Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information

Chemoselective and repetitive intermolecular cross-acyloin condensation reactions between a variety of aromatic and aliphatic aldehydes using a robust N-heterocyclic carbene catalyst

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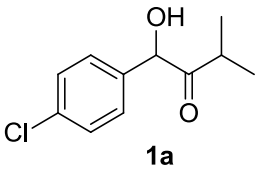
General Methods

Unless stated otherwise, reactions were carried out under a dry argon atmosphere in vacuum-flame dried glassware. Thin-layer chromatography (TLC) was performed on Merck silica gel 60 F254. Flash chromatography was performed using E. Merck silica gel (40-60 µm partical size). ^{1}H NMR spectra were recorded on a Varian at 300 MHz in $CDCl_3$ (δ 7.26 ppm), ^{13}C NMR spectral measurements were performed at 75 MHz using $CDCl_3$ (δ 77.0 ppm). The terms m, s, d, t, q, quint., and sept. represent multiplet, singlet, doublet, triplet, quadruplet, quintuplet, and septet, respectively, and the term br means a broad signal. Commercial grade reagents and solvents were used without further purification except as indicated below. THF was distilled from sodium-benzophenone prior to use. *m*-Xylene was distilled from P_2O_5. Gas chromatography analysis was performed by using Varian GC-450 model (Column VF-5ms).

General procedure for the synthesis of α-hydroxy ketones using N-heterocyclic carbene catalyst IId: To a suspension of aromatic aldehyde (0.5 mmol, 1.0 equiv), aliphatic aldehyde (7.5 mmol, 15 equiv), and N-heterocyclic carbene precatalyst **IId** (0.05 mmol, 10 mol%) in dry *m*-xylene (1 mL) was added to anhydrous Cs_2CO_3 (16.29 mg, 0.05 mmol). The reaction mixture was stirred at room temperature for 24 h, then quenched with distilled water and extracted with EtOAc (1 mL×3). The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated in vacuo. The residue was purified by flash chromatography (EtOAc:Hexanes = 1:20) to give α-hydroxy ketone as an colorless or yellow oil.

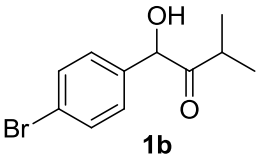
1-(4-chlorophenyl)-1-hydroxy-3-methylbutan-2-one (Table 2; Entry 1)



1a

The physical and spectral data were identical to those previously reported for this compound.[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.38-7.34 (m, 2H), 7.28-7.24 (m, 2H), 5.20 (d, *J* = 4.5 Hz, 1H), 4.38 (d, *J* = 4.5 Hz, 1H), 2.73-2.63 (m, 1H), 1.15 (d, *J* = 7.2 Hz, 3H), 0.85 (d, *J* = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.9, 136.4, 134.5, 129.0, 128.8, 77.5, 35.9, 19.2, 17.9 ppm.

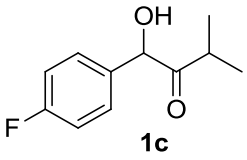
1-(4-bromophenyl)-1-hydroxy-3-methylbutan-2-one (Table 2; Entry 2)



1b

The physical and spectral data were identical to those previously reported for this compound.[1] ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.54-7.49 (m, 2H), 7.21-7.18 (m, 2H), 5.18 (d, *J* = 4.5 Hz, 1H), 4.38 (d, *J* = 4.5 Hz, 1H), 2.75-2.61 (m, 1H), 1.15 (d, *J* = 7.2 Hz, 3H), 0.85 (d, *J* = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.8, 136.9, 132.1, 129.2, 122.7, 77.6. 35.9, 19.3,17.9 ppm.

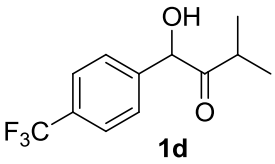
1-(4-fluorophenyl)-1-hydroxy-3-methylbutan-2-one (Table 2; Entry 3)



1c

By means of the general procedure described above, IR (film) ν_{max} 2975, 1711, 1602, 1508, 1467, 1230, 1126, 1092, 1013, 816 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.32-7.28 (m, 2H), 7.10-7.03 (m, 2H), 5.21 (d, *J* = 3.3 Hz, 1H), 4.40 (d, *J* = 3.9 Hz, 1H), 2.75-2.60 (m, 1H), 1.13 (d, *J* = 7.2 Hz, 3H), 0.85 (d, *J* = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 213.2, 162.8 (d, J_{CF} = 246.3 Hz), 133.8 (d, J_{CF} = 3.3 Hz), 129.3 (d, J_{CF} = 8.33 Hz), 115.9 (d, J_{CF} = 21.6 Hz), 77.5, 36.0, 19.3, 18.0; HRMS (EI) Calcd for $C_{12}H_{16}O_2$: 196.0900, found: 196.0902.

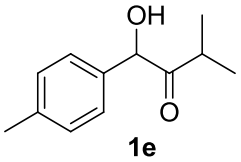
1-hydroxy-3-methyl-1-(4-(trifluoromethyl)phenyl)butan-2-one (Table 2; Entry 4)



1d

By means of the general procedure described above, IR (film) ν_{max} 2979, 1713, 1331, 1163, 1133, 1118, 1070, 1014, 819 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.65 (d, *J* = 8.1 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 5.28 (d, *J* = 4.5 Hz, 1H), 4.42 (d, *J* = 4.5 Hz, 1H), 2.76-2.62 (m, 1H), 1.17 (d, *J* = 6.9 Hz, 3H), 0.85 (d, *J* = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.6, 141.9 (q, J_{CF} = 1.3 Hz), 131.1, 130.7, 127.9, 123.8 (q, J_{CF} = 270 Hz), 125.9 (q, J_{CF} = 3.75 Hz), 77.7, 36.1, 29.7, 19.3, 17.9; HRMS (FAB) Calcd for $[C_{12}H_{14}F_3O_2]^+$: 247.0946, found: 247.0946.

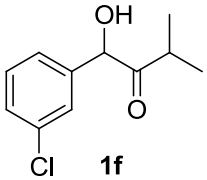
1-hydroxy-3-methyl-1-*p*-tolylbutan-2-one (Table 2; Entry 5)



1e

By means of the general procedure described above, IR (film) ν_{max} 2970, 1714, 1510, 1462, 1382, 1098, 1066, 1007, 802 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.18 (s, 4H), 5.19 (d, *J* = 4.87 Hz, 1H), 4.35 (d, *J* = 4.5 Hz, 1H), 2.74-2.64 (m, 1H), 2.35 (s, 3H), 1.13 (d, *J* = 6.9 Hz, 3H), 0.85 (d, *J* = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 213.7, 138.5, 134.9, 129.6, 127.5, 78.0, 35.9, 21.2, 19.4, 18.0 ppm; HRMS (EI) Calcd for $C_{12}H_{16}O_2$: 192.1150, found: 192.1151.

1-(3-chlorophenyl)-1-hydroxy-3-methylbutan-2-one (Table 2; Entry 6)

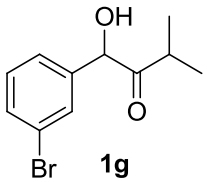


1f

By means of the general procedure described above, , IR (film) ν_{max} 2941, 2831, 1721, 1576, 1471, 1383, 1193, 1025, 788, 679 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.39-7.27 (m, 3H), 7.22-7.18 (m, 1H), 5.20 (s, 1H), 4.45 (s, 1H), 2.78-2.63 (m, 1H), 1.14 (d, *J* = 6.9 Hz, 3H),

0.86 (d, J = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.7, 139.9, 134.8, 130.1, 128.8, 127.6, 125.6, 77.5, 35.9, 19.3, 17.9 ppm; HRMS (EI) Calcd for $C_{11}H_{13}ClO_2$: 212.0604, found: 212.0605.

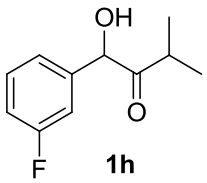
1-(3-bromophenyl)-1-hydroxy-3-methylbutan-2-one (Table 2; Entry 7)



1g

By means of the general procedure described above, IR (film) ν_{max} 2944, 2831, 1726, 1702, 1375, 1245, 1027, 850, 788, 667 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.49-7.45 (m, 2H), 7.26-7.24 (m, 2H), 5.18 (d, J = 4.2 Hz, 1H), 4.40 (d, J = 4.5 Hz, 1H), 2.78-2.63 (m, 1H), 1.16 (d, J = 7.2 Hz, 3H), 0.87 (d, J = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.7, 140.1, 131.8, 130.6, 130.5, 126.1, 123.0, 77.6, 36.0, 19.4, 17.9 ppm; HRMS (EI) Calcd for $C_{11}H_{13}BrO_2$: 256.0099, found: 256.0093.

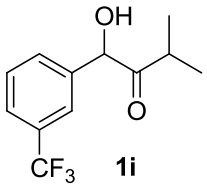
1-(3-fluorophenyl)-1-hydroxy-3-methylbutan-2-one (Table 2; Entry 8)



1h

By means of the general procedure described above, IR (film) ν_{max} 2975, 1706, 1590, 1483, 1452, 1247, 1123, 1016, 833, 784, 714, 687 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.39-7.31 (m, 1H), 7.14-7.10 (m, 1H), 7.07-7.00 (m, 2H), 5.21 (d, J = 4.5 Hz, 1H), 4.39 (d, J = 4.5 Hz, 1H), 2.79-2.64 (m, 1H), 1.16 (d, J = 7.2 Hz, 3H), 0.86 (d, J = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.8, 163.0 (d, J_{CF} = 246.2 Hz), 140.4 (d, J_{CF} = 6.9 Hz), 130.5 (d, J_{CF} = 8.1 Hz), 123.2 (d, J_{CF} = 3 Hz), 115.7 (d, J_{CF} = 21.1 Hz), 114.4 (d, J_{CF} = 21.9 Hz), 77.7 (d, J_{CF} = 1.9 Hz), 40.0, 19.3, 18.0 ppm; HRMS (EI) Calcd for $C_{11}H_{13}FO_2$: 196.0900, found: 196.0900.

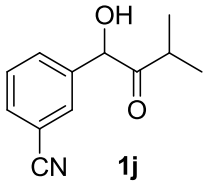
1-hydroxy-3-methyl-1-(3-(trifluoromethyl)phenyl)butan-2-one (Table 2; Entry 9)



1i

By means of the general procedure described above, IR (film) ν_{max} 2980, 2856, 1738, 1375, 1330, 1243, 1166, 1128, 1073, 1047, 780, 703, 664 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.63-7.59 (m, 2H), 7.55-7.51 (m 2H), 5.28 (d, J = 4.5 Hz, 1H), 4.44 (d, J = 4.5 Hz, 1H), 2.76-2.61 (m, 1H), 1.17 (d, J = 7.2 Hz, 3H), 0.85 (d, J = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.6, 139.0, 130.8, 131.4 (q, J_{CF} = 32.35 Hz), 130.8, 129.5, 125.5 (q, J_{CF} = 3.73 Hz) , 124.4 (q, J_{CF} = 3.8 Hz), 77.7, 36.1, 19.3, 17.9 ppm; HRMS (FAB) Calcd for $[C_{12}H_{14}F_3O_2]^+$: 247.0946, found: 247.0953.

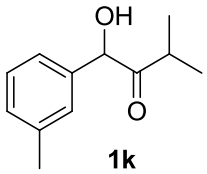
3-(1-hydroxy-3-methyl-2-oxobutyl)benzonitrile (Table 2; Entry 10)



1j

By means of the general procedure described above, IR (film) ν_{max} 2966, 2875, 2233, 1719, 1468, 1363, 1098, 1022, 900, 811, 706 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67-7.48 (m, 4H), 5.26 (d, J = 4.5 Hz, 1H), 4.44 (d, J = 4.5 HZ, 1H), 2.76-2.61 (m, 1H), 1.17 (d, J = 6.9 Hz, 3H), 0.86 (d, J = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.1, 139.6, 132.3, 131.7, 131.1, 129.8, 118.2, 113.1, 77.4, 36.0, 19.3, 17.9 ppm; HRMS (EI) Calcd for $C_{12}H_{13}NO_2$: 203.0946, found: 203.0949.

1-hydroxy-3-methyl-1-*m*-tolylbutan-2-one (Table 2; Entry 11)

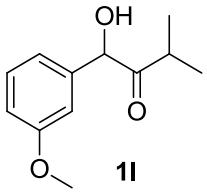


1k

By means of the general procedure described above, IR (film) ν_{max} 2977, 1739, 1712, 1374, 1241, 1046, 1020, 785, 712 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.28-7.08 (m, 4H), 5.19 (s, 1H), 4.38 (s, 1H), 2.78-2.63 (s, 1H), 2.35 (s, 3H), 1.31 (d, J = 7.2 Hz, 3H), 0.85 (d, J = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 213.6, 138.7, 137.8, 129.4, 128.8, 128.1, 124.7,

78.2, 35.9, 21.3, 19.4, 17.9 ppm; HRMS (EI) Calcd for $C_{12}H_{16}O_2$: 192.1150, found: 192.1146.

