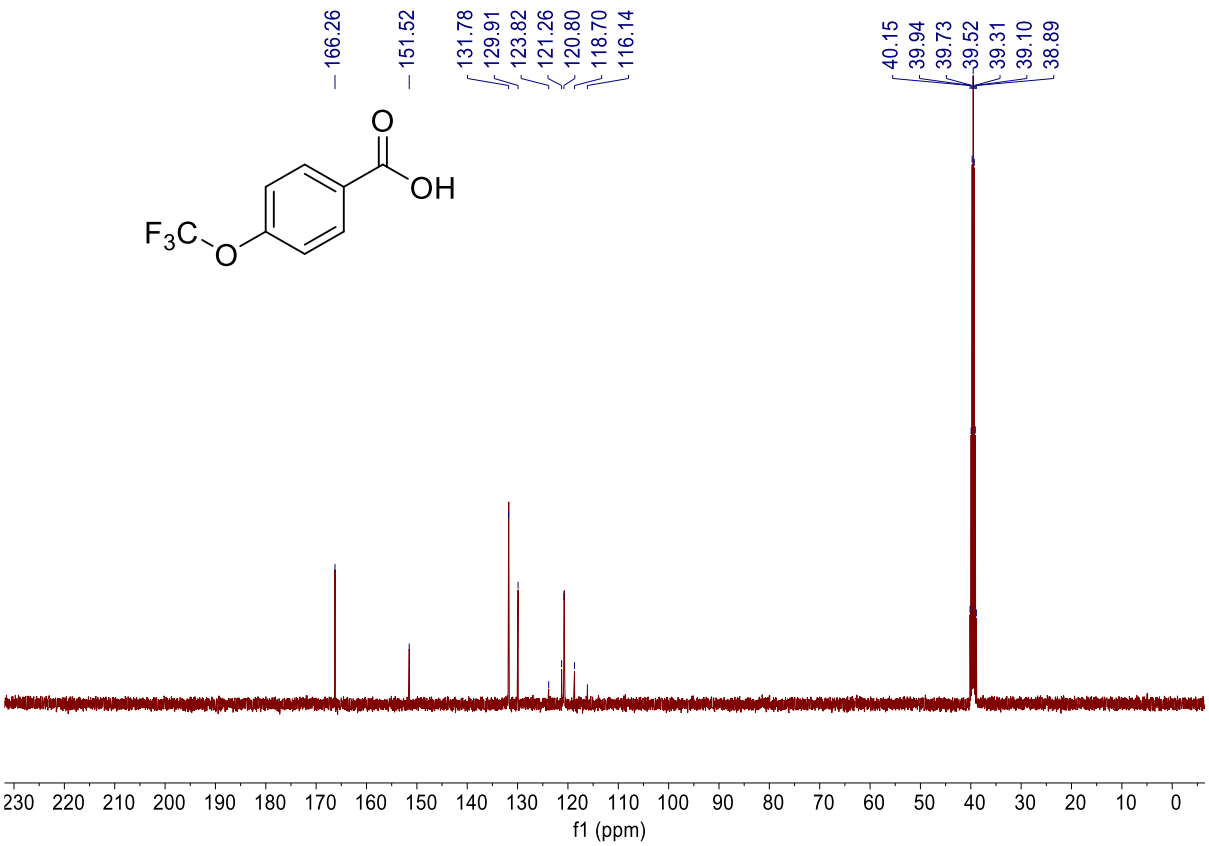
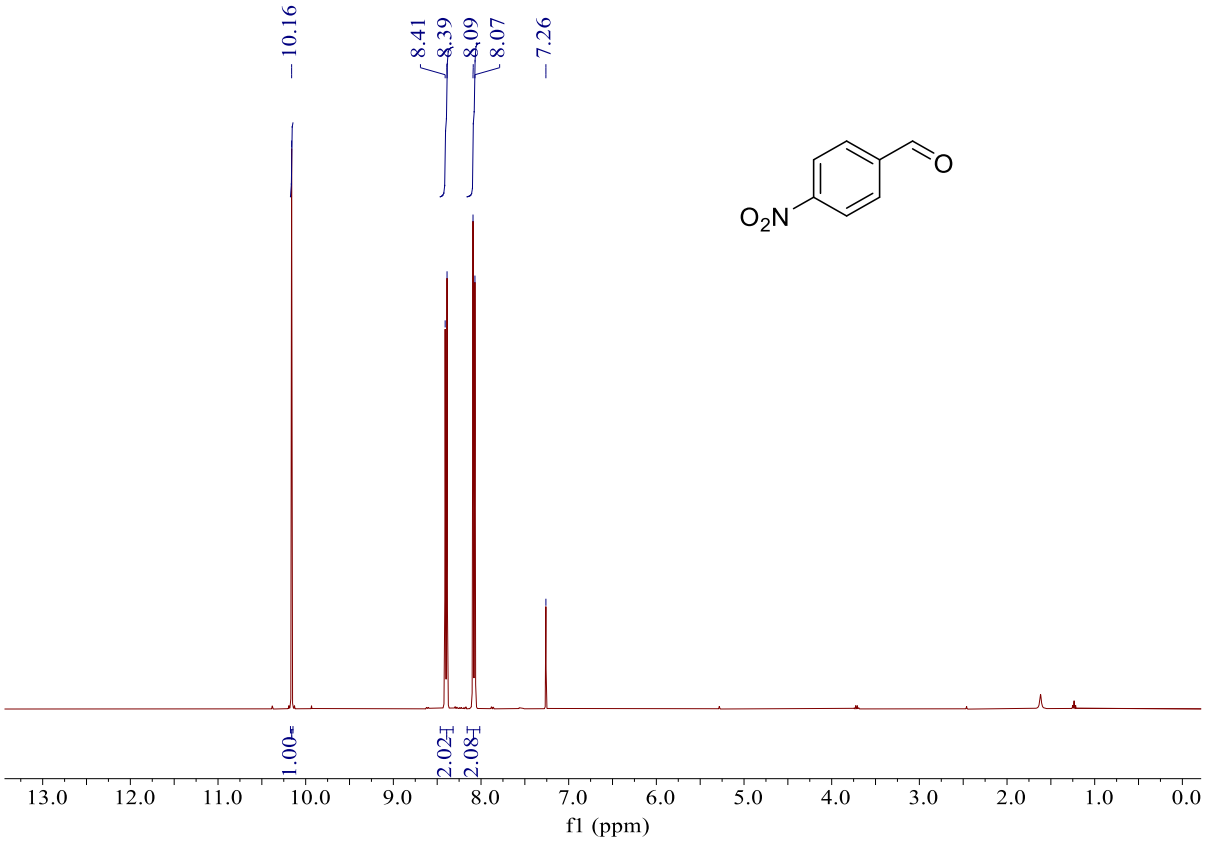
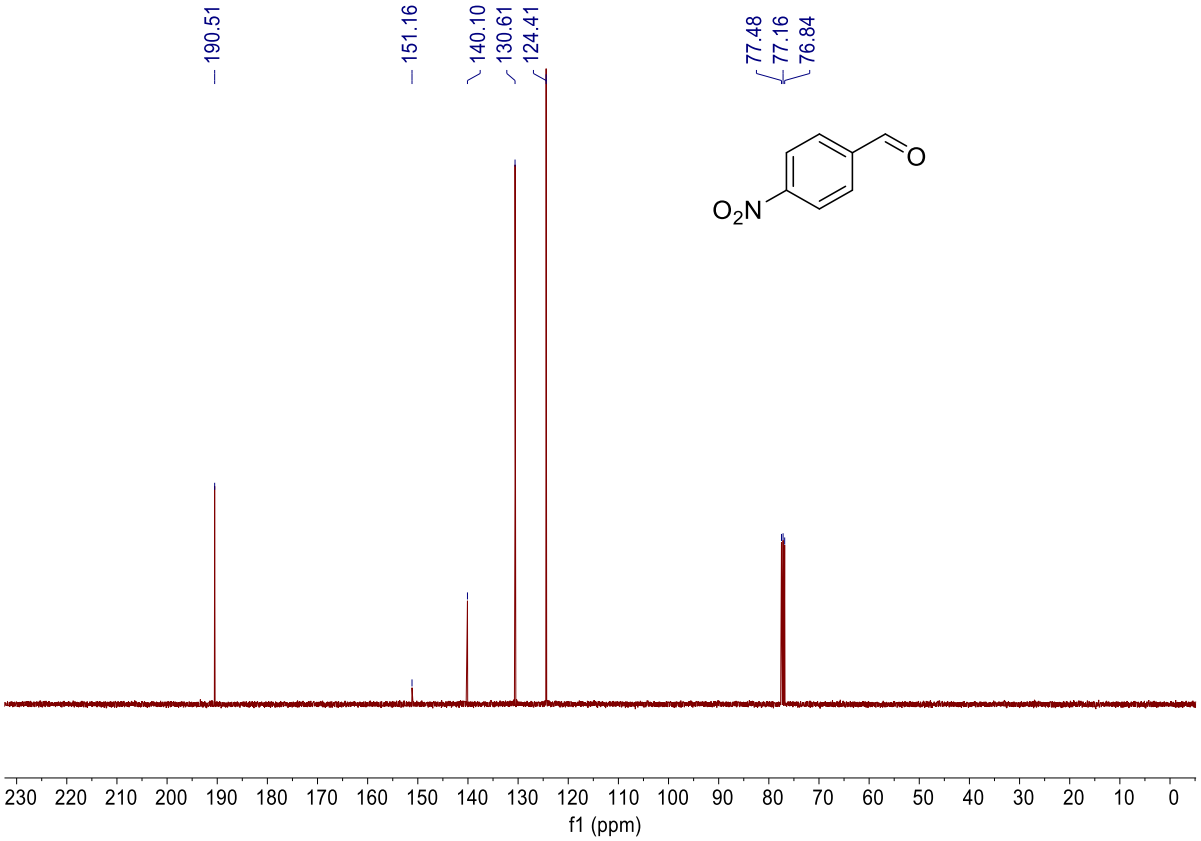


^{1}H NMR of compound 13a:



^{13}C NMR of compound 13a:



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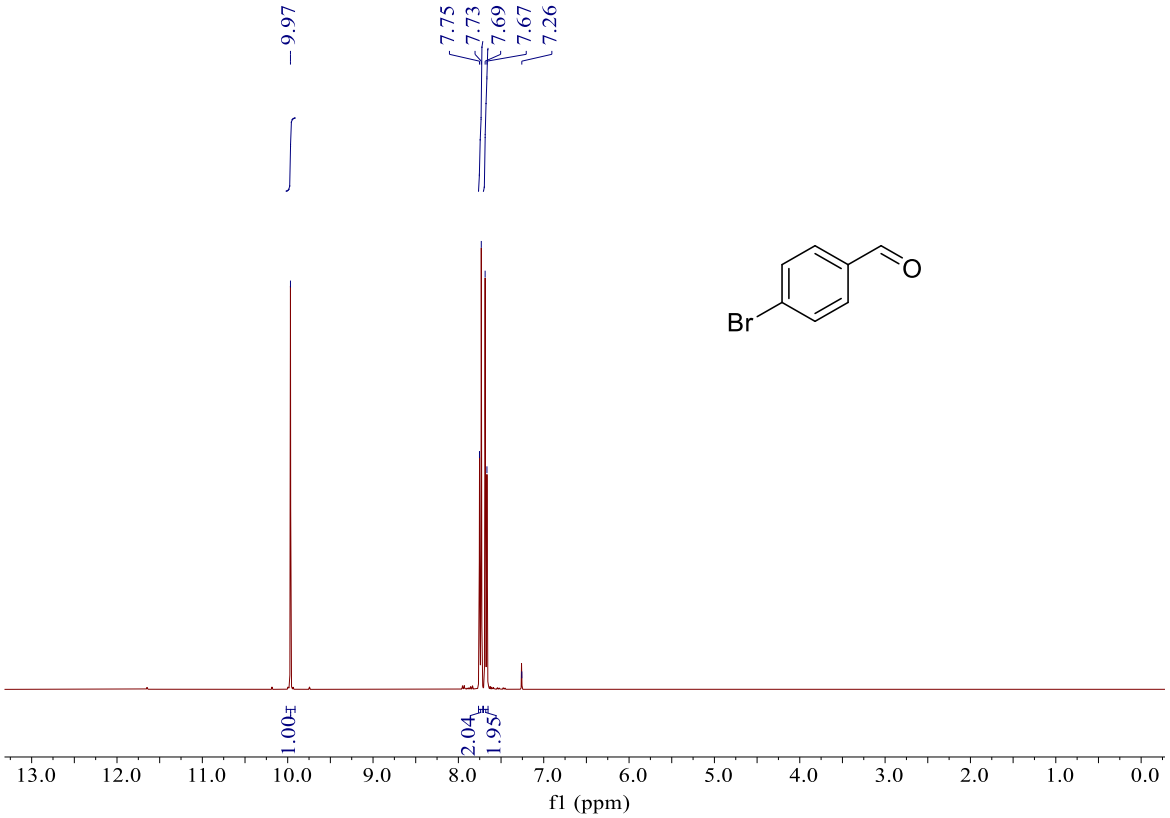
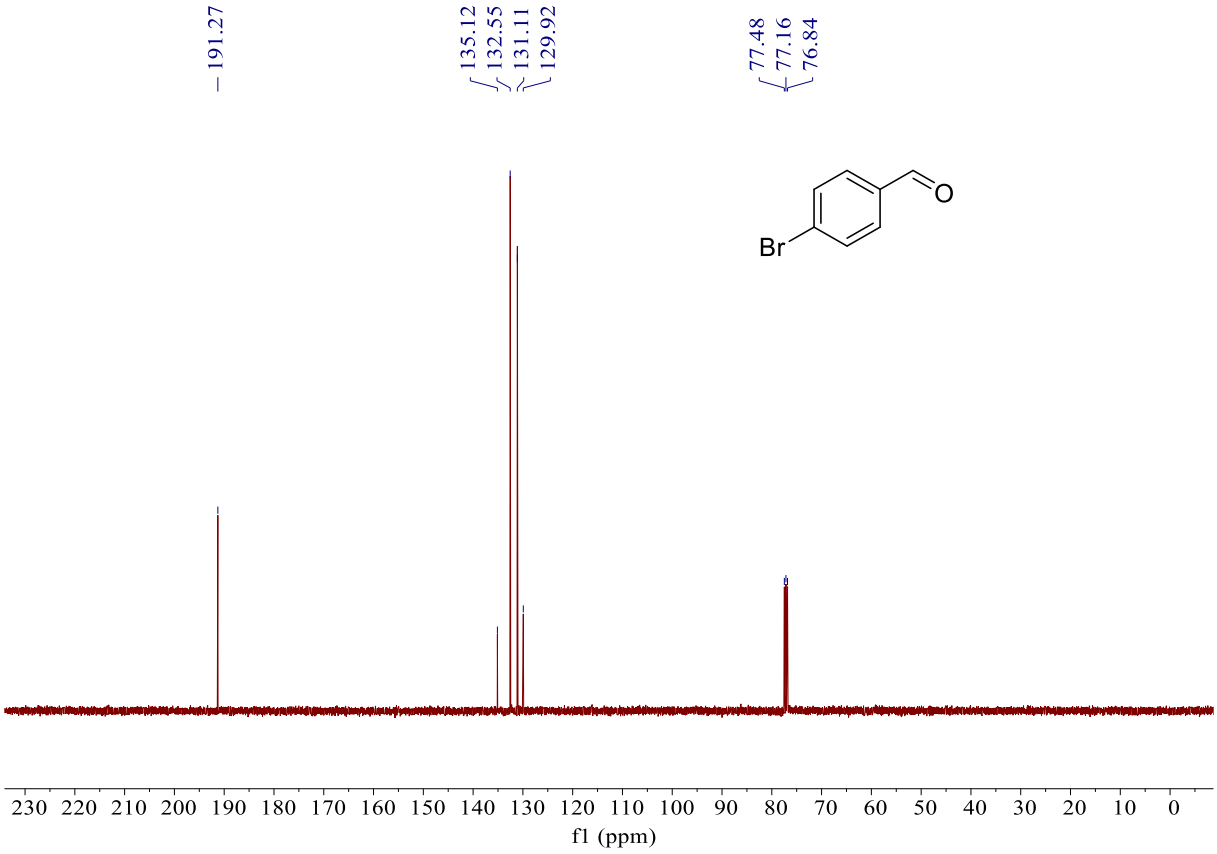


^{13}C NMR of compound 13b:



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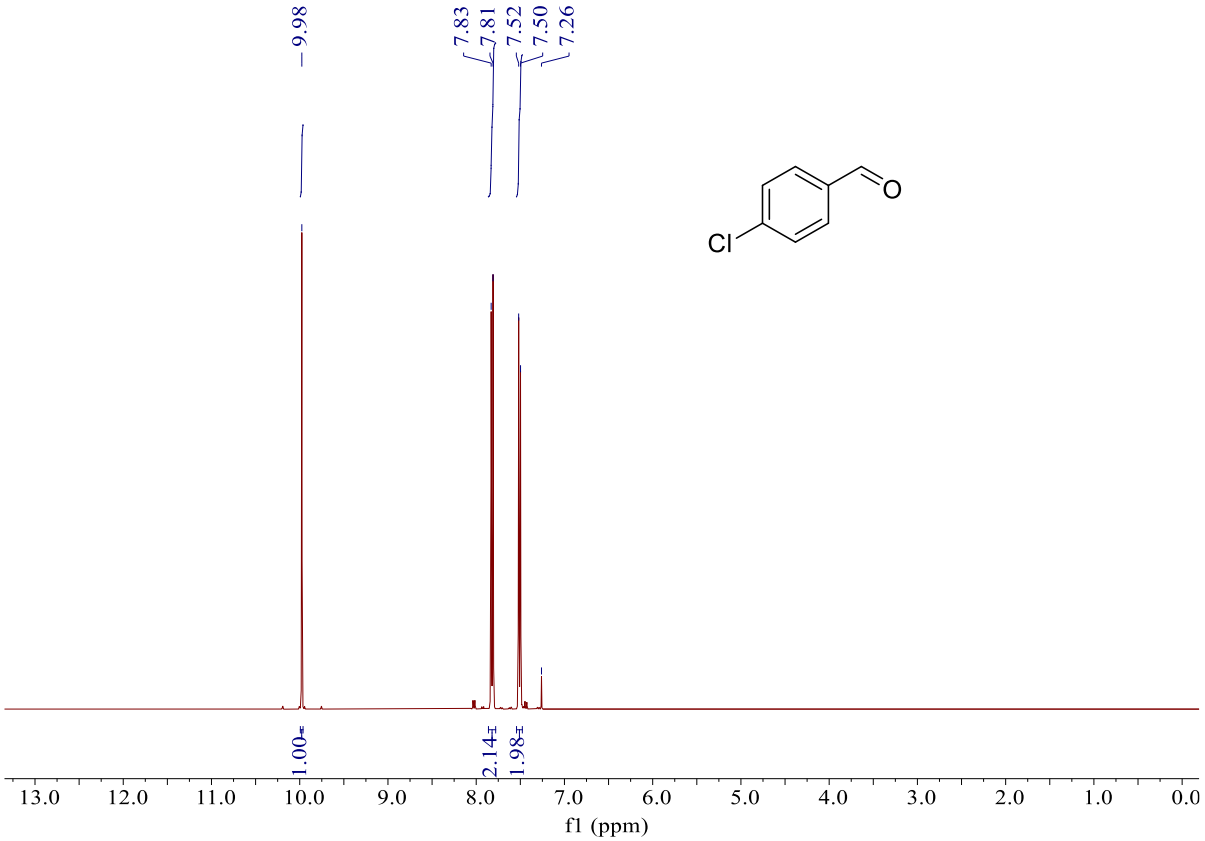