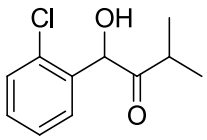
1-hydroxy-1-(3-methoxyphenyl)-3-methylbutan-2-one (Table 2; Entry 12)



1l

By means of the general procedure described above, IR (film) ν_{max} 2977, 1738, 1600, 1374, 1242, 1046, 1018, 784, 710 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.33-7.26 (m, 1H), 6.92-6.80 (m, 3H), 5.20 (d, *J* = 4.5 Hz, 1H), 4.38 (d, *J* = 4.8 Hz, 1H), 3.81 (s, 3H), 2.80-2.60 (m, 1H), 1.15 (d, *J* = 6.9 Hz, 3H), 0.86 (d, *J* = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 213.3, 159.9, 139.3, 129.9, 119.9, 114.2, 112.7, 78.1, 55.2, 35.8, 19.3, 17.9 ppm. HRMS (EI+) Calcd for $C_{12}H_{16}O_3$: 208.1099, found: 208.1104.

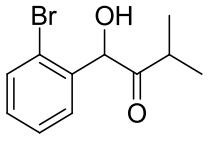
1-(2-chlorophenyl)-1-hydroxy-3-methylbutan-2-one (Table 2; Entry 13)



1m

The physical and spectral data were identical to those previously reported for this compound.[2] ^1H NMR (300 MHz, $CDCl_3$) δ: 7.47-7.39 (m, 1H), 7.31-7.20 (m, 3H), 5.73 (d, *J* = 4.5 Hz, 1H), 4.42 (d, *J* = 4.5 Hz, 1H), 2.78-2.63 (m, 1H), 1.17 (d, *J* = 6.9 Hz, 3H), 0.88 (d, *J* = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.8, 135.6, 133.7, 130.0, 129.8, 129.2, 127.4, 74.5, 36.1, 19.4, 17.8 ppm.

1-(2-bromophenyl)-1-hydroxy-3-methylbutan-2-one (Table 2; Entry 14)

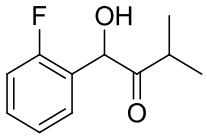


1n

The physical and spectral data were identical to those previously reported for this compound.[2] ^1H NMR (300 MHz, $CDCl_3$) δ: 7.64-7.59 (m, 1H), 7.36-7.29 (m, 1H), 7.24-7.16 (m, 2H), 5.74 (d, *J* = 4.5 Hz, 1H), 4.44 (d, *J* = 4.5 Hz, 1H), 2.78-2.60 (m, 1H), 1.18 (d, *J* = 7.2 Hz, 1H),

0.88 (d, J = 6.6 Hz, 1H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 212.8, 137.2, 133.2, 130.0, 129.2, 128.0, 124.0, 76.6, 36.1, 19.4, 17.8 ppm.

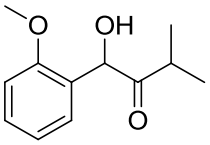
1-(2-fluorophenyl)-1-hydroxy-3-methylbutan-2-one (Table 2; Entry 15)



1o

By means of the general procedure described above, IR (film) ν_{max} 2977, 1716, 1491, 1458, 1375, 1238, 1015, 806, 760 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.37-7.07 (m, 5H), 5.56 (d, J = 4.8 Hz, 1H), 4.34 (d, J = 4.8 Hz, 1H), 2.76 -2.63 (m, 1H), 1.16 (d, J = 7.2 Hz, 3H), 0.90 (d, J = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 160.6 (d, J_{CF} = 246 Hz), 130.5 (d, J_{CF} = 8.25 Hz), 129.2 (d, J_{CF} = 3.5 Hz), 125.3 (d, J_{CF} = 20.25 Hz), 124.8 (d, J_{CF} = 3.52 Hz), 115.9 (d, J_{CF} = 21.6 Hz) ,71.7 (d, J_{CF} = 3.2 Hz), 36.1, 19.4, 18.0 ppm; HRMS (EI) Calcd for $C_{11}H_{13}FO_2$: 196.0900, found: 196.0895.

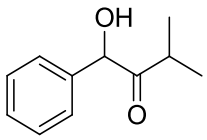
1-hydroxy-1-(2-methoxyphenyl)-3-methylbutan-2-one (Table 2; Entry 16)



1p

By means of the general procedure described above, IR (film) ν_{max} 2973, 1710, 1493, 1465, 1288, 1014, 911, 756, 733 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.34-7.27 (m, 1H), 7.23-7.19 (m, 1H), 6.99-6.89 (m, 2H), 5.53 (d, J = 4.8 Hz, 1H), 4.30 (d, J = 5.1 Hz, 1H), 3.83 (s, 3H), 2.74-2.60 (m, 1H), 1.11 (d, J = 6.9 Hz, 3H), 0.89 (d, J = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 213.6, 156.9, 129.8, 129.2, 126.4, 120.9, 110.9, 73.1, 55.3, 35.6, 19.4, 17.8 ppm; HRMS (EI) Calcd for $C_{12}H_{16}O_3$: 208.1099, found: 208.1101.

1-hydroxy-3-methyl-1-phenylbutan-2-one (Table 2; Entry 17)

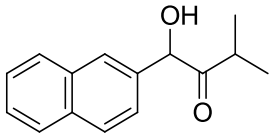


1q

The physical and spectral data were identical to those previously reported for this compound.[1]

^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.42-7.28 (m,5H), 5.22 (d, *J* = 4.5 Hz, 1H), 4.39 (d, *J* = 4.5 HZ, 1H), 2.78-2.63 (m, 1H), 1.14 (d, *J* = 7.2 Hz, 3H), 0.84 (d, *J* = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 213.4, 137.8, 128.9, 128.6, 127.5, 78.2, 35.9, 19.3, 17.9 ppm.

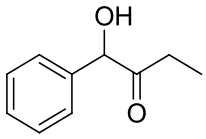
1-hydroxy-3-methyl-1-(naphthalen-2-yl)butan-2-one (Table 2; Entry 18)



1r

By means of the general procedure described above, IR (film) ν_{max} 2970, 1706, 1246, 1222, 1106, 1017, 818, 749, 677 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.90-7.80 (m, 4H), 7.54-7.50 (m, 2H), 7.37-7.32 (m, 1H), 5.40 (d, *J* = 4.8 Hz, 1H), 4.49 (d, *J* = 4.5 Hz, 1H), 2.80-2.70 (m, 1H), 1.17 (d, *J* = 6.9 Hz, 3H), 0.82 (d, J = 6.6 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 213.5, 135.2, 133.2, 133.2, 128.8, 127.9, 127.7, 127.3, 126.4, 124.4, 78.3, 35.9, 19.3, 17.9 ppm. HRMS (EI) Calcd for $C_{15}H_{16}O_2$: 228.1150, found: 228.1149.

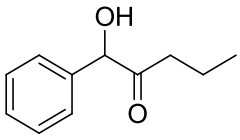
1-hydroxy-1-phenylbutan-2-one (Table 2; Entry 19)



1s

The physical and spectral data were identical to those previously reported for this compound.[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.42-7.29 (m, 5H), 5.10 (d, *J* = 4.2 Hz, 1H), 4.36 (d, *J* = 4.2 Hz, 1H), 2.49-2.26 (m, 2H), 1.01 (t, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 210.1, 138.3, 129.0, 128.7, 127.3, 79.5, 31.2, 7.6 ppm.

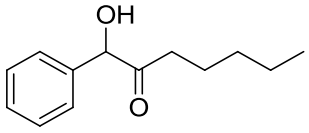
1-hydroxy-1-phenylpentan-2-one (Table 2; Entry 20)



1t

The physical and spectral data were identical to those previously reported for this compound.[4] ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.42-7.30 (m, 5H), 5.08 (d, *J* = 4.2 Hz, 1H), 4.40 (d, *J* = 4.2 Hz, 1H), 2.46 -2.22 (m, 2H), 1.68-1.44 (m, 2H), 0.80 (t, *J* = 7.5 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 209.5, 138.0, 128.9, 128.6, 127.4, 79.7, 39.7, 17.1, 13.5 ppm.

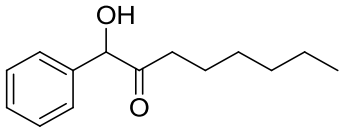
1-hydroxy-1-phenylheptan-2-one (Table 2; Entry 21)



1u

The physical and spectral data were identical to those previously reported for this compound.[5] ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.42-7.29 (m, 5H), 5.08 (d, *J* = 4.2 Hz, 1H), 4.38 (d, *J* = 4.5 Hz, 1H), 2.43-2.23 (m, 2H), 1.59-1.19 (m, 2H), 1.27-1.07 (m, 4H), 0.82 (t, *J* = 6.9 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 209.6, 138.0, 128.9, 128.6, 127.4, 79.6, 37.7, 31.0, 23.3, 22.2, 13.8 ppm.

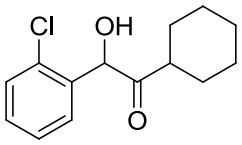
1-hydroxy-1-phenyloctan-2-one (Table 2; Entry 22)



1v

By means of the general procedure described above, IR (film) ν_{max} 2934, 2860, 1716, 1494, 1375, 1193, 1028, 701 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.42-7.29 (m, 5H), 5.08 (d, *J* = 4.2 Hz, 1H), 4.38 (d, *J* = 4.5 Hz, 1H), 2.43-2.23 (m, 2H), 1.59-1.13 (m, 8H), 0.83 (t, *J* = 7.1 Hz, 3H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 209.6, 138.0, 128.9, 128.6, 127.4, 79.6, 37.8,31.3, 28.6, 23.6, 22.3, 13.9 ppm; HRMS (EI) Calcd for $C_{14}H_{20}O_2$: 220.1463, found: 220.1463.

2-(2-chlorophenyl)-1-cyclohexyl-2-hydroxyethanone (Table 2; Entry 24)

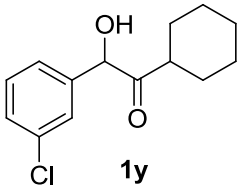


1x

By means of the general procedure described above, IR (film) ν_{max} 2935, 2924, 2855, 1721, 1447, 1133, 1045, 994, 766, 709 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.46-7.39 (m, 1H), 7.32-7.18 (m, 3H), 5.71 (d, *J* = 4.5 Hz, 1H), 4.43 (d, *J* = 4.8 Hz, 1H), 2.44 (tt, *J* = 11.1 Hz, 3.6 Hz, 1H), 1.97-1.92 (m, 1H), 1.80-1.50 (m, 3H), 1.48-0.98 (m, 6H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 211.8, 135.6, 133.7, 130.0, 129.7, 129.1, 127.4, 74.4, 46.1, 29.8, 27.7, 25.7,

25.5, 24.9 ppm. HRMS (FAB) Calcd for $[C_{14}H_{18}ClO_2]^+$: 253.0990, found: 253.0995.

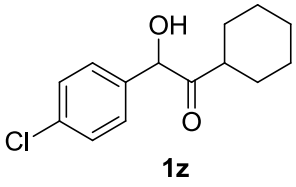
2-(3-chlorophenyl)-1-cyclohexyl-2-hydroxyethanone (Table 2; Entry 25)



1y

By means of the general procedure described above, IR (film) ν_{max} 2933, 2860, 1706, 1445, 1193, 1126, 993, 768, 694, 619 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.38-7.28 (m, 3H), 7.24-7.17 (m, 1H), 5.16 (d, *J* = 3.9 Hz, 1H), 4.42 (d, *J* = 4.5 Hz, 1H), 2.42 (tt, *J* = 11.1 Hz, 3.6 Hz, 1H), 1.89-1.61 (m, 4H), 1.44-0.82 (m, 6H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 211.7, 139.8, 134.8, 130.1, 128.9, 127.7, 125.7, 77.6, 46.0, 29.6, 27.8, 25.6, 25.4, 24.9 ppm; HRMS (EI) Calcd for $C_{14}H_{17}ClO_2$: 252.0917, found: 252.0917.

2-(4-chlorophenyl)-1-cyclohexyl-2-hydroxyethanone (Table 2; Entry 26)

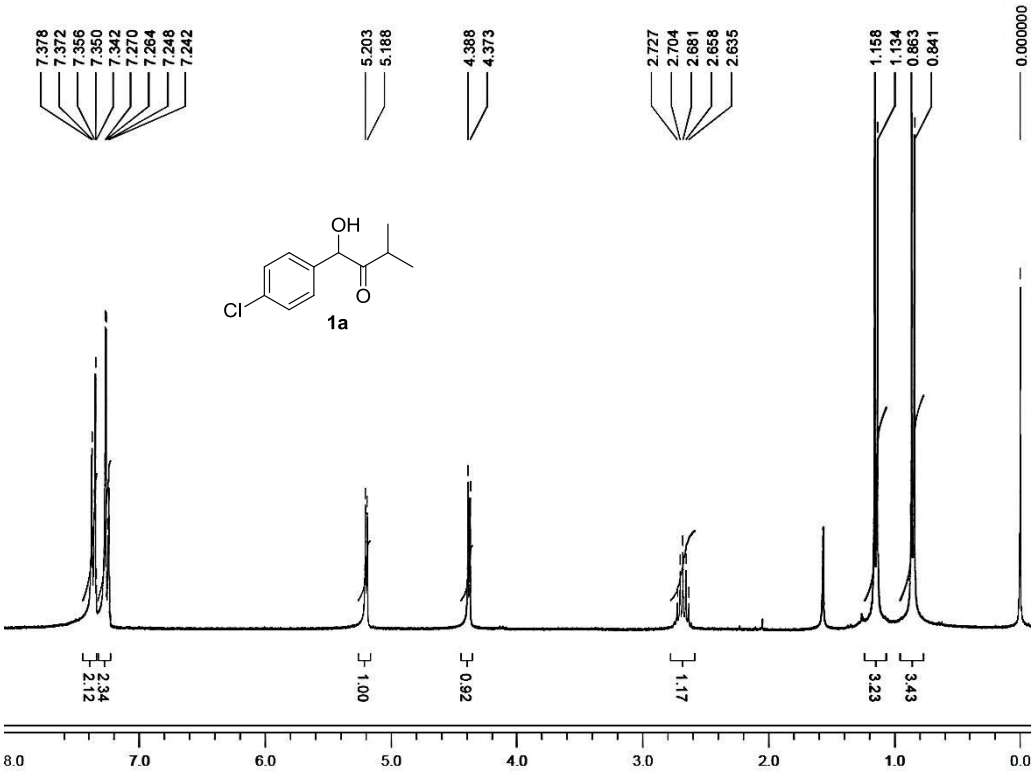


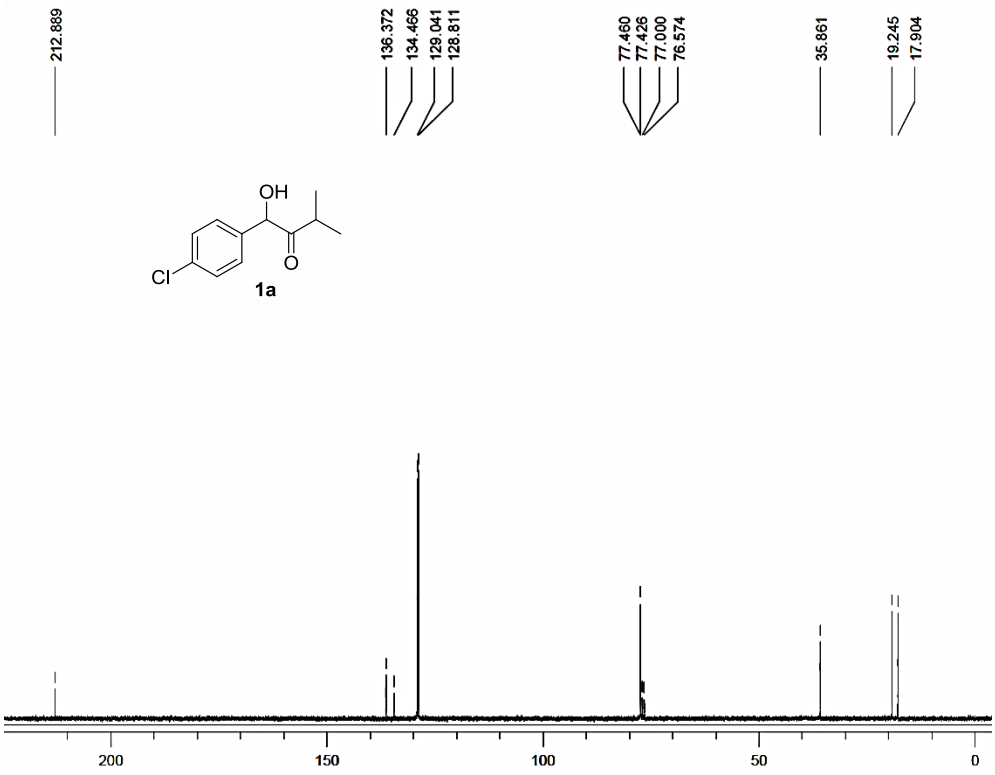
1z

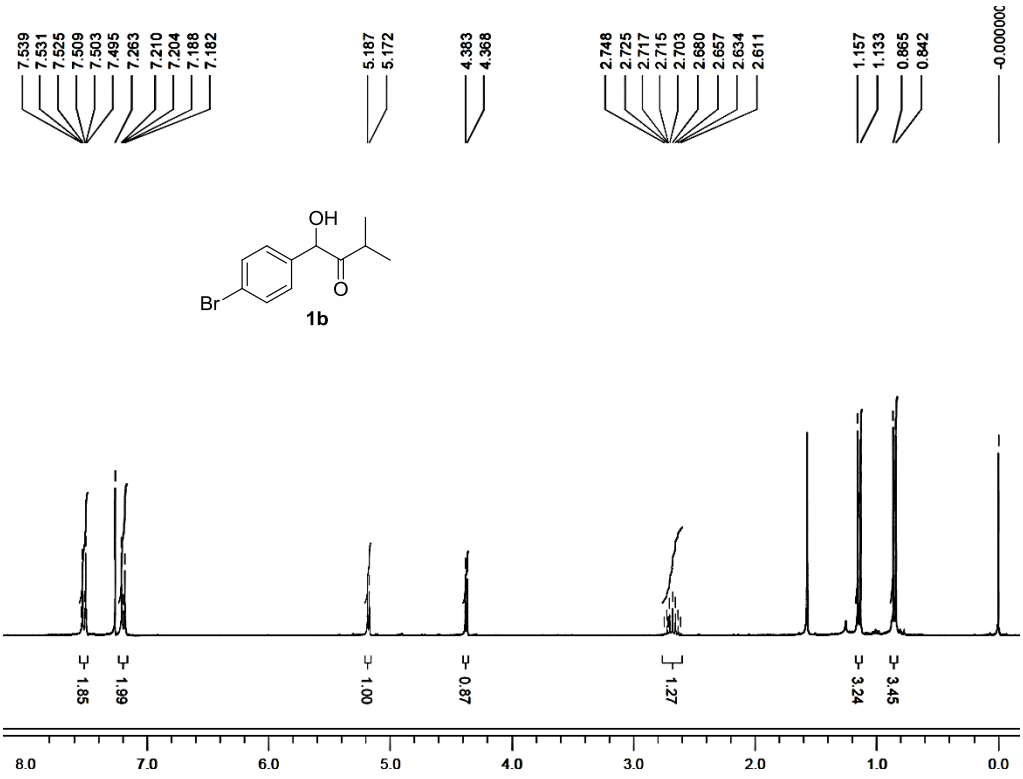
By means of the general procedure described above, IR (film) ν_{max} 2935, 2852, 1707, 1491, 1448, 1094, 995, 823, 772, 645, 625 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ: 7.38-7.36 (m, 2H), 7.27-7.22 (m, 2H), 5.16 (d, *J* = 4.5 Hz, 1H), 4.42 (d, *J* = 4.8 Hz, 1H), 2.45-2.34 (m, 1H), 1.88-1.61 (m, 4H), 1.43-0.96 (m, 7H) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) δ: 211.9, 136.3, 134.5, 128.9, 77.5, 45.9, 29.5, 27.8, 25.6, 25.4, 24.9 ppm; HRMS (EI) Calcd for $C_{14}H_{17}ClO_2$: 252.0917, found: 252.0911.

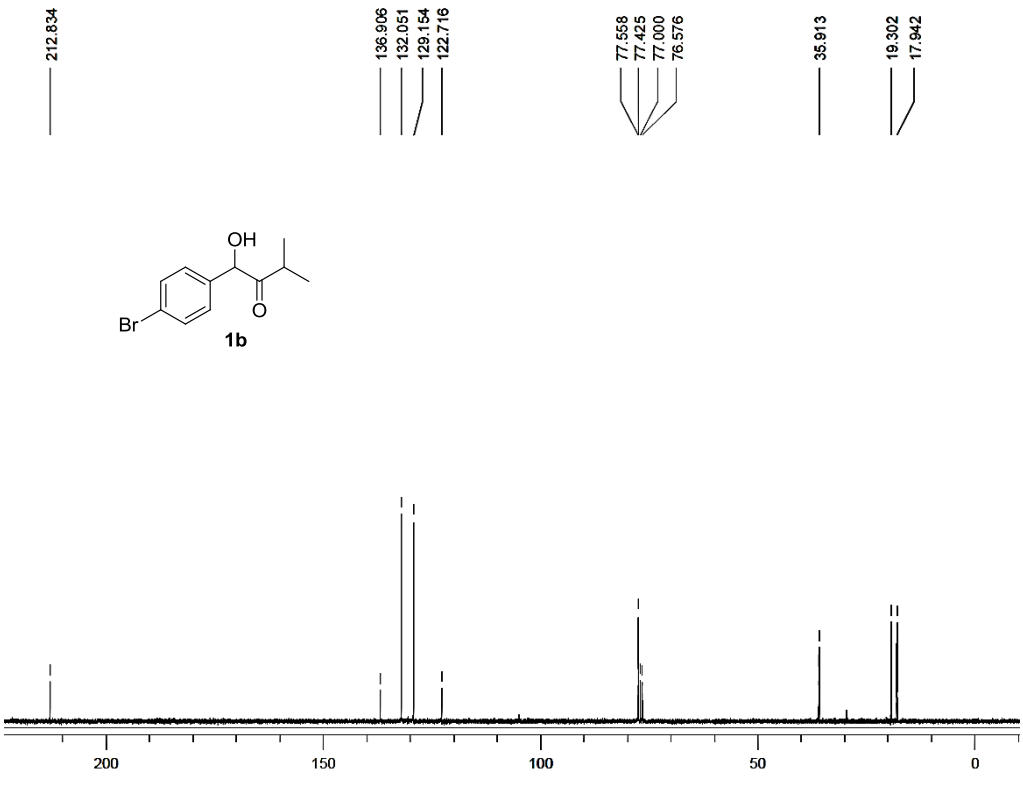
References:

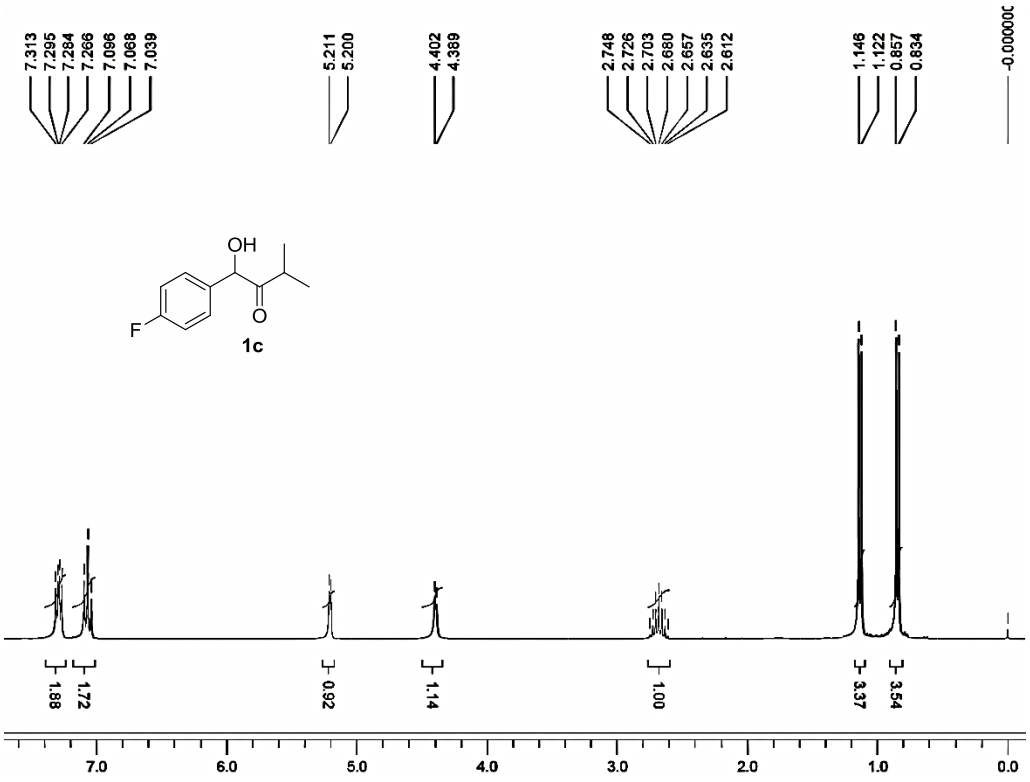
1. C. A. Rose, S. Gundala, S. J. Connon and K. Zeitler, *Synthesis* 2011, **2**, 190-198.
2. S. E. O'Toole, C. A. Rose, S. Gundala, K. Zeitler and S. J. Connon, *J. Org. Chem*., 2011, **76**, 347-357.
3. C.-T. Chen, J.-Q. Kao, S. B. Salunke and Y.-H. Lin, *Org. Lett*., 2011, **13**, 26-29.
4. D. Gocke, C. L. Nguyen, M. Pohl, T. Stillger, L. Walter and M. Müller, *Adv. Synth. Catal*., 2007, **349**, 1425-1435.
5. H. Okada, T. Mori, Y. Saikawa and M. Nakata, *Tetrahedron Lett*., 2009, **50**, 1276-1278.