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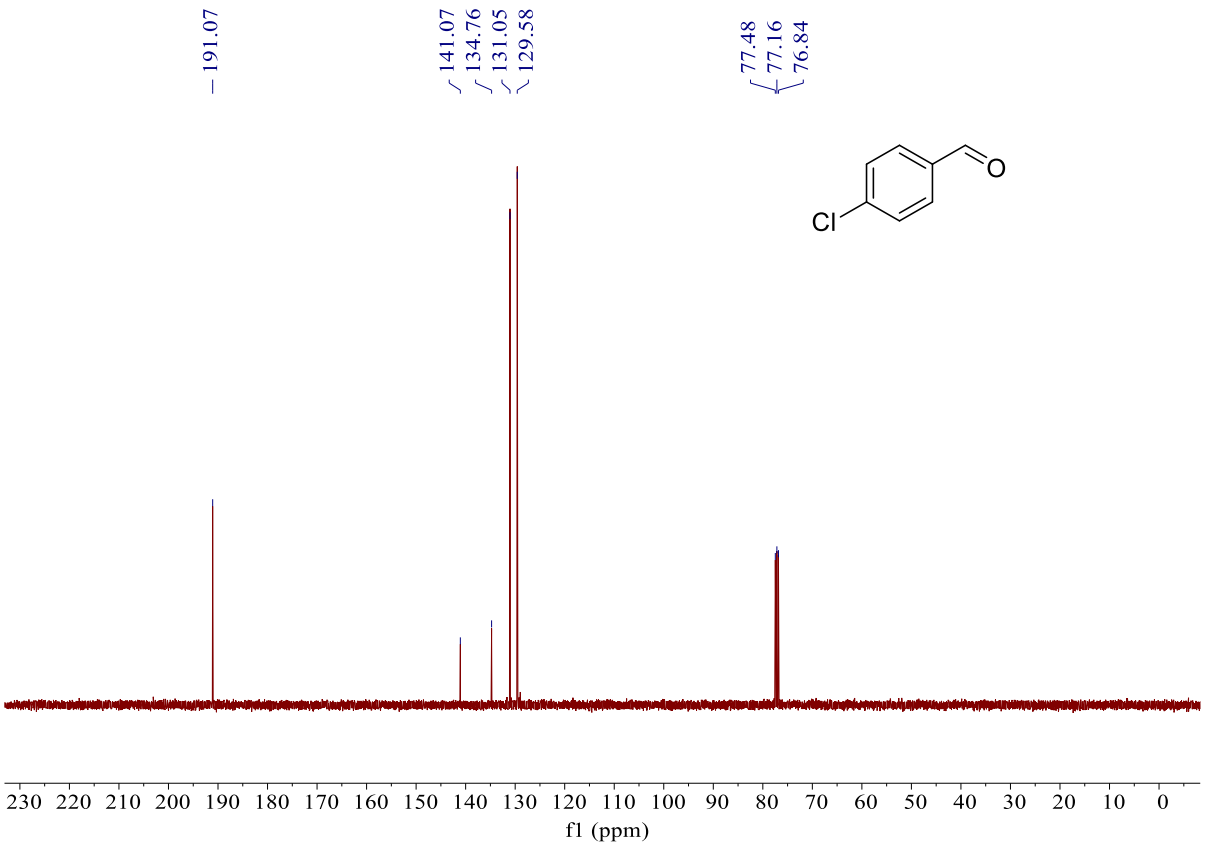
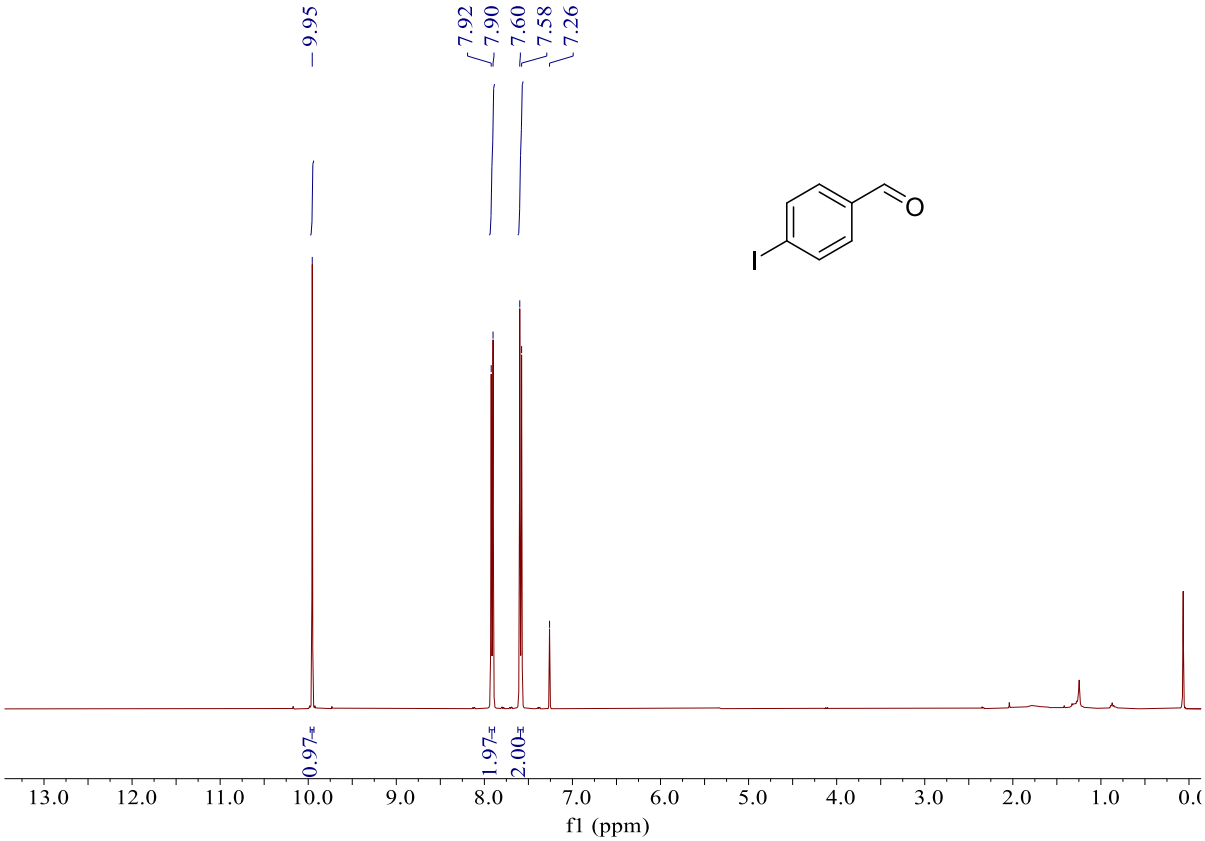
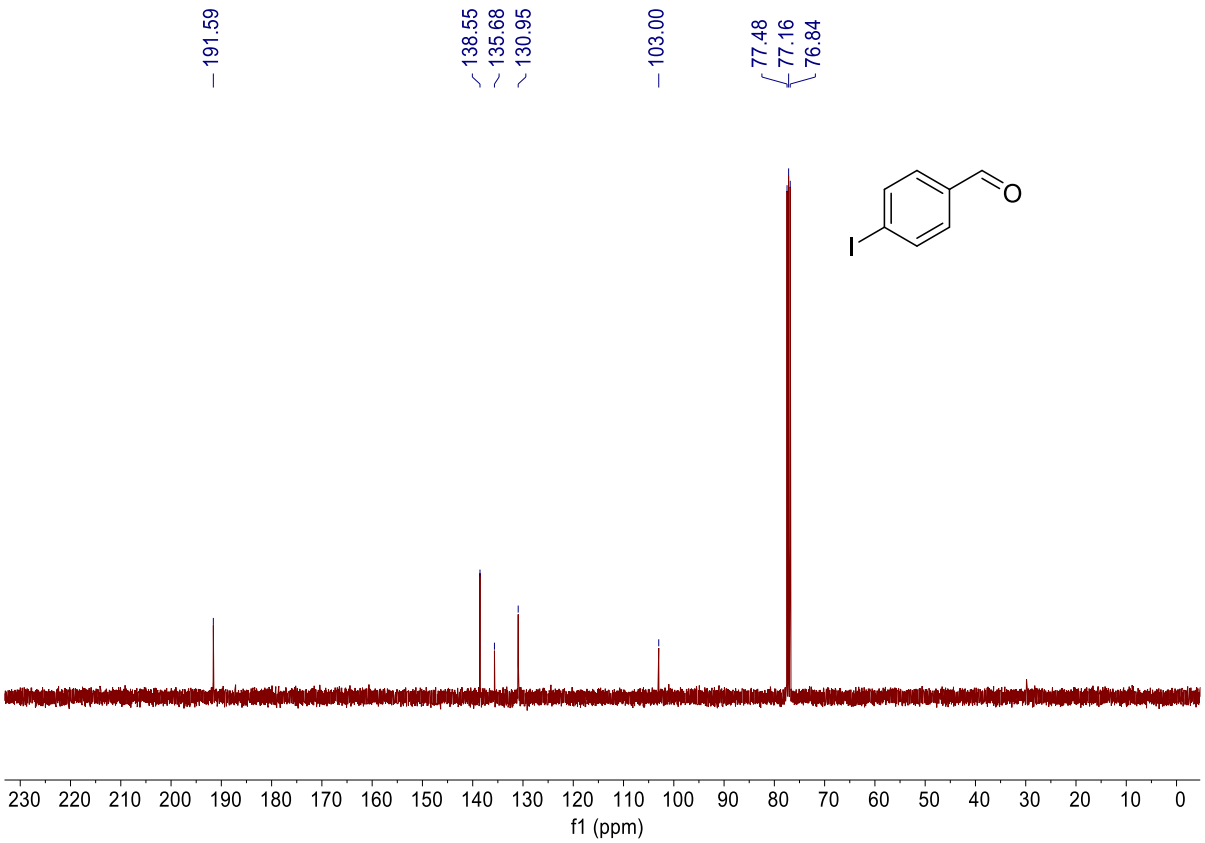


^{1}H NMR of compound 13d:



^{13}C NMR of compound 13d:



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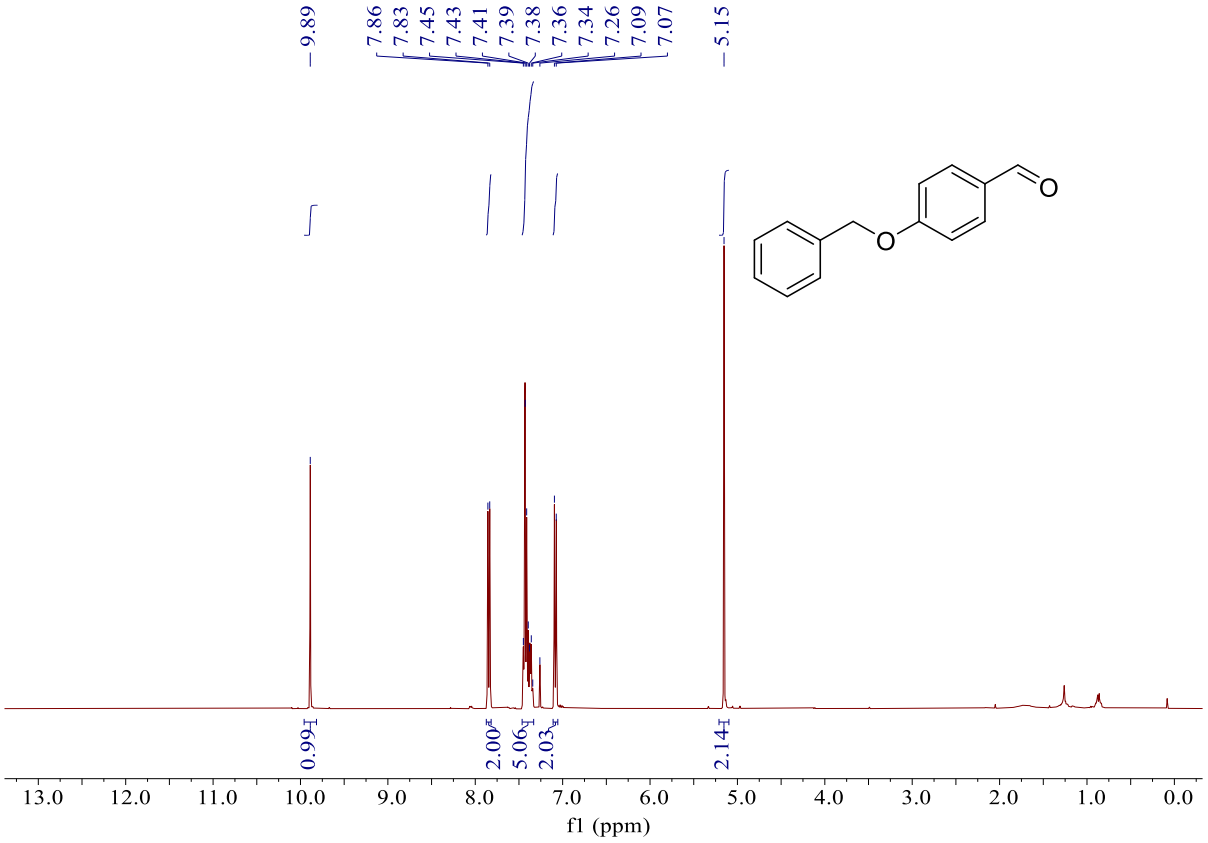

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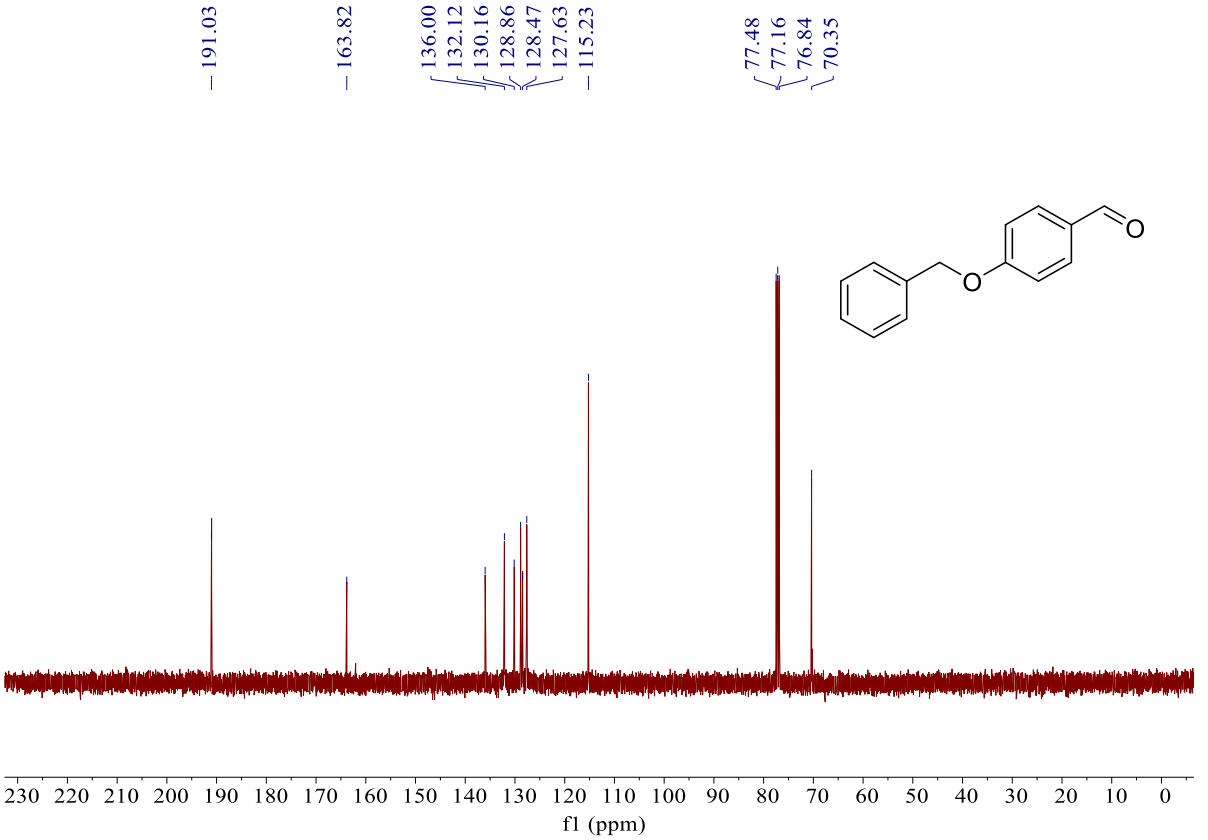

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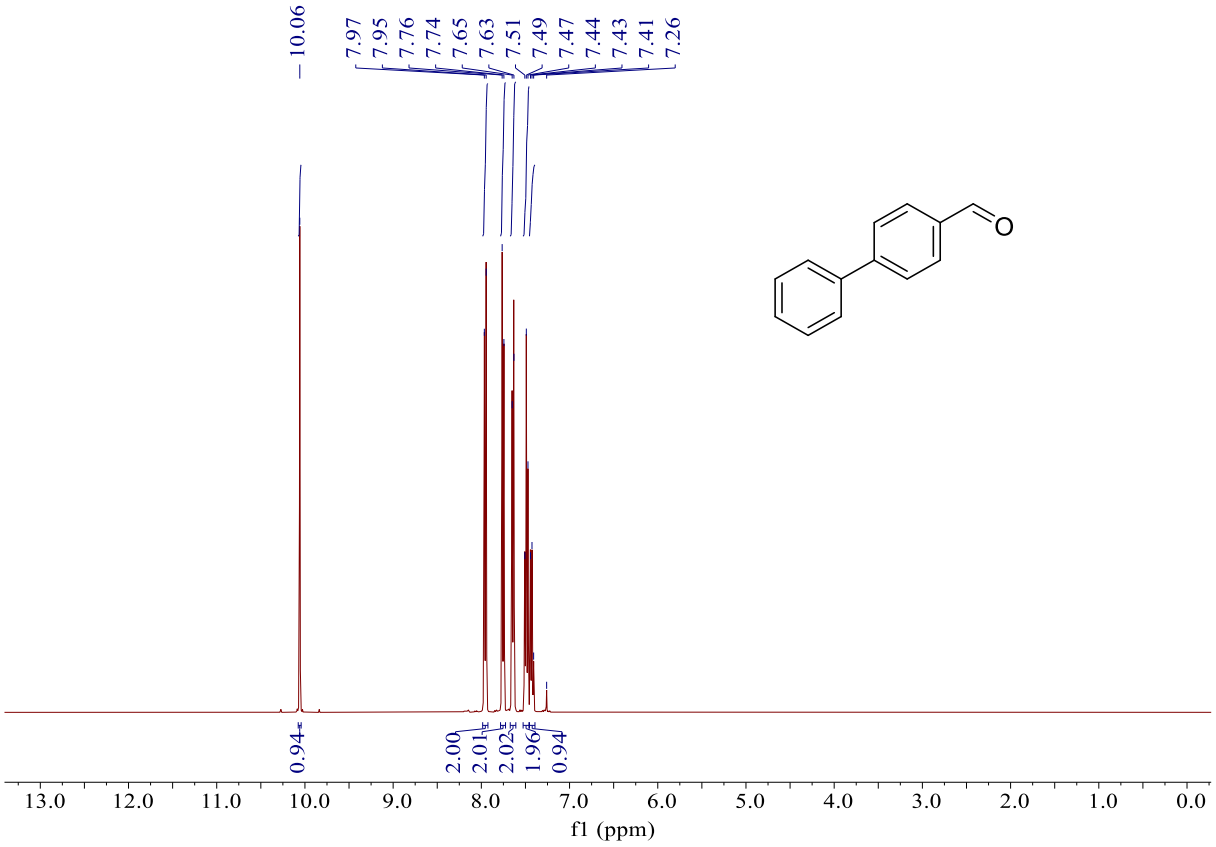
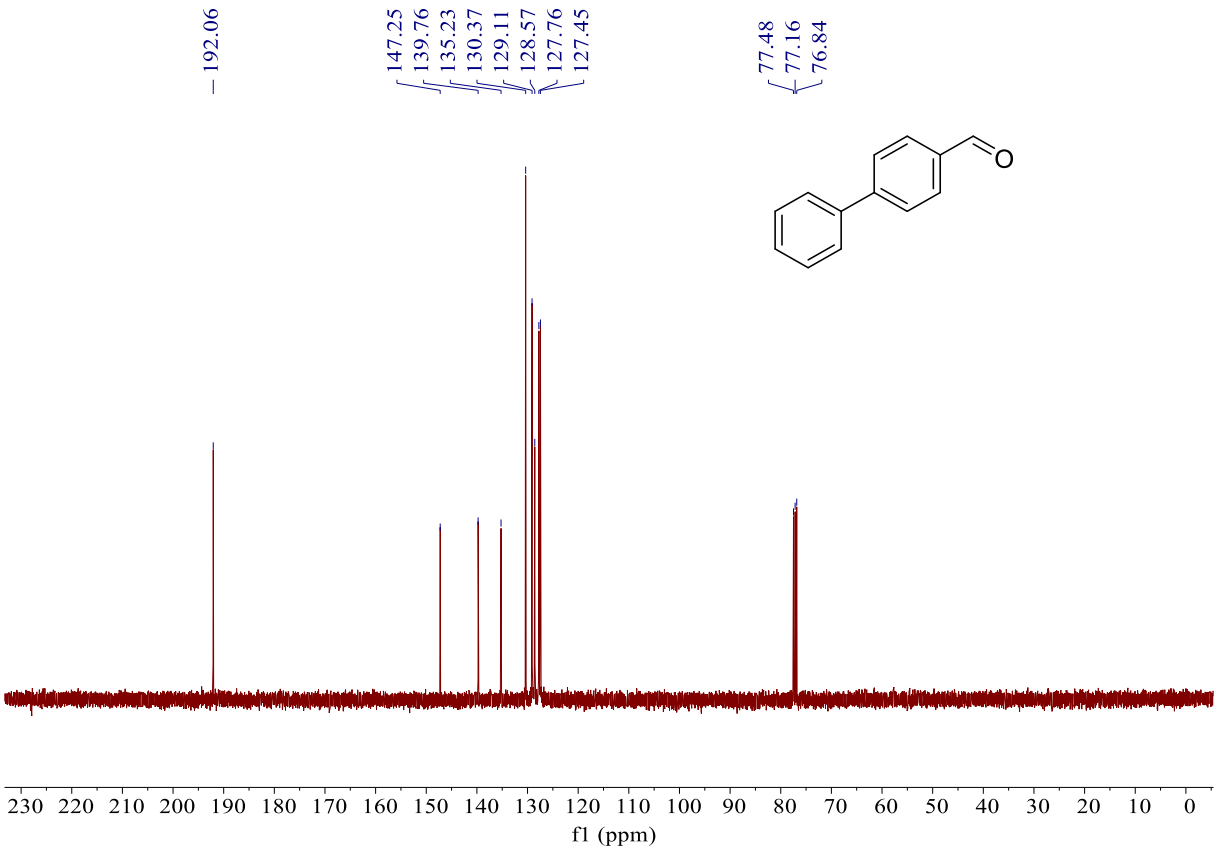
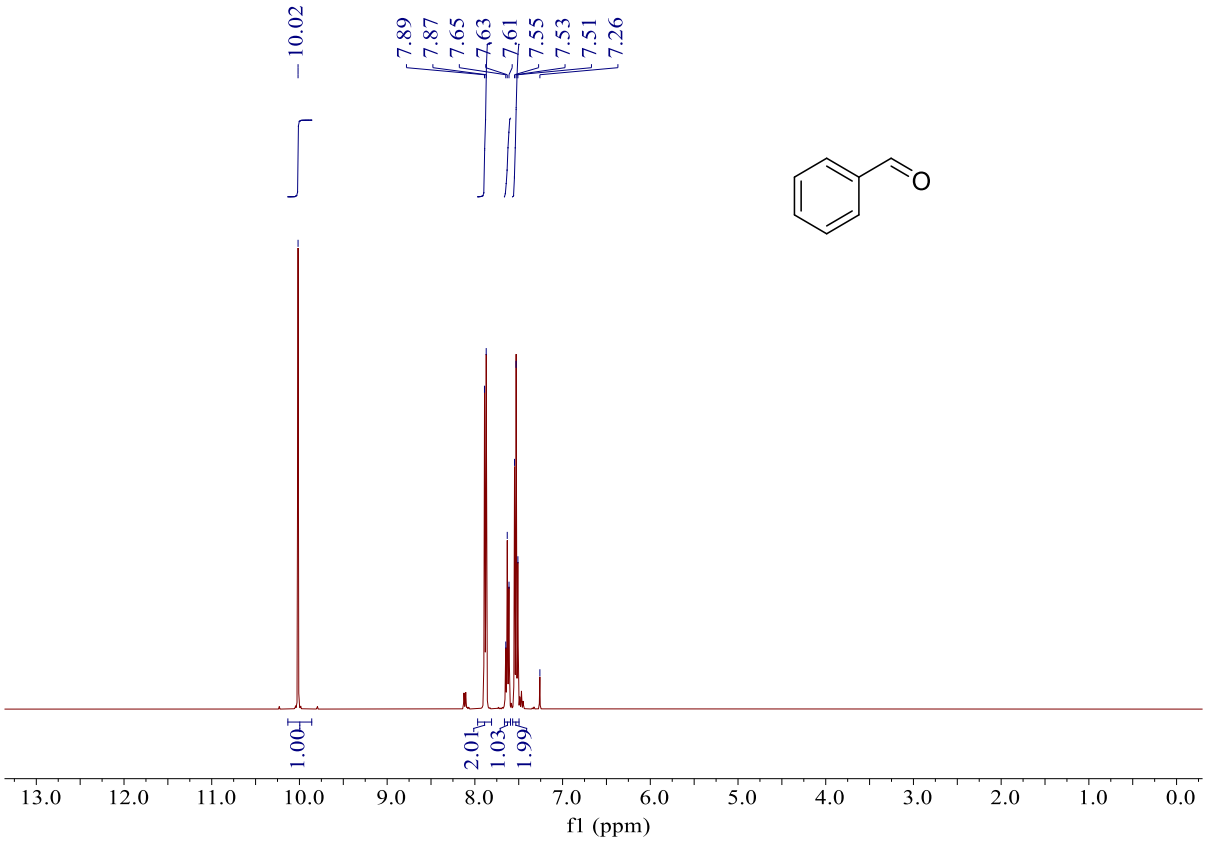