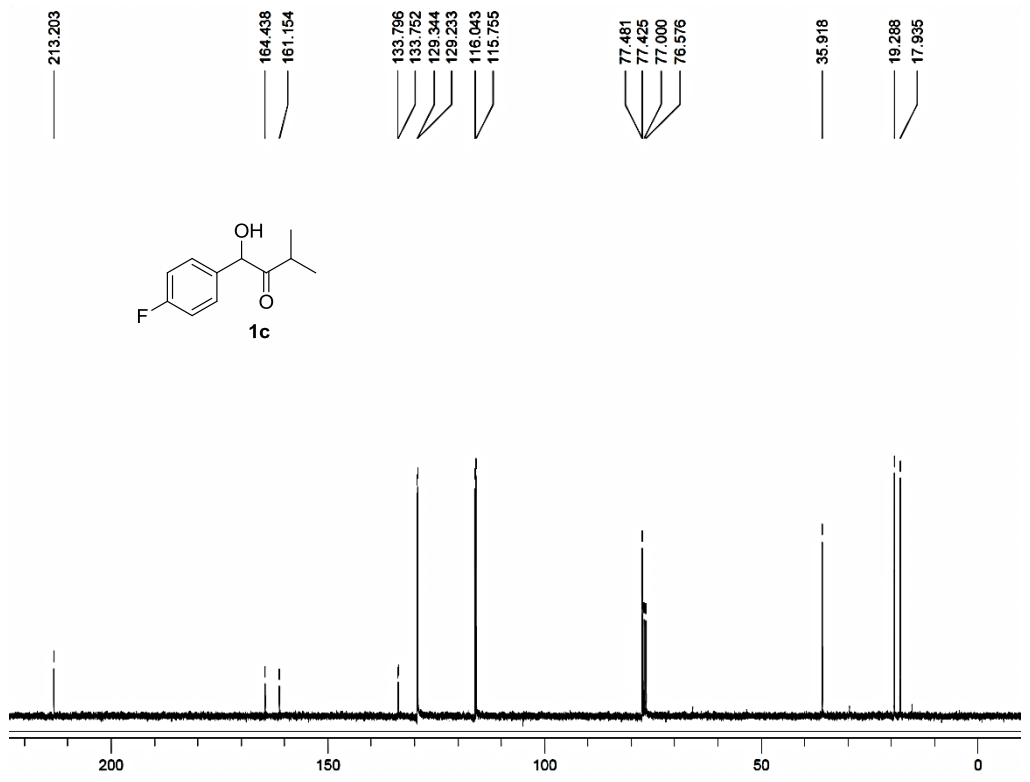


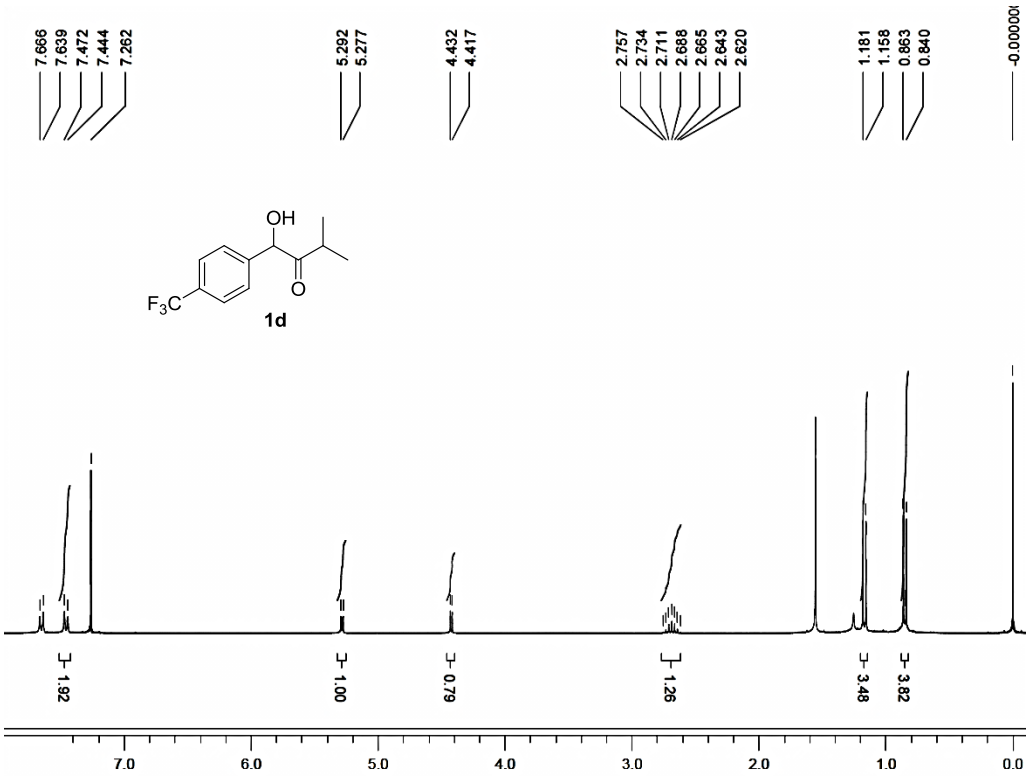


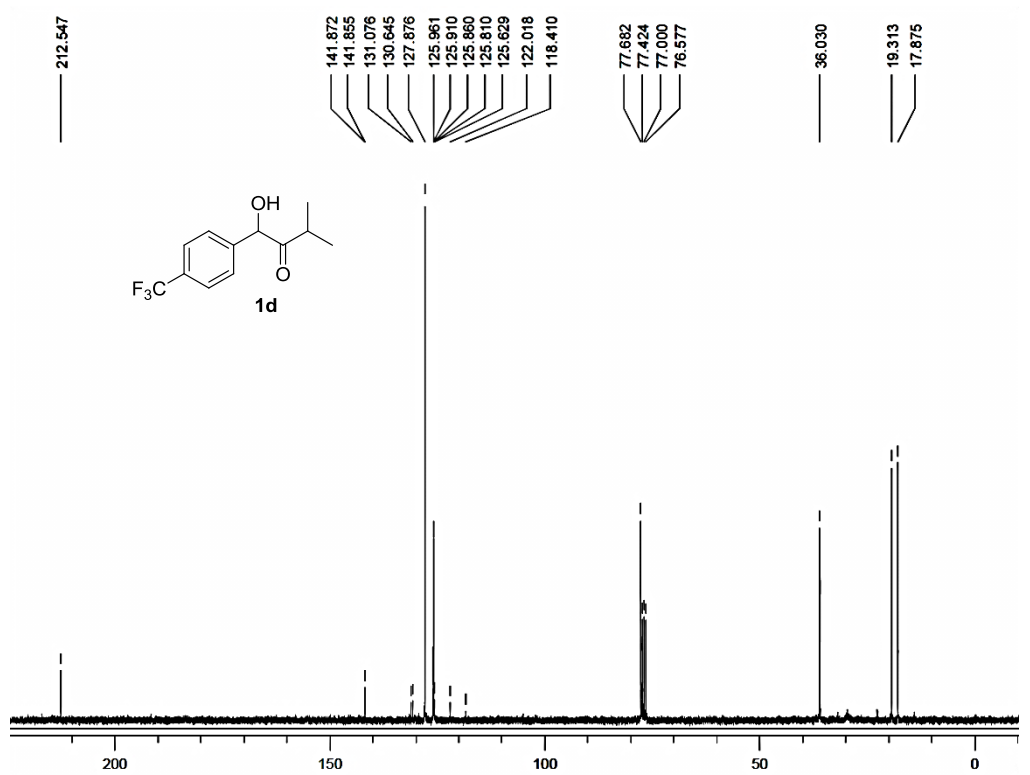


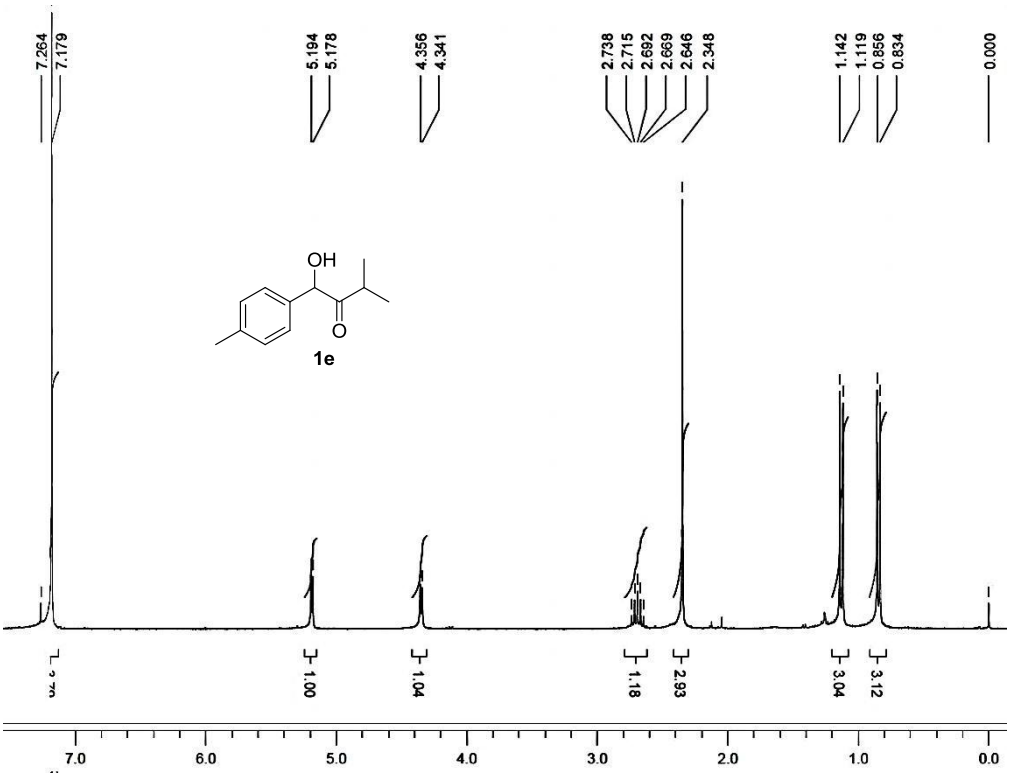


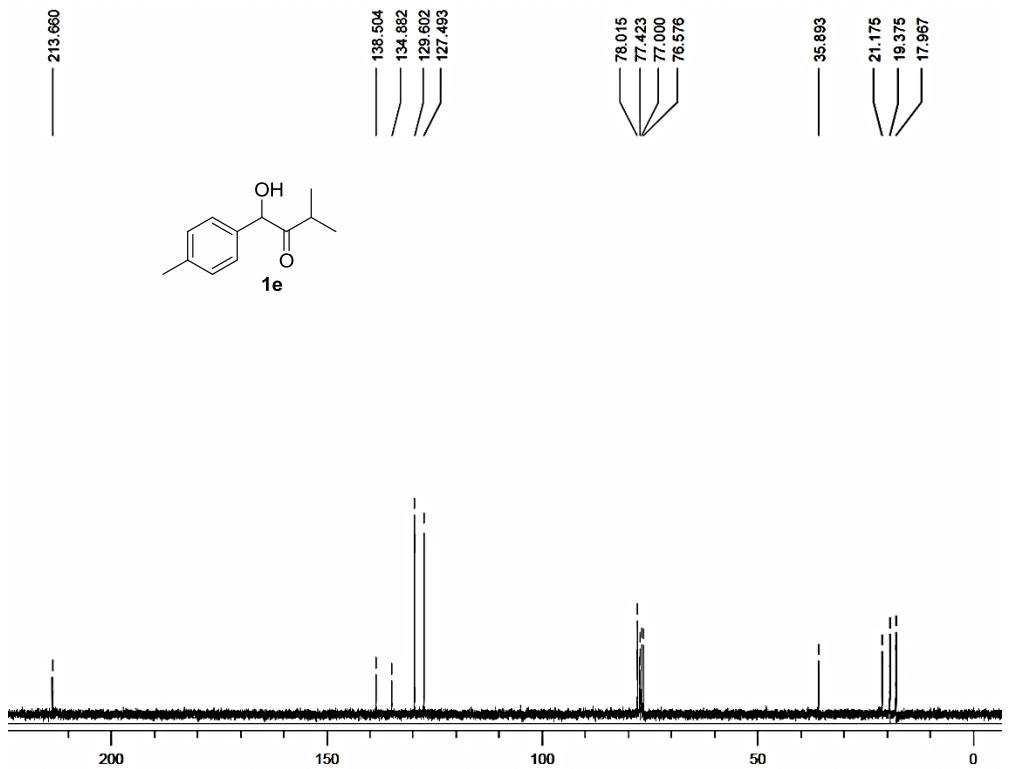


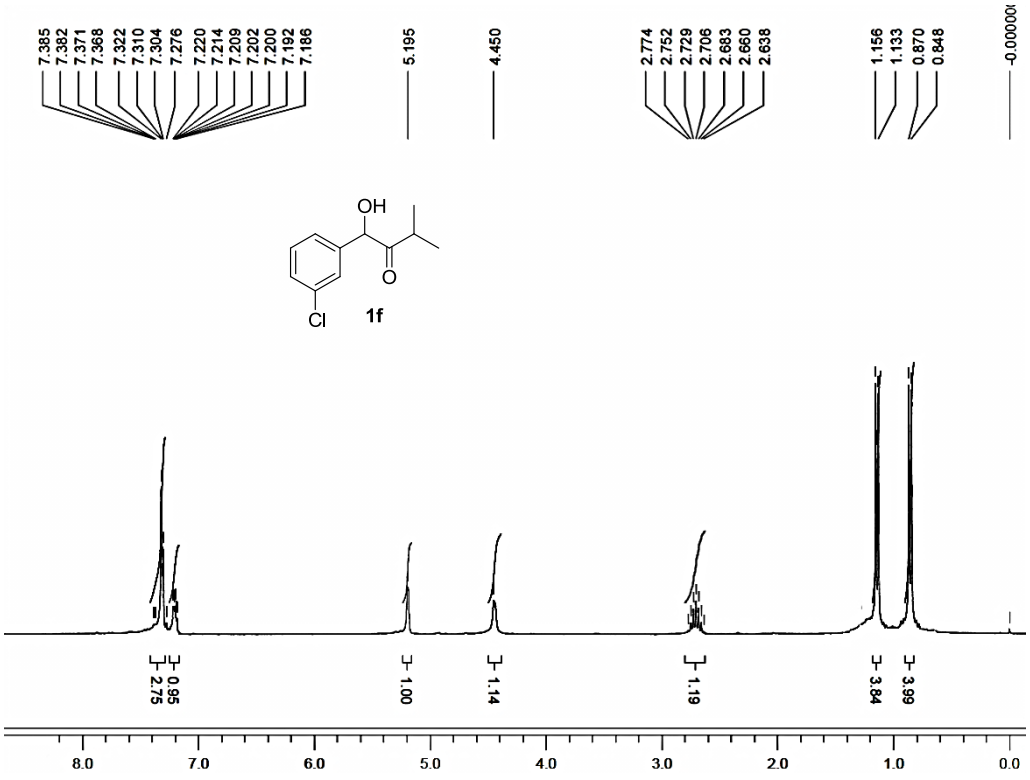


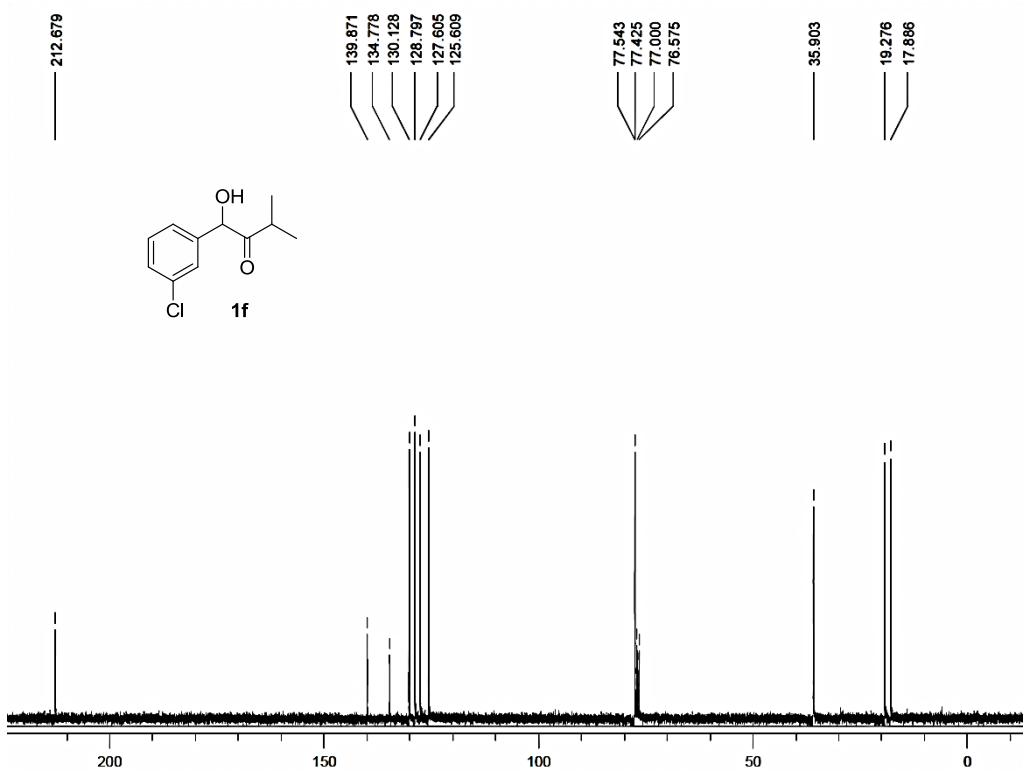


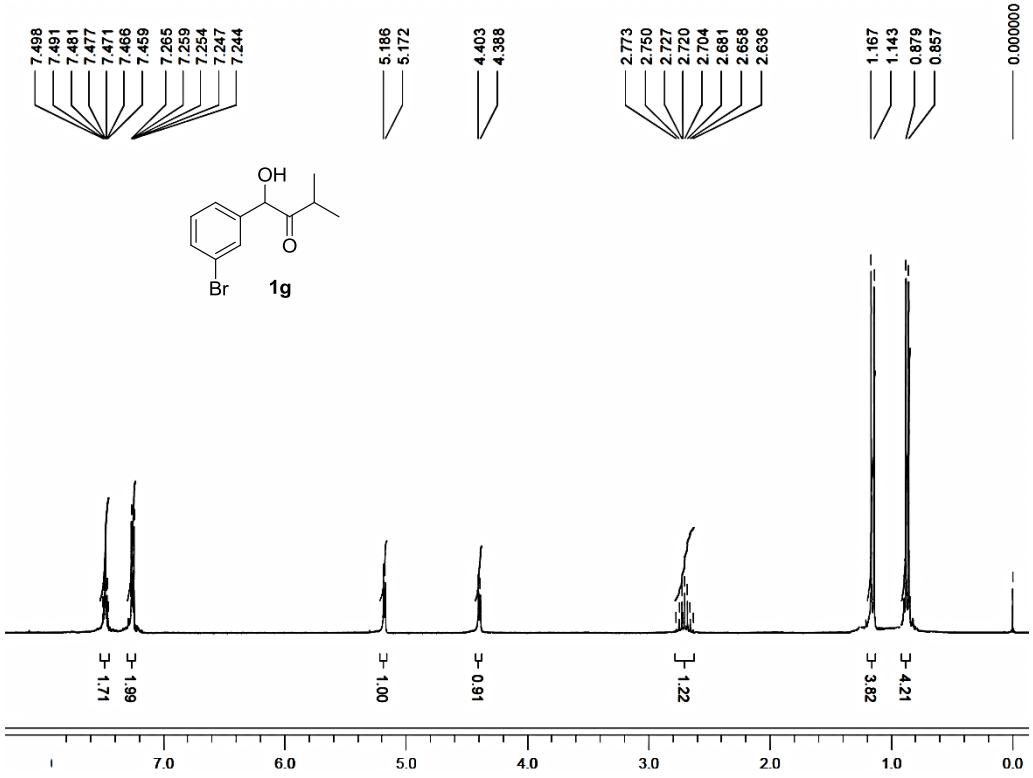


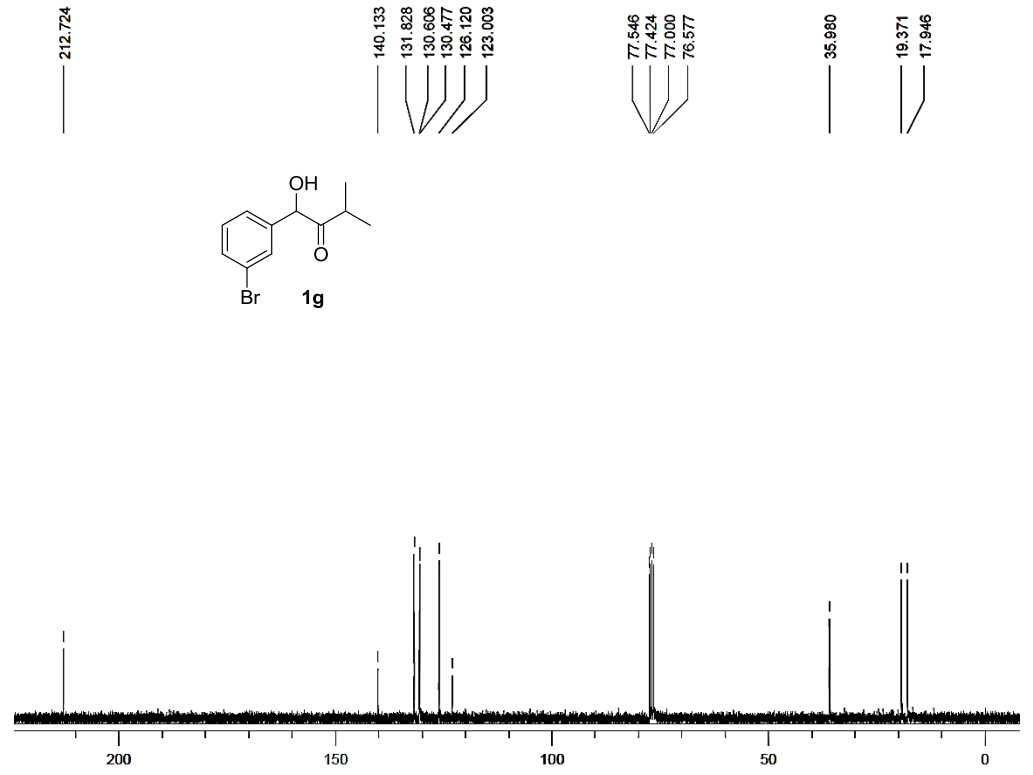


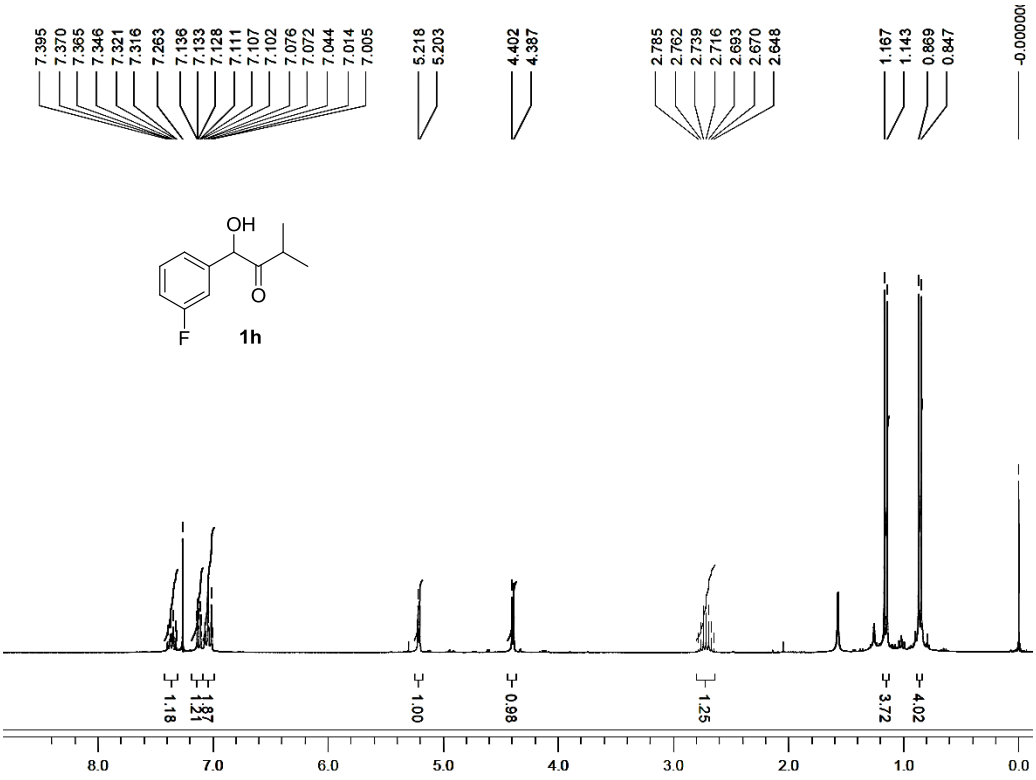


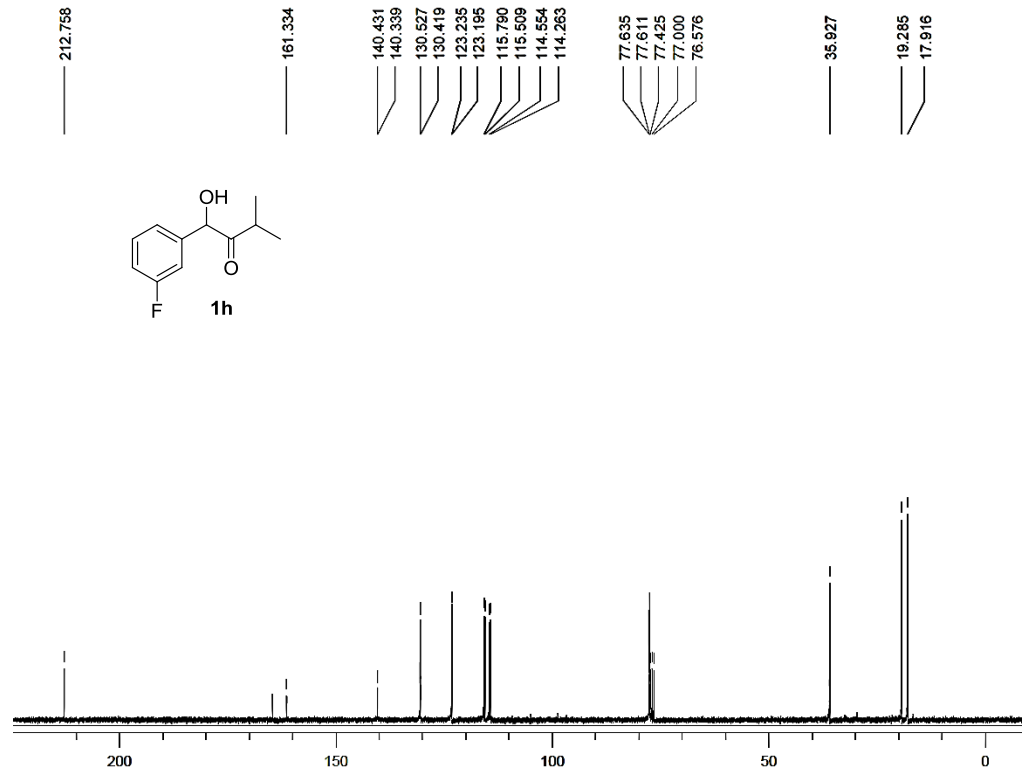