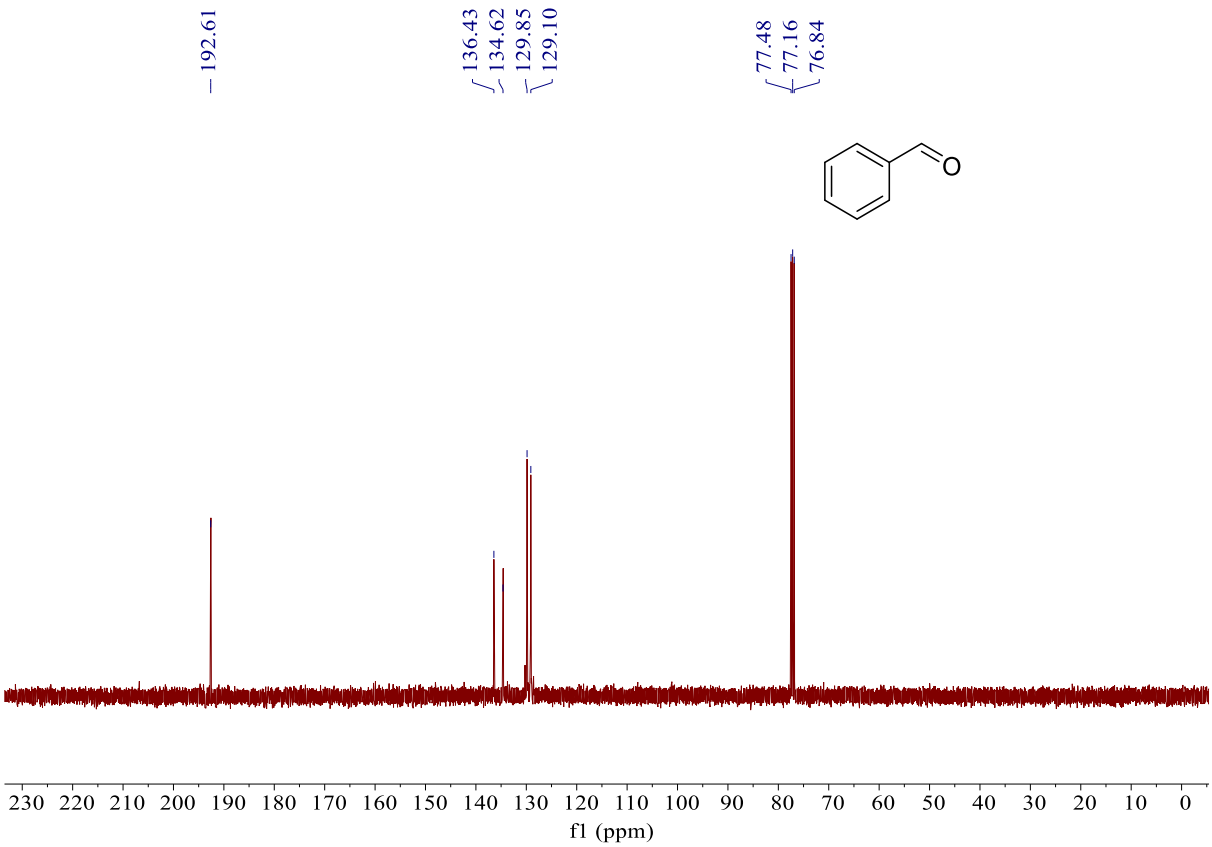
^{13}C NMR of compound 13f:



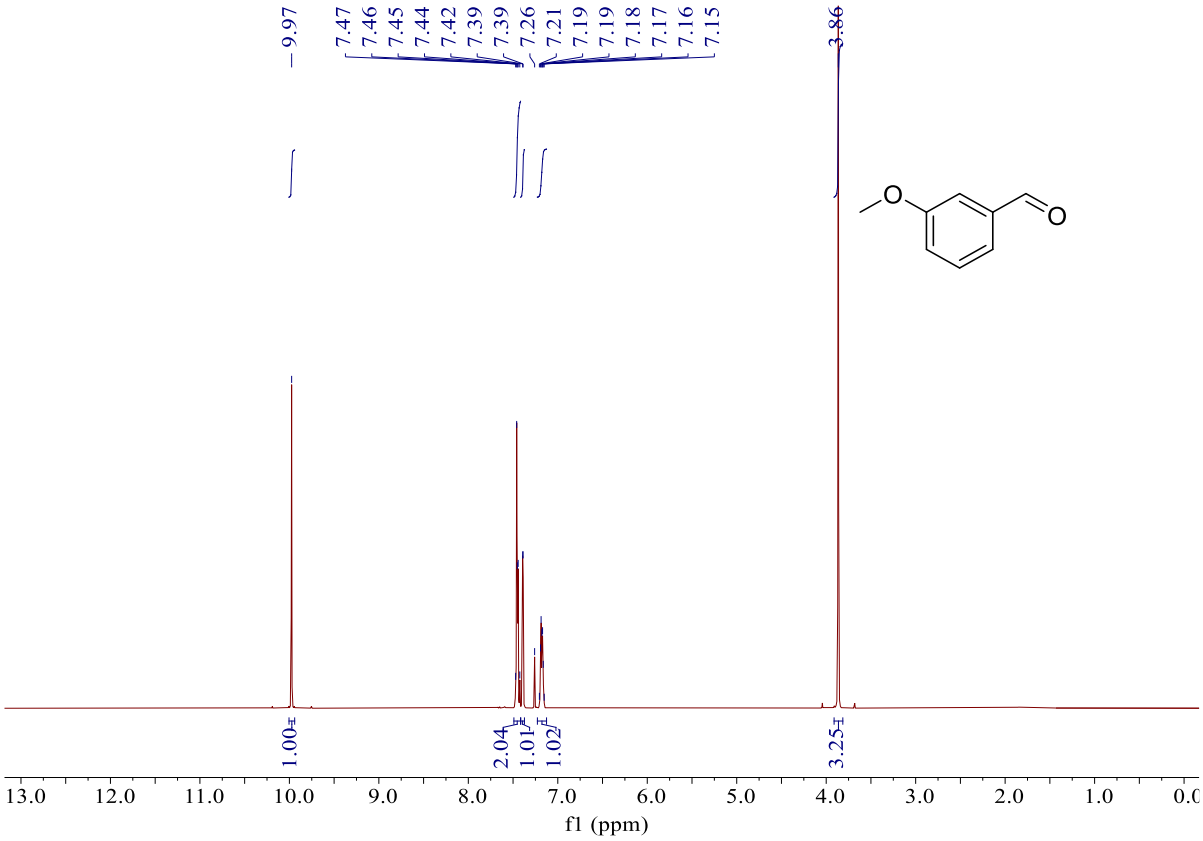
^{1}H NMR of compound 13g:



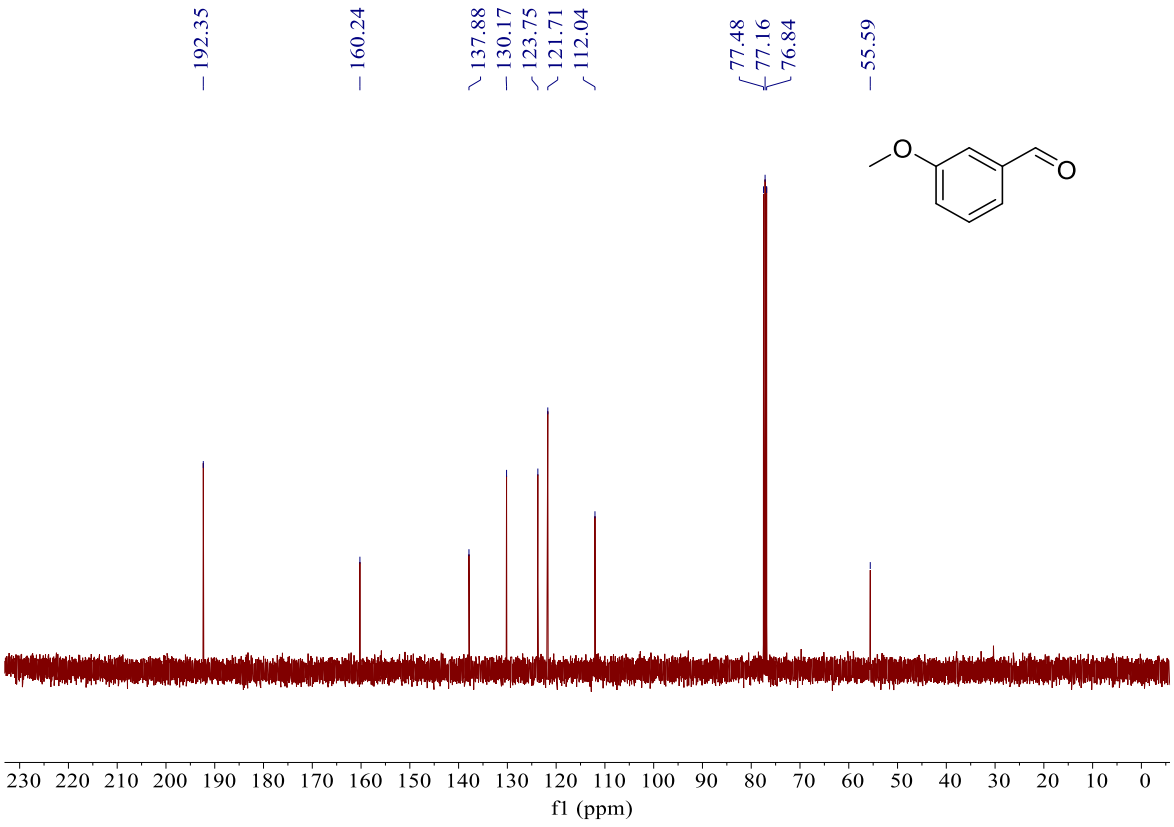
^{13}C NMR of compound 13g:



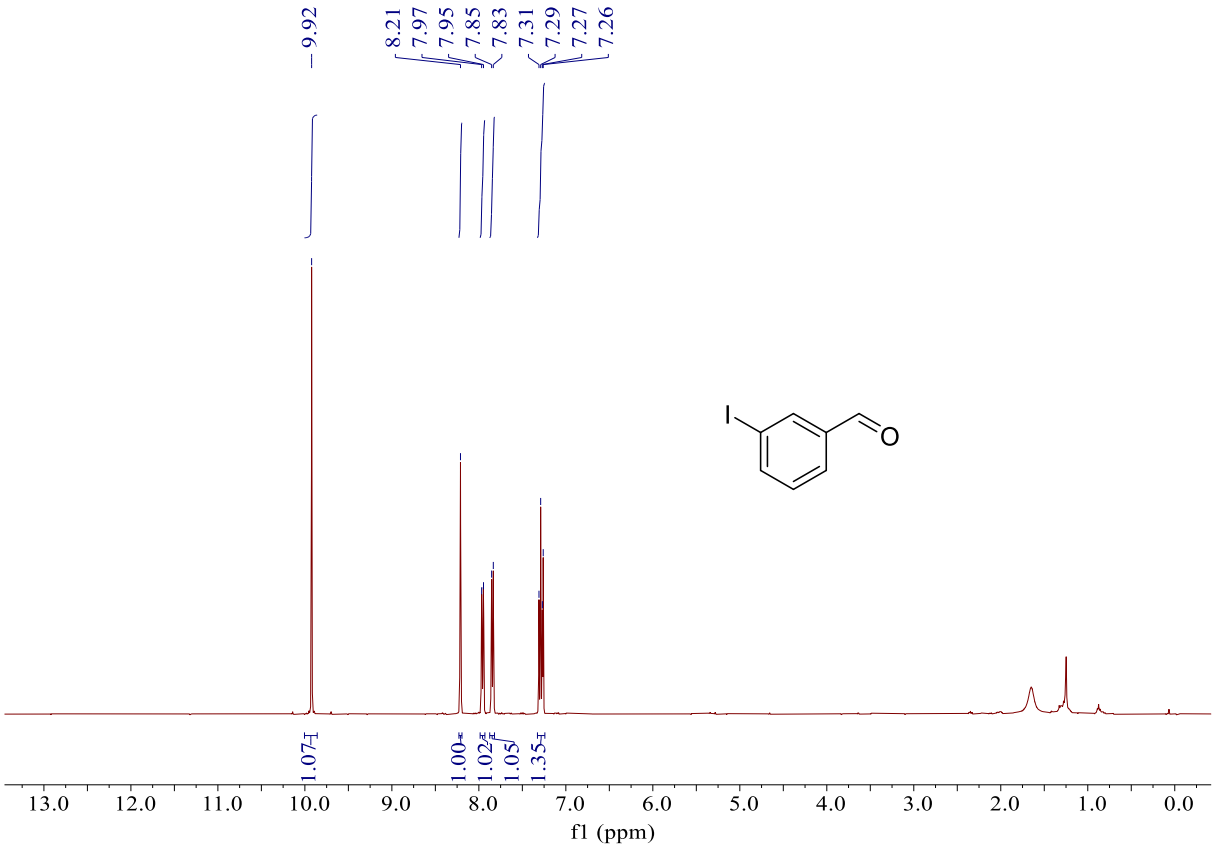
^{1}H NMR of compound 13h:



^{13}C NMR of compound 13h:



^{1}H NMR of compound 13i:



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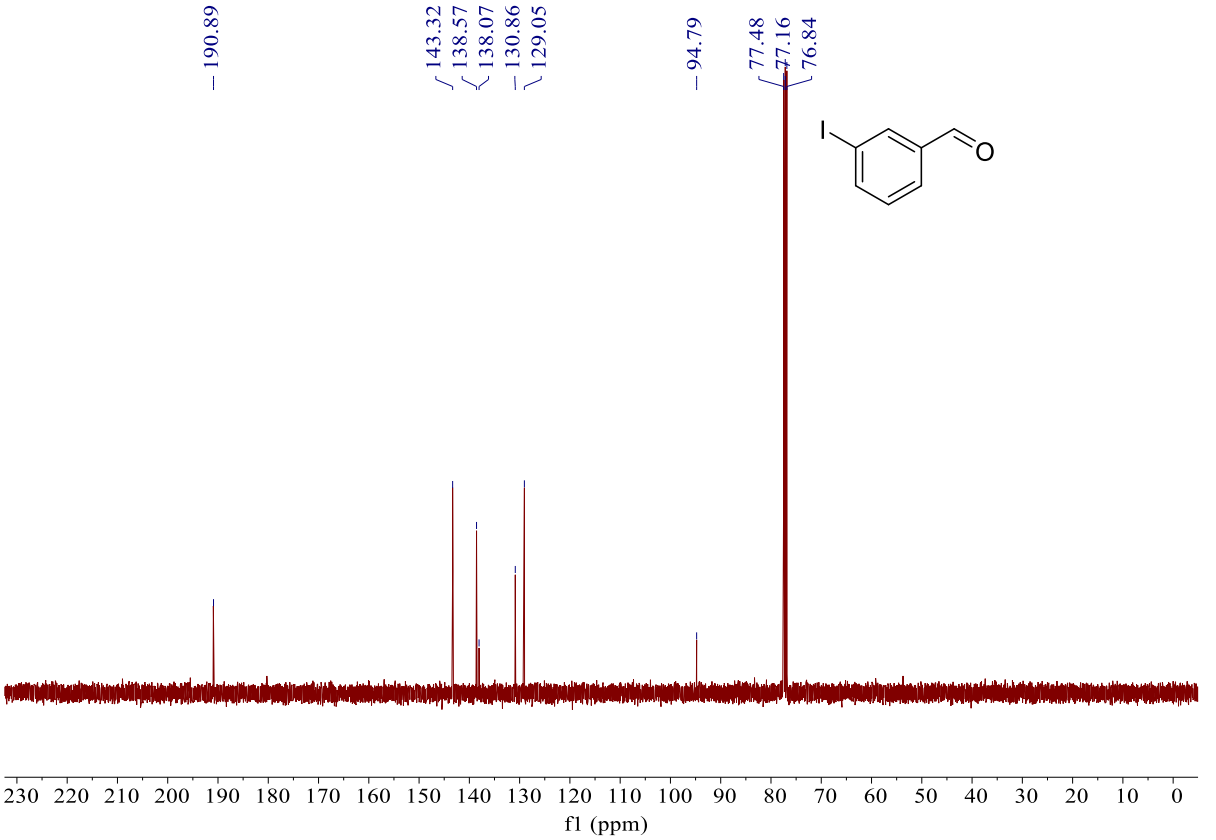

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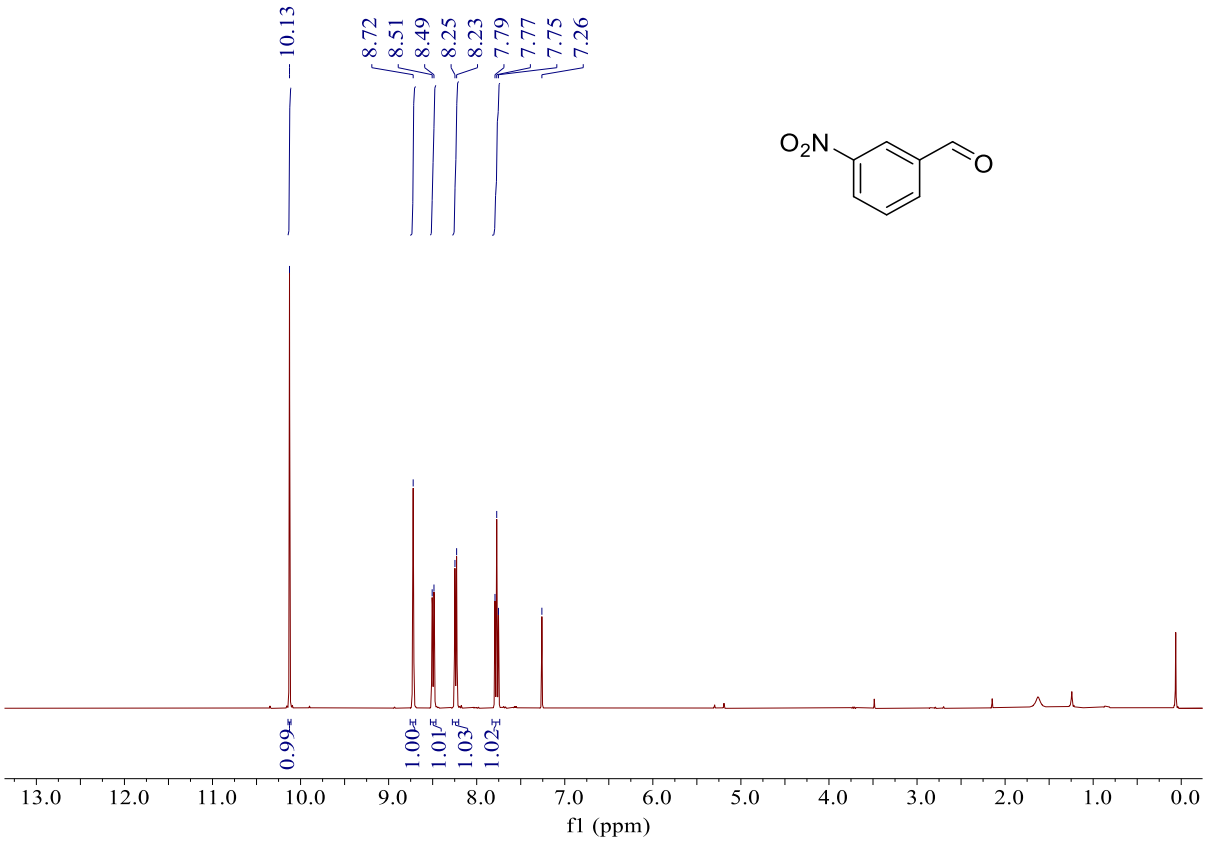