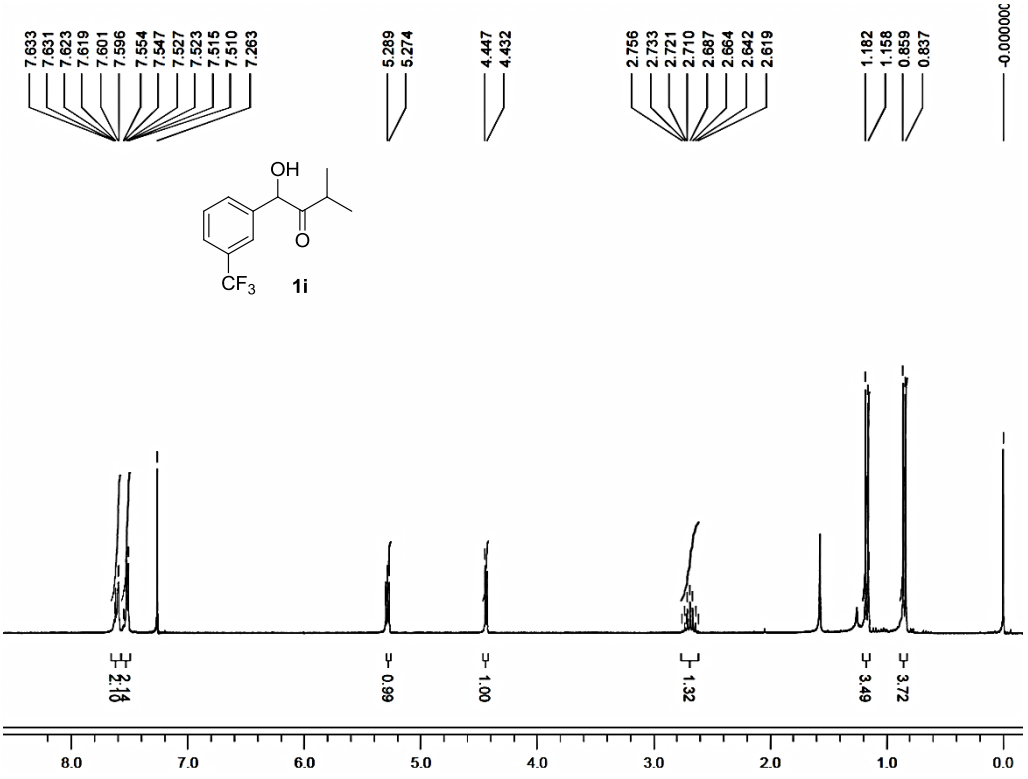


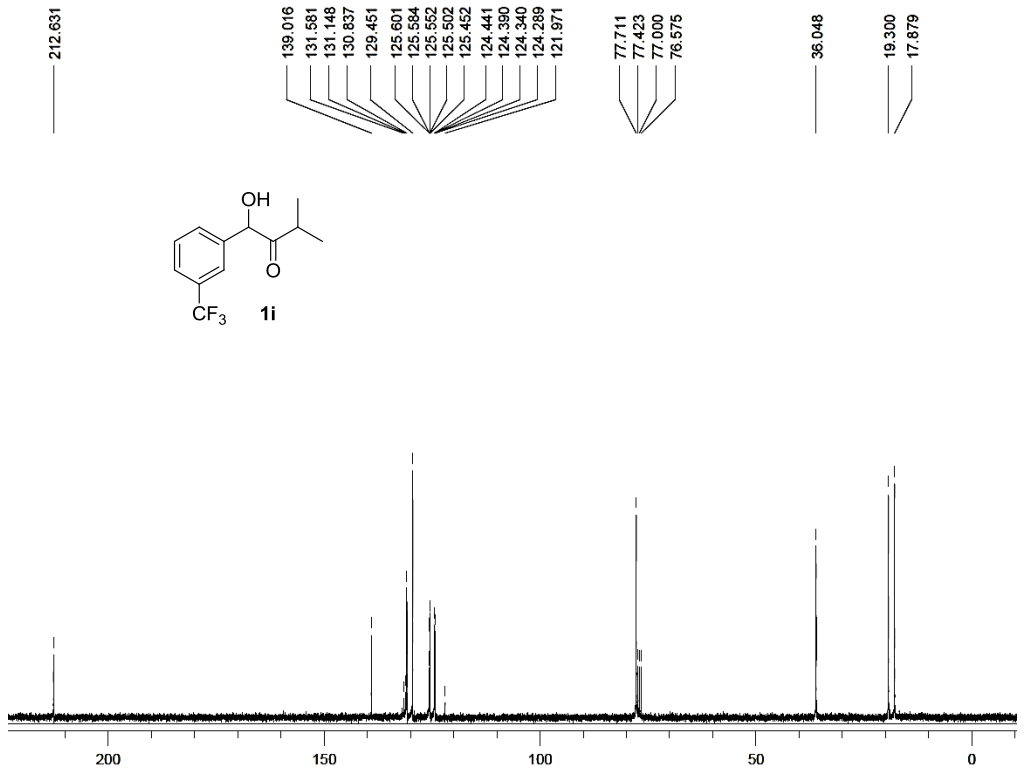


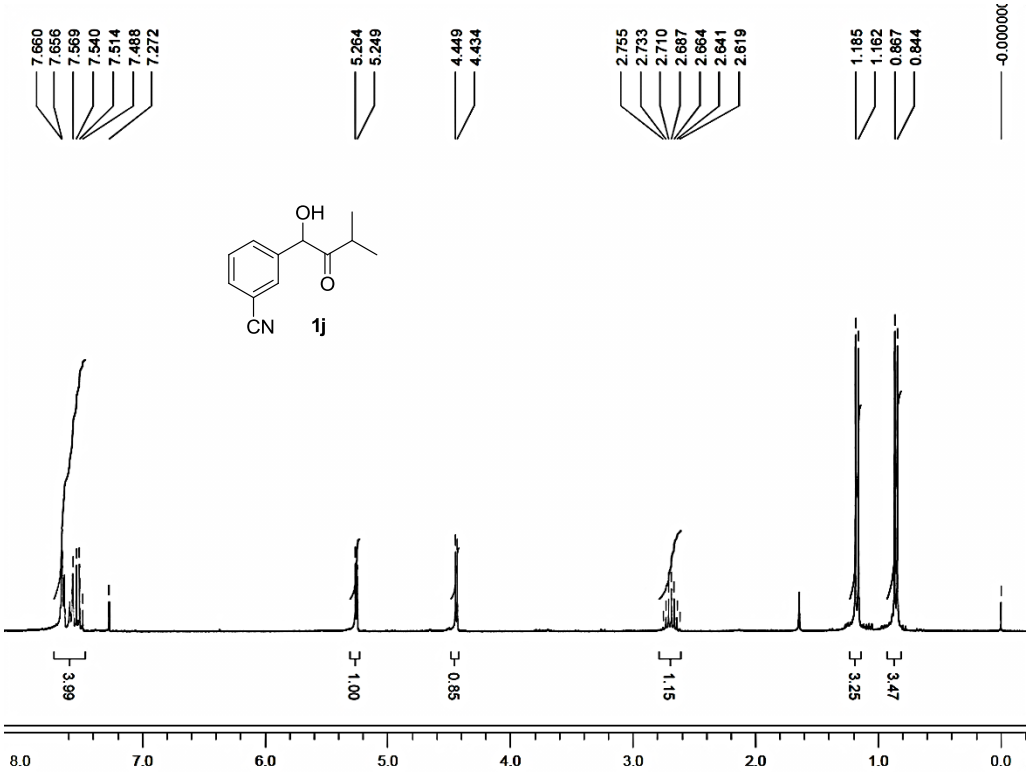


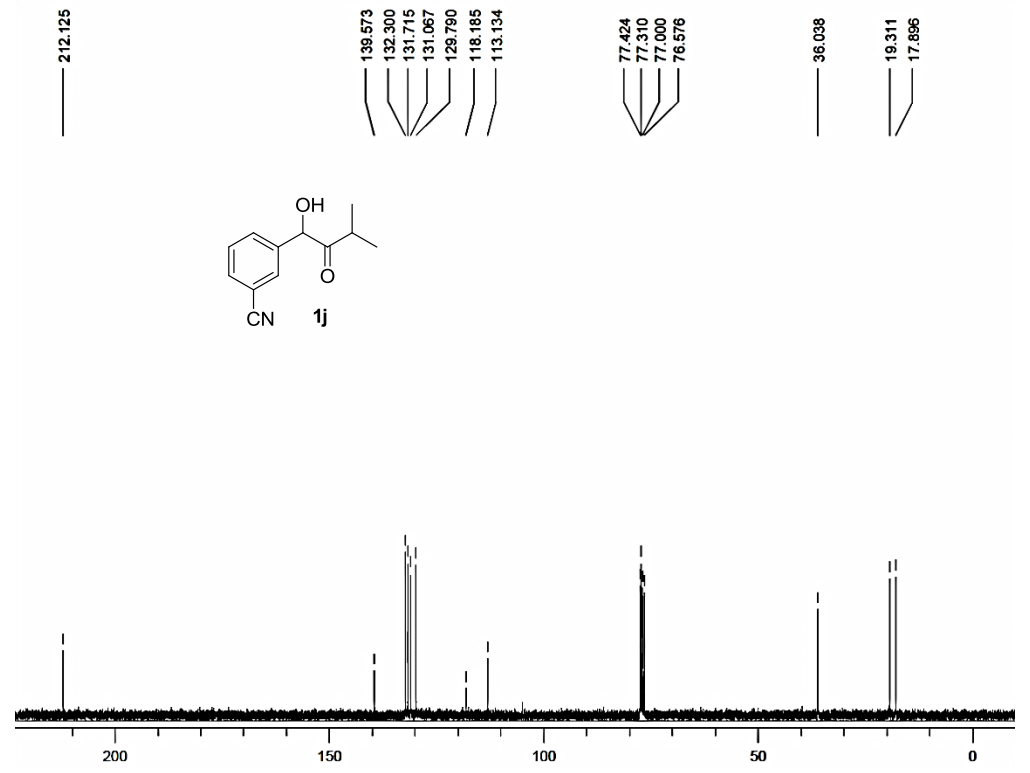


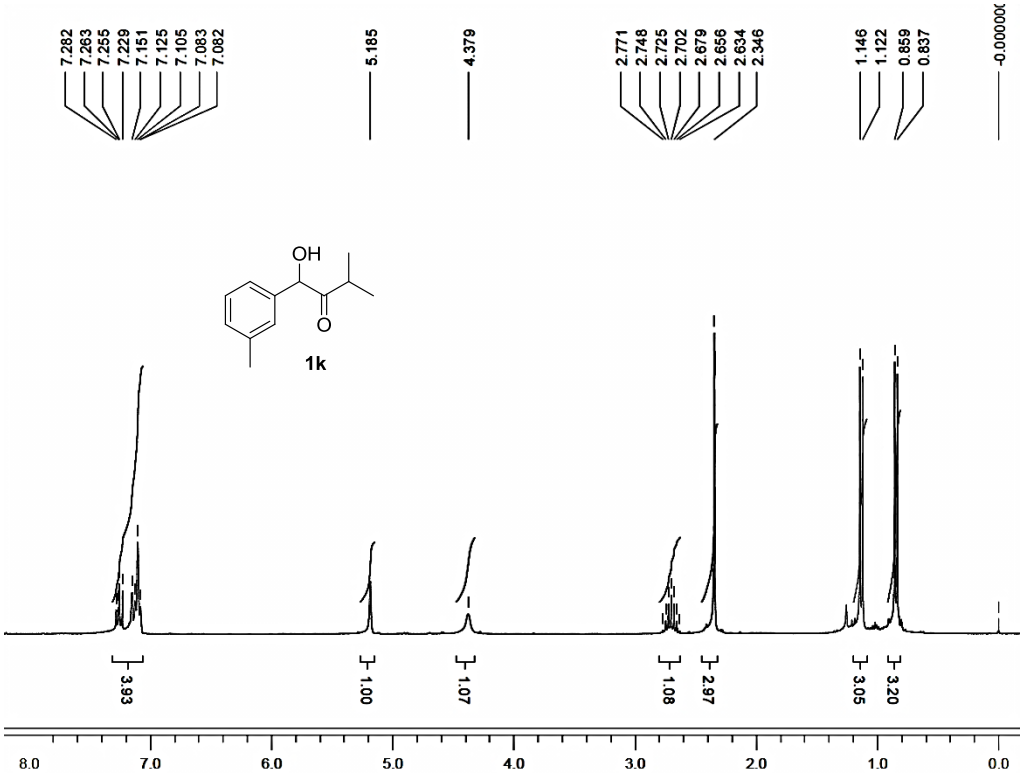


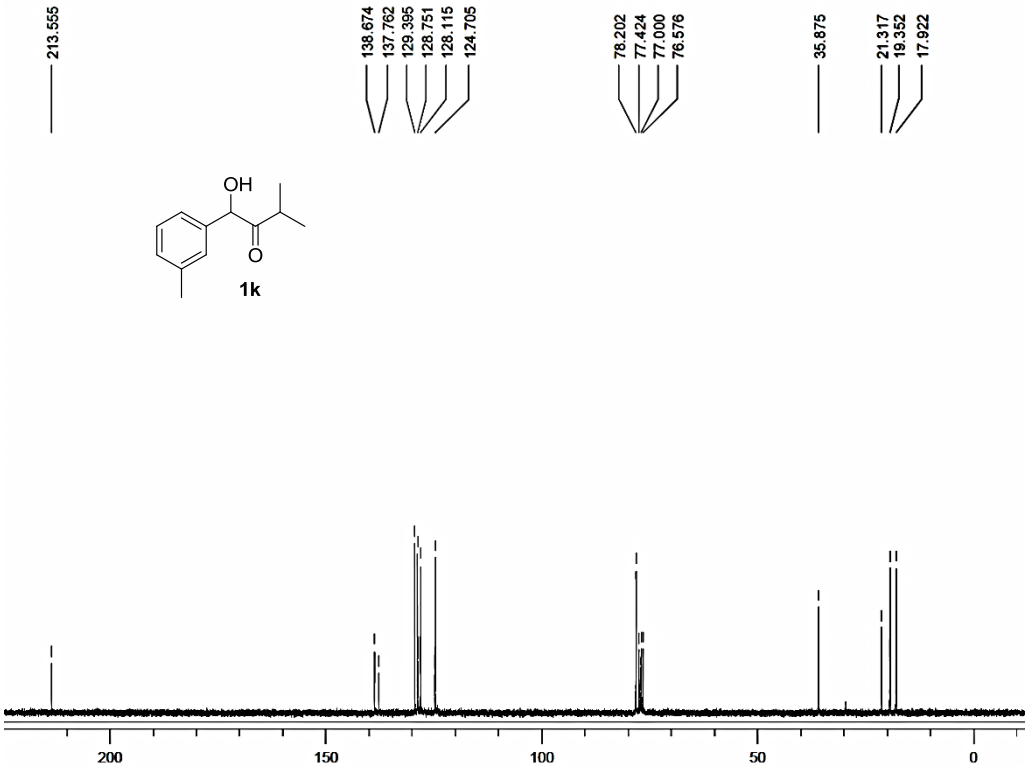


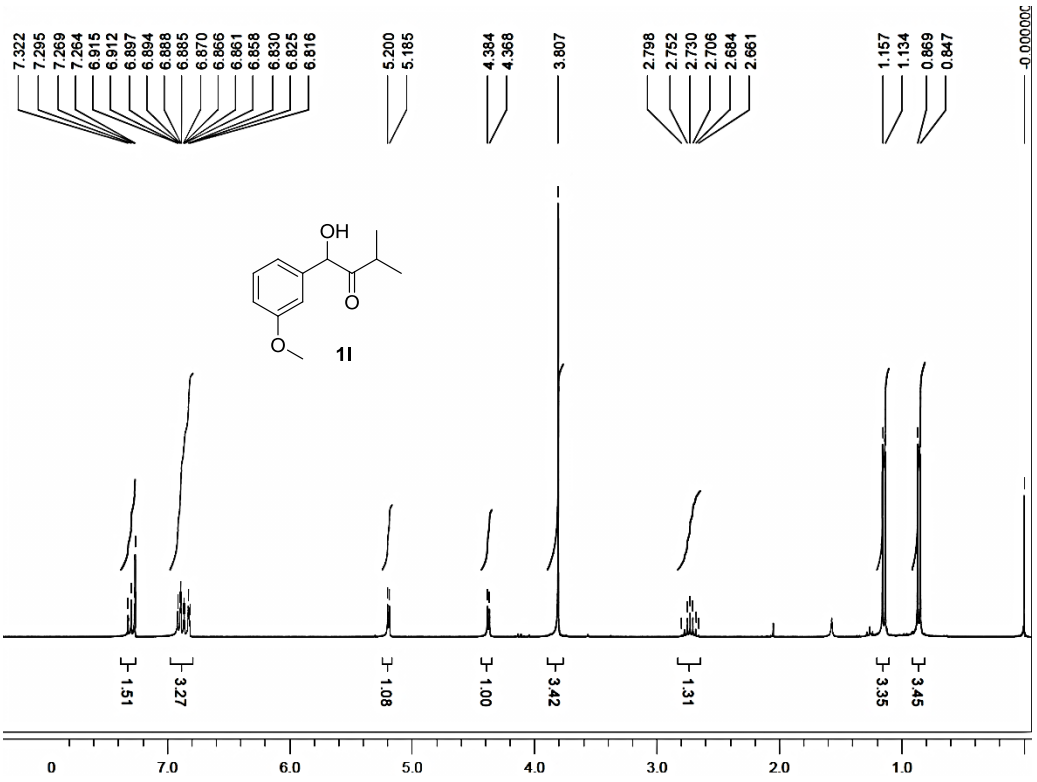


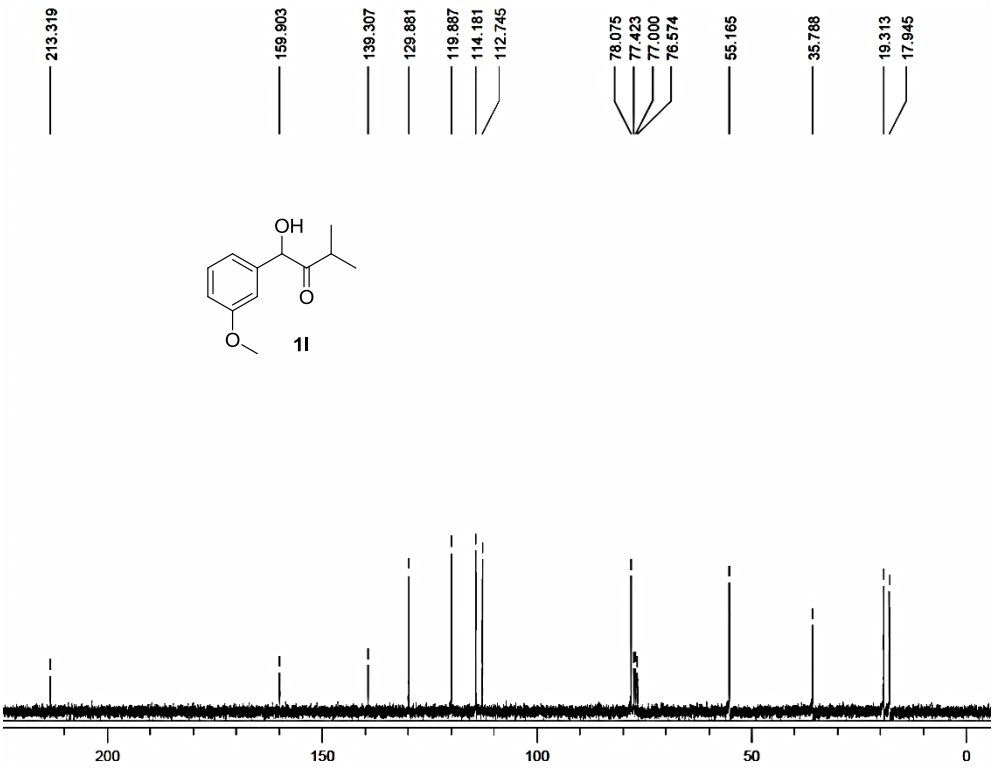


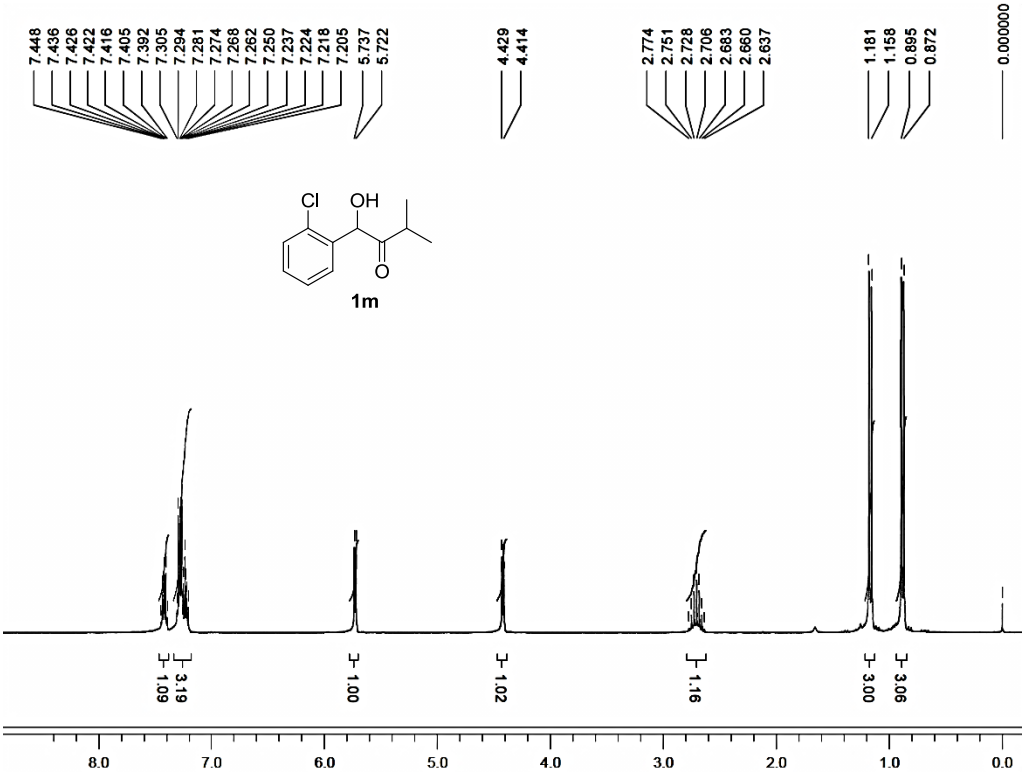


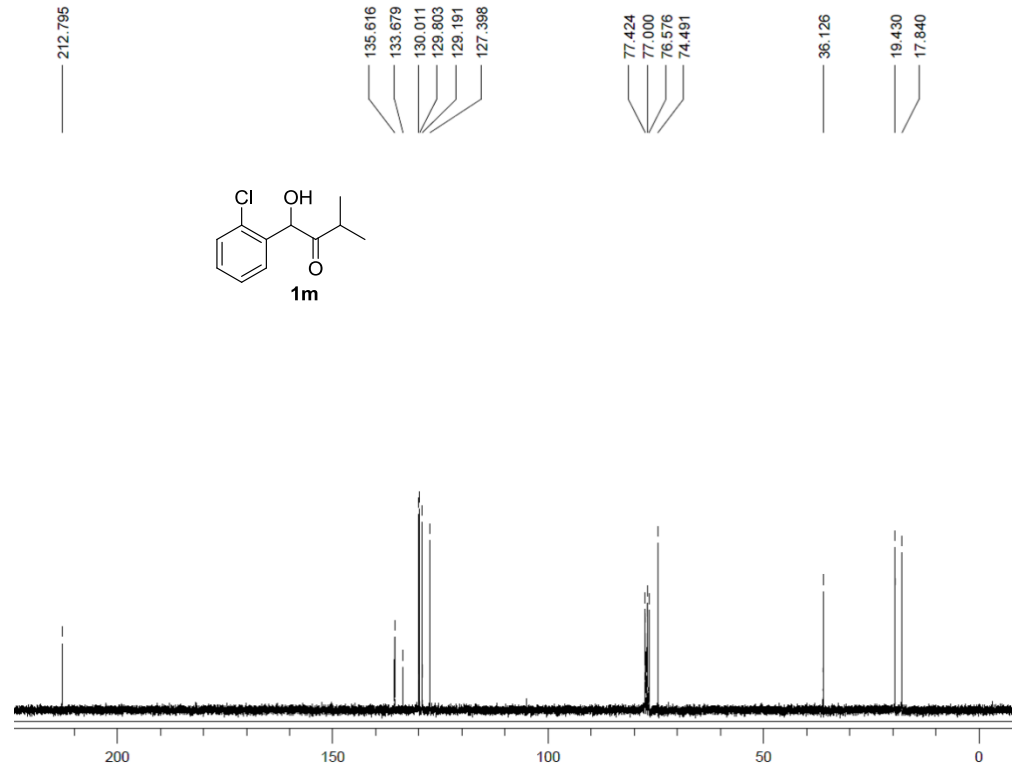


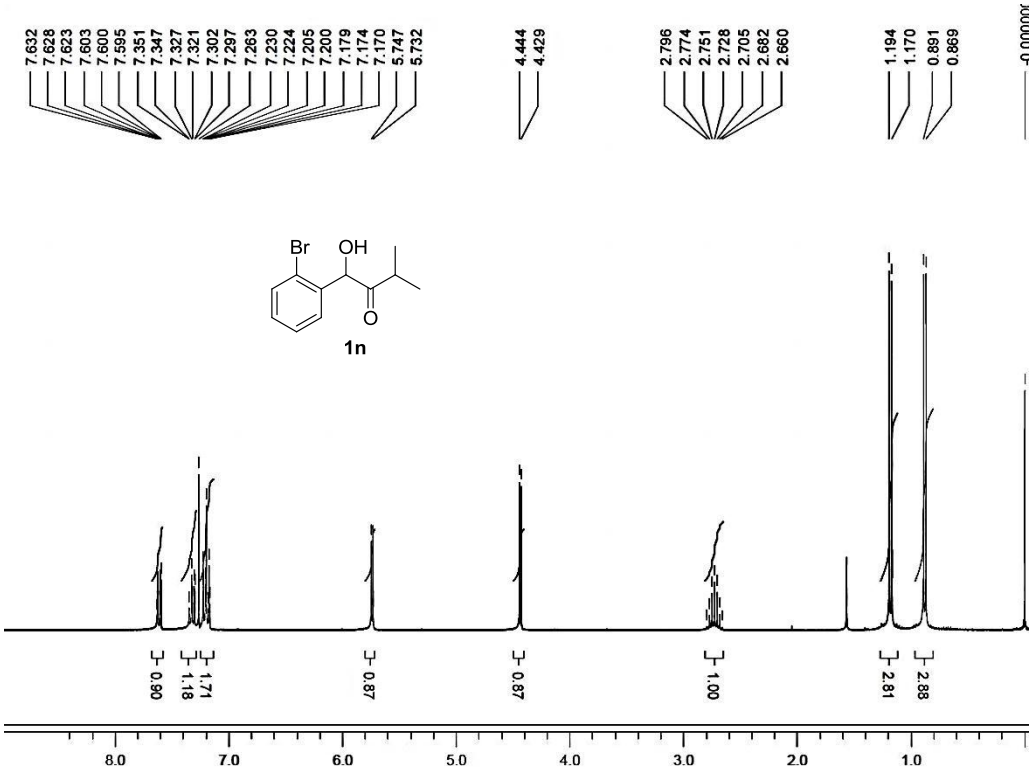