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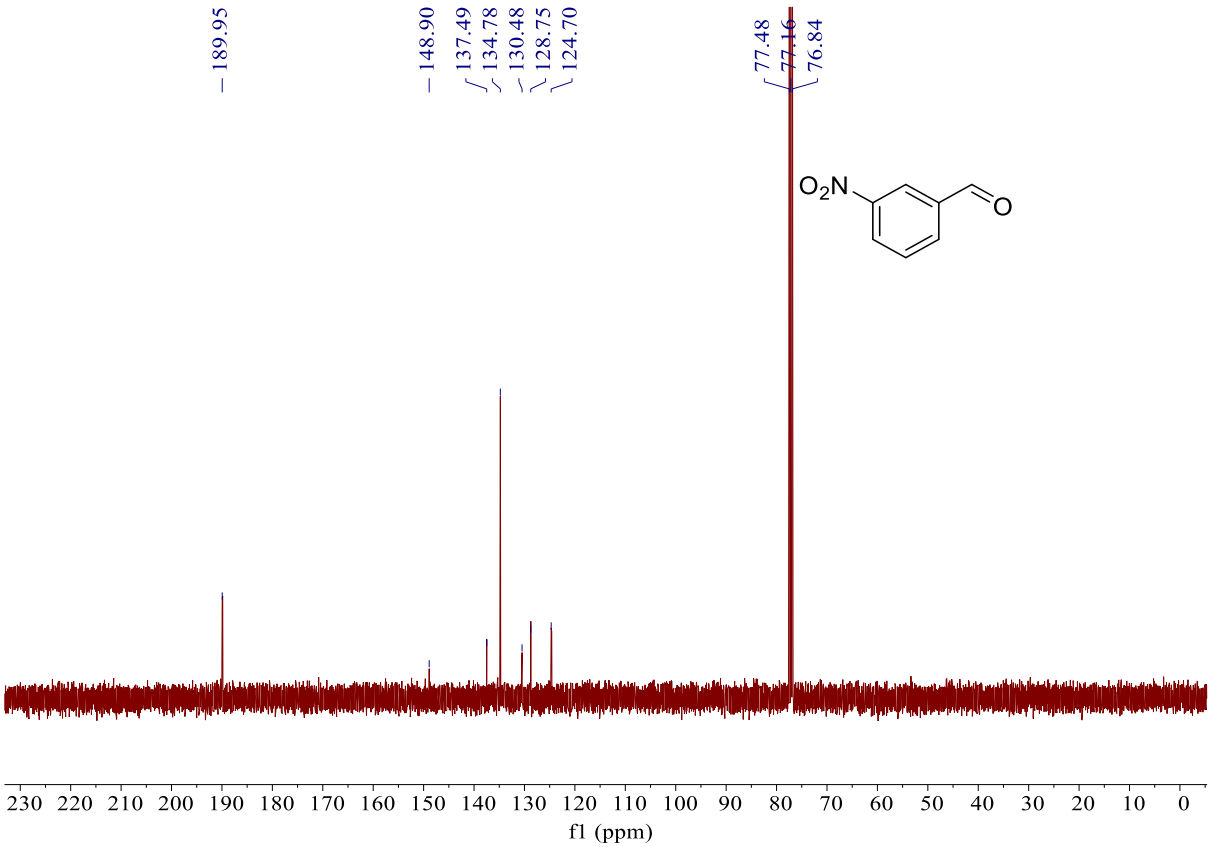
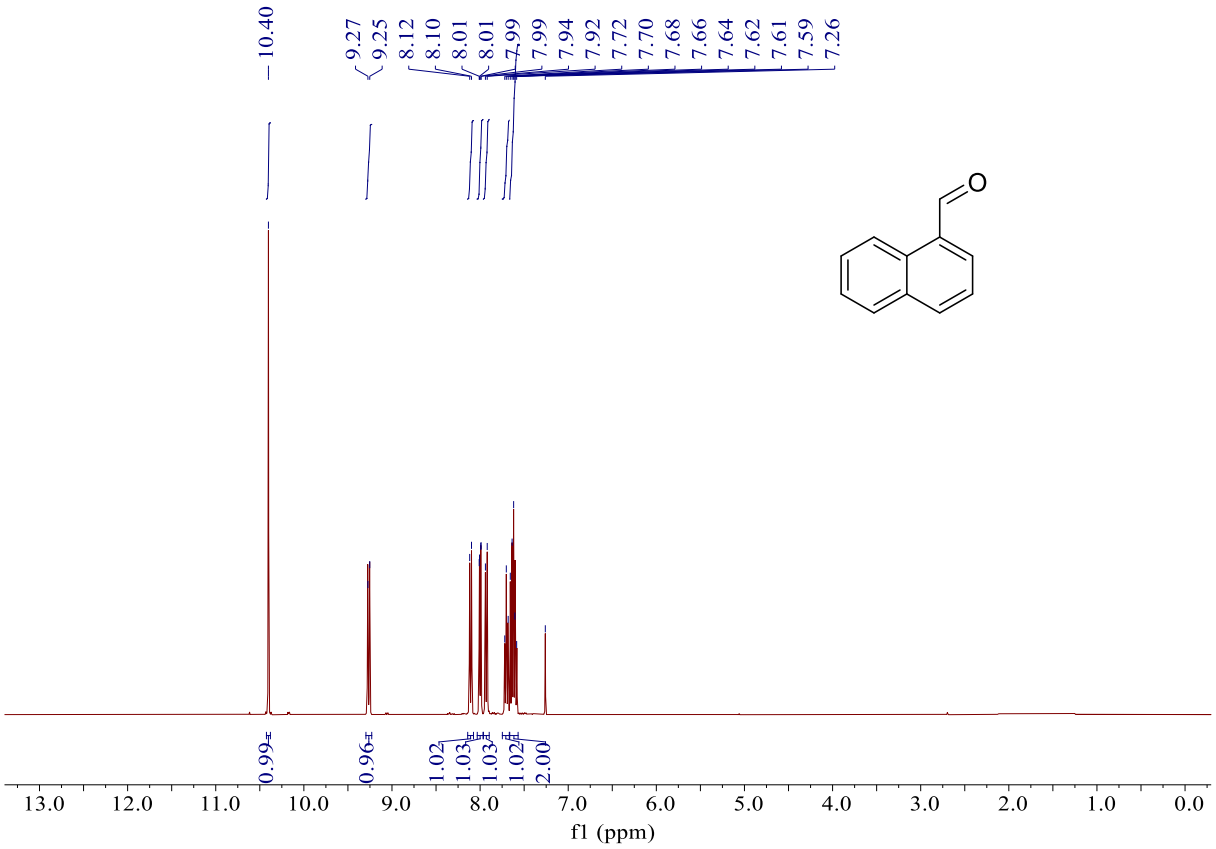


^{1}H NMR of compound 13k:



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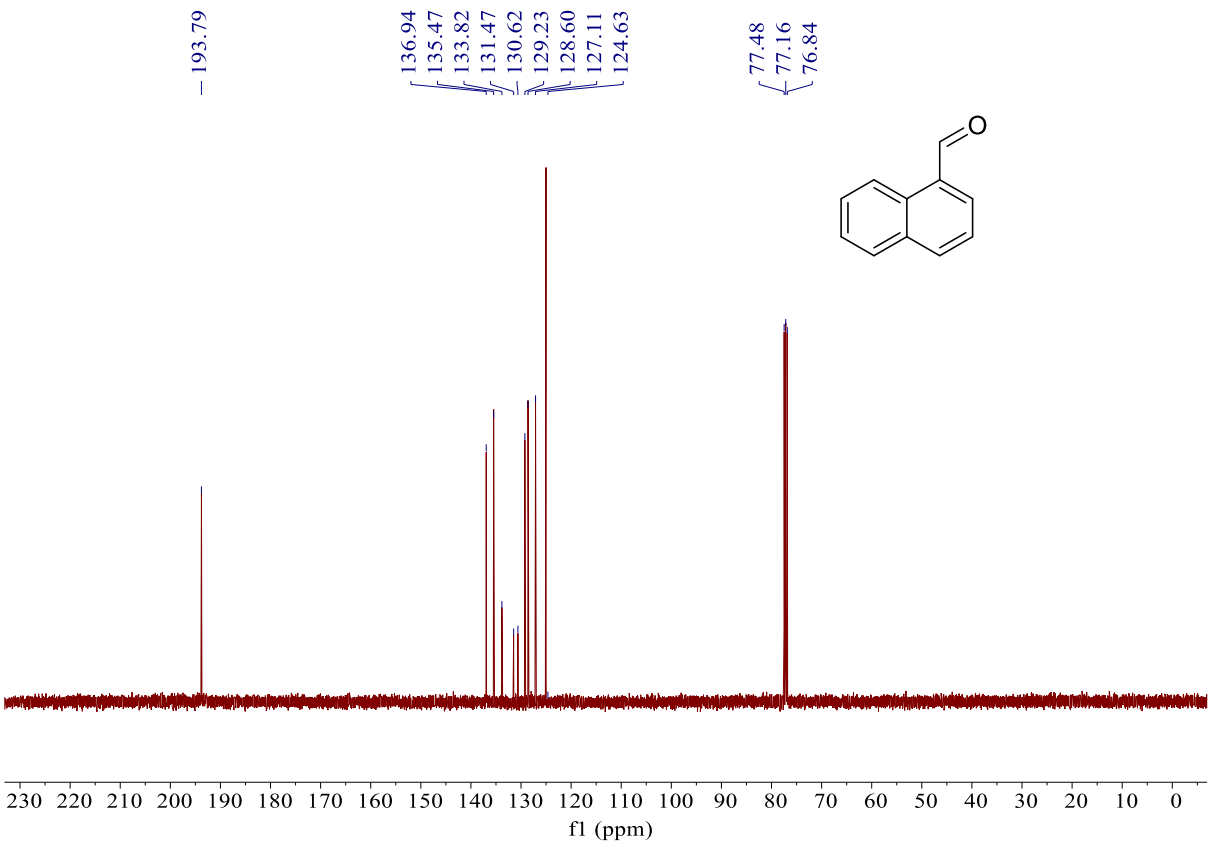
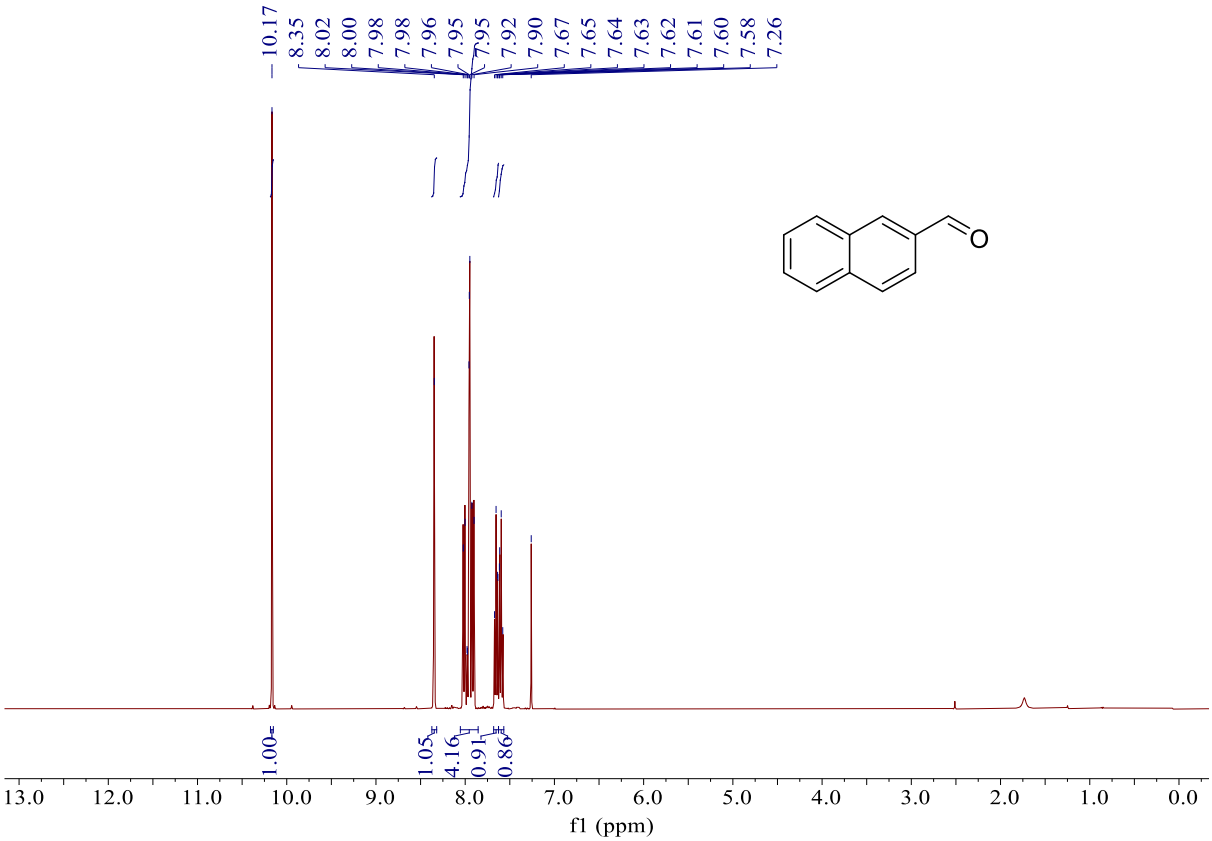


^{1}H NMR of compound 13l:



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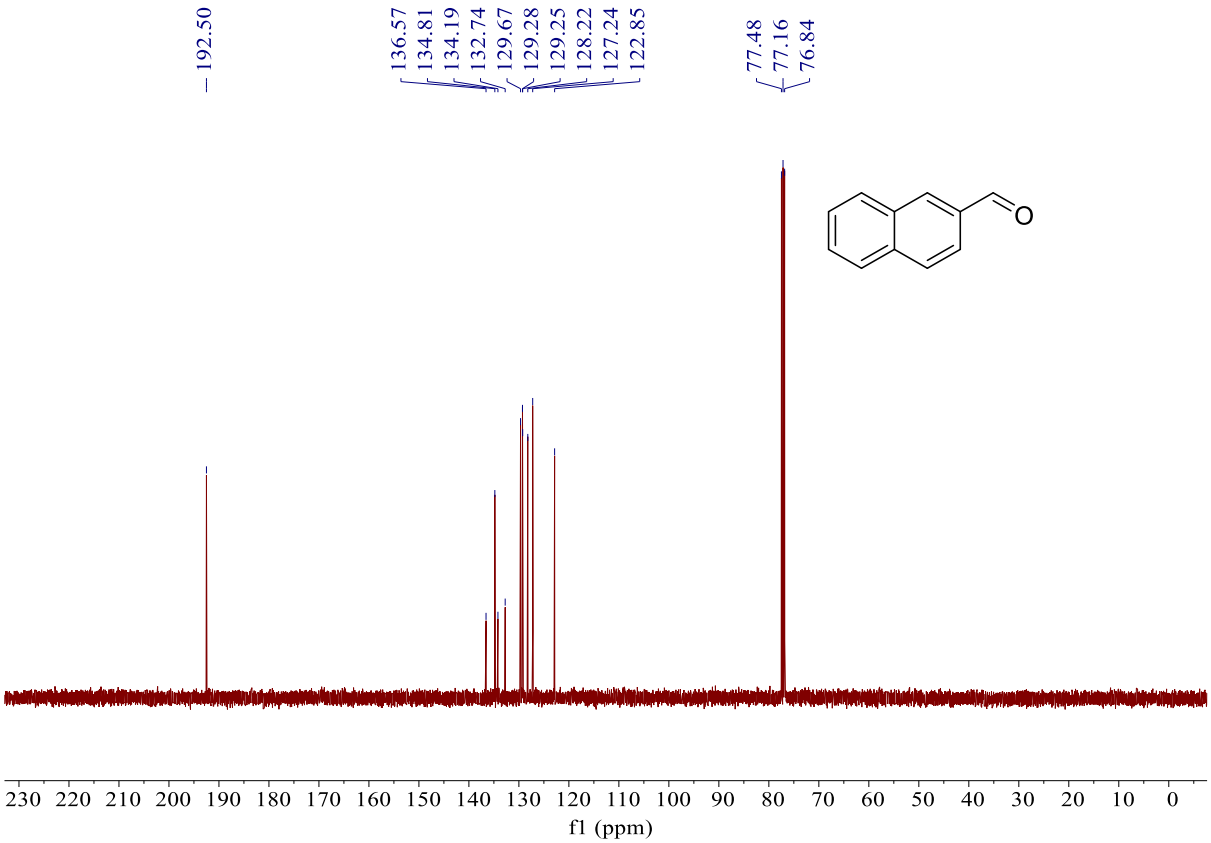

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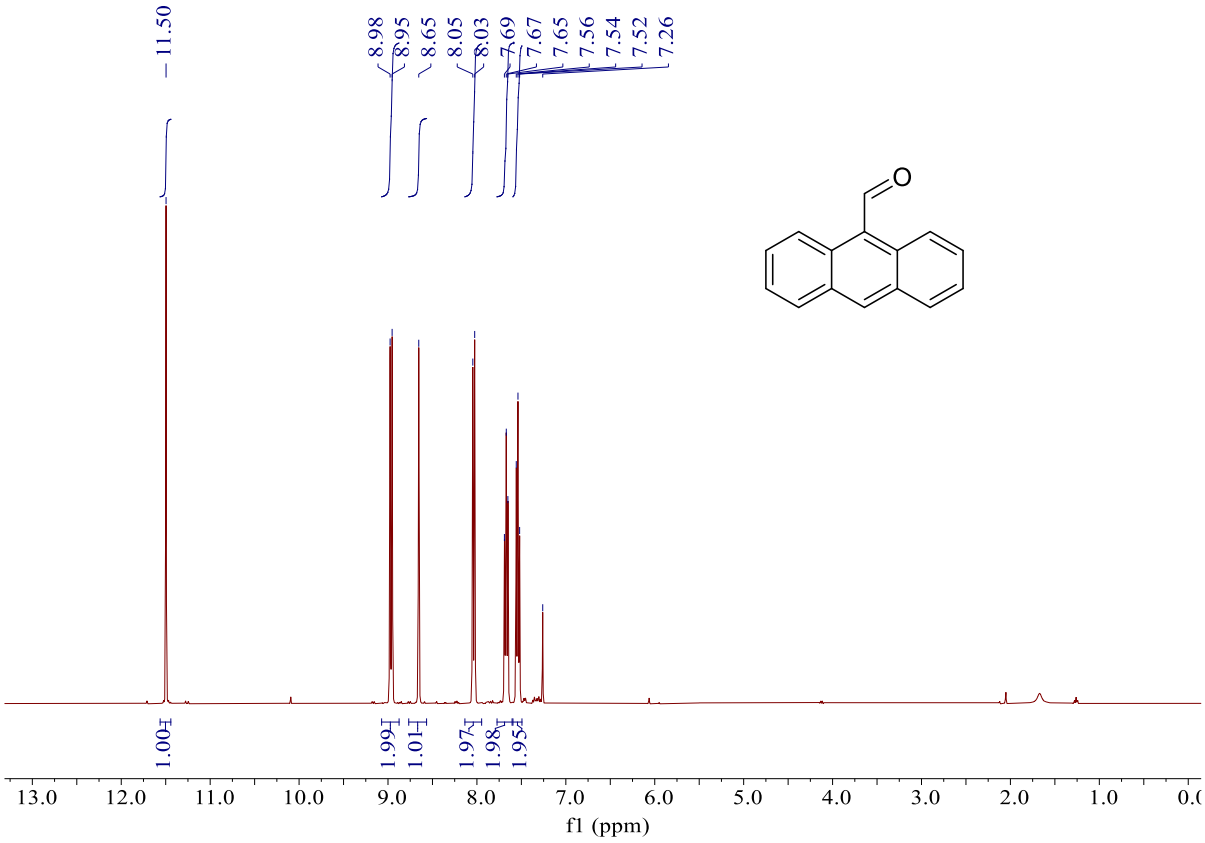