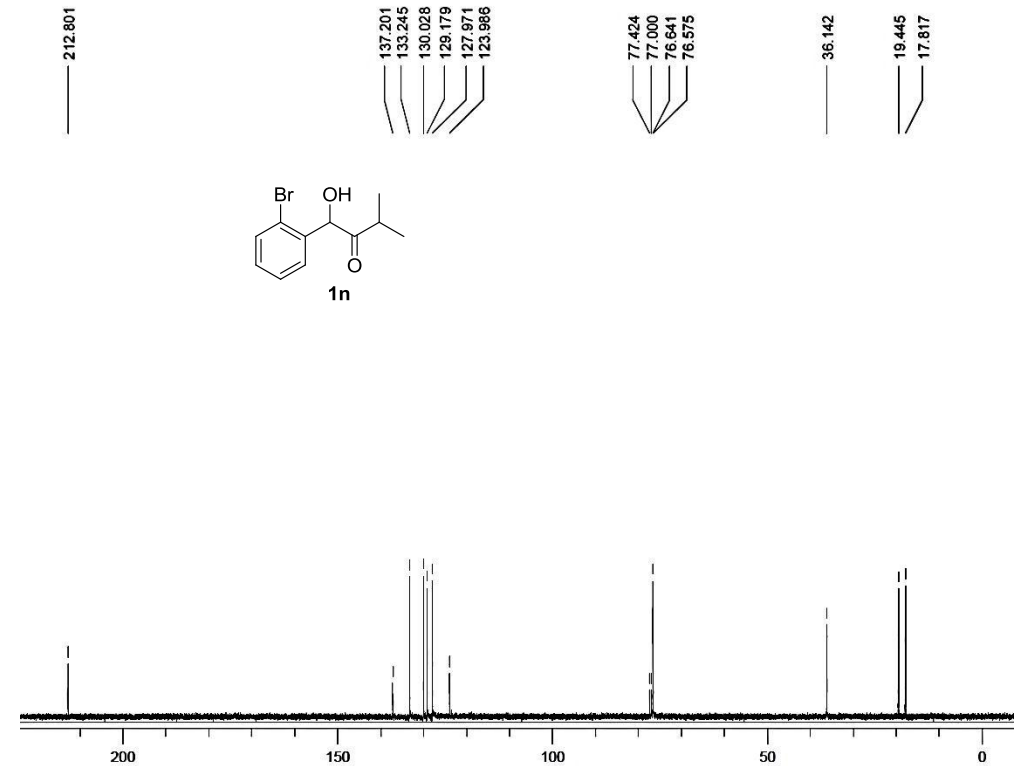


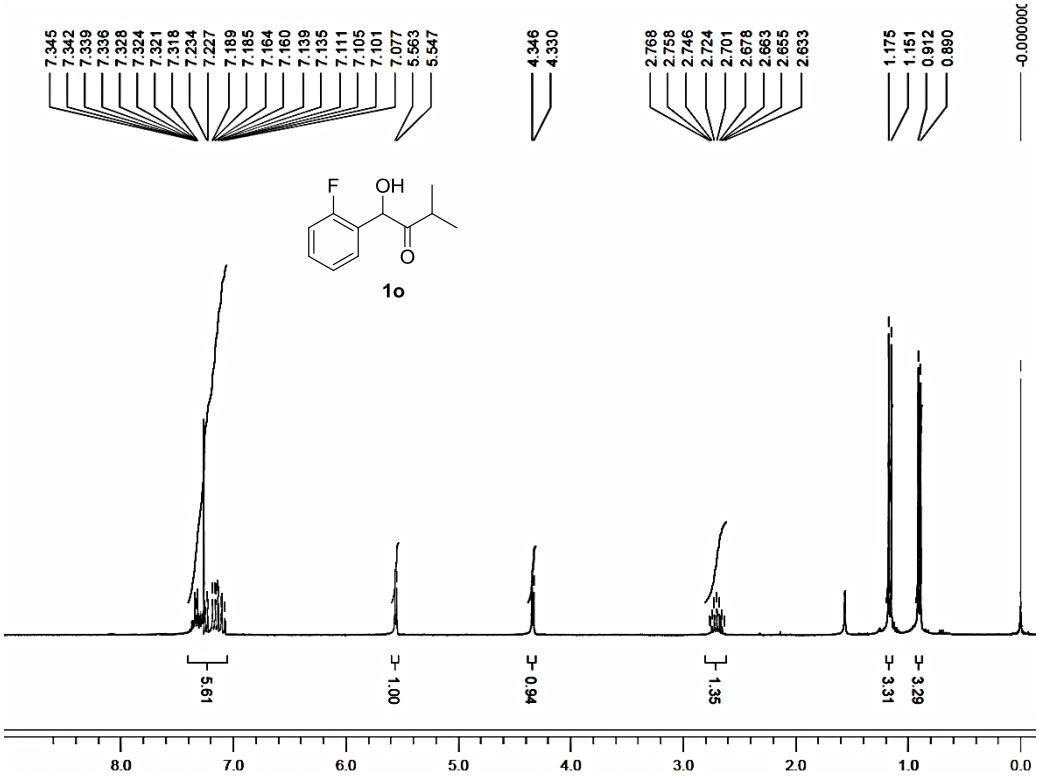








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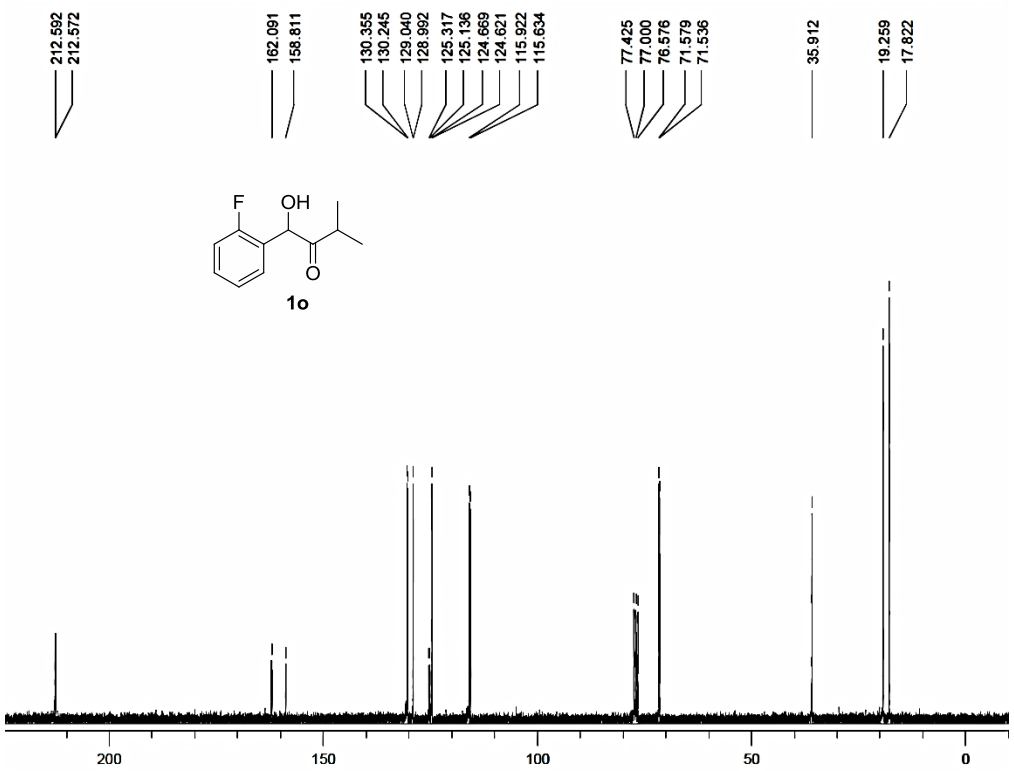
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