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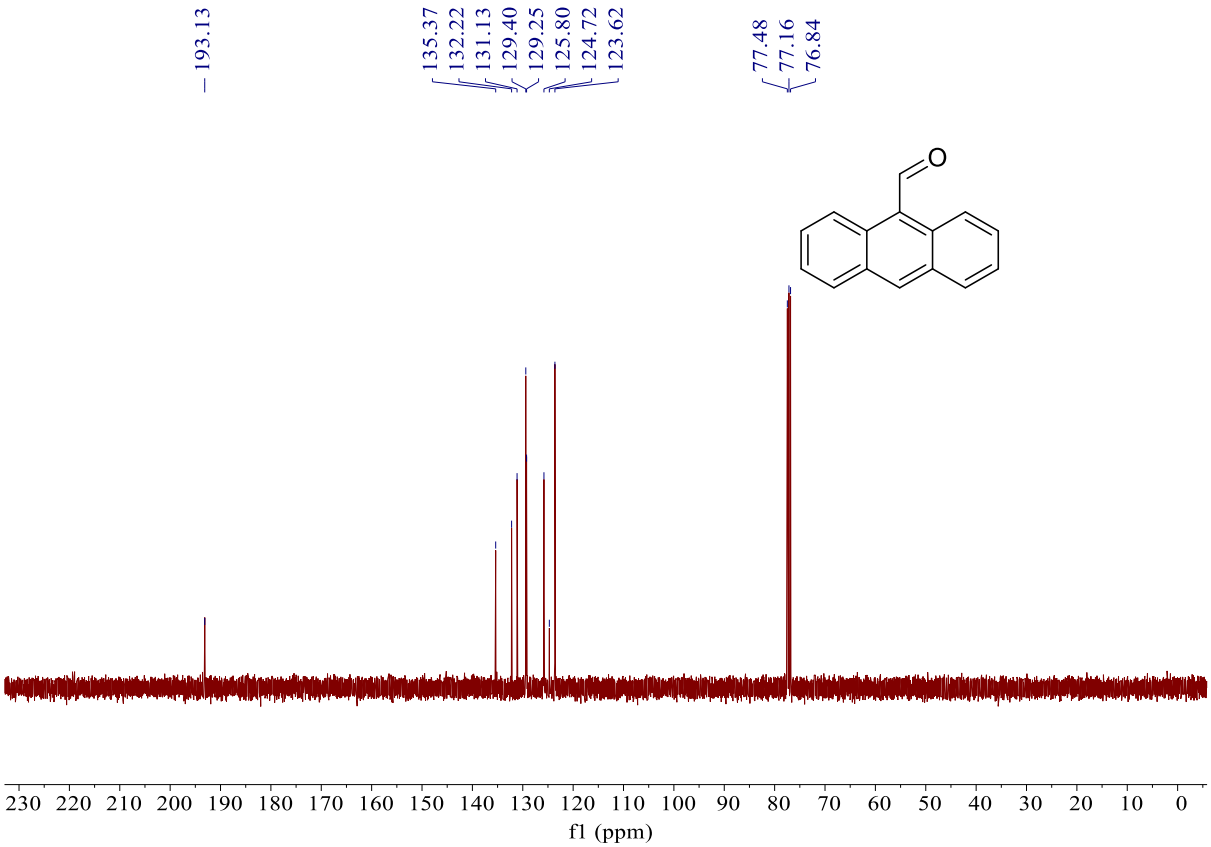
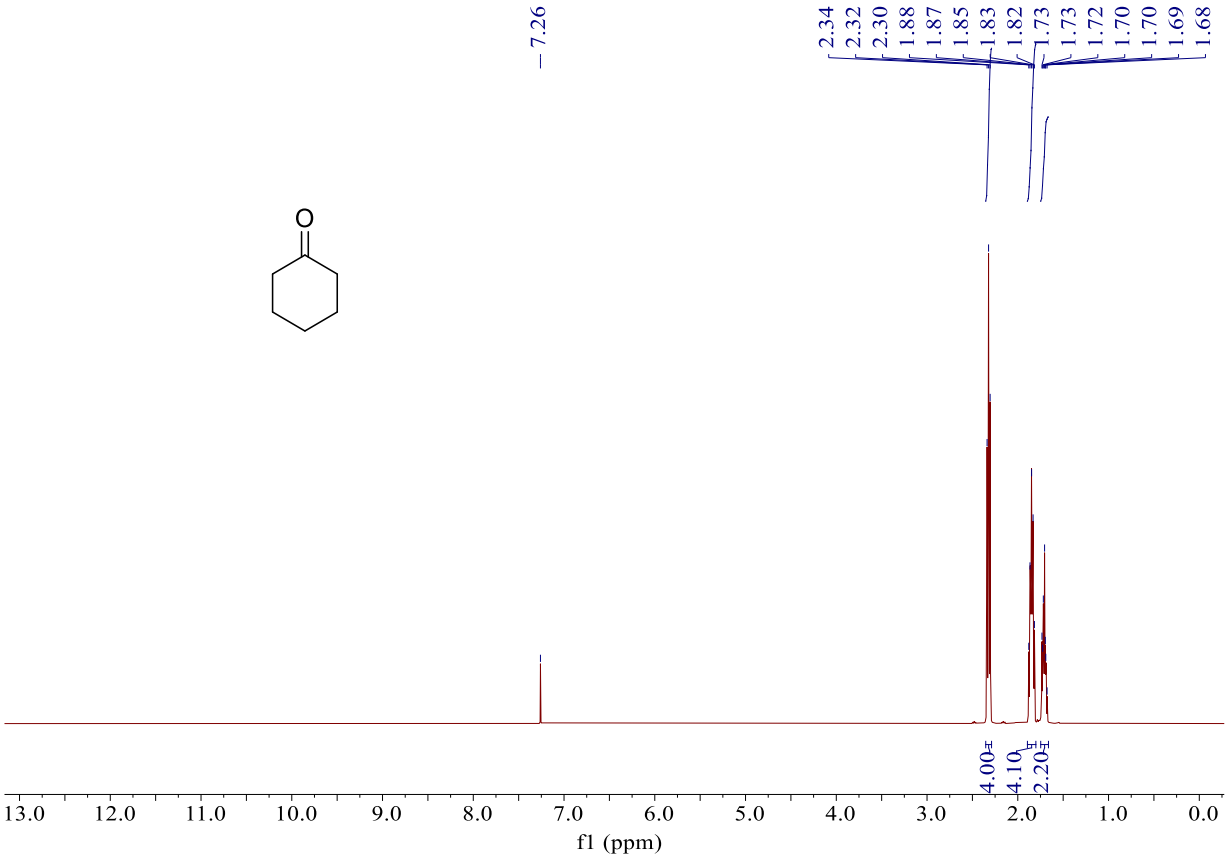
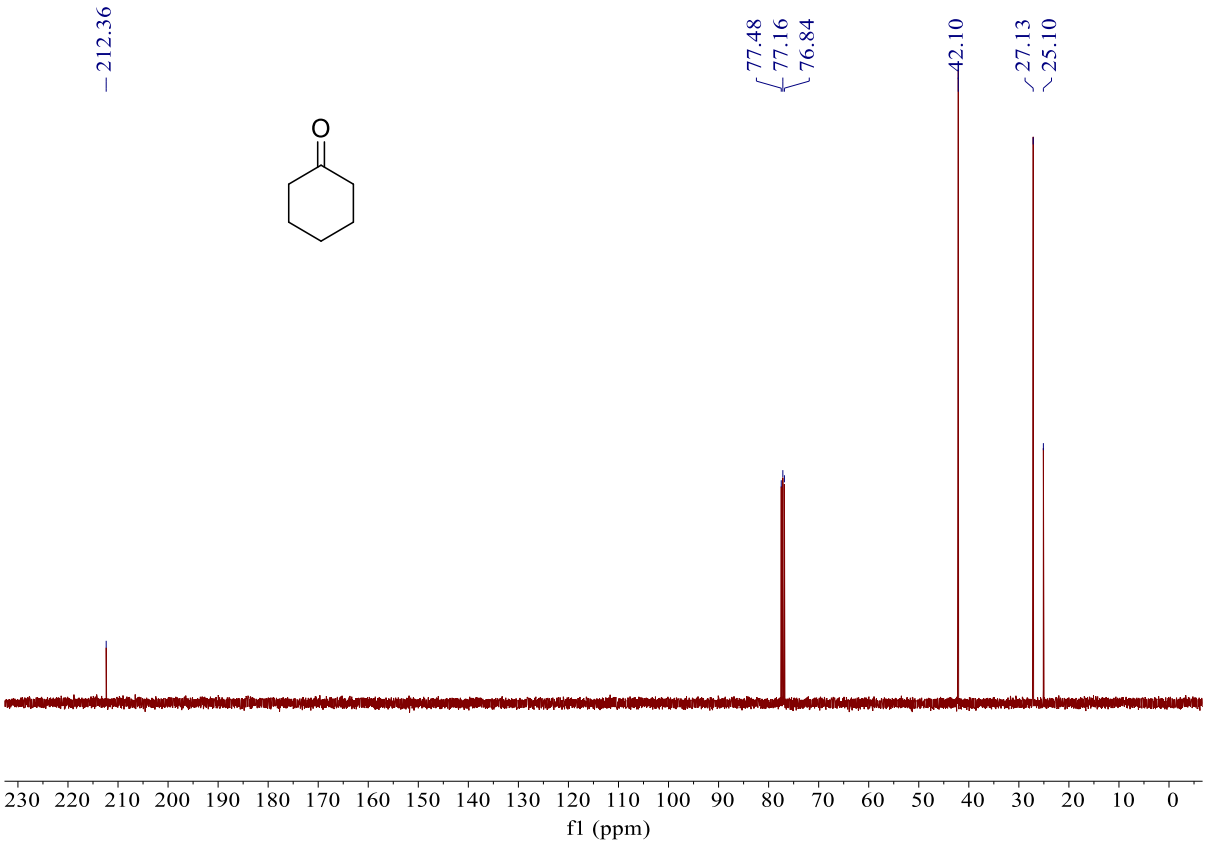


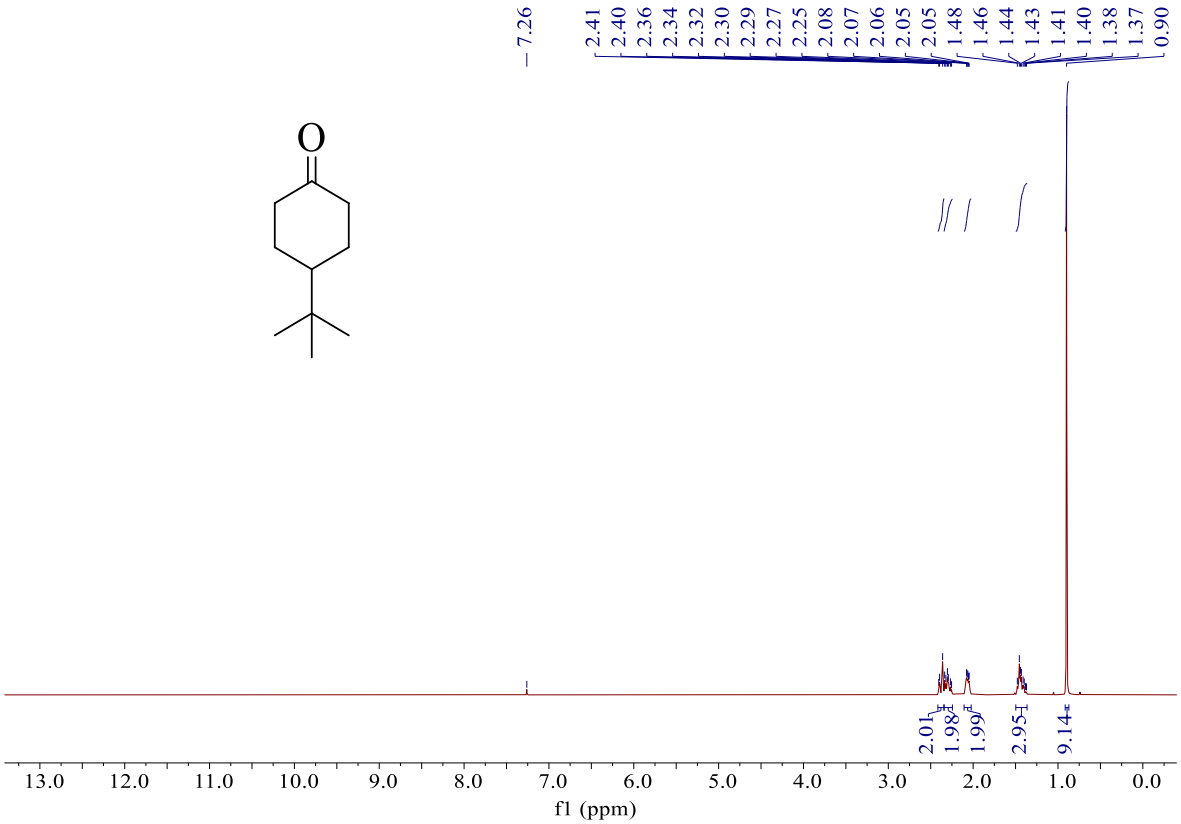
^{1}H NMR of compound 16a:



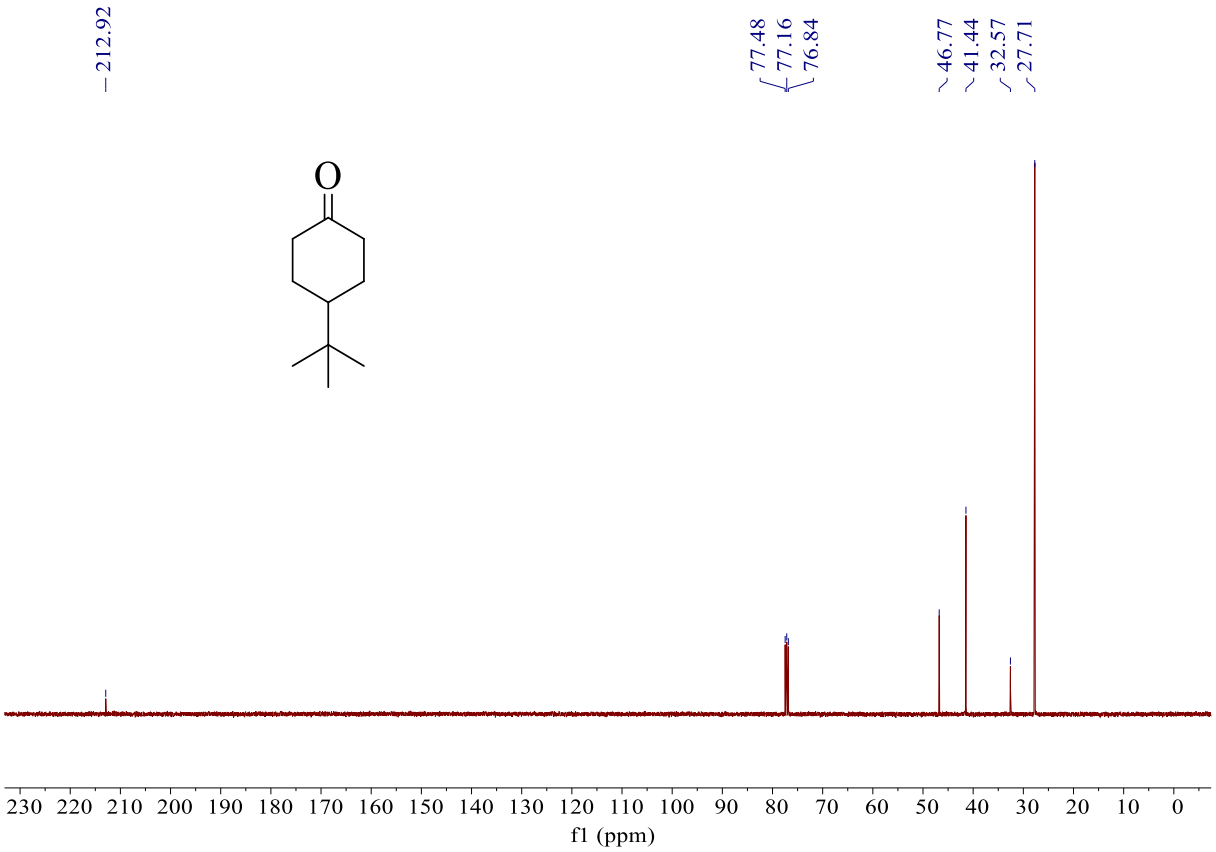
^{13}C NMR of compound 16a:



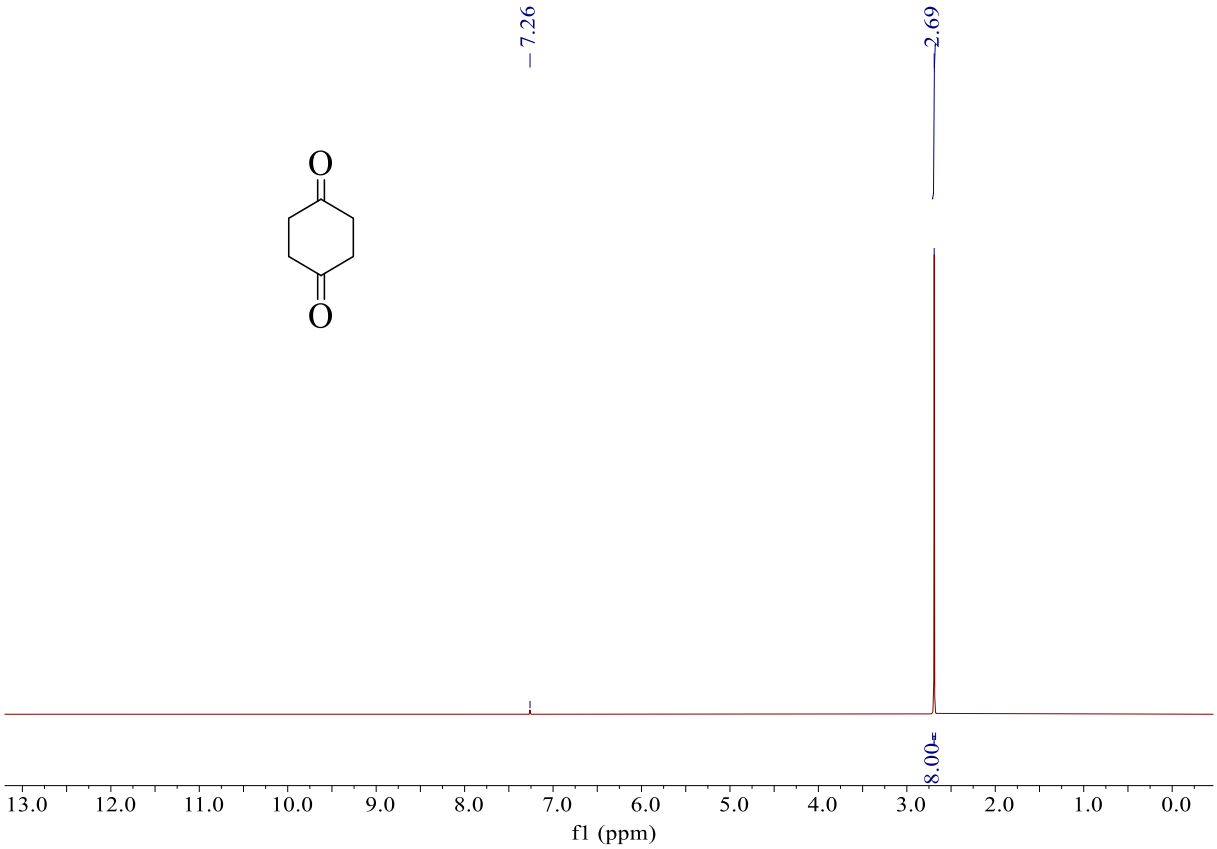
^{1}H NMR of compound 16b:



^{13}C NMR of compound 16b:



^{1}H NMR of compound 16c:



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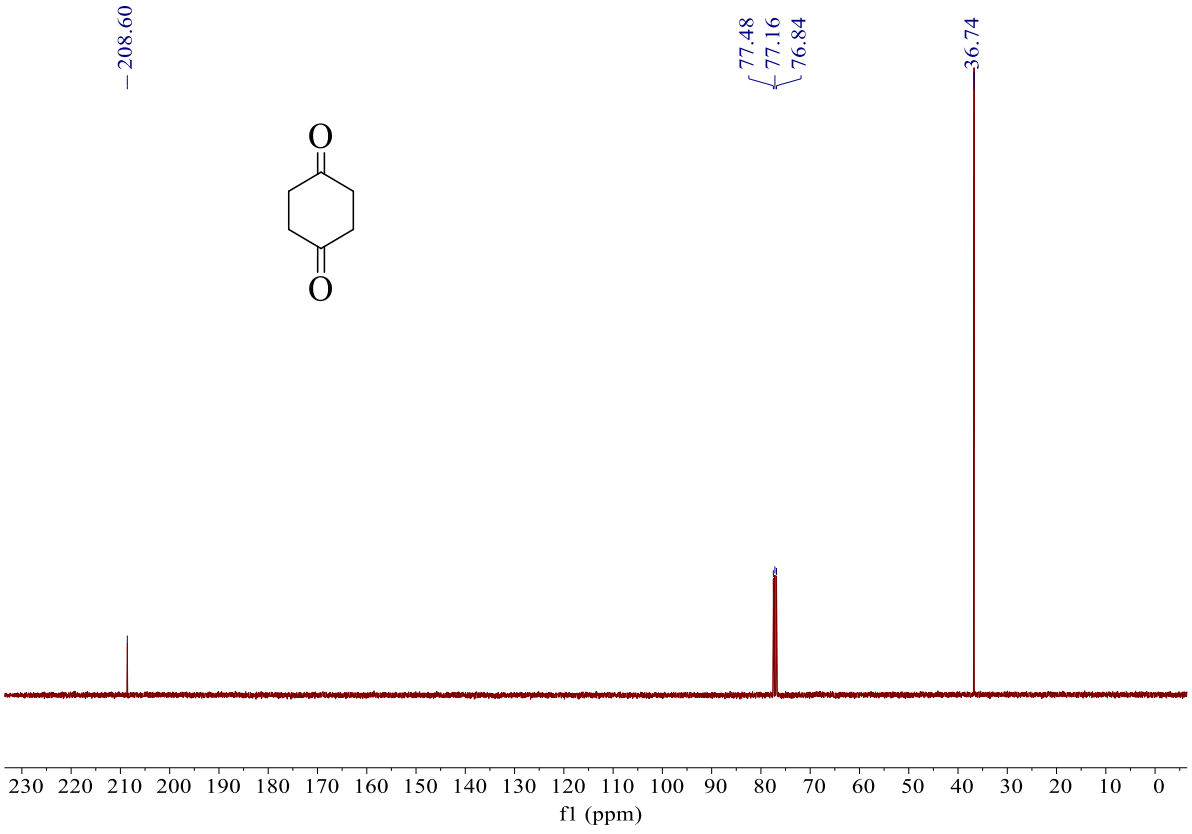
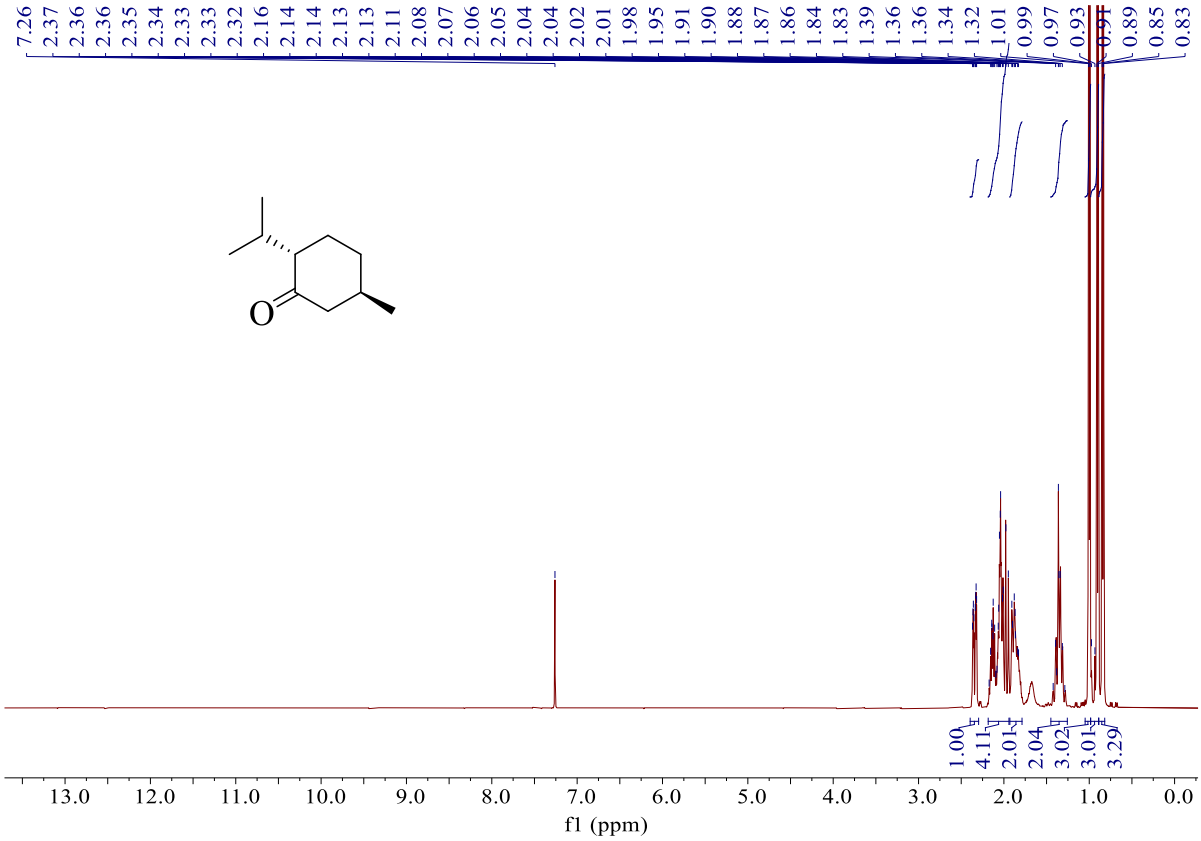
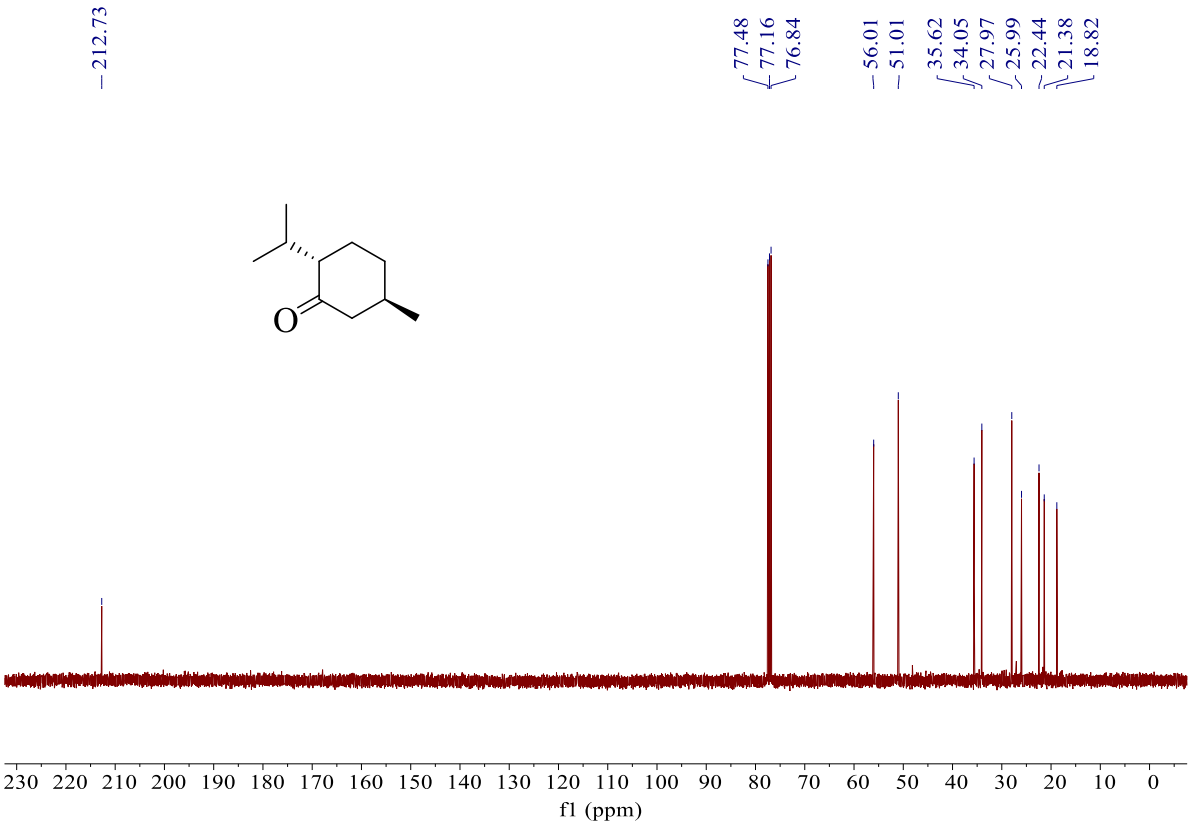


^{1}H NMR of compound 16d:



^{13}C NMR of compound 16d:



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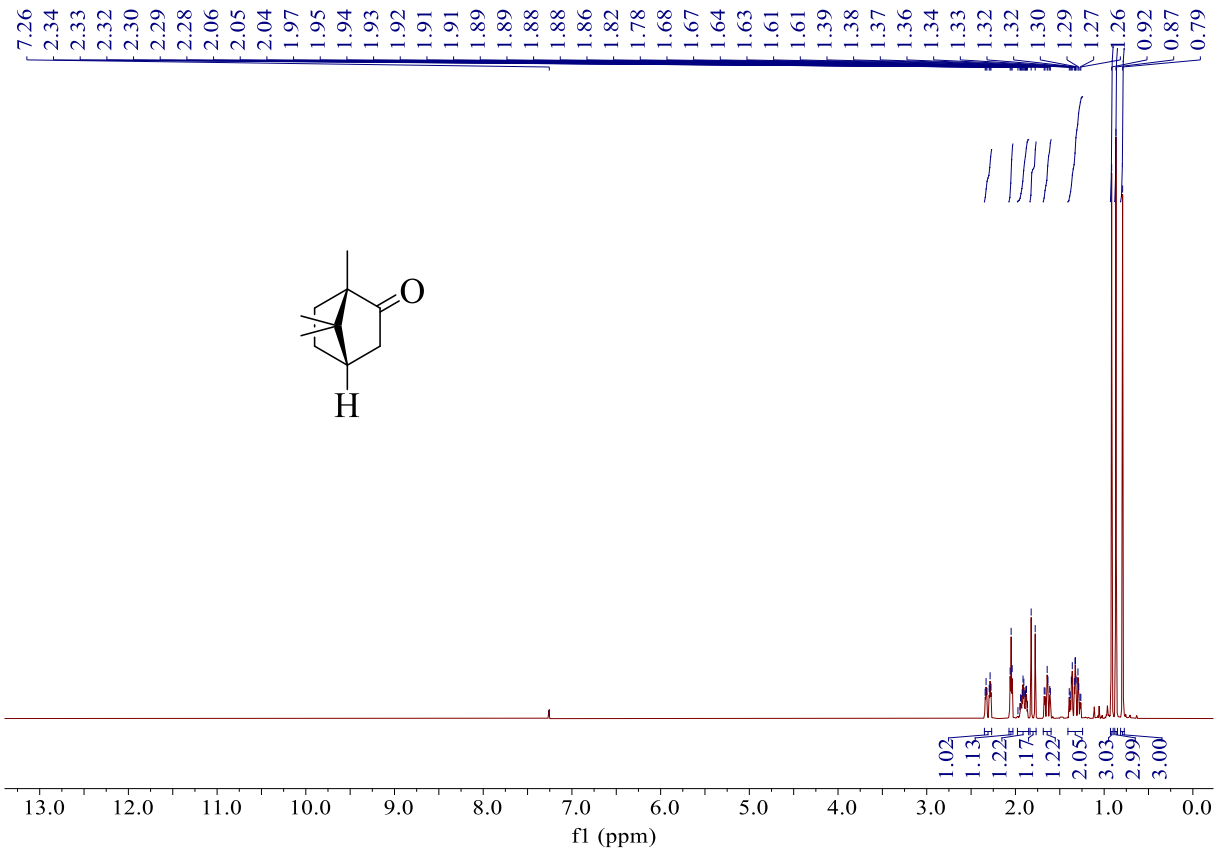

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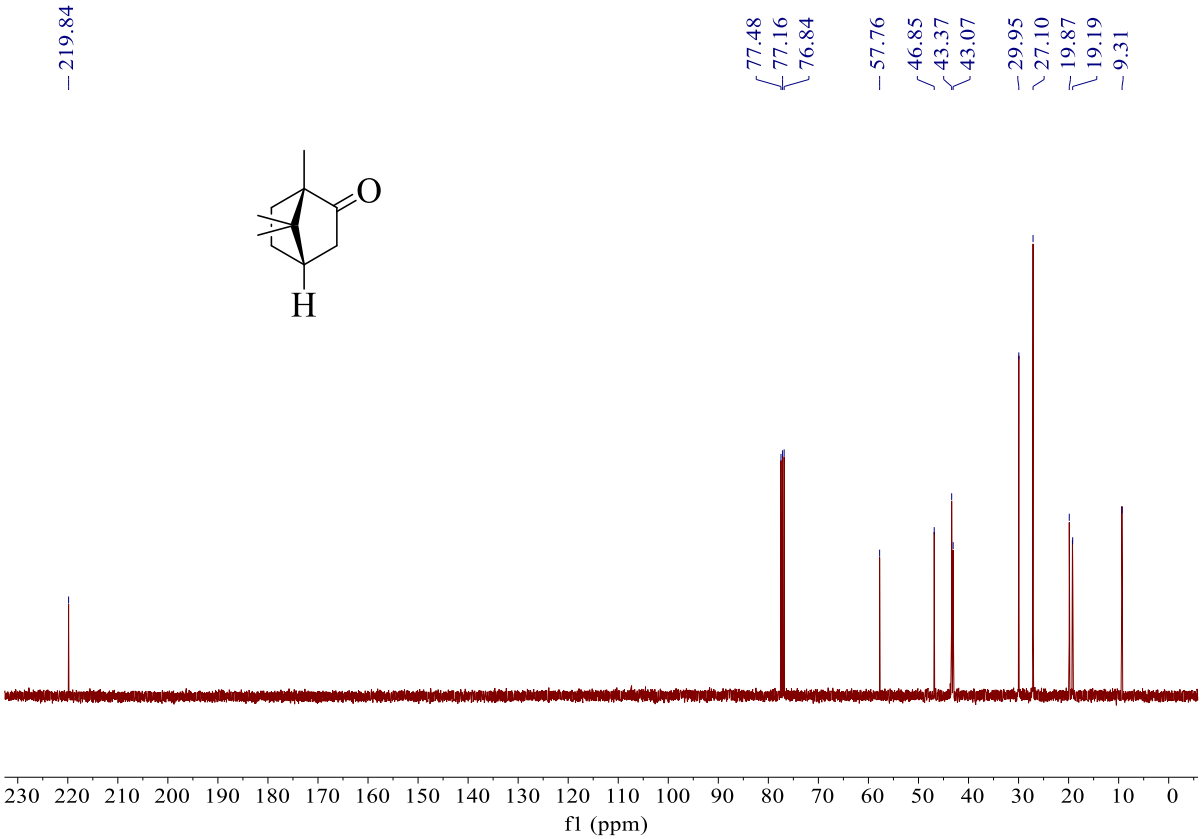
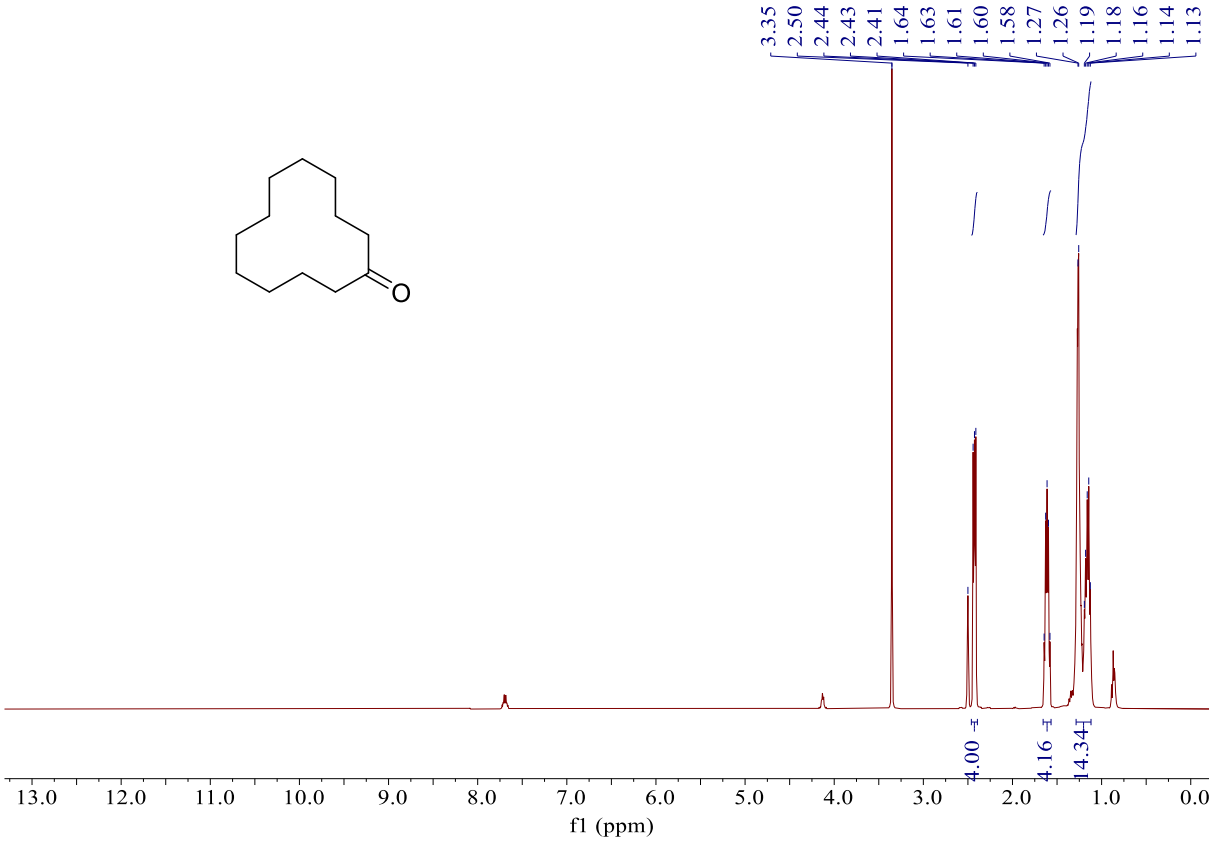
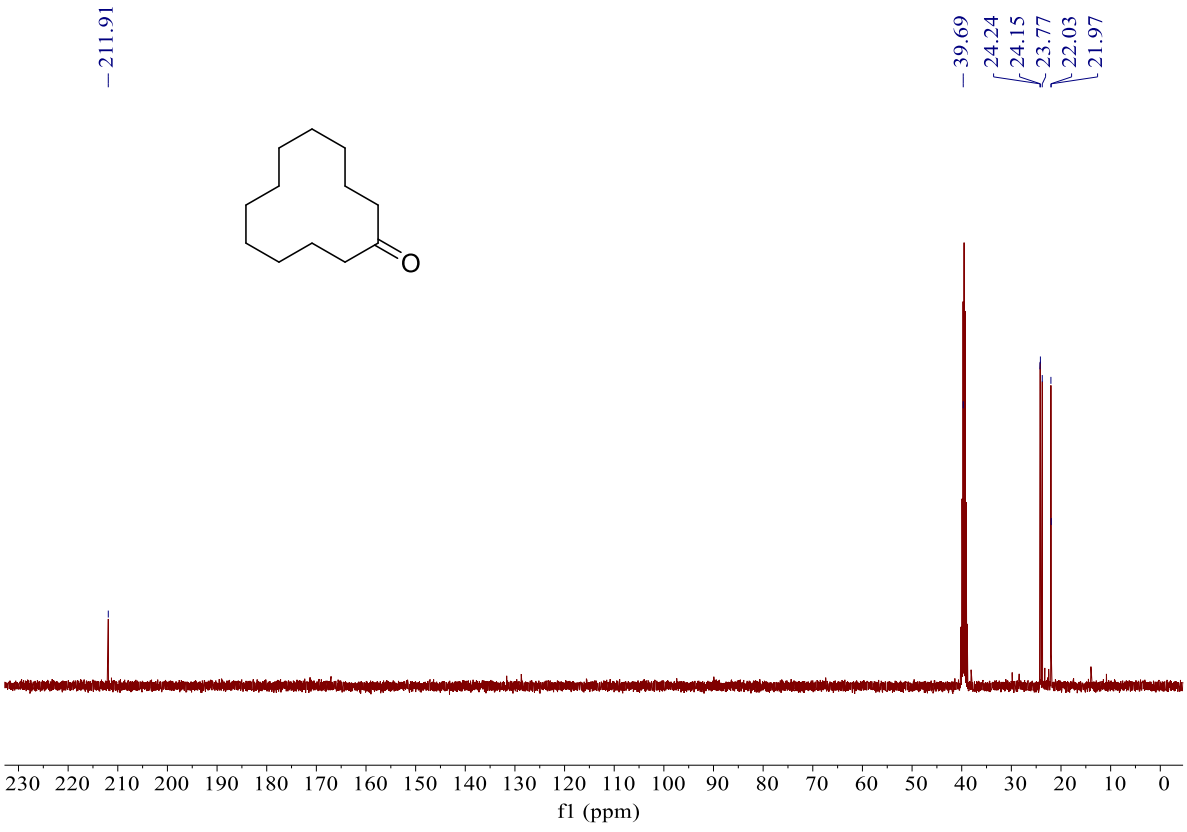


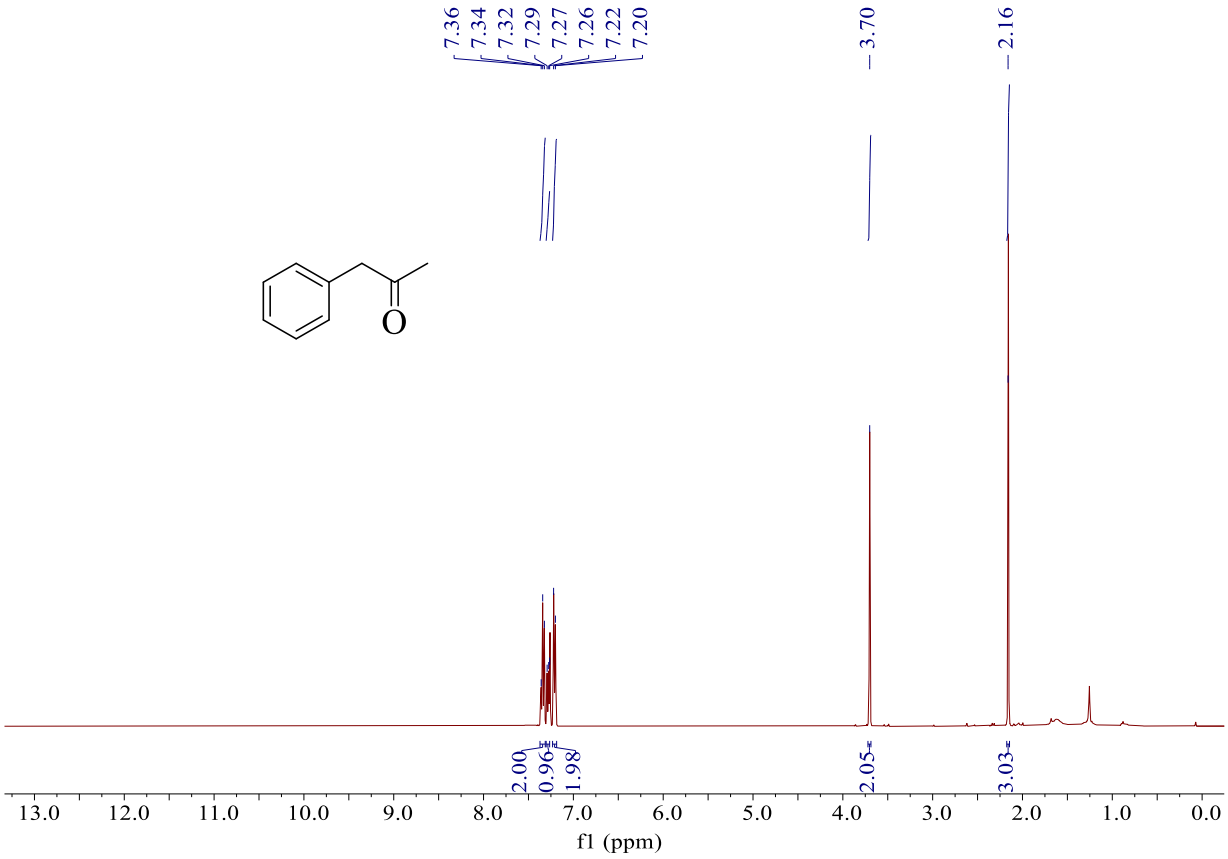
^{1}H NMR of compound 16f:



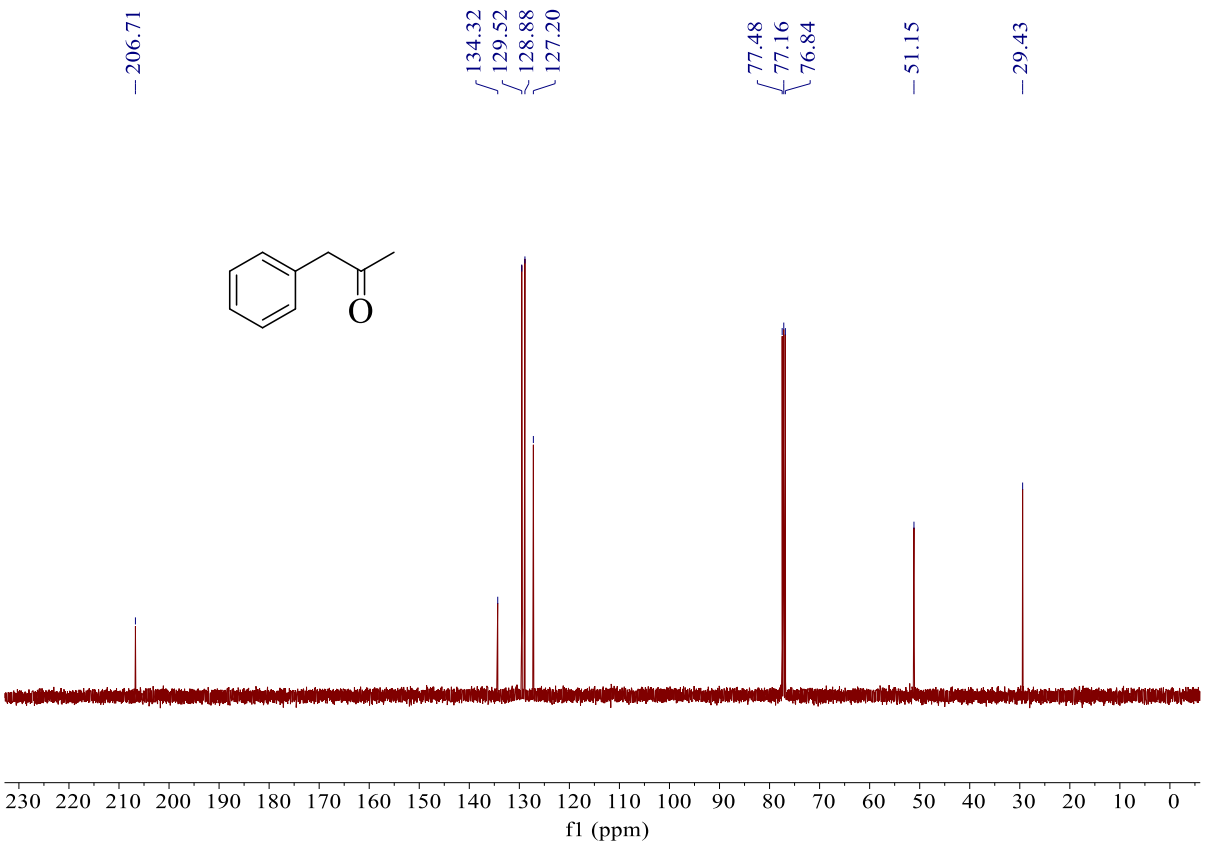
^{13}C NMR of compound 16f



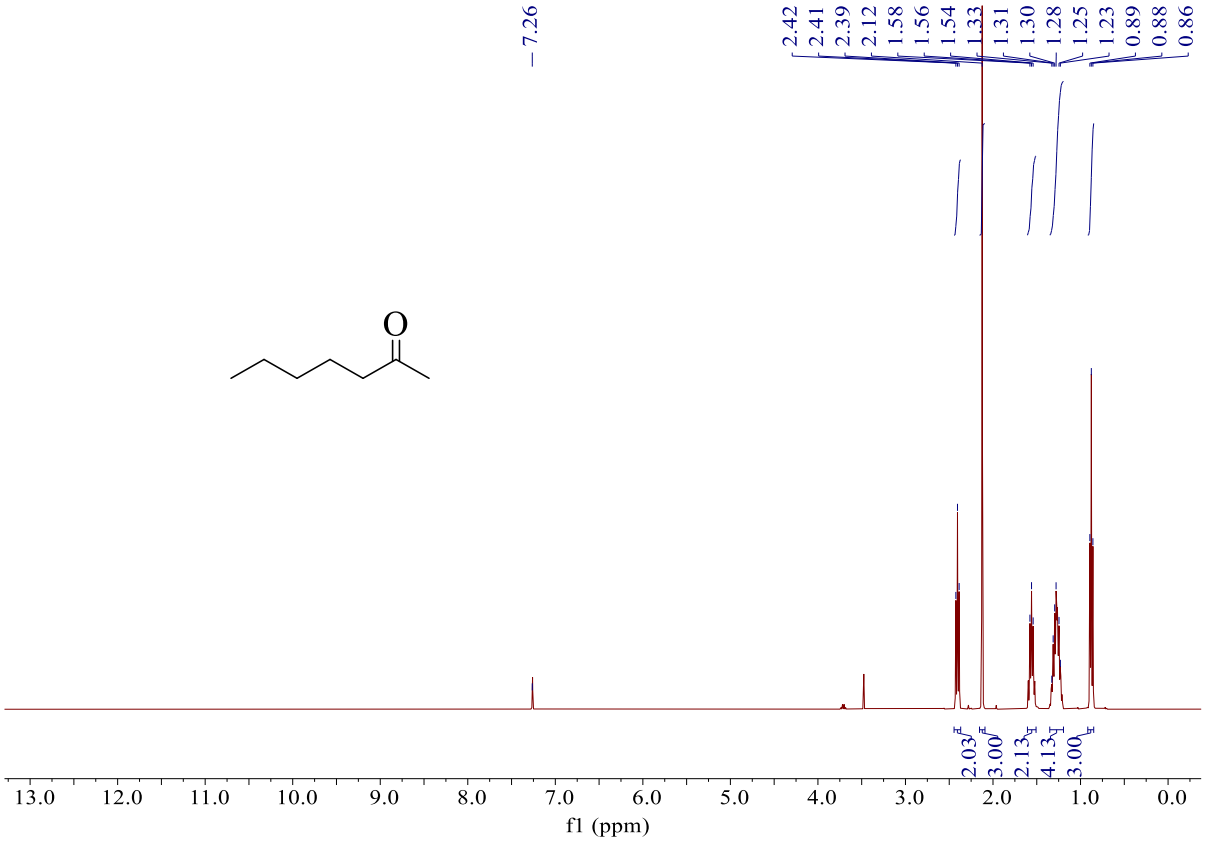
^{1}H NMR of compound 16g:



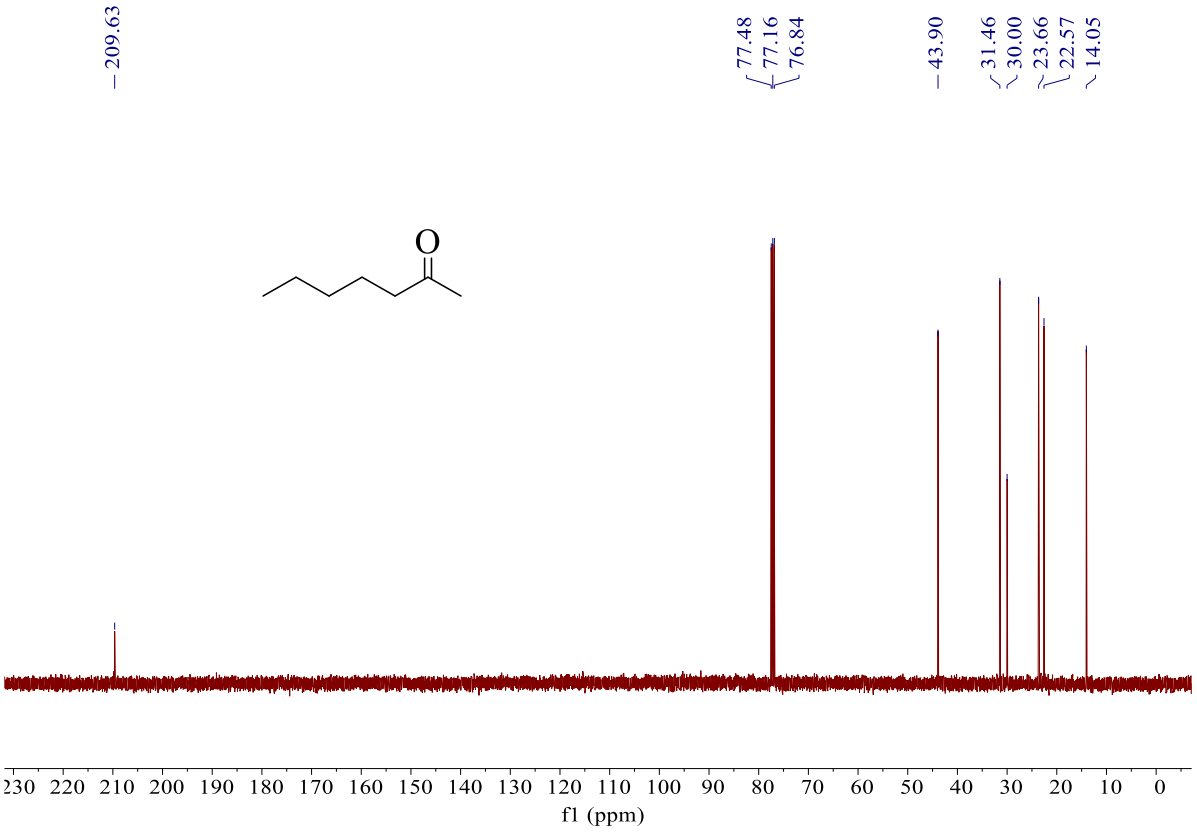
^{13}C NMR of compound 16g:



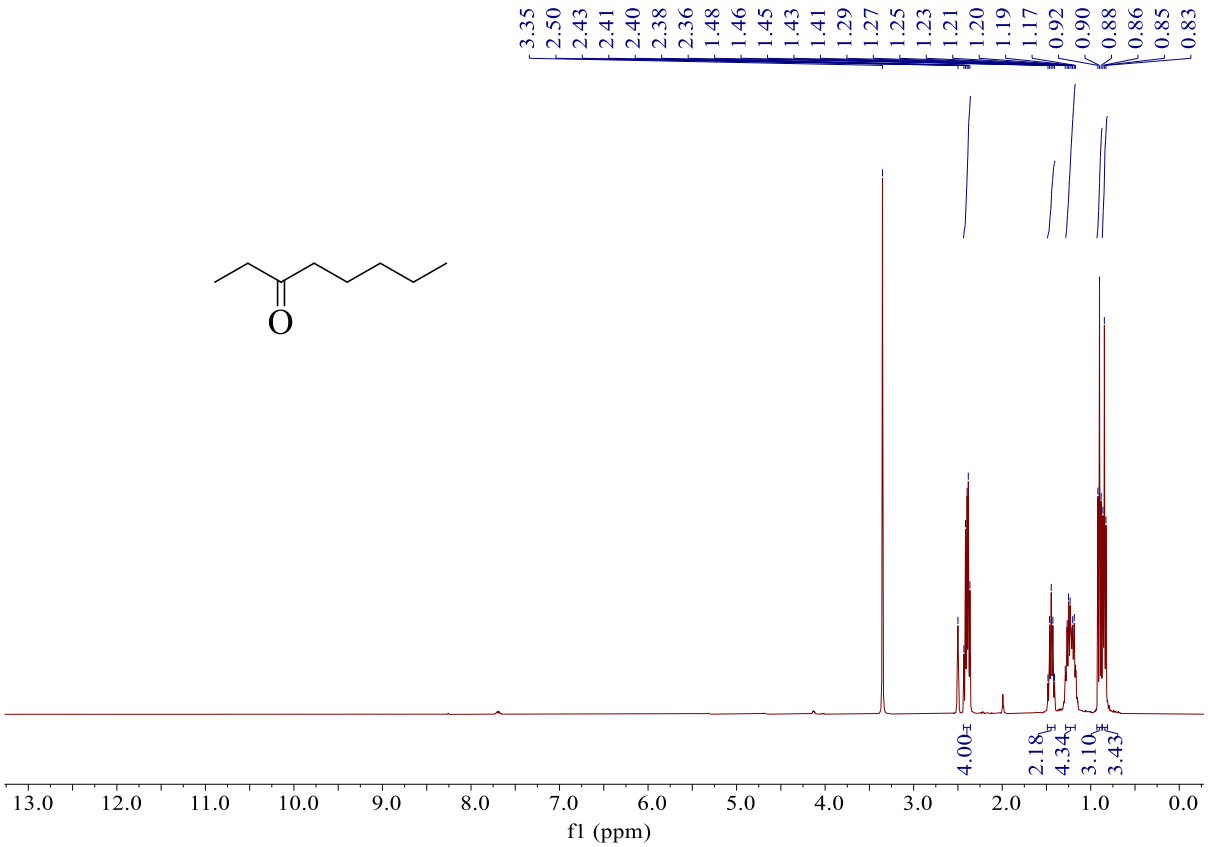
^{1}H NMR of compound 16h:



^{13}C NMR of compound 16h:



^{1}H NMR of compound 16i:



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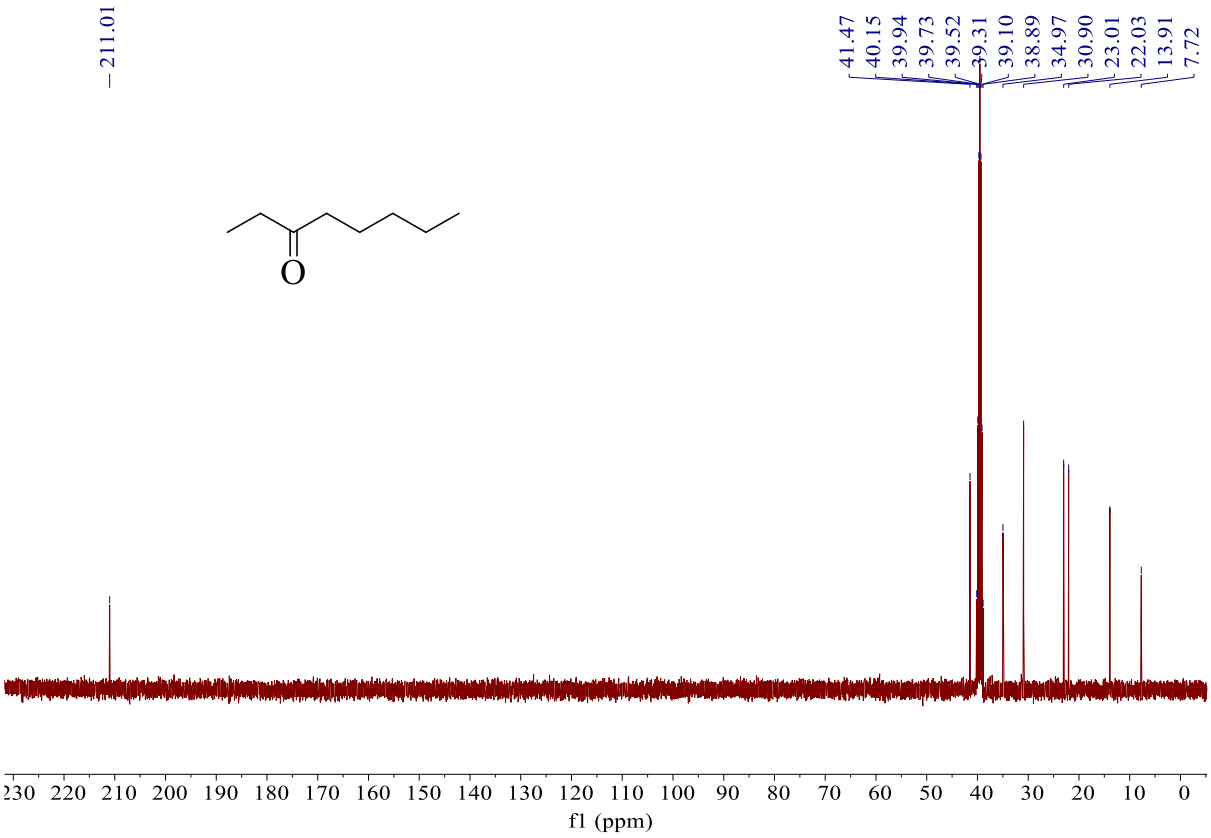

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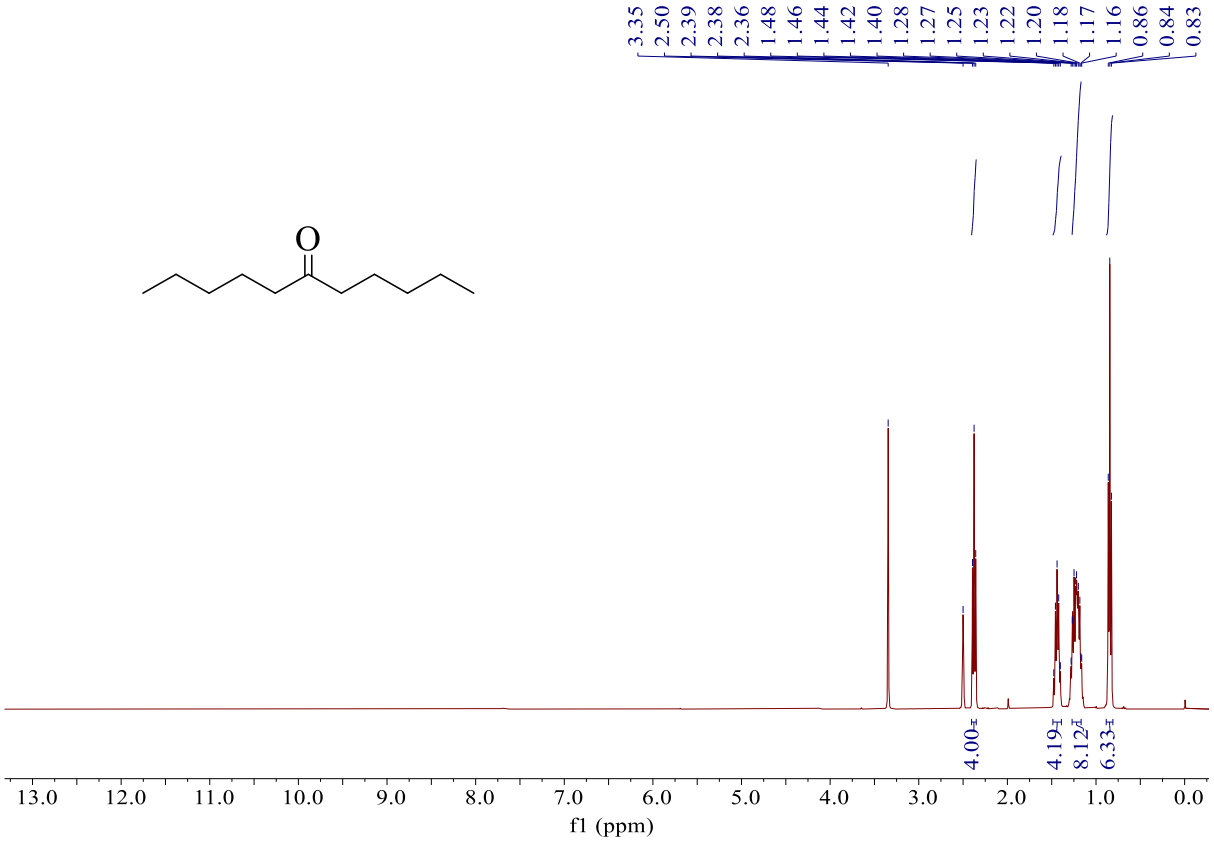

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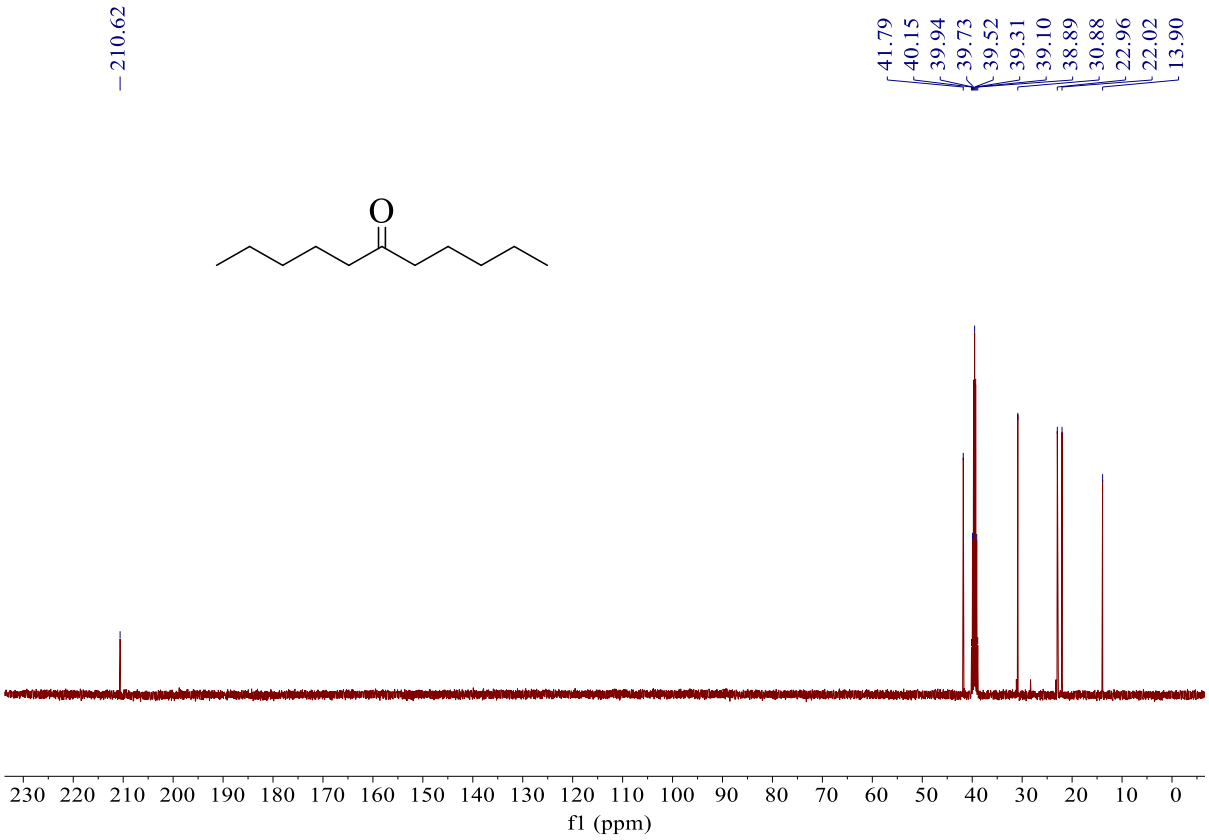
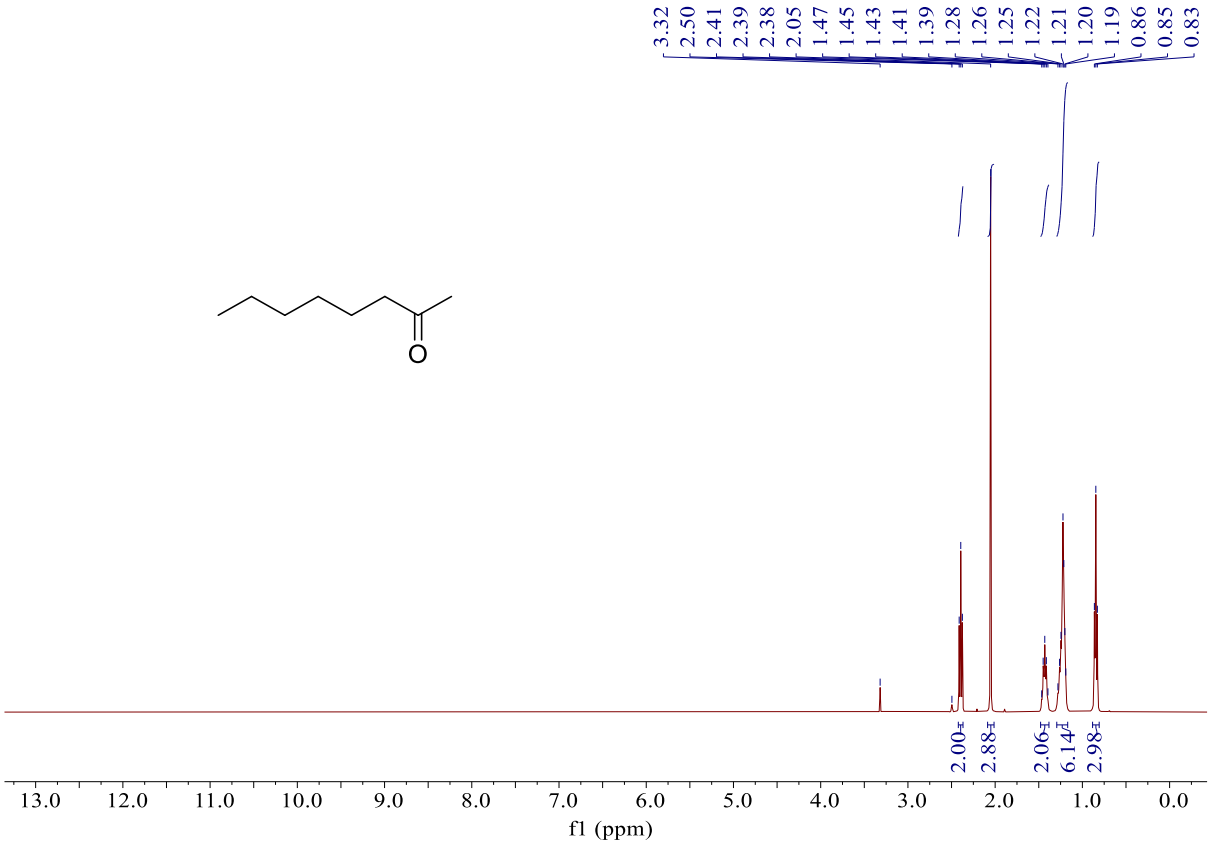
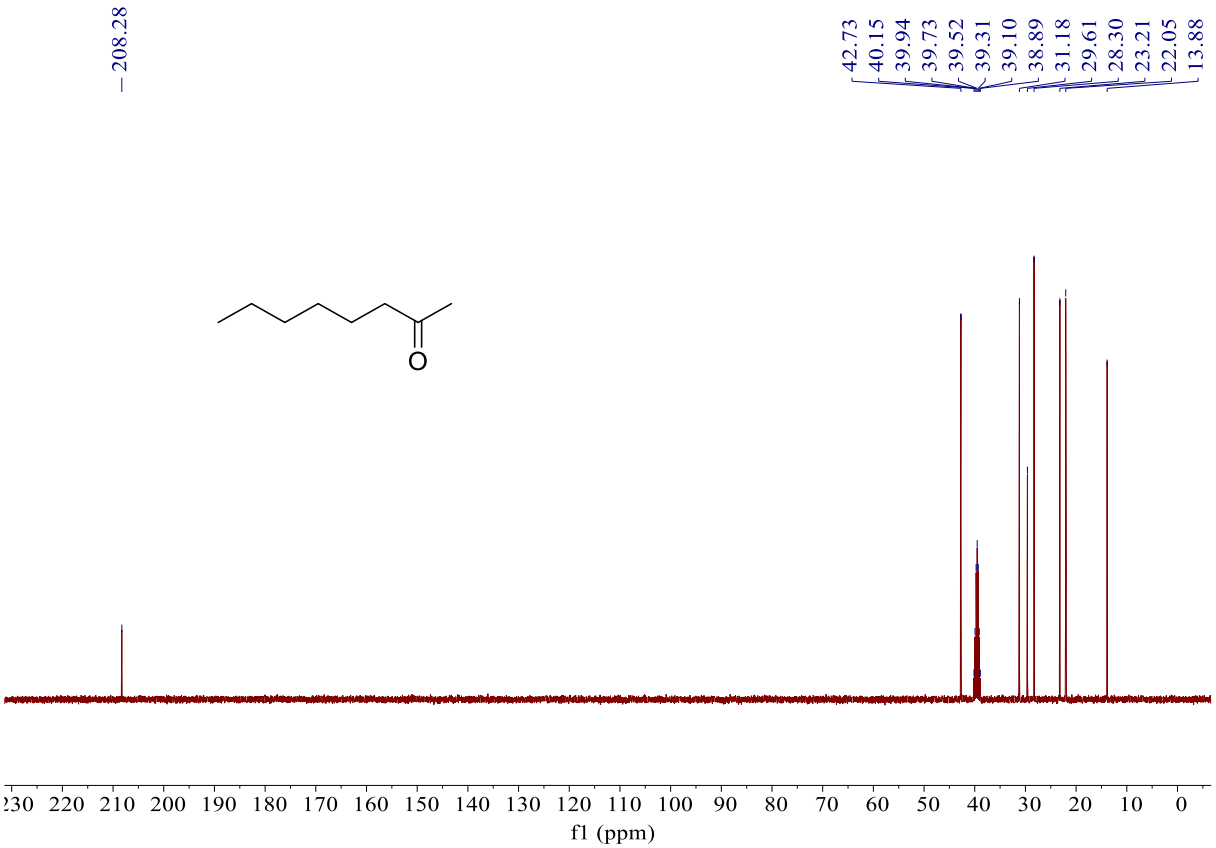


^{1}H NMR of compound 16k:



^{13}C NMR of compound 16k:



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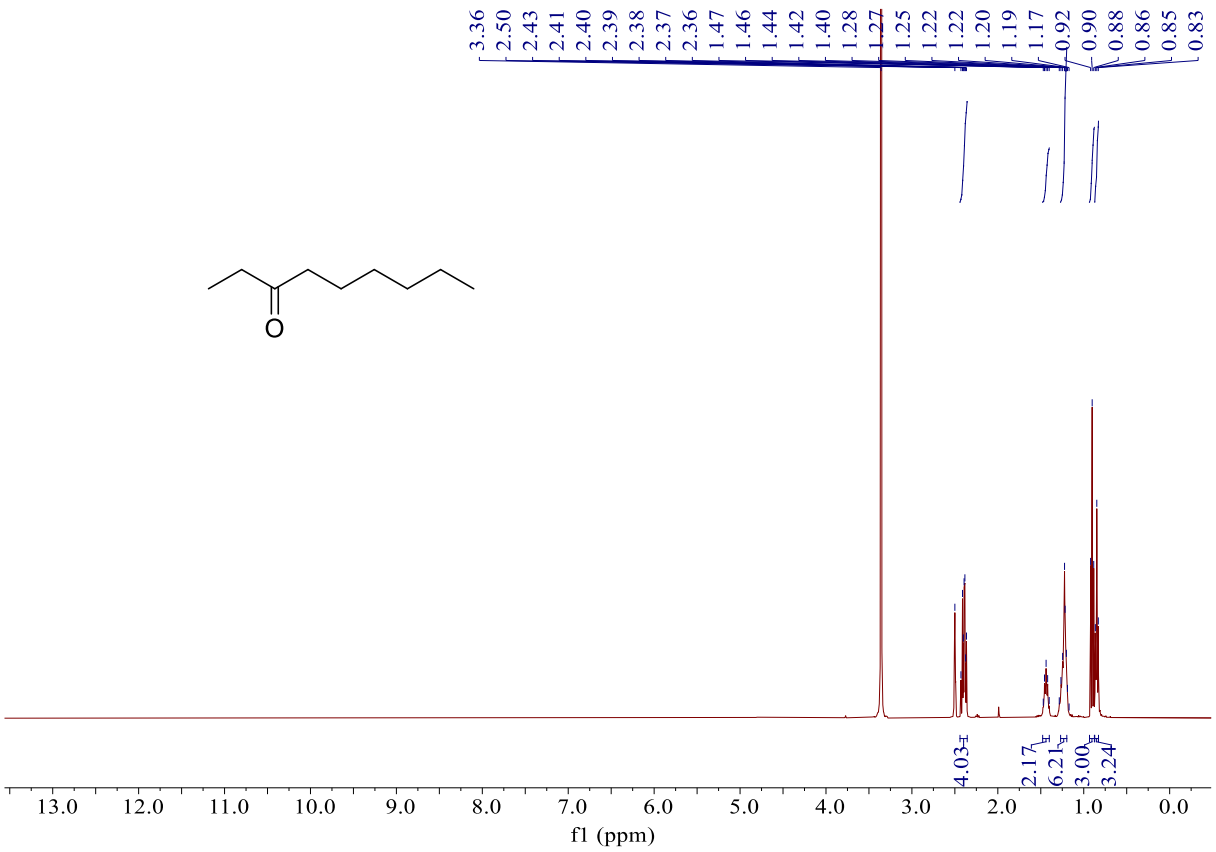
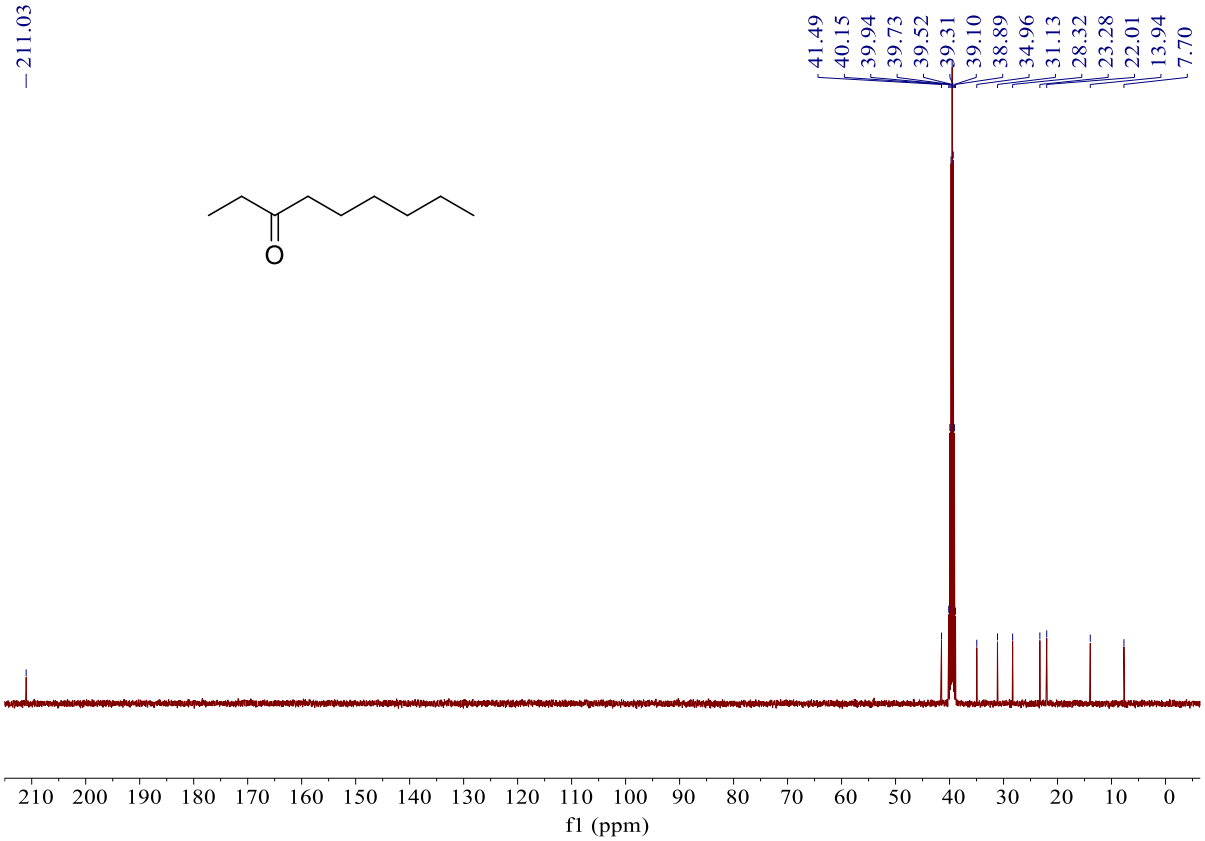
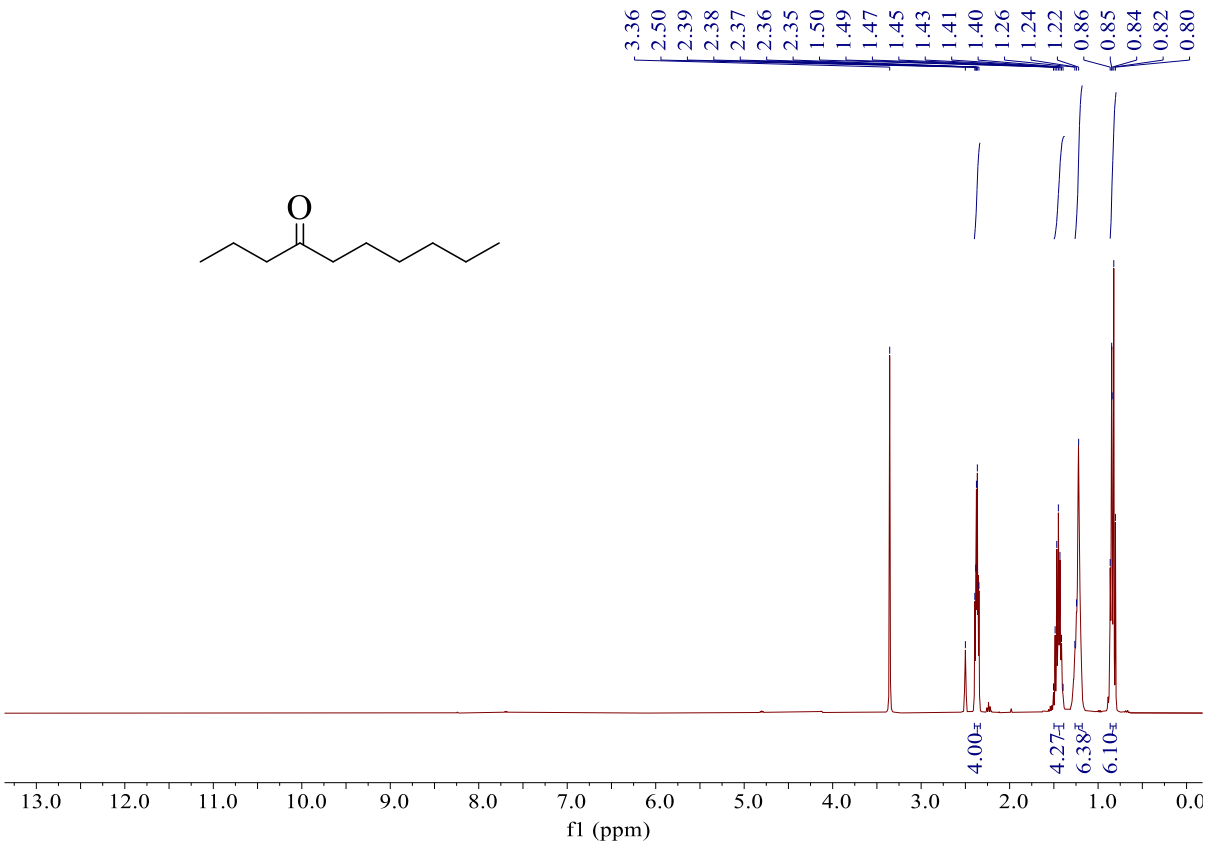


^{13}C NMR of compound 16l:



^{1}H NMR of compound 16m:



^{13}C NMR of compound 16m: