Supporting Information

Efficient synthesis of tertiary α-hydroxy ketones through CO_2-promoted regioselective hydration of propargylic alcohols

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A. General methods

^{1}H and ^{13}C NMR spectra were recorded using a Bruker Avance 400 MHz NMR spectrometer. The chemical shifts are referenced to signals at 7.26 and 77.0 ppm, respectively, and chloroform is solvent with TMS as the internal standard. Mass spectra were recorded on a Shimadzu GCMS-QP5050A spectrometer at an ionization voltage of 70 eV equipped with a DB-WAX capillary column (internal diameter: 0.25 mm, length: 30 m). GC–MS was obtained using electron ionization. The data of HRMS was carried out on a high-resolution mass spectrometer (LCMS-IT-TOF). IR spectra were obtained either as potassium bromide pellets or as liquid films between two potassium bromide pellets with a Bruker Vector 22 spectrometer. Melting points were determined with a Büchi Melting Point B-545 instrument. Compounds **1b-p** were synthesized according to the literature procedures.[1] Other substrates were commercially purchased and used without further purification.

B. General procedure for the preparation of α-hydroxy ketones (2a-2t)

To a 15 mL polytetrafluoroethylene (PTFE) reaction vessel, the mixture of propargylic alcohol **1** (0.5 mmol), AgOAc (0.05 mmol), DBU (0.25 mmol), H_2O (0.3 mL) and MeCN (1.0 mL) was added successively. The vessel was fixed into a stainless steel autoclave with a pressure-regulating system. Then the autoclave was sealed and CO_2 was introduced from a cylinder. The reaction was carried out at the selected temperature under magnetic stirring for 24 h and the pressure was kept constant during the reaction. As the reaction was completed, the vessel was cooled with an ice bath and the pressure was released slowly to atmospheric pressure. The residual material was diluted with diethyl ether (30 mL), dried over anhydrous $MgSO_4$ and then filtered. The volatile compounds were removed in *vacuo* and the crude residue was separated by column chromatography on silica gel to give the desired product **2a-2t**.

C. Procedure for the preparation of the cyclic carbonate 6a from 1a

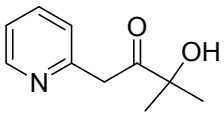
To a 15 mL polytetrafluoroethylene (PTFE) reaction vessel, the mixture of propargylic alcohol **1a** (0.5 mmol), AgOAc (0.05 mmol), DBU (0.25 mmol), and extra dry MeCN (1.0 mL) was added successively. The vessel was fixed into a stainless steel autoclave with a

pressure-regulating system. Then the autoclave was sealed and CO_2 was introduced from a cylinder. The reaction was carried out at 25°C under magnetic stirring for 24 h and the pressure was kept constant during the reaction. As the reaction was completed, the vessel was cooled with an ice bath and the pressure was released slowly to atmospheric pressure. The residual material was diluted with diethyl ether and then filtered. The volatile compounds were removed in *vacuo* and the crude residue was separated by column chromatography on silica gel (petroleum / EtOAc = 4:1) to give **6a** in 96% yield.

D. Procedure for the preparation of 2a from 6a

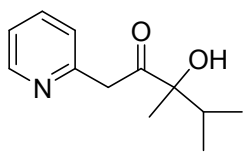
To a 15 mL Schlenk tube, the mixture of **6a** (0.24 mmol), DBU (0.5 equiv.), H_2O (0.3 mL) and MeCN (1.0 mL) was added successively. After sealing the tube, the reaction was carried out at 90°C under magnetic stirring for 18 h. As the reaction was completed, the residual material was diluted with diethyl ether (30 mL), dried over anhydrous $MgSO_4$ and then filtered. The volatile compounds were removed in *vacuo* and the crude residue was separated by column chromatography on silica gel (petroleum / EtOAc = 2:1) to give **2a** in 95% yield.

E. Analytical data



3-hydroxy-3-methyl-1-(pyridin-2-yl)butan-2-one (2a)

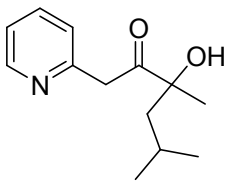
1a (80.5mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 °C under 2 MPa of CO_2 for 24 h. **2a** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) as a pale yellow oil in 91% yield (81.4 mg, 0.455 mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 89:11 ratio, and only NMR data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.50 (d, J = 4.4 Hz, 1 H), 7.69 (td, J = 7.7, 1.6 Hz, 1 H), 7.26 (d, J = 7.8 Hz, 1 H), 7.21 (dd, J = 7.1, 5.2 Hz, 1 H), 4.16 (s, 2 H), 1.37 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.6, 155.3, 148.7, 137.4, 124.3, 122.1, 76.79, 46.6, 26.8. IR (KBr): 3475, 2981, 2879, 1715, 1595, 1460, 1313, 1157, 989, 750 cm^{-1}. HRMS EI (m/z): calcd for $C_{10}H_{14}NO_2$, 180.1019 $[M + H]^+$; found 180.1029.



3-hydroxy-3,4-dimethyl-1-(pyridin-2-yl)pentan-2-one (2b)

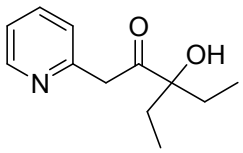
1b (94.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 °C under 2 MPa of CO_2 for 24 h. **2b** was obtained after purification by column

chromatography on silica gel (petroleum / EtOAc = 2:1) as a pale yellow oil in 77% yield (72.8 mg, 0.385 mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 89:11 ratio, and only NMR data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.50 (d, J = 4.4 Hz, 1 H), 7.68 (t, J = 7.7 Hz, 1 H), 7.26 (d, J = 7.8 Hz, 1 H), 7.22 – 7.16 (m, 1 H), 4.19 (d, J = 13.9 Hz, 1 H), 4.01 (d, J = 13.9 Hz, 1 H), 2.13 – 2.02 (m, 1 H), 1.31 (s, 3 H), 1.01 (d, J = 6.7 Hz, 3 H), 0.80 (d, J = 6.8 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.7, 154.8, 148.7, 137.2, 124.3, 122.0, 81.2, 47.0, 34.8, 23.5, 17.1, 16.1. IR (KBr): 3495, 2969, 2877, 1715, 1593, 1438, 1308, 1153, 1027, 751 cm^{-1}. HRMS EI (m/z): calcd for $C_{12}H_{18}NO_2$, 208.1332 $[M + H]^+$; found 208.1330.



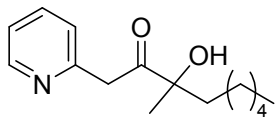
3-hydroxy-3,5-dimethyl-1-(pyridin-2-yl)hexan-2-one (2c)

1c (101.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 °C under 2 MPa of CO_2 for 24 h. **2a** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) as a pale yellow oil in 80% yield (88.4 mg, 0.400 mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 90:10 ratio, and only NMR data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.55 – 8.46 (m, 1 H), 7.69 (tt, J = 7.7, 1.9 Hz, 1 H), 7.27 (d, J = 7.8 Hz, 1 H), 7.21 (t, J = 5.6 Hz, 1 H), 4.22 (dd, J = 13.7, 1.6 Hz, 2 H), 4.07 (dd, J = 13.7, 1.3 Hz, 1 H), 1.82 – 1.75 (m, 1 H), 1.74 – 1.69 (m, 1 H), 1.65 – 1.60 (m, 1 H), 1.32 (d, J = 1.7 Hz, 3 H), 0.96 (dd, J = 6.5, 1.8 Hz, 3 H), 0.85 (dd, J = 6.5, 1.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ =212.8, 154.9, 148.7, 137.4, 124.4, 122.1, 79.7, 48.1, 47.1, 26.7, 24.4, 24.2, 23.9. IR (KBr): 3494, 2956, 2870, 1713, 1640, 1595, 1474, 1367, 1276, 1153, 1030, 750 cm^{-1}. HRMS EI (m/z): calcd for $C_{13}H_{20}NO_2$, 222.1489 $[M + H]^+$; found 222.1486.



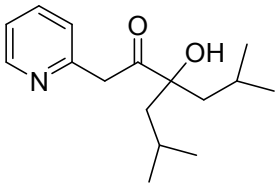
3-ethyl-3-hydroxy-1-(pyridin-2-yl)pentan-2-one (2d)

1d (94.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 °C under 2 MPa of CO_2 for 24 h. **2d** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) as a pale yellow oil in 88% yield (91.1 mg, 0.440 mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 89:11 ratio, and only NMR data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.51 (s, 1 H), 7.68 (d, J = 1.7 Hz, 1 H), 7.23 (d, J = 25.0 Hz, 2 H), 4.06 (s, 2 H), 1.87 – 1.78 (m, 2 H), 1.76 – 1.67 (m, 2 H), 0.84 – 0.75 (m, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ =211.9, 154.5, 148.8, 137.1, 124.4, 122.0, 82.5, 47.0, 31.2, 7.6. IR (KBr): 3310, 2972, 2881, 1714, 1591, 1462, 1278, 1174, 975, 749 cm^{-1}. HRMS EI (m/z): calcd for $C_{12}H_{18}NO_2$, 208.1332 $[M + H]^+$; found 208.1338.



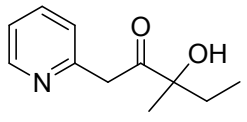
3-hydroxy-3-methyl-1-(pyridin-2-yl)nonan-2-one (2e)

1e (115.5mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 °C under 2 MPa of CO_2 for 24 h. **2e** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) as a pale yellow oil in 62% yield (77.2 mg, 0.310 mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 90:10 ratio, and only NMR data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.51 (s, 1 H), 7.68 (t, J = 7.7 Hz, 1 H), 7.26 (d, J = 7.7 Hz, 1 H), 7.23 – 7.16 (m, 1 H), 4.17 (d, J = 13.7 Hz, 1 H), 4.07 (d, J = 13.6 Hz, 1 H), 1.80 – 1.73 (m, 1 H), 1.68 – 1.60 (m, 1 H), 1.45 – 1.39 (m, 1 H), 1.34 (s, 3 H), 1.29 – 1.21 (m, 6 H), 1.14 – 1.04 (m, 1 H), 0.86 (t, J = 6.5 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ =212.5, 154.9, 148.6, 137.3, 124.3, 122.0, 79.3, 46.9, 39.8, 31.6, 29.4, 25.6, 23.4, 22.4, 13.9. IR (KBr): 3495, 2929, 2855, 1714, 1594, 1472, 1436, 1153, 750 cm^{-1}. HRMS EI (m/z): calcd for $C_{15}H_{24}NO_2$, 250.1802 $[M + H]^+$; found 250.1797.



3-hydroxy-3-isobutyl-5-methyl-1-(pyridin-2-yl)hexan-2-one (2f)

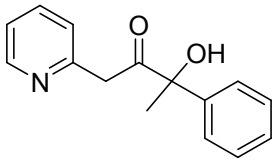
1f (122.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 °C under 2 MPa of CO_2 for 24 h. **2f** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a pale yellow oil in 71% yield (93.4 mg, 0.355mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 87:13 ratio, and only NMR data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.52 (s, 1 H), 7.68 (t, J = 7.7 Hz, 1 H), 7.30 (d, J = 7.9 Hz, 1 H), 7.24 – 7.17 (m, 1 H), 4.13 (s, 2 H), 1.71 – 1.63 (m, 6 H), 0.95 (d, J = 5.9 Hz, 6 H), 0.82 (d, J = 5.9 Hz, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.6, 154.4, 148.8, 137.1, 124.4, 122.2, 82.9, 48.0, 47.1, 24.4, 24.2. IR (KBr): 3231, 2957, 2868, 1711, 1648, 1591, 1474, 1387, 1322, 1173, 1058, 998, 752, 510 cm^{-1}. HRMS EI (m/z): calcd for $C_{16}H_{26}NO_2$, 264.1958 $[M + H]^+$; found 264.1957.



3-hydroxy-3-methyl-1-(pyridin-2-yl)pentan-2-one (2g)

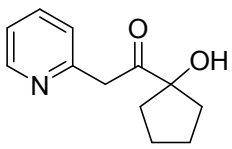
1g (87.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 °C under 2 MPa of CO_2 for 24 h. **2g** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) as a pale yellow oil in 80% yield (77.2 mg, 0.400 mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 90:10 ratio, and only NMR data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.51 (d, J = 4.4 Hz, 1 H), 7.69 (t, J = 7.7 Hz, 1 H), 7.26 (d, J = 7.7 Hz, 1 H), 7.23 – 7.17 (m, 1 H), 4.12 (q, J = 13.7 Hz, 2 H),

1.86 – 1.77 (m, 1 H), 1.73 – 1.65 (m, 1 H), 1.34 (s, 3 H), 0.86 (t, J = 7.4 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.5, 154.9, 148.7, 137.4, 124.4, 122.0, 79.5, 46.9, 32.4, 25.2, 7.8. IR (KBr): 3498, 2986, 2938, 1723, 1648, 1596, 1480, 1381, 1322, 1153, 996, 935, 751 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{16}NO_2$, 194.1176 $[M + H]^+$; found 194.1173.



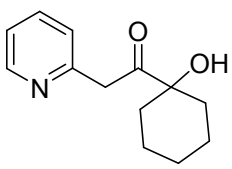
3-hydroxy-3-phenyl-1-(pyridin-2-yl)butan-2-one (2h)

1h (111.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 ^{o}C under 2 MPa of CO_2 for 24 h. **2h** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) as brown oil in 69% yield (83.1 mg, 0.345 mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 89:11 ratio, and only NMR data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.45 (d, J = 4.5 Hz, 1 H), 7.63 – 7.54 (m, 3 H), 7.34 (t, J = 7.5 Hz, 2 H), 7.26 (t, J = 7.2 Hz, 1 H), 7.20 – 7.06 (m, 2 H), 4.27 (d, J = 13.8 Hz, 1 H), 3.72 (d, J = 13.8 Hz, 1 H), 1.73 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 208.8, 155.3, 148.5, 141.9, 137.5, 128.3, 127.5, 125.5, 124.3, 122.0, 80.4, 46.3, 26.8. IR (KBr): 3467, 3059, 2978, 2932, 1720, 1635, 1596, 1475, 1441, 1366, 1319, 1154, 1071, 1030, 753, 701 cm^{-1}. HRMS EI (m/z): calcd for $C_{15}H_{16}NO_2$, 242.1176 $[M + H]^+$; found 242.1173.



1-(1-hydroxycyclopentyl)-2-(pyridin-2-yl)ethanone (2i)

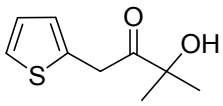
1i (93.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 ^{o}C under 2 MPa of CO_2 for 24 h. **2i** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) as a pale yellow oil in 82% yield (84.1 mg, 0.410 mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 94:6 ratio, and only NMR data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.48 (s, 1 H), 7.67 (t, J = 7.6 Hz, 1 H), 7.25 (d, J = 7.7 Hz, 1 H), 7.19 (dd, J = 6.4, 5.6 Hz, 1 H), 4.16 (s, 2 H), 2.00 – 1.85 (m, 4 H), 1.77 – 1.67 (m, 4 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.9, 155.2, 148.6, 137.5, 124.3, 122.1, 87.4, 47.9, 39.7, 25.0. IR (KBr): 3499, 2971, 2872, 1711, 1594, 1475, 1436, 1319, 1173, 1015, 751 cm^{-1}. HRMS EI (m/z): calcd for $C_{12}H_{16}NO_2$, 206.1176 $[M + H]^+$; found 206.1173.



1-(1-hydroxycyclohexyl)-2-(pyridin-2-yl)ethanone (2j)

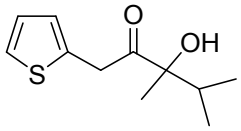
1j (100.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 90 ^{o}C under 2 MPa of CO_2 for 24 h. **2j** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) as a pale yellow oil in 78% yield (85.4 mg, 0.390 mmol). In $CDCl_3$ solution keto and enol tautomers are present in a 91:9 ratio, and only NMR

data for the major ketone form is given. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.49 (s, 1 H), 7.71 – 7.64 (m, 1 H), 7.27 – 7.16 (m, 2 H), 4.14 (s, 2 H), 1.75 – 1.66 (m, 4 H), 1.60 – 1.53 (m, 4 H), 1.41 – 1.10 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.9, 155.5, 148.6, 137.5, 124.4, 122.0, 78.5, 47.0, 34.2, 25.3, 21.0. IR (KBr): 3445, 2930, 2856, 1710, 1595, 1476, 1439, 1265, 1158, 990, 751 cm^{-1}. HRMS EI (m/z): calcd for $C_{13}H_{18}NO_2$, 220.1332 $[M + H]^+$; found 220.1330.



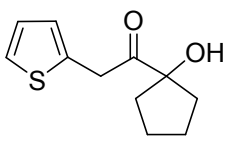
3-hydroxy-3-methyl-1-(thiophen-2-yl)butan-2-one (2k)

1k (83.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **2k** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a brown oil in 76% yield (69.9 mg, 0.380 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.22 (dd, J = 5.2, 1.1 Hz, 1 H), 6.97 (dd, J = 5.1, 3.5 Hz, 1 H), 6.90 (dd, J = 3.3, 0.8 Hz, 1 H), 4.10 (s, 2 H), 1.44 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 210.4, 134.6, 126.9, 126.8, 125.1, 76.7, 36.5, 26.5. IR (KBr): 3518, 2977, 2933, 1715, 1465, 1362, 1194, 1049, 968, 700 cm^{-1}. HRMS EI (m/z): calcd for $C_9H_{12}NaO_2S$, 207.0450 $[M + Na]^+$; found 207.0447.



3-hydroxy-3,4-dimethyl-1-(thiophen-2-yl)pentan-2-one (2l)

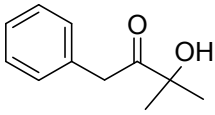
1l (97.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **2l** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a brown oil in 70% yield (74.2 mg, 0.350 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.23 (dd, J = 5.1, 1.1 Hz, 1 H), 6.97 (dd, J = 5.1, 3.5 Hz, 2 H), 6.91 (d, J = 2.6 Hz, 1 H), 4.05 (s, 2 H), 2.07 (dt, J = 13.5, 6.8 Hz, 1 H), 1.38 (s, 3 H), 1.04 (d, J = 6.7 Hz, 3 H), 0.76 (d, J = 6.8 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 210.9, 134.4, 127.0, 126.8, 125.2, 81.1, 36.8, 34.7, 23.3, 17.2, 15.8. IR (KBr): 3498, 2928, 2853, 1707, 1466, 1386, 1278, 1096, 1039, 748 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{16}NaO_2S$, 235.0763 $[M + Na]^+$; found 235.0768.



1-(1-hydroxycyclopentyl)-2-(thiophen-2-yl)ethanone (2m)

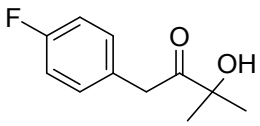
1m (96.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **2m** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a brown oil in 51% yield (53.6 mg, 0.255 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.25 (d, J = 5.1 Hz, 1 H), 7.02 – 6.96 (m, 1 H), 6.93 (d, J = 3.1 Hz, 1 H), 4.10 (s, 2 H), 2.15 – 2.06 (m, 2 H), 2.02 – 1.93 (m, 2 H), 1.89 – 1.82 (m, 2 H), 1.80 – 1.73 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 210.0, 134.9, 126.8, 126.8, 125.1, 87.4, 39.1, 37.0, 25.3. IR

(KBr): 3480, 2941, 2835, 1710, 1423, 1231, 1209, 743 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{14}NaO_2S$, 233.0607 $[M + Na]^+$; found 233.0601.



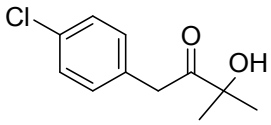
3-hydroxy-3-methyl-1-phenylbutan-2-one ($2n_1$)[2]

$1n_1$ (80.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **$2n_1$** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a yellow oil in 67% yield (59.6 mg, 0.335 mmol). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.33 (t, J = 7.2 Hz, 2 H), 7.28 (d, J = 7.0 Hz, 1 H), 7.20 (d, J = 7.3 Hz, 2 H), 3.88 (s, 2 H), 1.44 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 211.8, 133.6, 129.5, 128.6, 127.1, 76.7, 42.4, 26.6. IR (KBr): 3499, 2980, 1713, 1365, 1276, 1190, 1051, 969, 751 cm^{-1}. MS (EI) m/z: 59, 65, 91, 120, 135, 150, 161, 178.



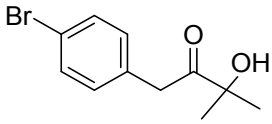
1-(4-fluorophenyl)-3-hydroxy-3-methylbutan-2-one ($2n_2$)

$1n_2$ (89.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **$2n_2$** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a yellow oil in 87% yield (85.2 mg, 0.435 mmol). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.15 (dd, J = 7.2, 5.6 Hz, 2 H), 7.01 (td, J = 8.6, 1.6 Hz, 2 H), 3.85 (s, 2 H), 1.43 (d, J = 1.8 Hz, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 211.6, 161.9, 131.0, 129.3, 115.4, 76.6, 41.5, 26.5. IR (KBr): 3512, 2978, 2935, 1723, 1605, 1511, 1465, 1364, 1225, 1225, 1159, 1054, 970, 825, 793, 521 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{13}FNaO_2$, 219.0792 $[M + Na]^+$; found 219.0791.



1-(4-chlorophenyl)-3-hydroxy-3-methylbutan-2-one ($2n_3$)

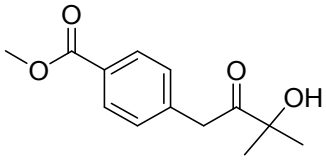
$1n_3$ (97.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **$2n_3$** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a yellow solid in 78% yield (82.7 mg, 0.390 mmol); mp: 49-50 °C. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.27 (d, J = 8.4 Hz, 2 H), 7.11 (d, J = 8.4 Hz, 2 H), 3.85 (s, 2 H), 1.40 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 211.4, 132.8, 132.1, 130.8, 128.8, 128.5, 76.6, 41.6, 26.4. IR (KBr): 3501, 2977, 2934, 1720, 1364, 1322, 1190, 1094, 1053, 970, 805, 775, 495 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{13}ClNaO_2$, 235.0496 $[M + Na]^+$; found 235.0491.



1-(4-bromophenyl)-3-hydroxy-3-methylbutan-2-one ($2n_4$)[3]

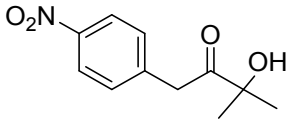
$1n_4$ (119.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **$2n_4$** was obtained after purification by

column chromatography on silica gel (petroleum / EtOAc = 10:1) in 76% yield (90.4 mg, 0.380 mmol). Yellow solid; mp 57-58 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.47 – 7.42 (m, 2 H), 7.07 (d, J = 8.4 Hz, 2 H), 3.83 (s, 2 H), 1.43 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 211.2, 132.6, 131.62, 131.2, 121.1, 76.7, 41.7, 26.49. IR (KBr): 3510, 2978, 2933, 1715, 1489, 1364, 1322, 1191, 1049, 1012, 969, 771, 673, 489 cm^{-1}. MS (EI) m/z: 59, 89, 170, 198, 213, 256.



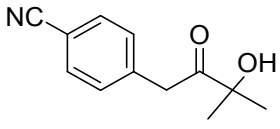
methyl 4-(3-hydroxy-3-methyl-2-oxobutyl)benzoate ($2n_5$)

$1n_5$ (109.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **$2n_5$** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) in 85% yield (100.3 mg, 0.425 mmol). Yellow solid; mp 63-64 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.99 (d, J = 8.2 Hz, 2 H), 7.27 (d, J = 8.1 Hz, 2 H), 3.95 (s, 2 H), 3.90 (s, 3 H), 1.43 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 211.1, 166.8, 139.0, 129.7, 129.6, 128.9, 76.8, 52.0, 42.4, 26.5. IR (KBr): 3525, 2980, 1729, 1611, 1438, 1367, 1277, 1186, 1112, 1052, 968, 747, 564, 482 cm^{-1}. HRMS EI (m/z): calcd for $C_{13}H_{16}NaO_4$, 259.0941 $[M + Na]^+$; found 259.0940.



3-hydroxy-3-methyl-1-(4-nitrophenyl)butan-2-one ($2n_6$)

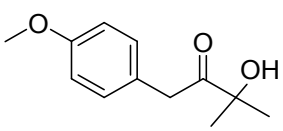
$1n_6$ (102.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **$2n_6$** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) in 80% yield (89.2 mg, 0.400 mmol). Brown solid; mp 103-104 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.19 (dd, J = 8.6, 1.8 Hz, 2H), 7.38 (d, J = 8.4 Hz, 2H), 4.03 (s, 2H), 1.47 (d, J = 1.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 210.4, 147.1, 141.3, 130.5, 123.7, 76.9, 42.1, 26.6. IR (KBr): 3538, 3081, 2978, 2935, 1709, 1604, 1515, 1348, 1189, 1110, 970, 859, 734 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{13}NaO_4$, 246.0737 $[M + Na]^+$; found 246.0731.



4-(3-hydroxy-3-methyl-2-oxobutyl)benzonitrile ($2n_7$)

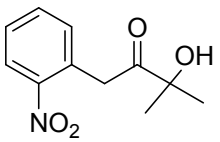
$1n_7$ (92.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **$2n_7$** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a yellow oil in 90% yield (81.2 mg, 0.450 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.62 (d, J = 7.5 Hz, 2 H), 7.32 (d, J = 7.8 Hz, 2 H), 3.98 (s, 2 H), 1.45 (s, 6 H). ^{13}C NMR (101 MHz, $CDCl_3$): δ = 210.6, 139.3, 132.2, 130.4, 118.6, 110.9,

76.8, 42.3, 26.5. IR (KBr): 3470, 2981, 2231, 2028, 1716, 1635, 1380, 1101, 741, 617 cm^{-1}. HRMS EI (m/z): calcd for $C_{12}H_{13}NNaO_2$, 226.0838 $[M + Na]^+$; found 226.0844.



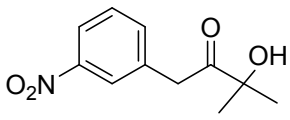
3-hydroxy-1-(4-methoxyphenyl)-3-methylbutan-2-one ($2n_8$)[4]

$1n_8$ (95.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **$2n_8$** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a yellow oil in 61% yield (63.4 mg, 0.305 mmol). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.12 (d, J = 8.1 Hz, 2 H), 6.87 (d, J = 7.9 Hz, 2 H), 3.82 (s, 2 H), 3.80 (s, 3 H), 1.43 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.1, 158.7, 130.4, 125.5, 114.1, 76.6, 55.2, 41.5, 26.6. IR (KBr): 3495, 2976, 2934, 1714, 1612, 1513, 1464, 1248, 1179, 1034, 789 cm^{-1}. MS (EI) m/z: 59, 77, 91, 107, 121, 150, 208.



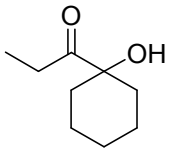
3-hydroxy-3-methyl-1-(2-nitrophenyl)butan-2-one (2o)

1o (102.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **2o** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a brown oil in 77% yield (85.9 mg, 0.385 mmol). ^1H NMR (400 MHz, $CDCl_3$): δ = 8.13 (d, J = 8.2 Hz, 1 H), 7.60 (td, J = 7.5, 1.0 Hz, 1 H), 7.51 – 7.45 (m, 1 H), 7.27 (d, J = 6.2 Hz, 1 H), 4.38 (s, 2 H), 1.52 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 210.1, 148.8, 133.6, 133.5, 130.0, 128.5, 125.3, 76.8, 41.9, 26.9. IR (KBr): 3502, 3070, 2977, 2934, 1726, 1612, 1532, 1351, 1190, 1057, 968, 865, 788, 564 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{13}NaO_4$, 246.0737 $[M + Na]^+$; found 246.0739.



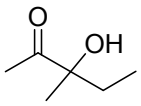
3-hydroxy-3-methyl-1-(3-nitrophenyl)butan-2-one (2p)

1p (102.5 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **2p** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a brown oil in 89% yield (99.2 mg, 0.445 mmol). ^1H NMR (400 MHz, $CDCl_3$): δ = 8.17 – 8.11 (m, 1 H), 8.08 (s, 1 H), 7.56 – 7.49 (m, 2 H), 4.04 (s, 2 H), 1.48 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 210.6, 148.3, 135.9, 135.7, 129.4, 124.6, 122.2, 76.8, 41.8, 26.6. IR (KBr): 3548, 3093, 2978, 2935, 1723, 1533, 1481, 1375, 1193, 1053, 970, 930, 806, 729, 677, 560 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{13}NaO_4$, 246.0737 $[M + Na]^+$; found 246.0736.



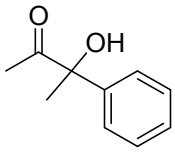
1-(1-hydroxycyclohexyl)propan-1-one (2q)[5]

1q (69.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **2q** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a pale yellow oil in 46% yield (35.9 mg, 0.230 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.59 (q, J = 7.2 Hz, 2 H), 1.74 – 1.63 (m, 6 H), 1.52 – 1.43 (m, 2 H), 1.36 – 1.19 (m, 2 H), 1.10 (t, J = 7.2 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 215.4, 77.9, 34.0, 28.9, 25.3, 21.1, 7.9. IR (KBr): 3465, 2027, 1635, 1384, 1102, 617 cm^{-1}. MS (EI) m/z: 55, 79, 81, 99, 109, 156.



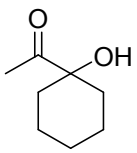
3-hydroxy-3-methylpentan-2-one (2r)[6]

1r (49.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **2r** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a pale yellow oil in 78% yield (45.2 mg, 0.390 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.23 (s, 3 H), 1.77 (q, J = 7.4 Hz, 2 H), 1.38 (s, 3 H), 0.85 (t, J = 7.4 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.4, 79.0, 32.2, 25.1, 23.6, 7.6. IR (KBr): 3320, 2945, 2853, 1739, 1668, 11463, 1383, 1260, 1090, 1021, 799, 749 cm^{-1}. MS (EI) m/z: 55, 73, 87, 101, 116.



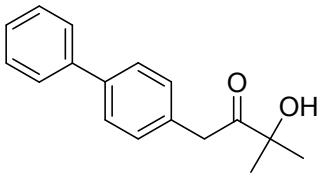
3-hydroxy-3-phenylbutan-2-one (2s)[7]

1s (73.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **2s** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as a brown oil in 72% yield (59.0 mg, 0.360 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.44 (d, J = 7.4 Hz, 2 H), 7.37 (t, J = 7.4 Hz, 2 H), 7.31 (t, J = 7.1 Hz, 1 H), 2.08 (s, 3 H), 1.78 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 209.6, 141.4, 128.7, 128.0, 125.9, 79.8, 24.0, 23.4. IR (KBr): 3464, 2981, 2934, 1711, 1448, 1355, 1198, 1135, 1169, 914, 762, 701, 555 cm^{-1}. MS (EI) m/z: 51, 77, 105, 121, 147, 164.



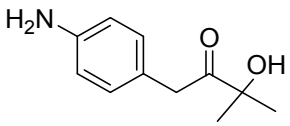
1-(1-hydroxycyclohexyl)ethanone (2t)[8]

1t (62.0 mg, 0.5 mmol) was used and the reaction mixture was stirred at 120 °C under 2 MPa of CO_2 for 24 h. **2t** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) as yellow oil in 82% yield (58.2 mg, 0.410 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.24 (s, 3 H), 1.73 – 1.64 (m, 6 H), 1.53 – 1.46 (m, 2 H), 1.39 – 1.16 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.7, 78.0, 33.8, 25.3, 23.7, 21.0. IR (KBr): 3394, 2924, 2852, 1714, 1670, 1464, 1408, 1260, 1095, 1020, 801, 749 cm^{-1}. MS (EI) m/z: 55, 79, 81, 99, 109, 125, 142.



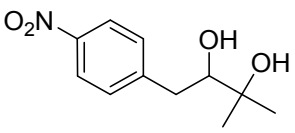
1-(biphenyl-4-yl)-3-hydroxy-3-methylbutan-2-one (3)

2n$_4$ (59.5 mg, 0.25 mmol) was used and **3** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 10:1) in 82% yield (52.0 mg, 0.205 mmol). Yellow solid; mp 84-85 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.55 (t, J = 8.4 Hz, 4 H), 7.40 (t, J = 7.6 Hz, 2 H), 7.31 (t, J = 7.3 Hz, 1 H), 7.25 (d, J = 8.1 Hz, 2 H), 3.89 (s, 2 H), 1.43 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 211.8, 140.6, 139.9, 132.6, 129.8, 128.7, 127.2, 127.2, 126.9, 76.6, 42.0, 26.5. IR (KBr): 3450, 3057, 2979, 2936, 1697, 1488, 1371, 1267, 1194, 1057, 968, 754, 693 cm^{-1}. HRMS EI (m/z): calcd for $C_{17}H_{18}NaO_2$, 277.1199 [M + Na]$^+$; found 277.1208.



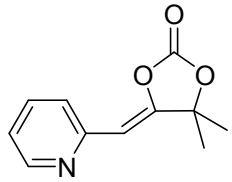
1-(4-aminophenyl)-3-hydroxy-3-methylbutan-2-one (4)

2n$_6$ (55.7 mg, 0.25 mmol) was used and **4** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) in 95% yield (45.8 mg, 0.237 mmol). Brown solid; mp 106-107 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.98 (d, J = 8.1 Hz, 2 H), 6.64 (d, J = 8.1 Hz, 2 H), 3.75 (s, 2 H), 3.65 (s, 3 H), 1.41 (s, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 212.4, 145.4, 130.2, 123.2, 115.3, 76.5, 41.6, 26.6. IR (KBr): 3491, 2976, 2029, 1711, 1631, 1518, 1210, 1101, 741, 616 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{16}NO_2$, 194.1176 [M + H]$^+$; found 194.1187.



3-methyl-1-(4-nitrophenyl)butane-2,3-diol (5)

2n$_6$ (55.7 mg, 0.25 mmol) was used and **5** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 2:1) in 70% yield (39.4 mg, 0.175 mmol). Yellow solid; mp 116-117 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.16 (d, J = 8.1 Hz, 2 H), 7.43 (d, J = 8.2 Hz, 2 H), 3.63 (d, J = 10.5 Hz, 1 H), 2.95 (d, J = 13.8 Hz, 1 H), 2.71 (dd, J = 13.6, 10.9 Hz, 1 H), 2.11 (s, 2 H), 1.30 (d, J = 13.6 Hz, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 147.4, 146.7, 130.1, 123.6, 78.8, 72.9, 38.0, 26.5, 23.8. IR (KBr): 3465, 2980, 1635, 1517, 1346, 1104, 689, 616 cm^{-1}. HRMS EI (m/z): calcd for $C_{11}H_{15}NNaO_4$, 248.0893 [M + Na]$^+$; found 248.0886.



(Z)-4,4-dimethyl-5-((pyridin-2-yl)methylene)-1,3-dioxolan-2-one (6a)[9]

2a (40.3 mg, 0.25 mmol) was used and **6a** was obtained after purification by column chromatography on silica gel (petroleum / EtOAc = 4:1) as a yellow oil in 96% yield (49.2 mg, 0.240 mmol). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.54 (d, J = 4.4 Hz, 1 H), 7.85 (d, J = 8.1 Hz, 1 H), 7.70 (td, J = 8.0, 1.5 Hz, 1 H), 7.13-7.16 (m, 1 H), 5.81 (s, 1 H), 1.70 (s, 7 H). ^{13}C

NMR (100 MHz, $CDCl_3$): δ = 153.6, 151.9, 150.8, 149.2, 136.6, 123.5, 122.0, 102.8, 85.8, 27.5. MS (EI) m/z: 64, 78, 92, 118, 132, 146, 160, 205.

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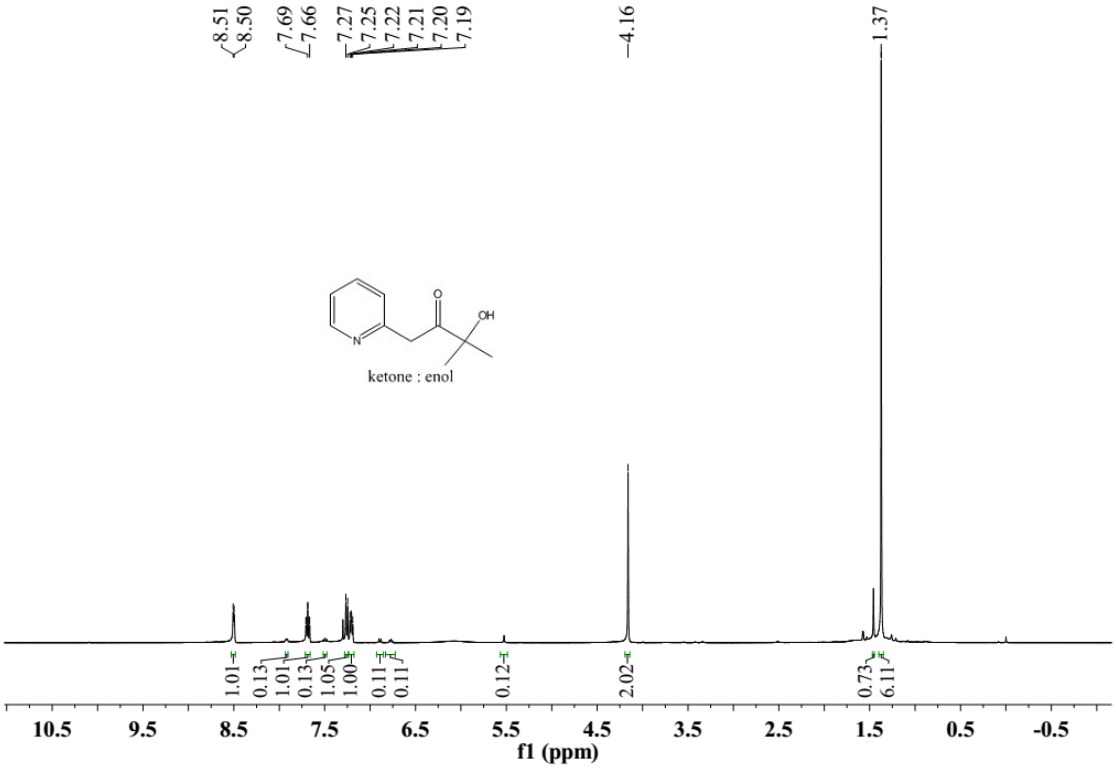
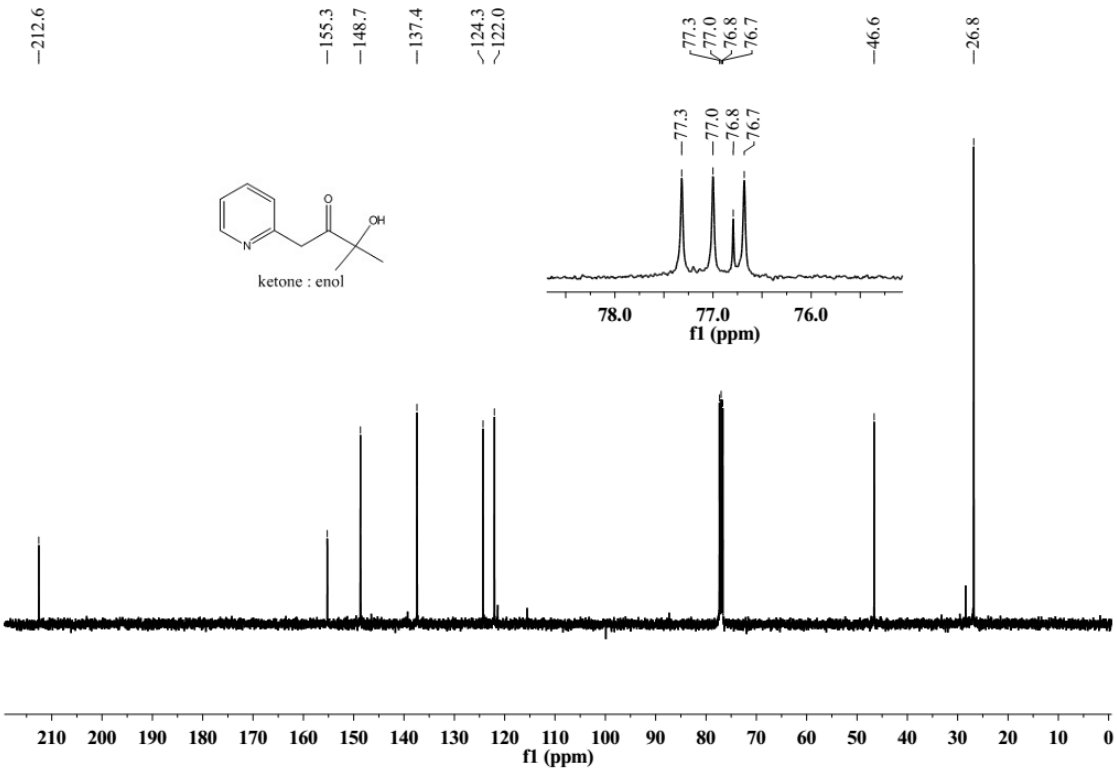
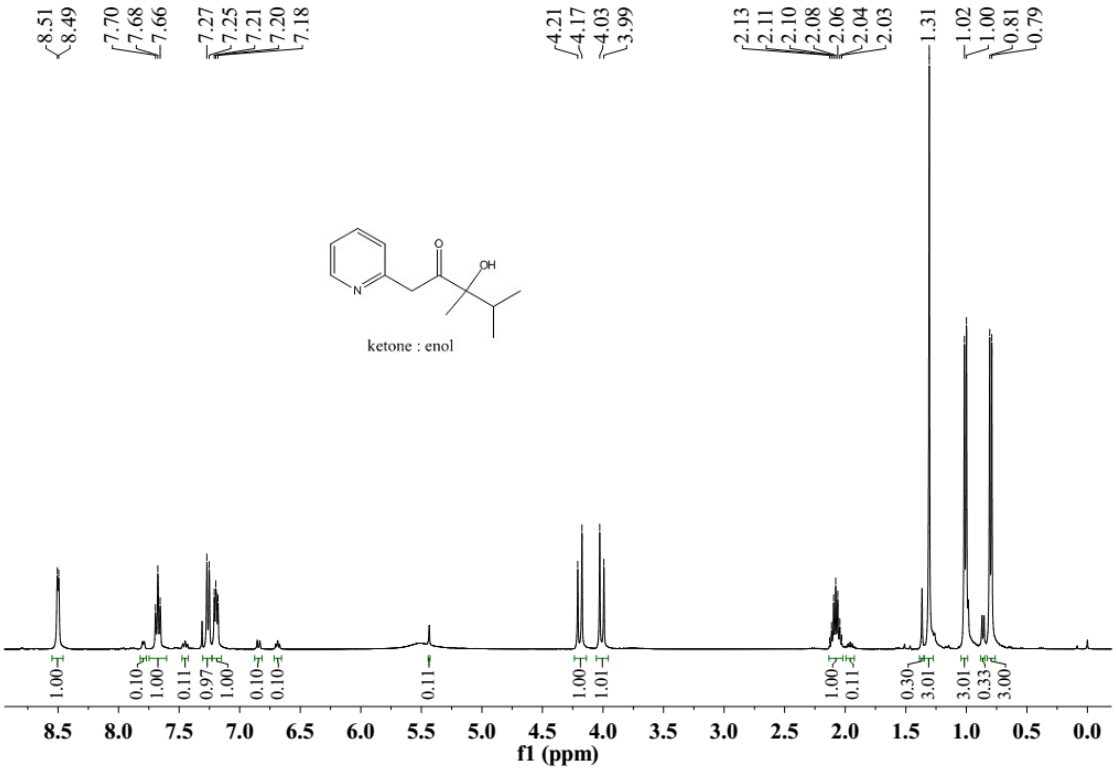
F. NMR Spectra

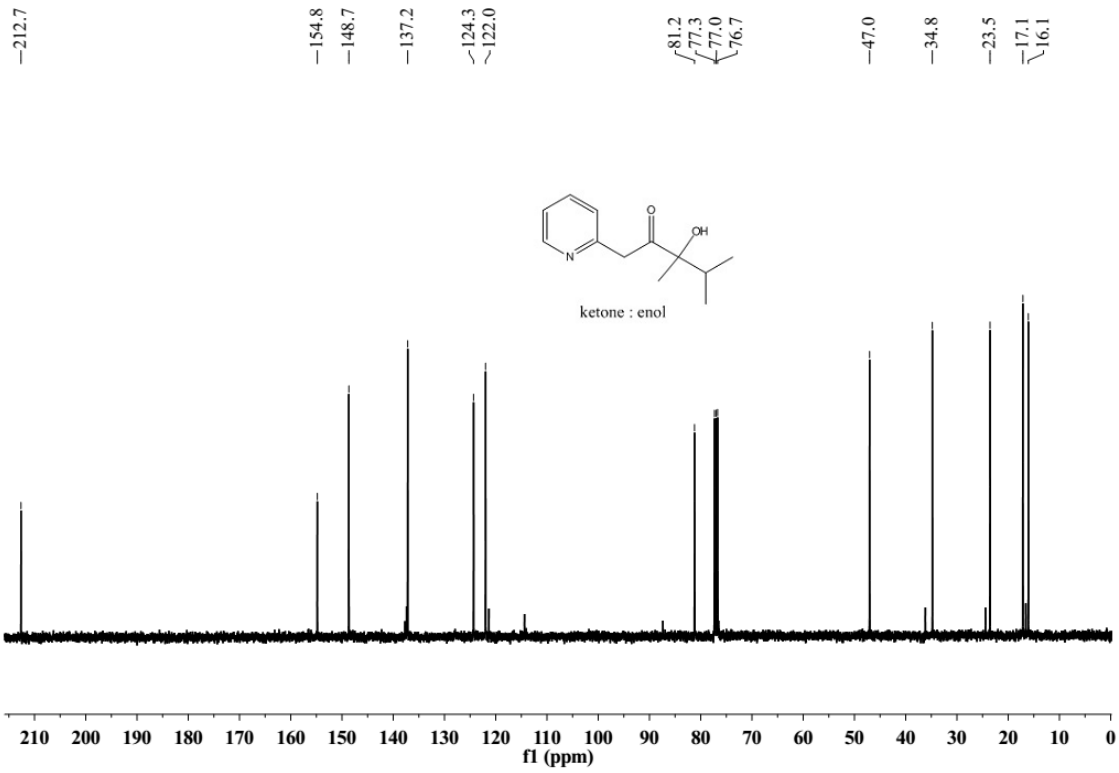
3-hydroxy-3-methyl-1-(pyridin-2-yl)butan-2-one (2a)



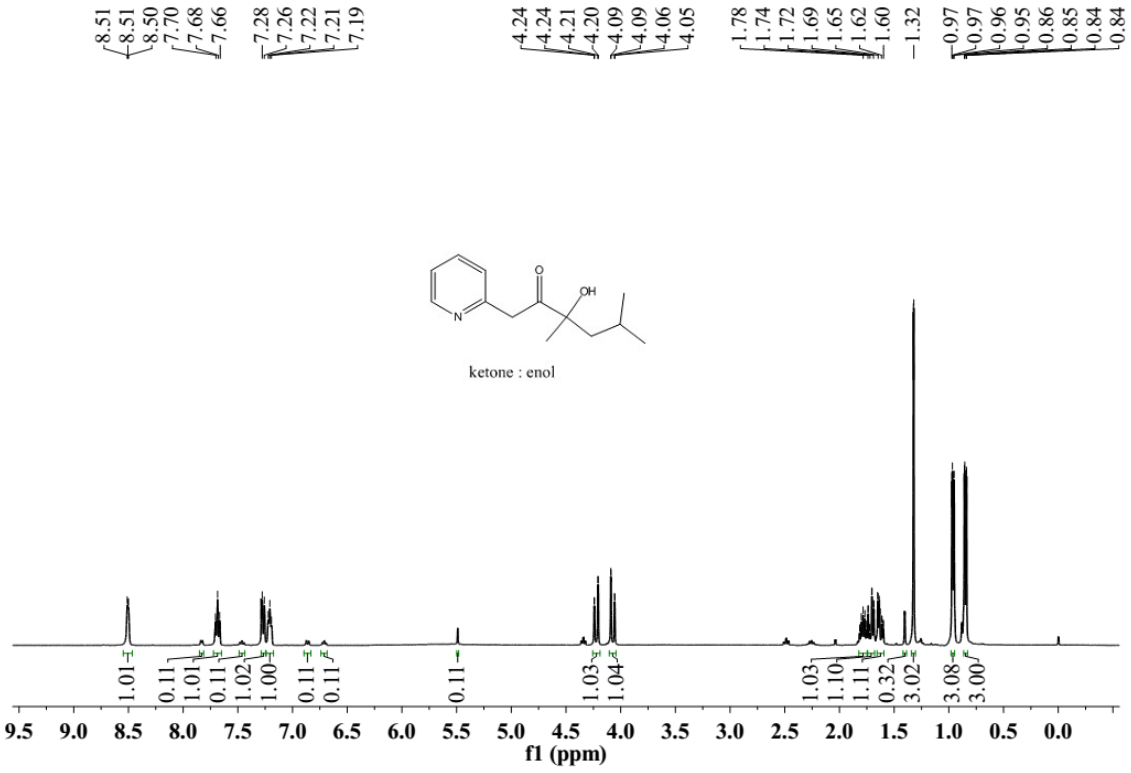


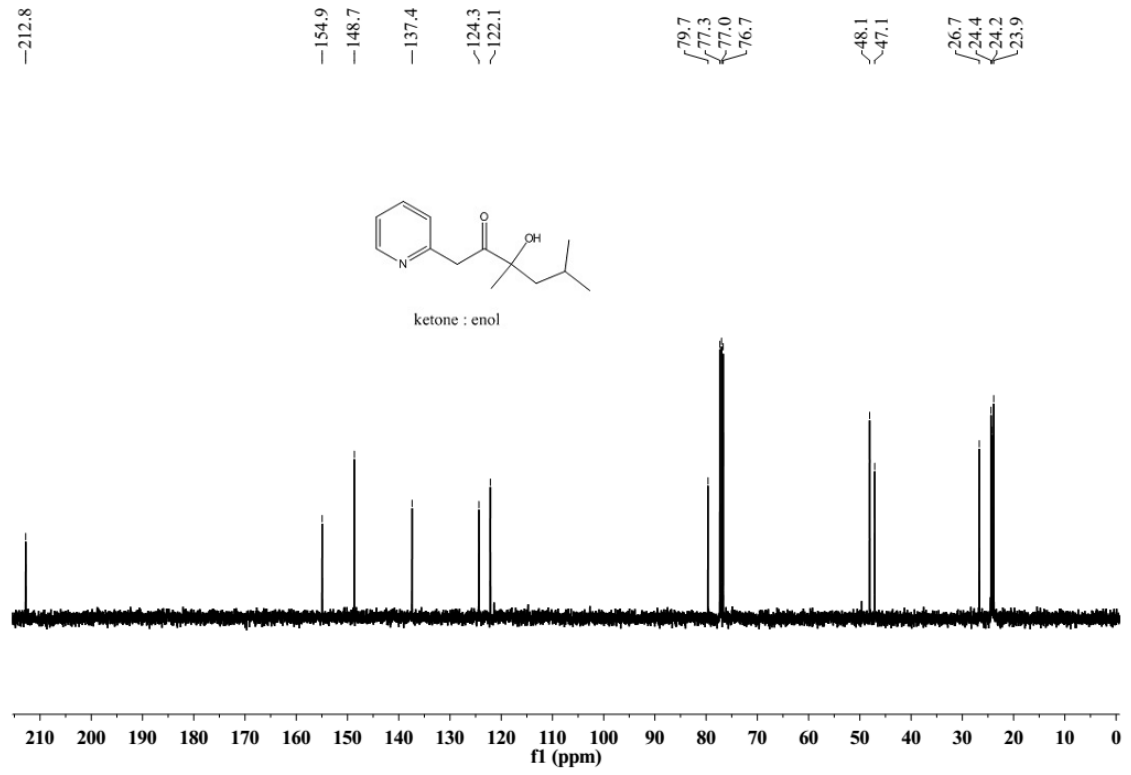
3-hydroxy-3,4-dimethyl-1-(pyridin-2-yl)pentan-2-one (2b)



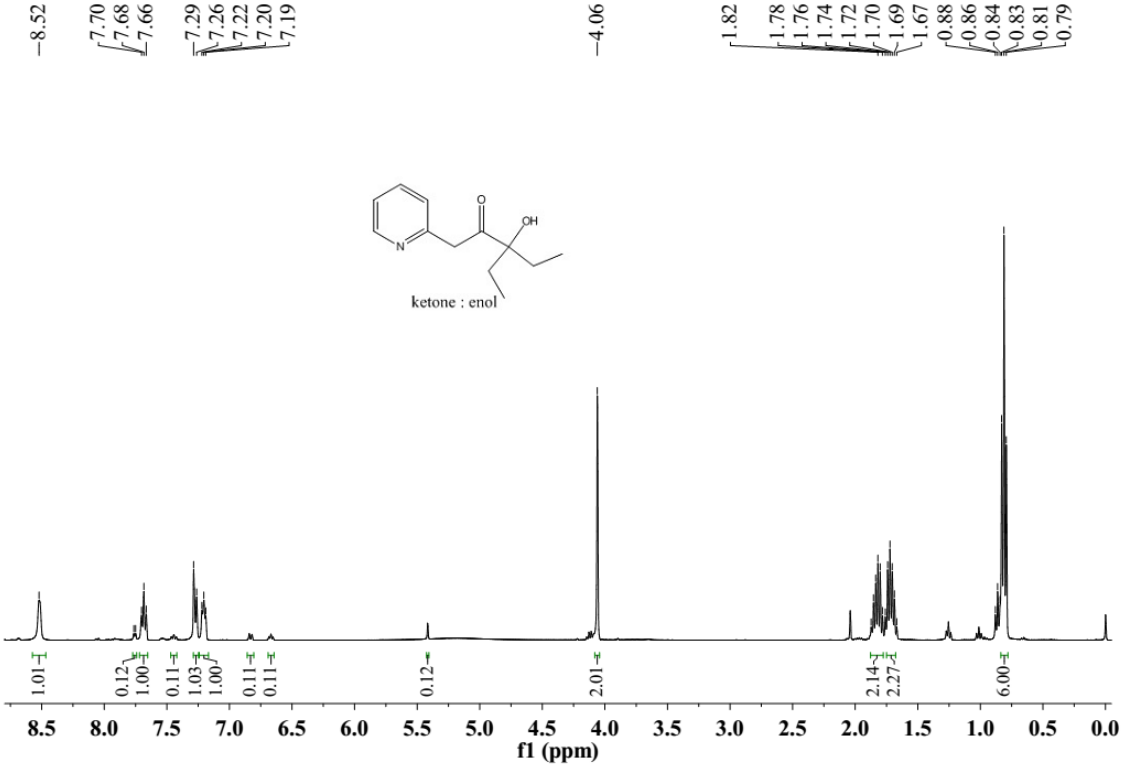


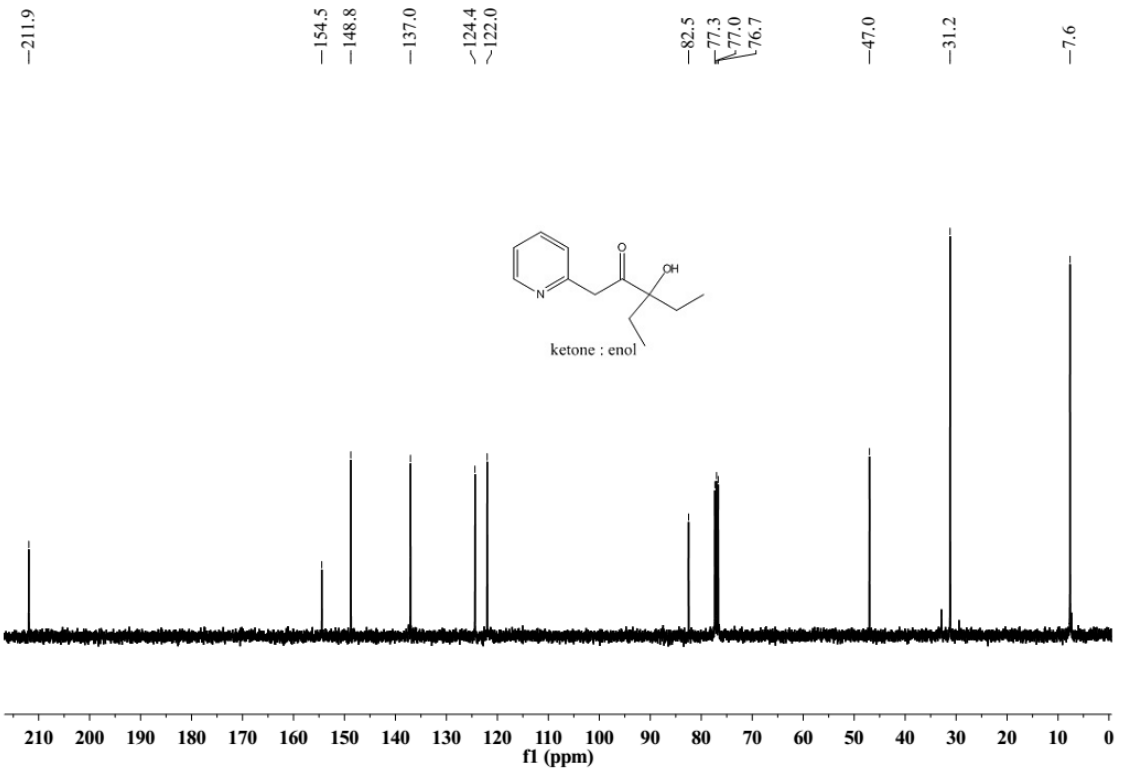
3-hydroxy-3,5-dimethyl-1-(pyridin-2-yl)hexan-2-one (2c)





3-ethyl-3-hydroxy-1-(pyridin-2-yl)pentan-2-one (2d)





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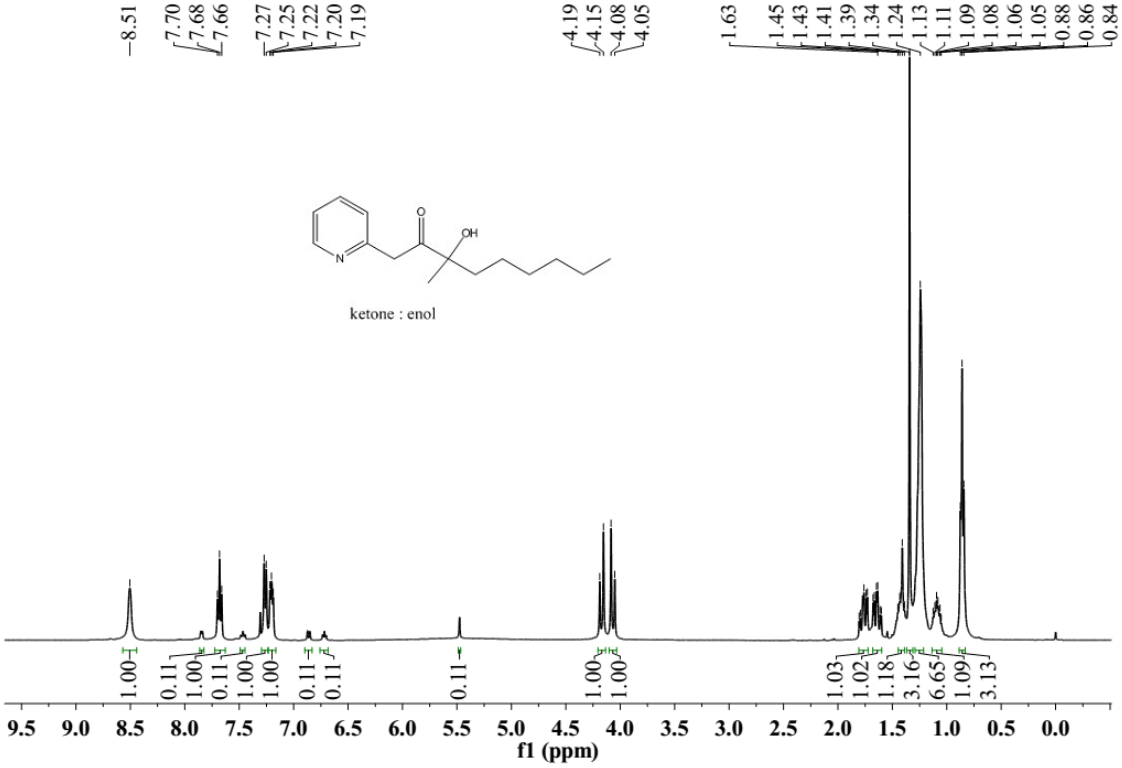

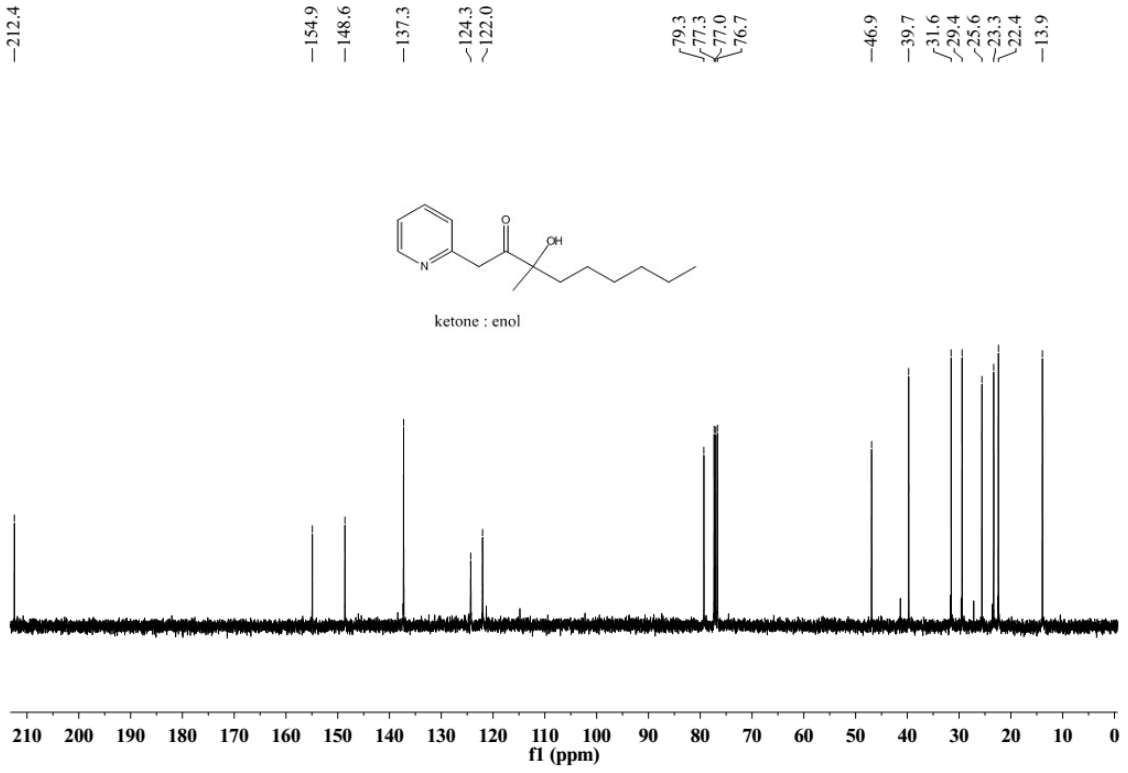

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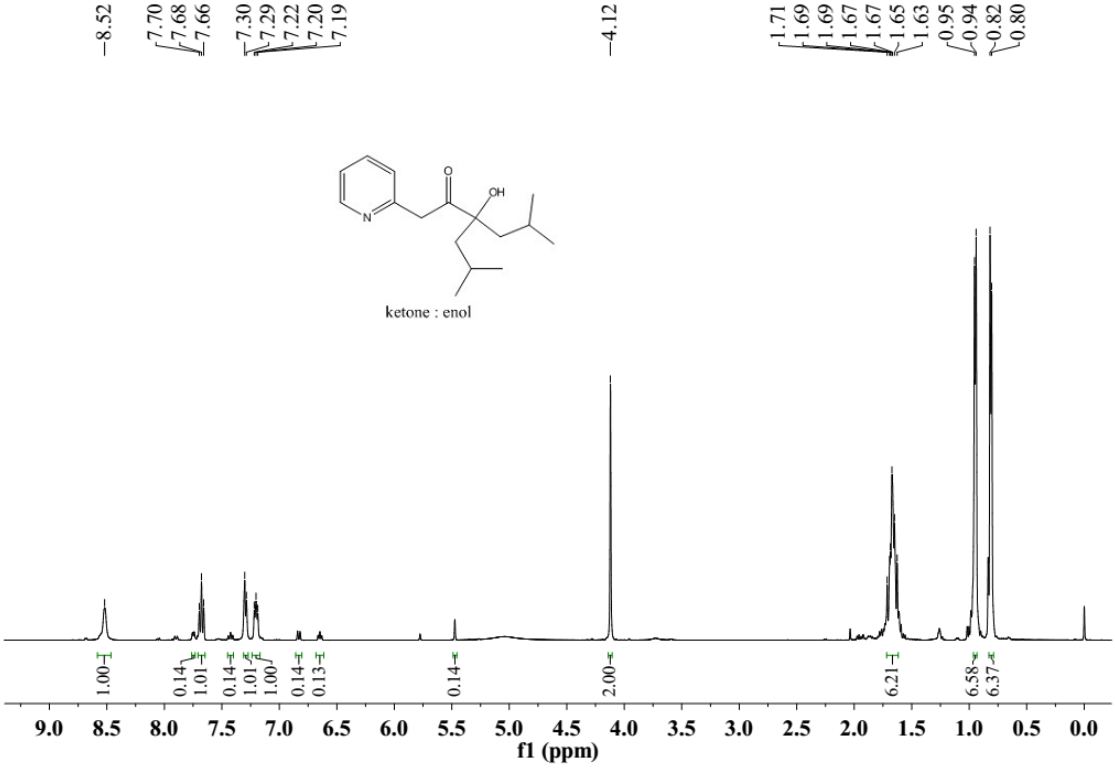
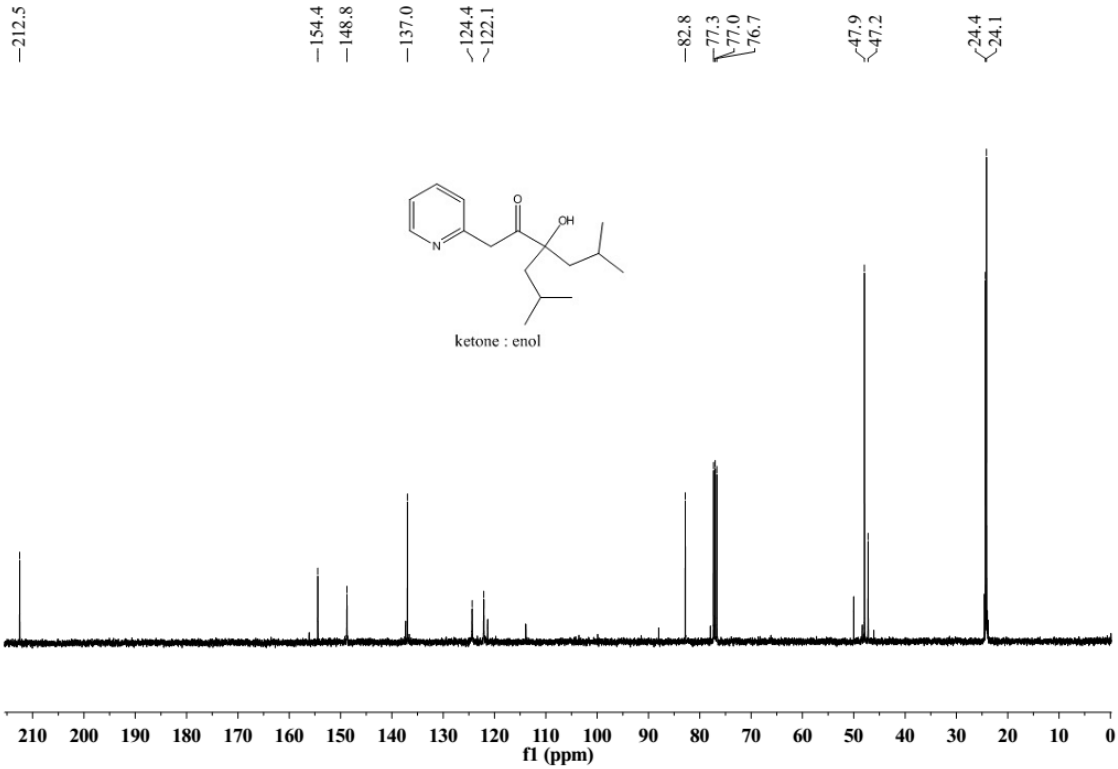
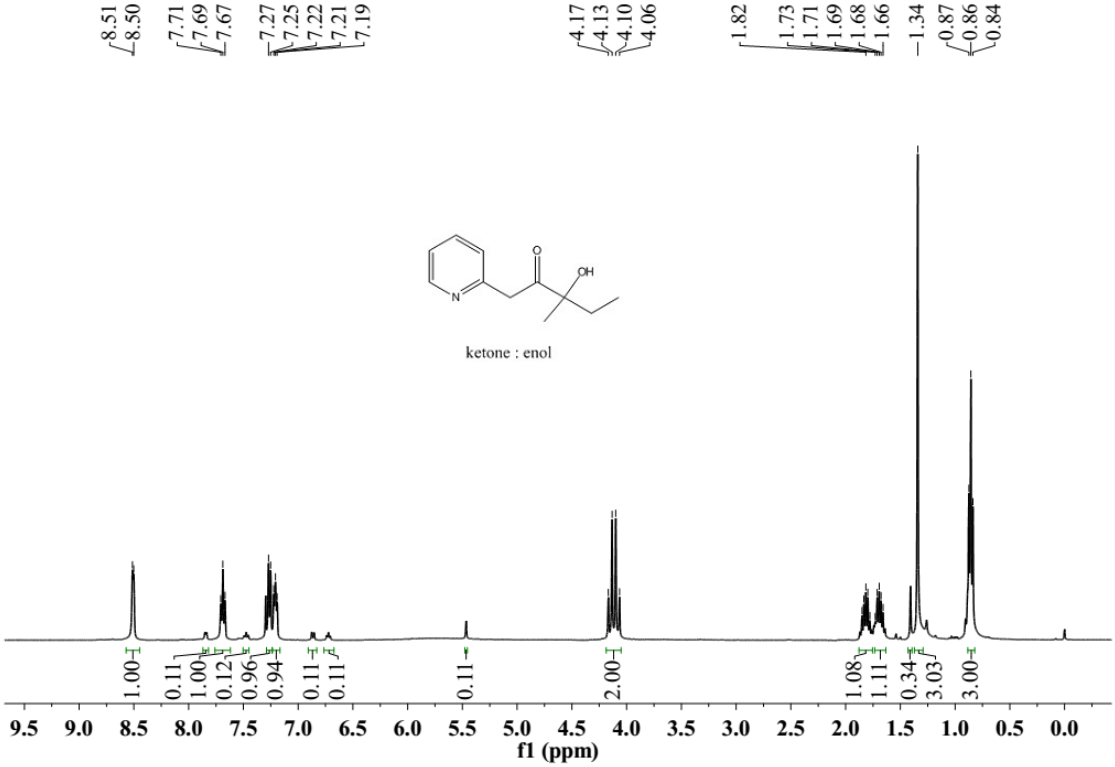
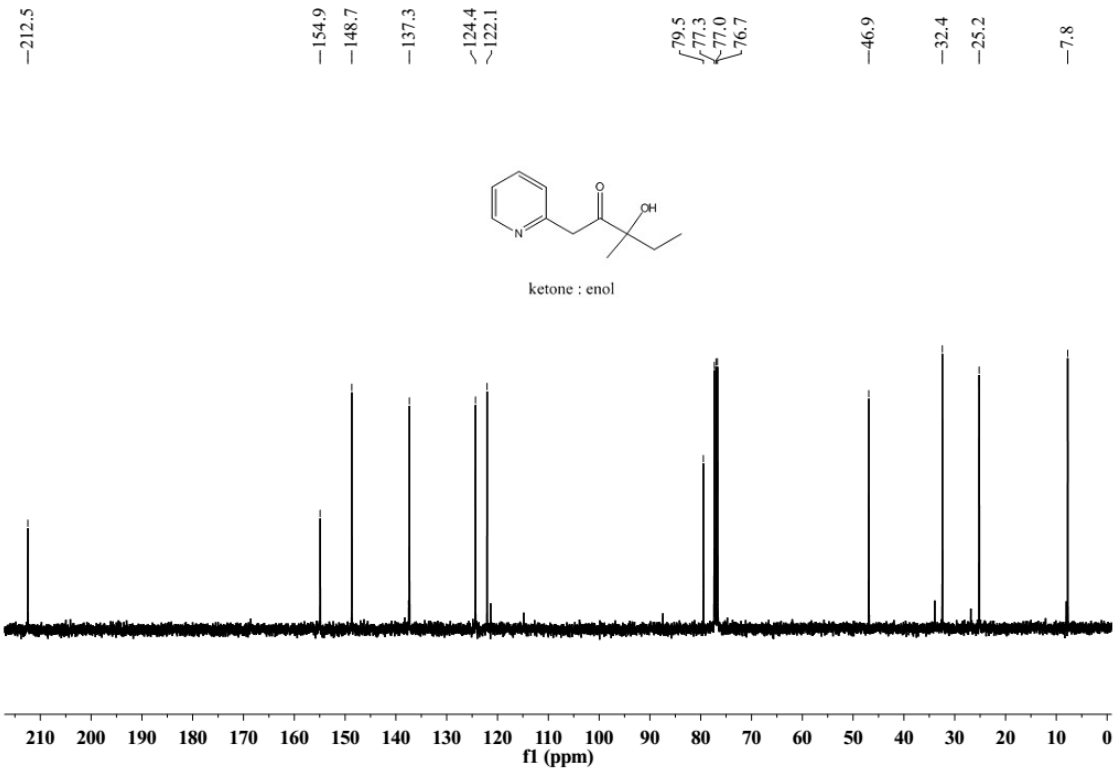




3-hydroxy-3-methyl-1-(pyridin-2-yl)pentan-2-one (2g)





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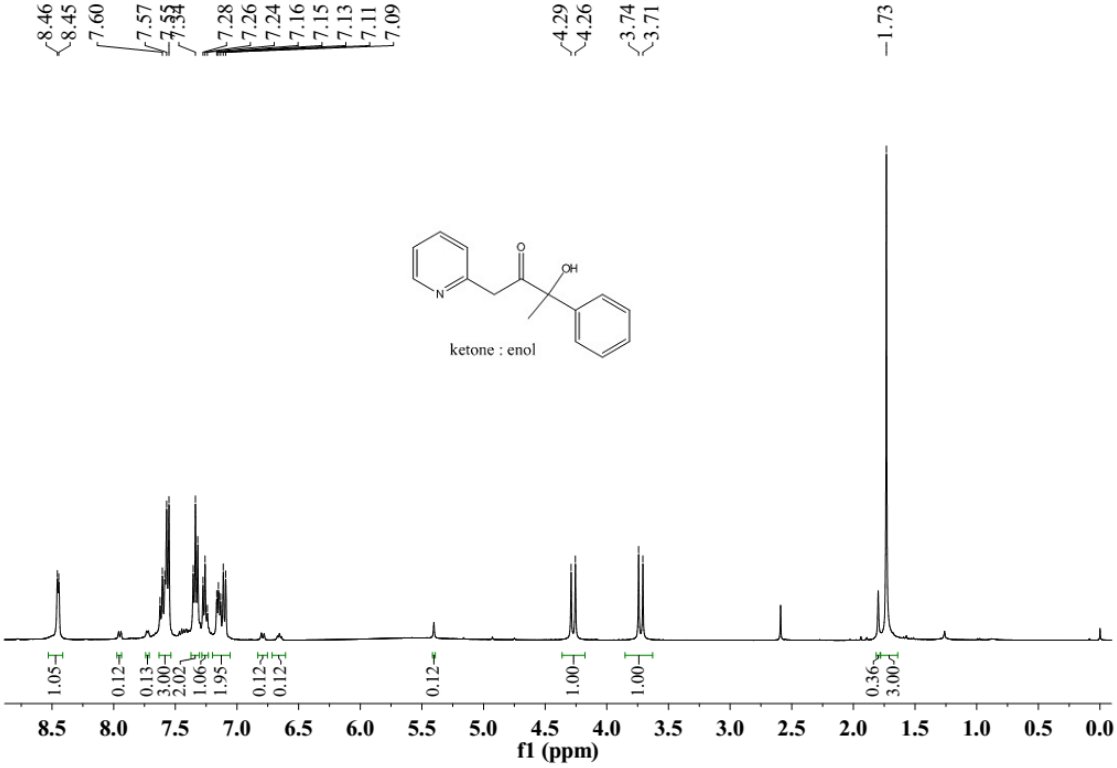
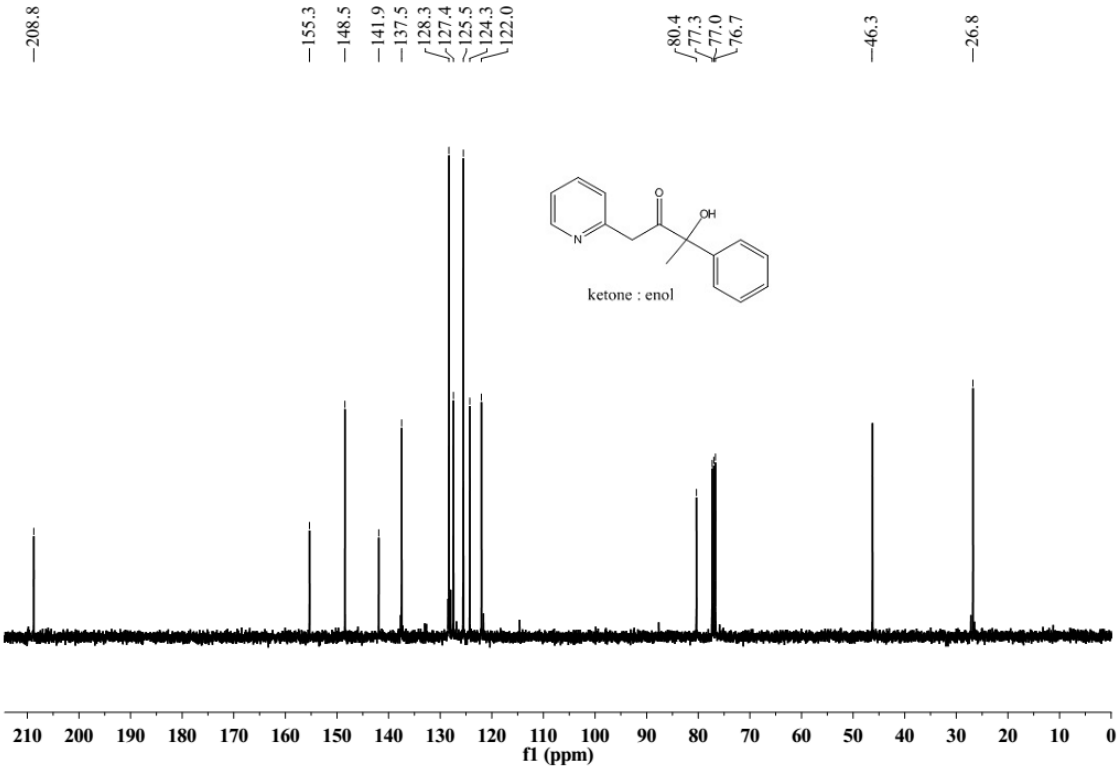
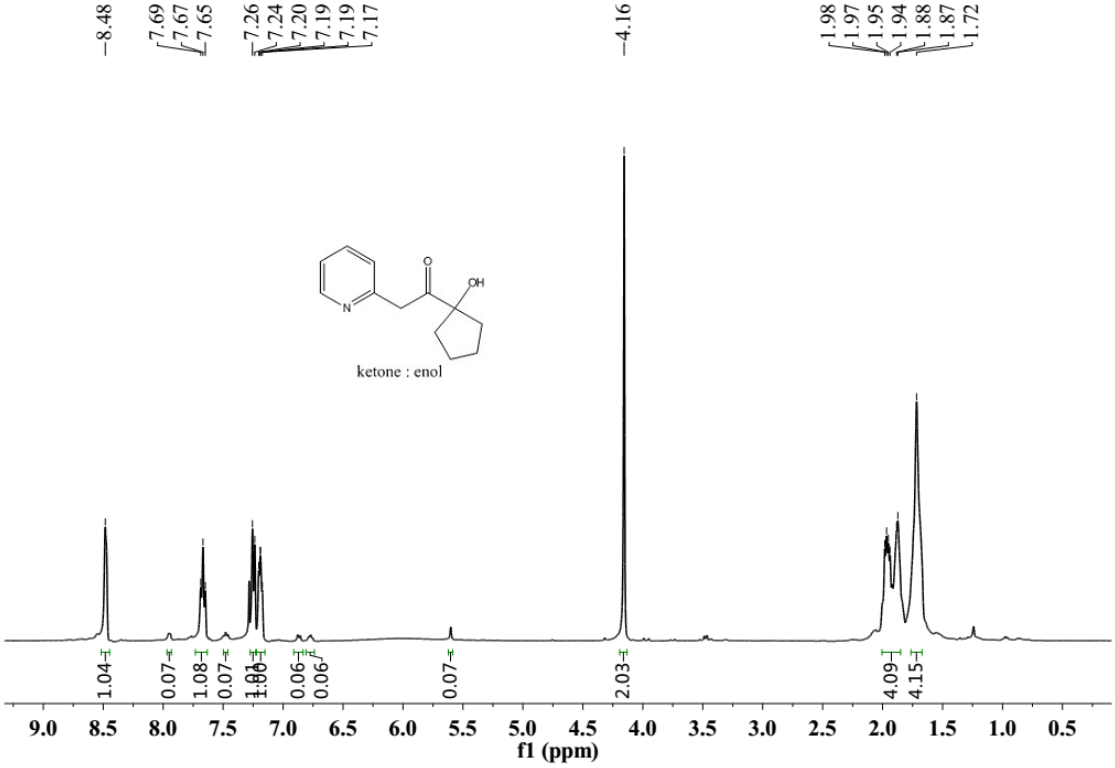
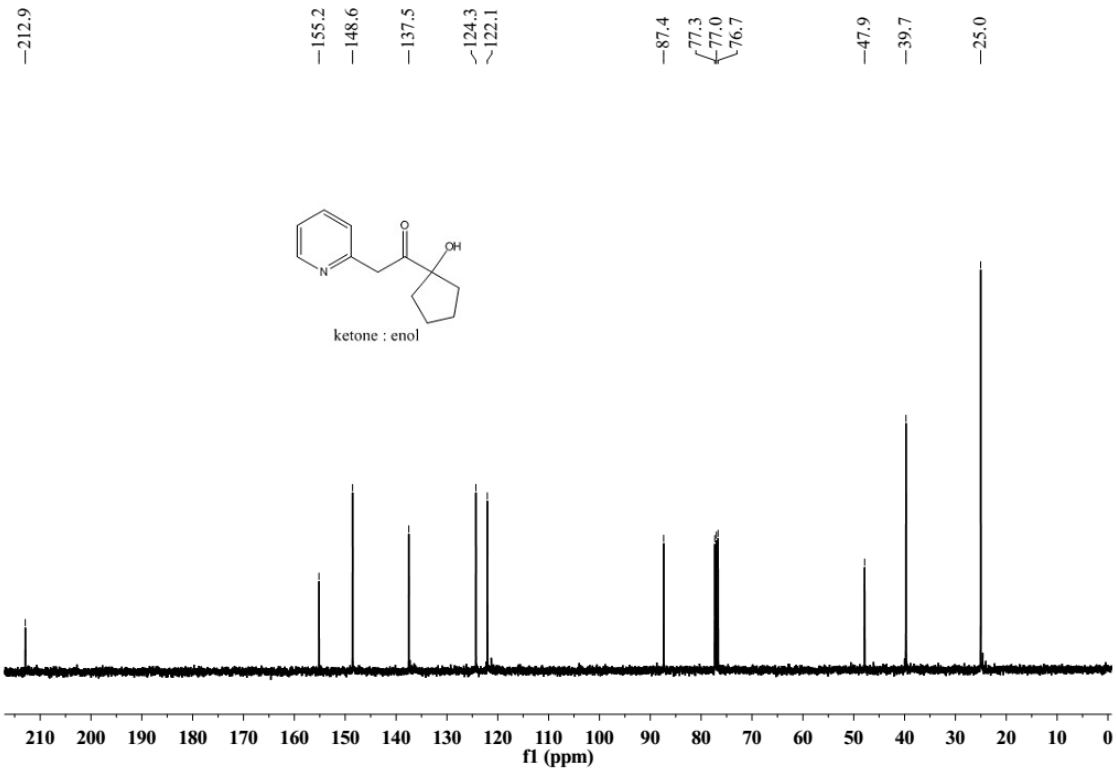


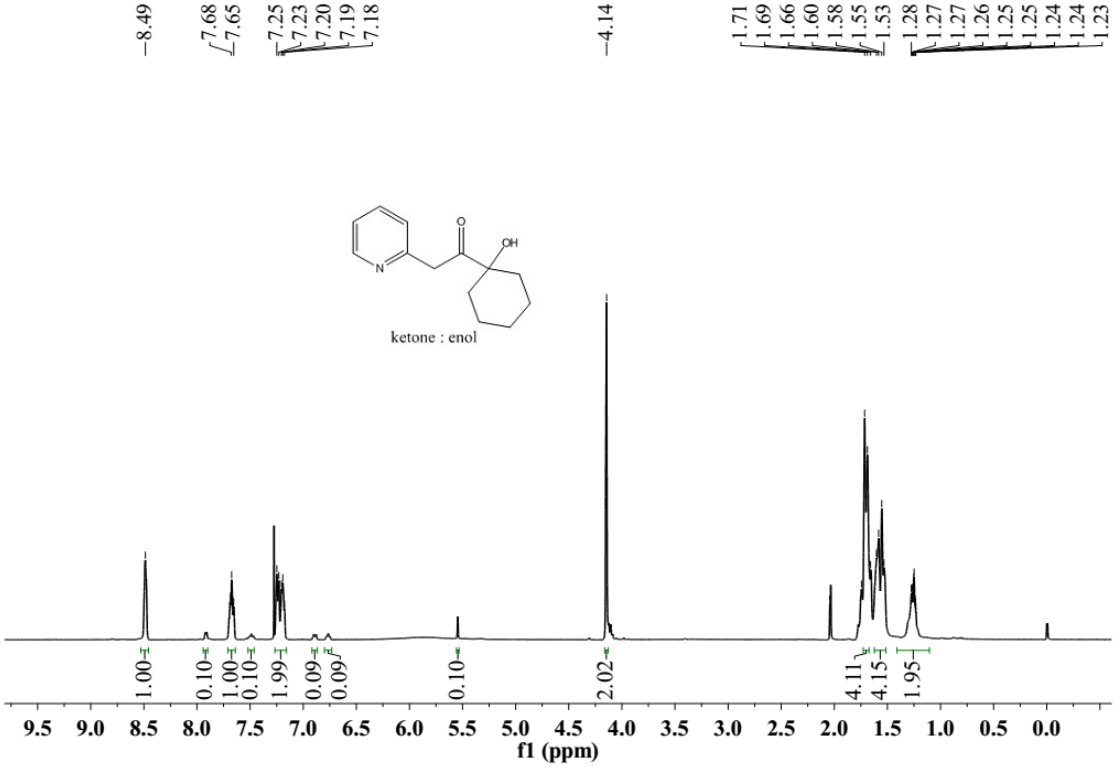


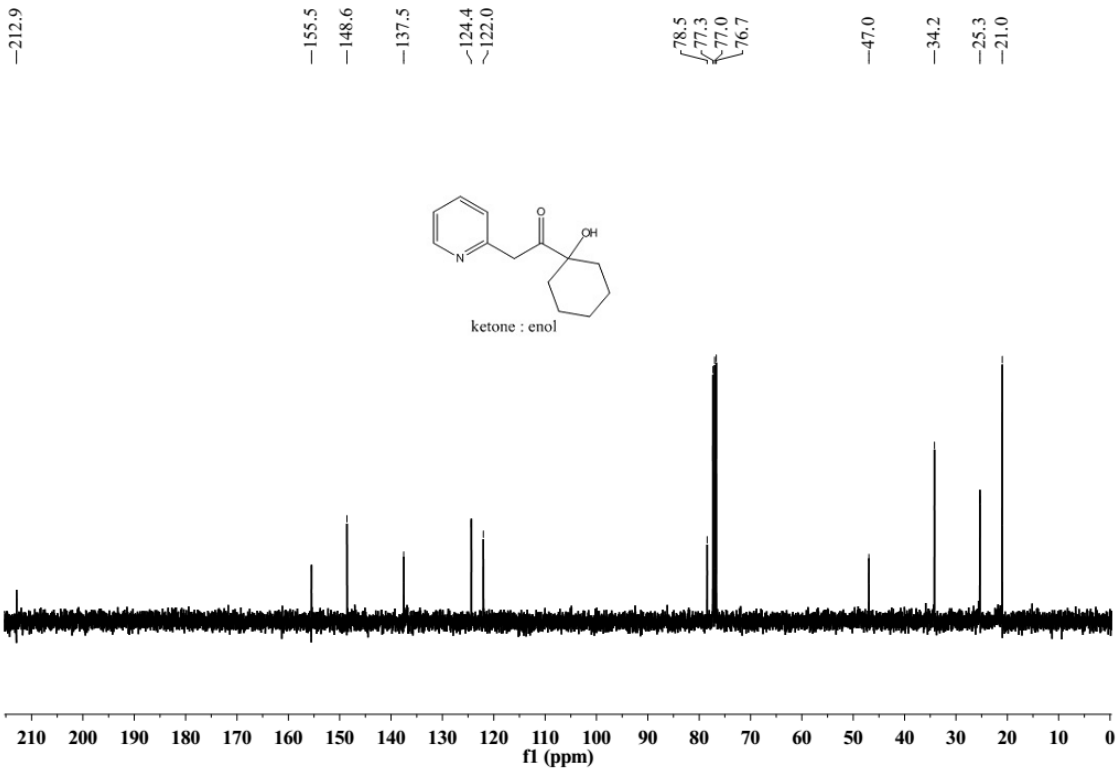
1-(1-hydroxycyclopentyl)-2-(pyridin-2-yl)ethanone (2i)



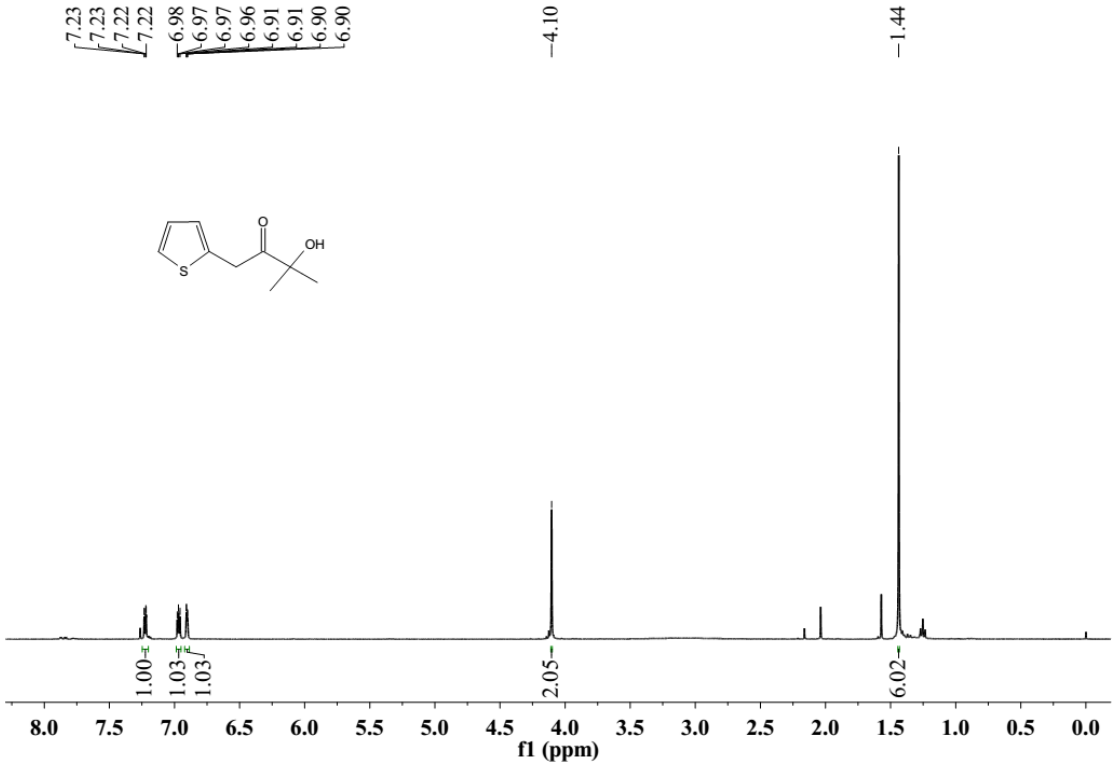


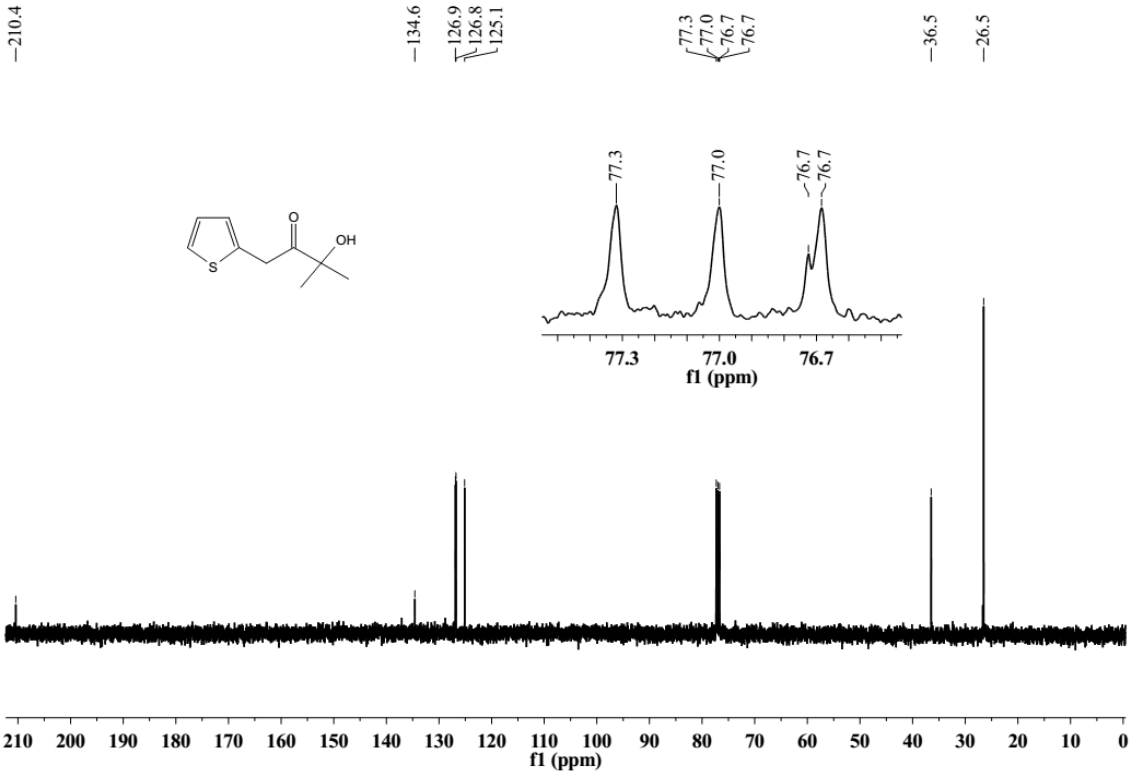
1-(1-hydroxycyclohexyl)-2-(pyridin-2-yl)ethanone (2j)





3-hydroxy-3-methyl-1-(thiophen-2-yl)butan-2-one (2k)





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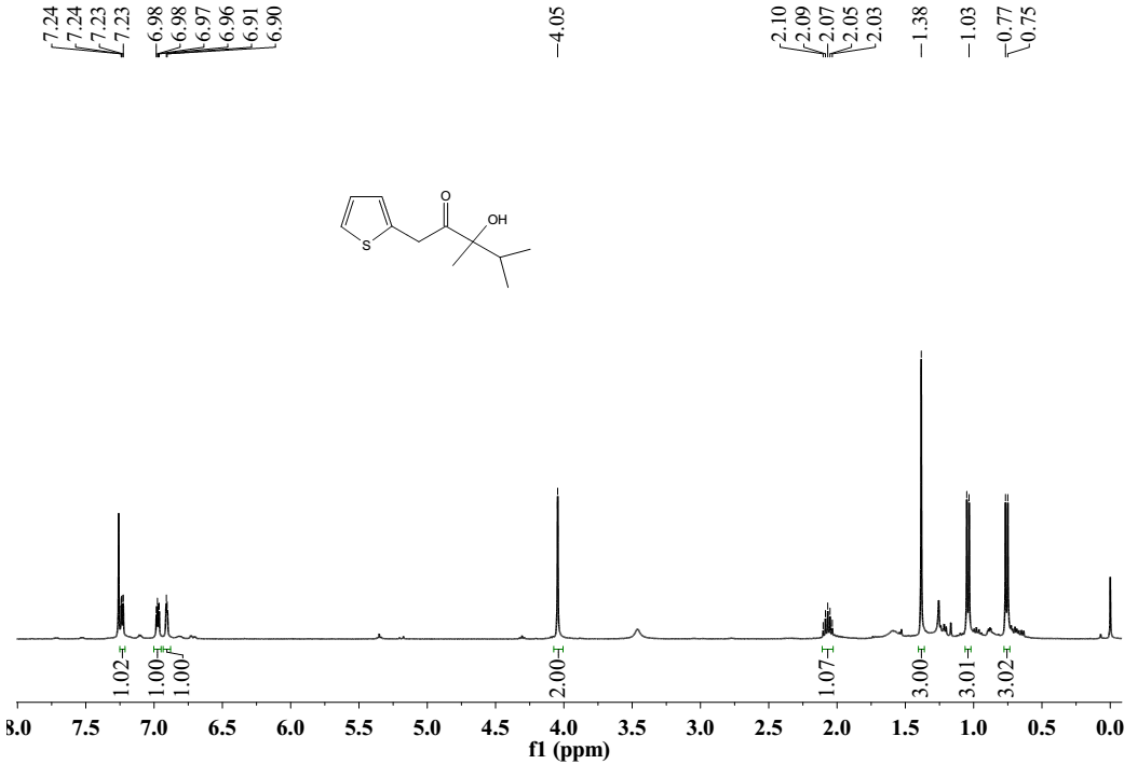
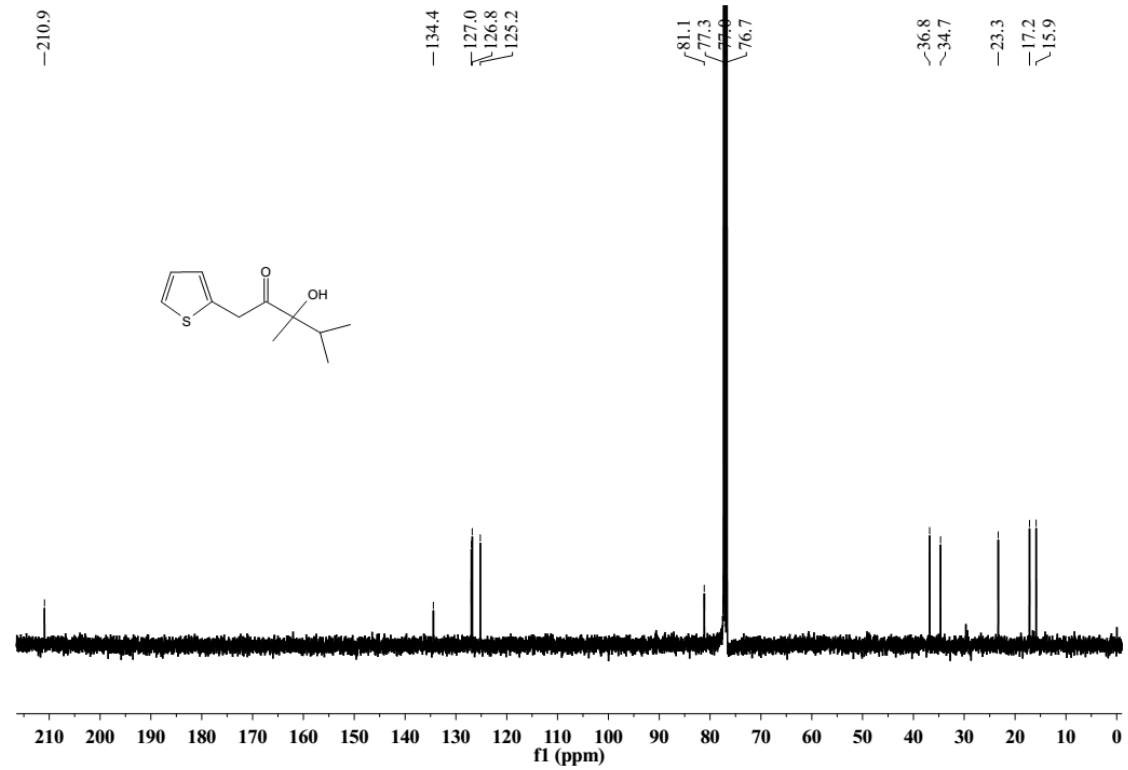
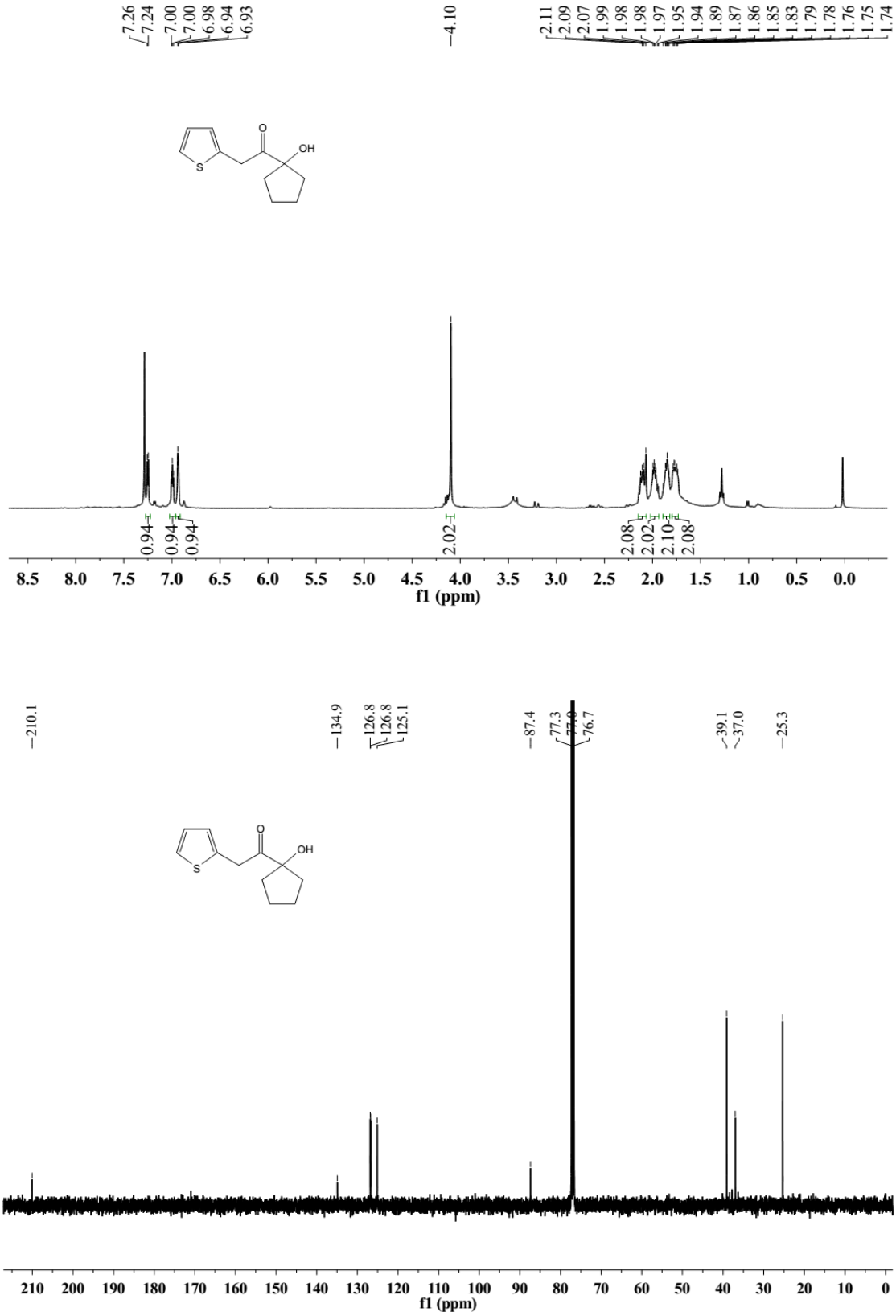
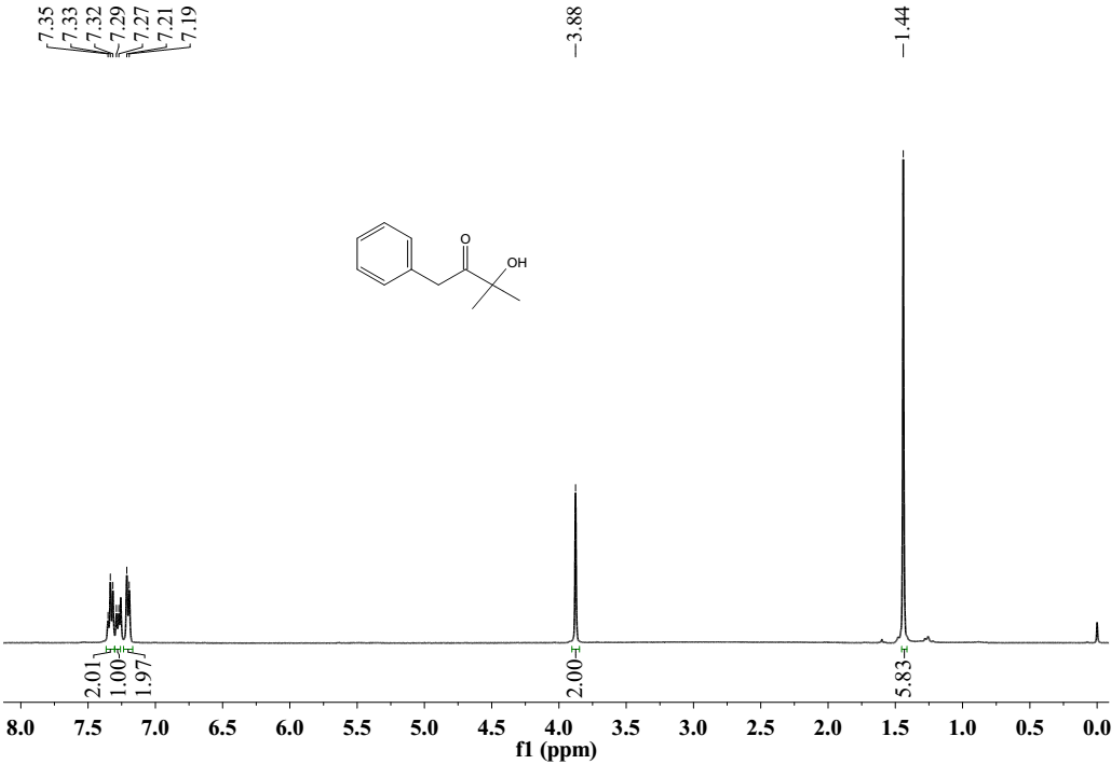


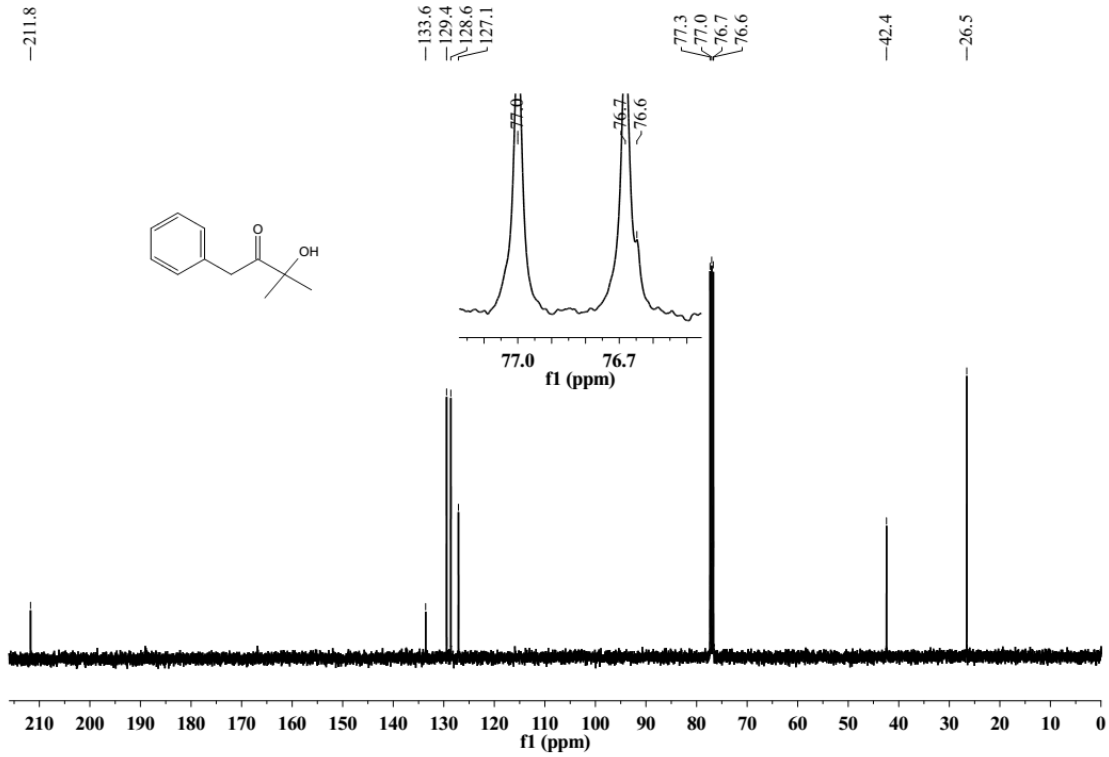


1-(1-hydroxycyclopentyl)-2-(thiophen-2-yl)ethanone (2m)

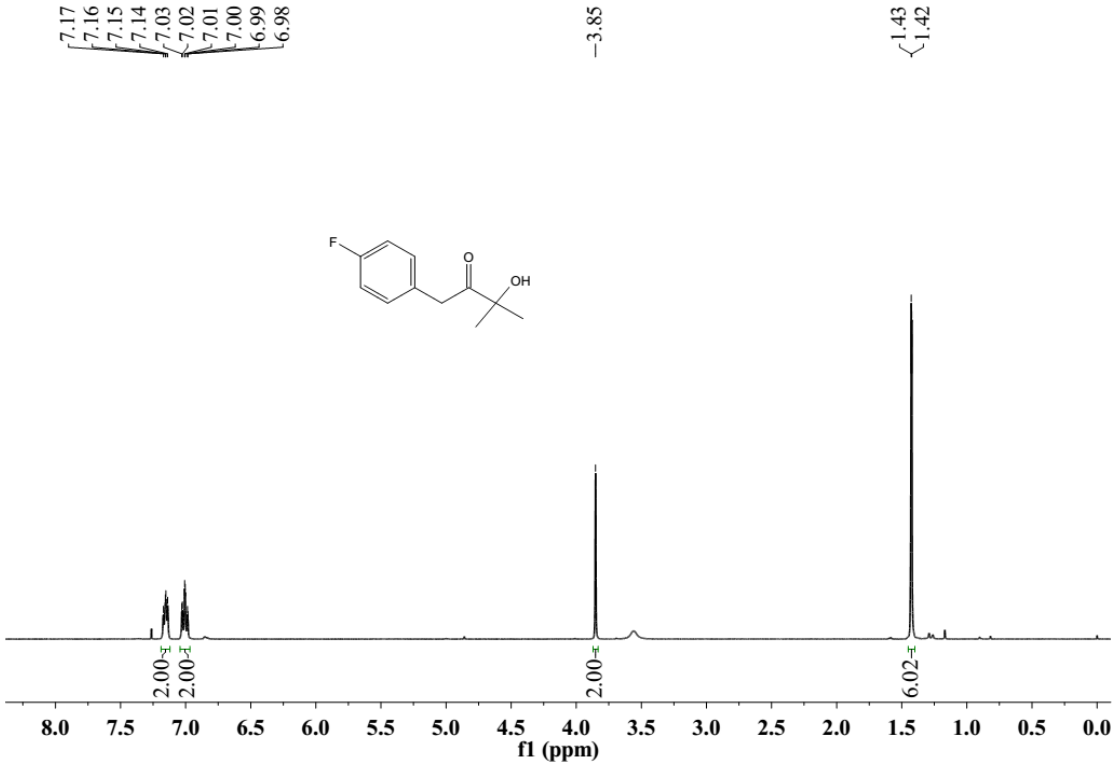


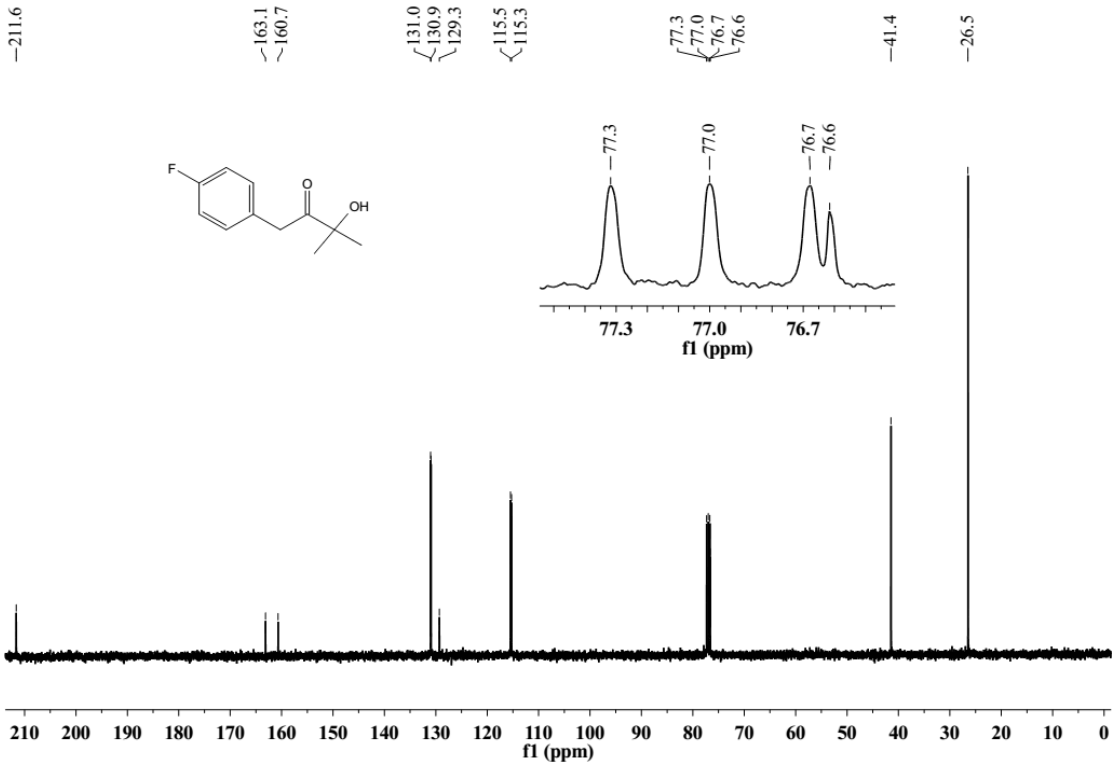
3-hydroxy-3-methyl-1-phenylbutan-2-one ($2n_1$)



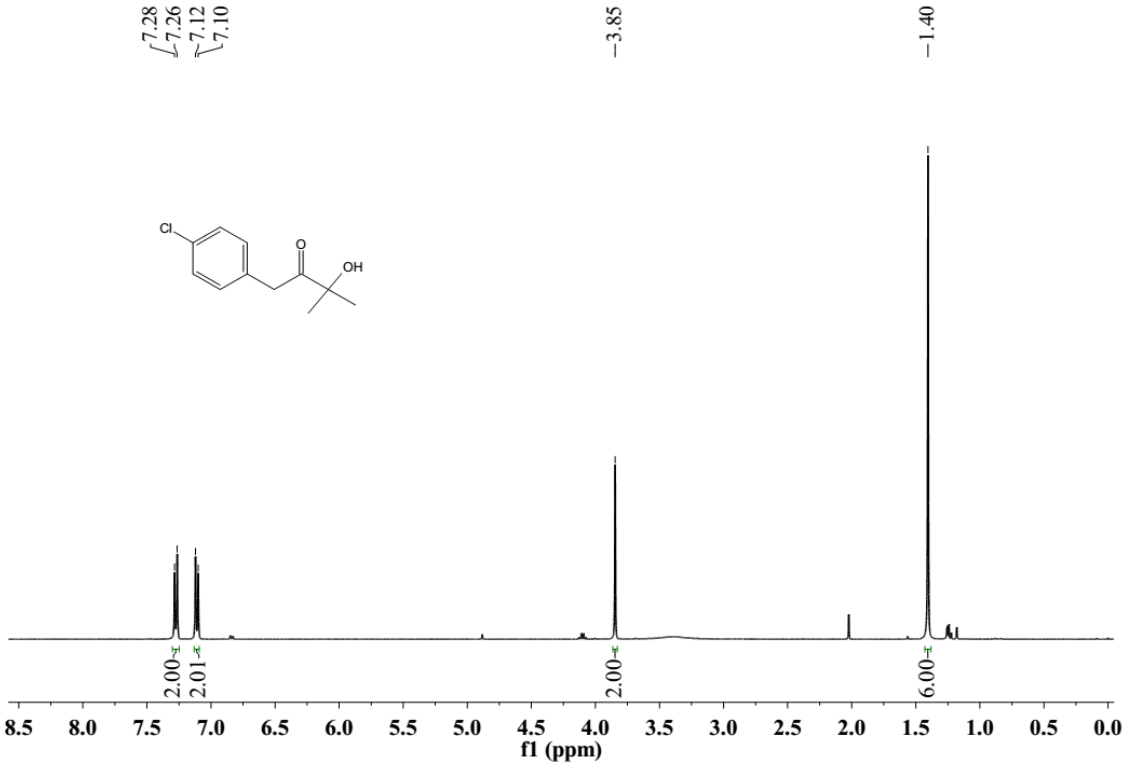


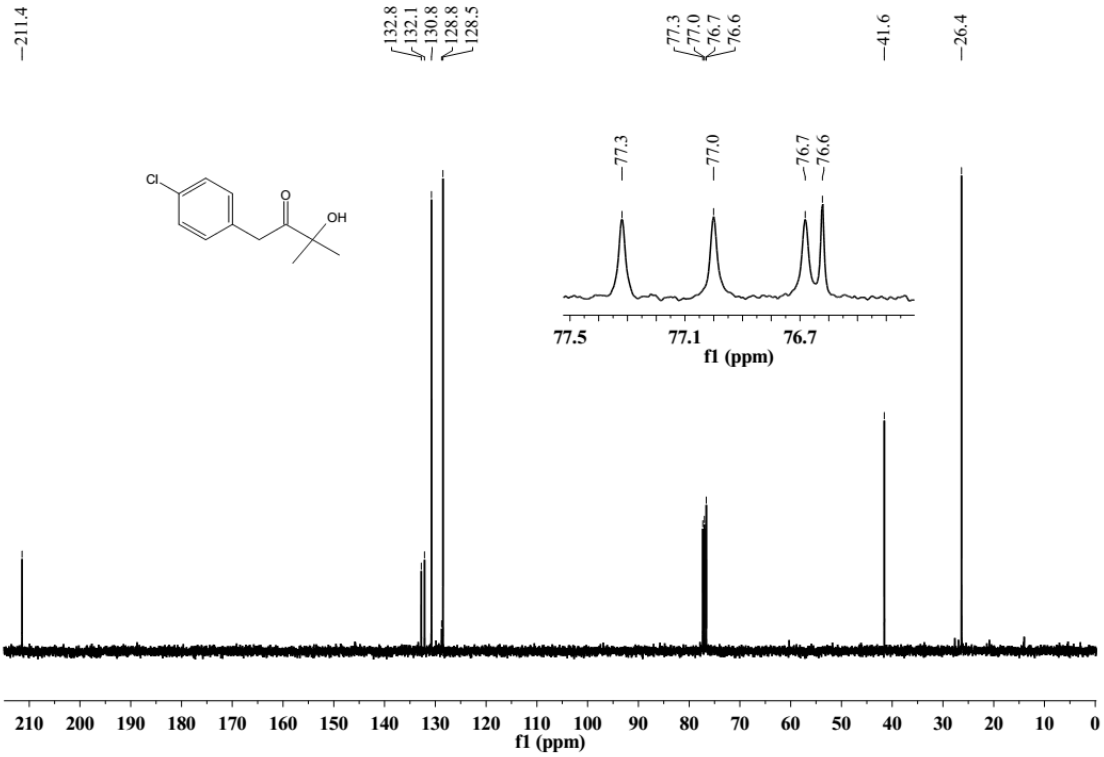
1-(4-fluorophenyl)-3-hydroxy-3-methylbutan-2-one ($2n_2$)



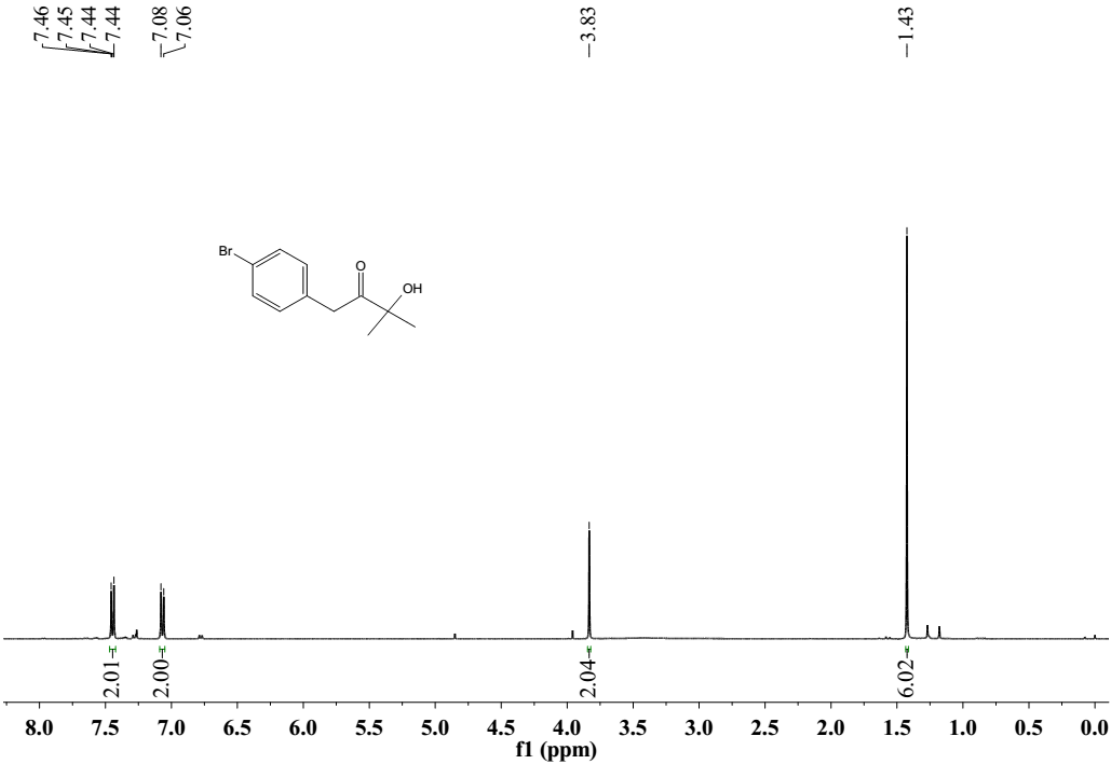


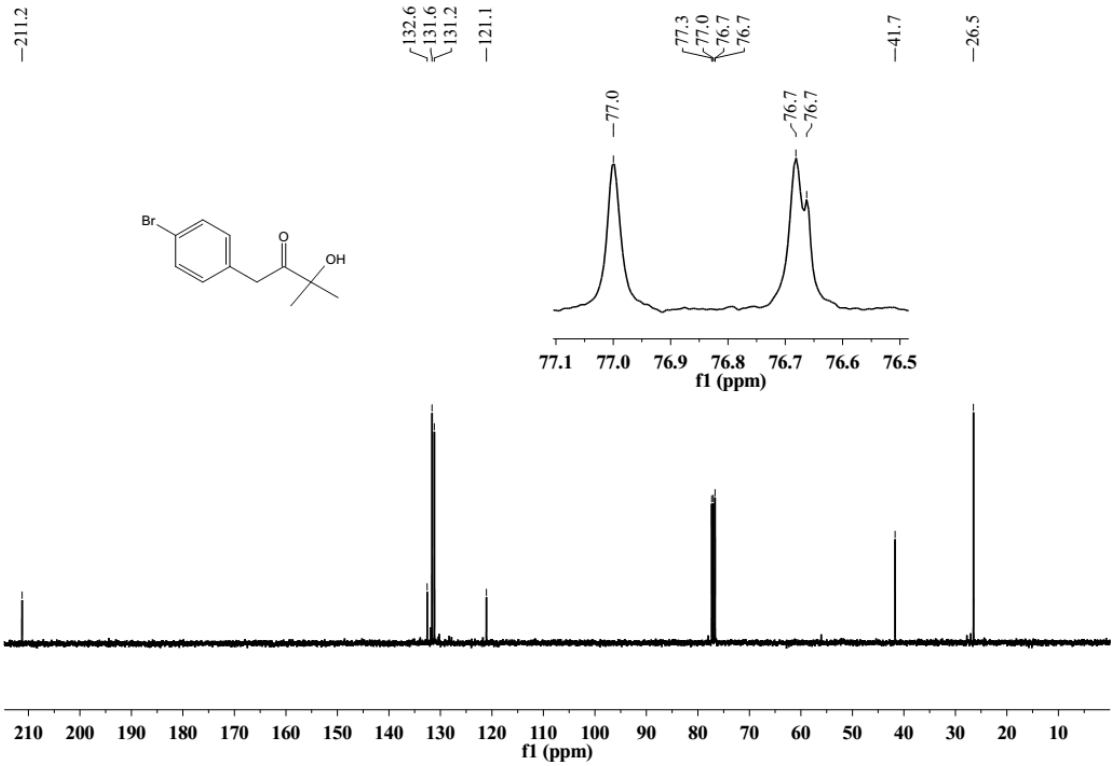
1-(4-chlorophenyl)-3-hydroxy-3-methylbutan-2-one ($2n_3$)



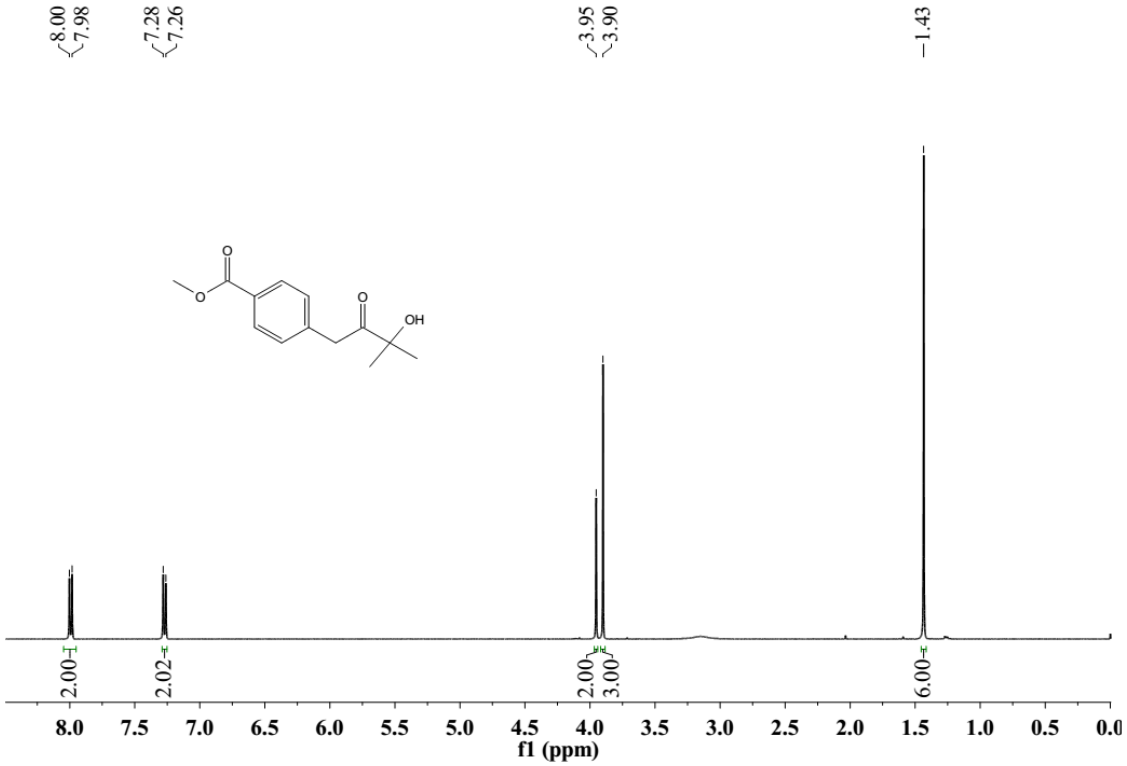


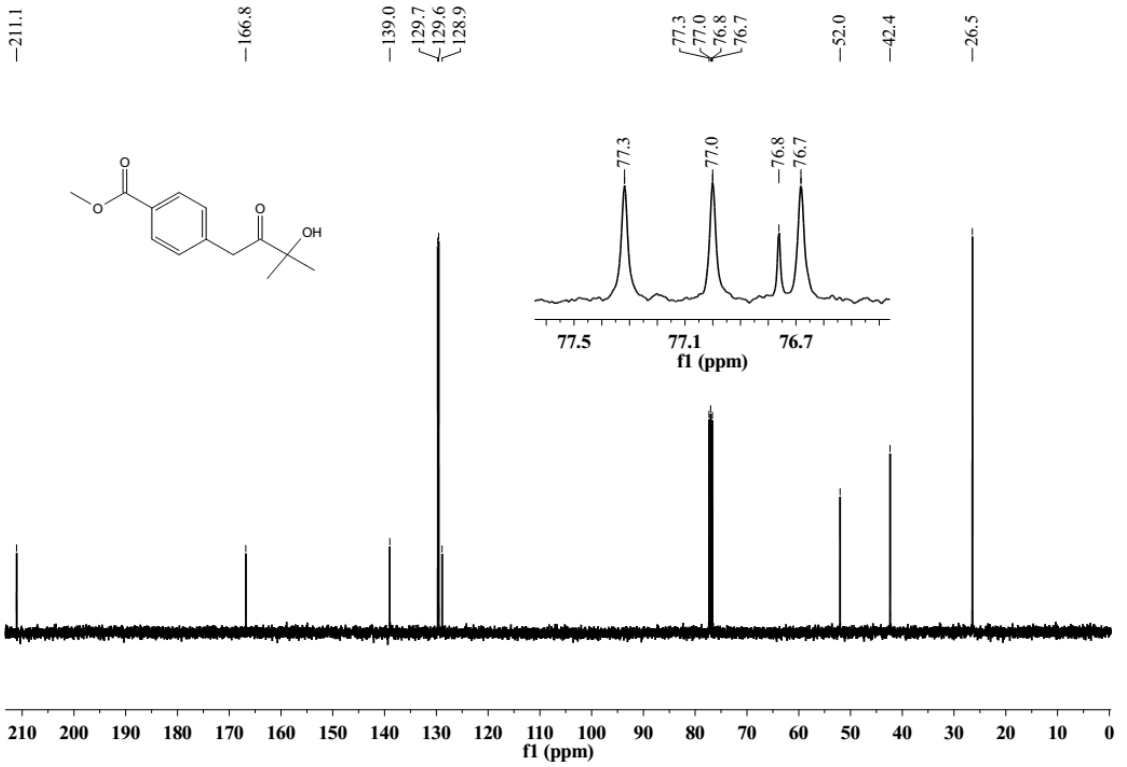
1-(4-bromophenyl)-3-hydroxy-3-methylbutan-2-one ($2n_4$)



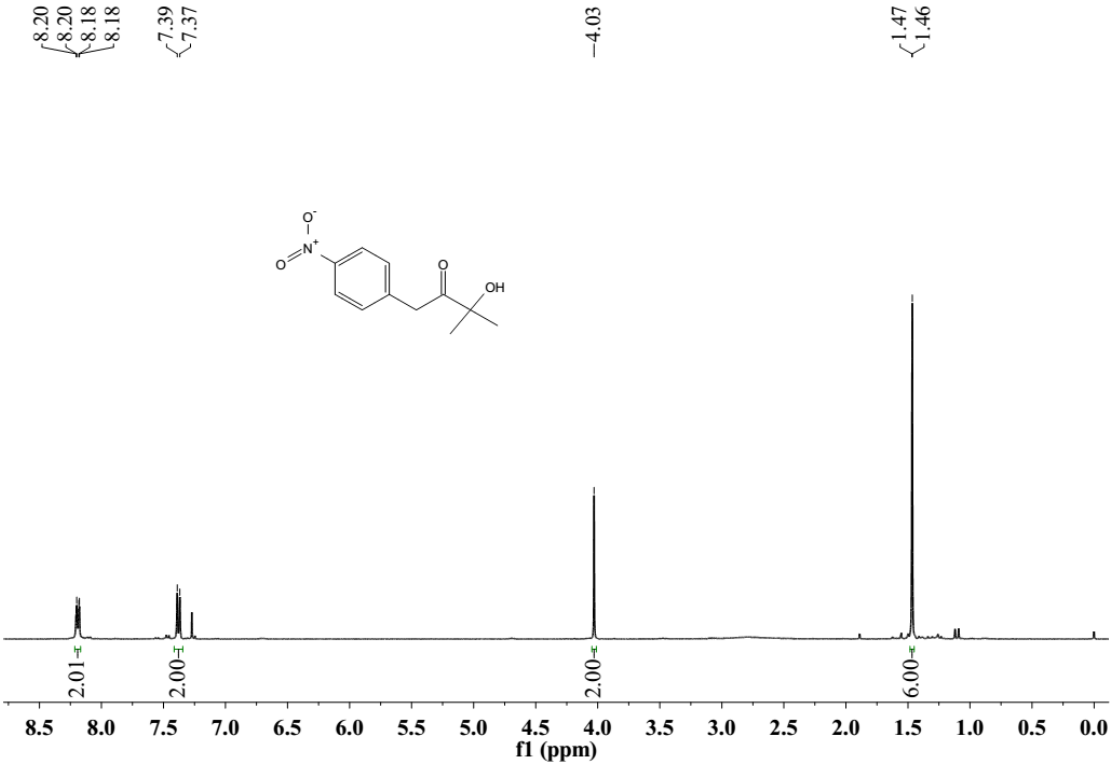


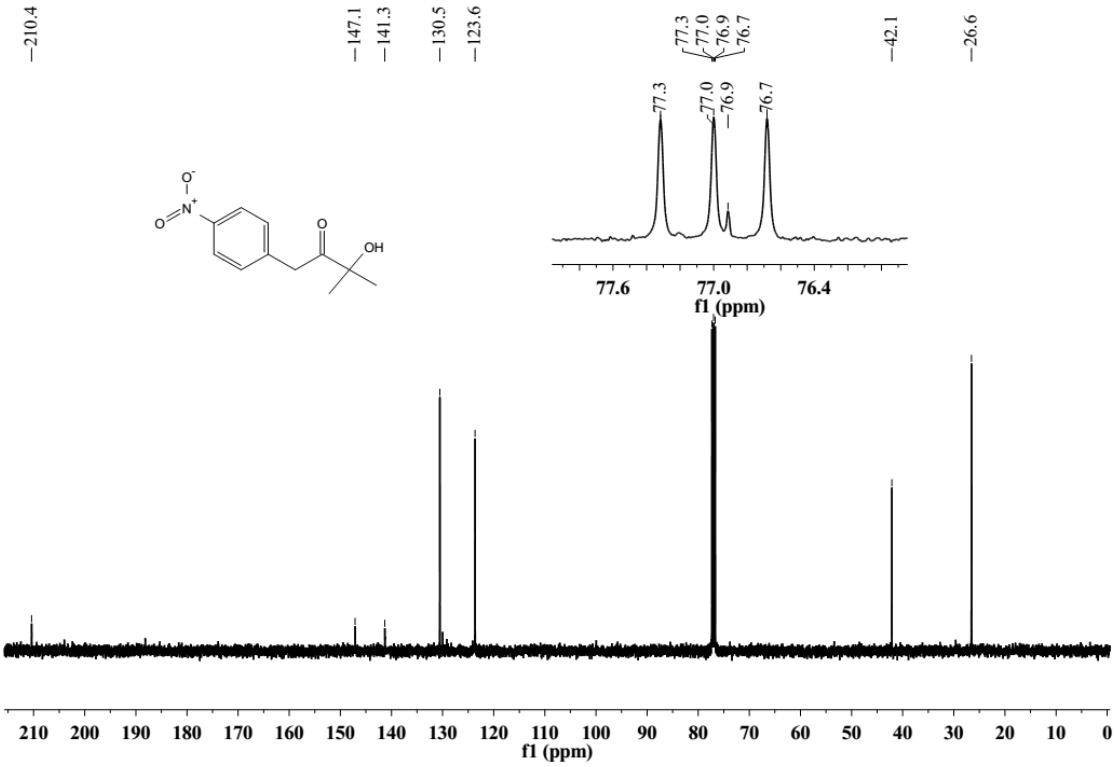
methyl 4-(3-hydroxy-3-methyl-2-oxobutyl)benzoate ($2n_5$)



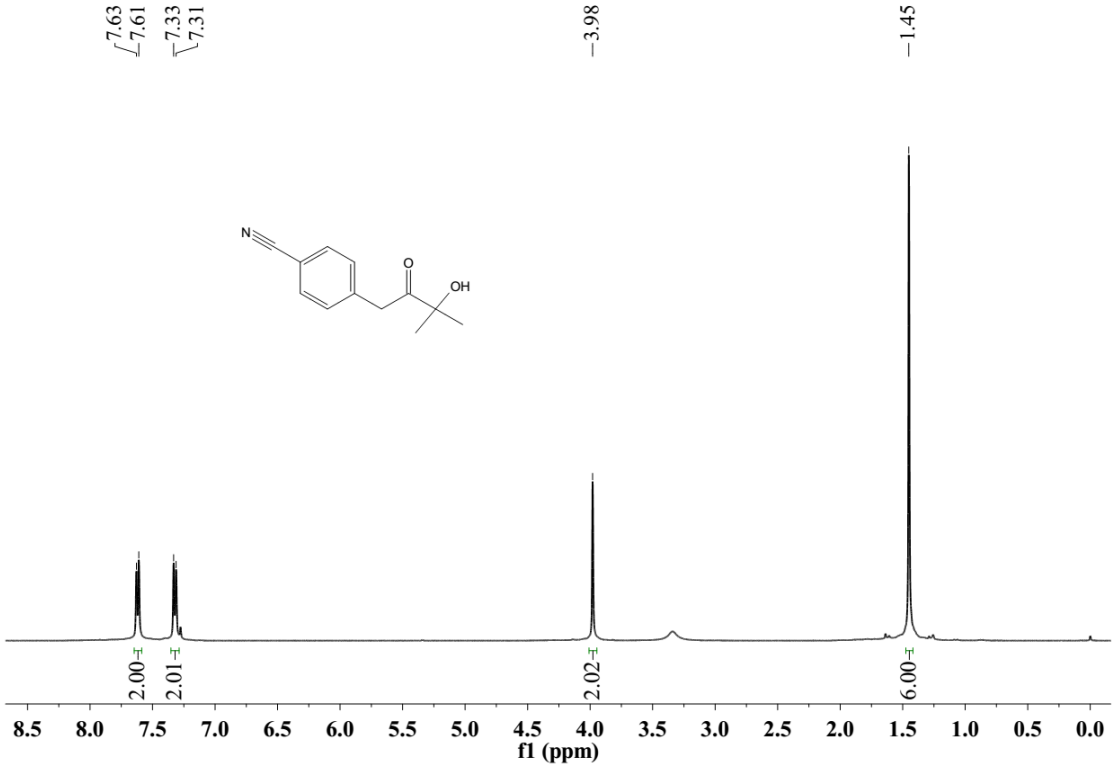


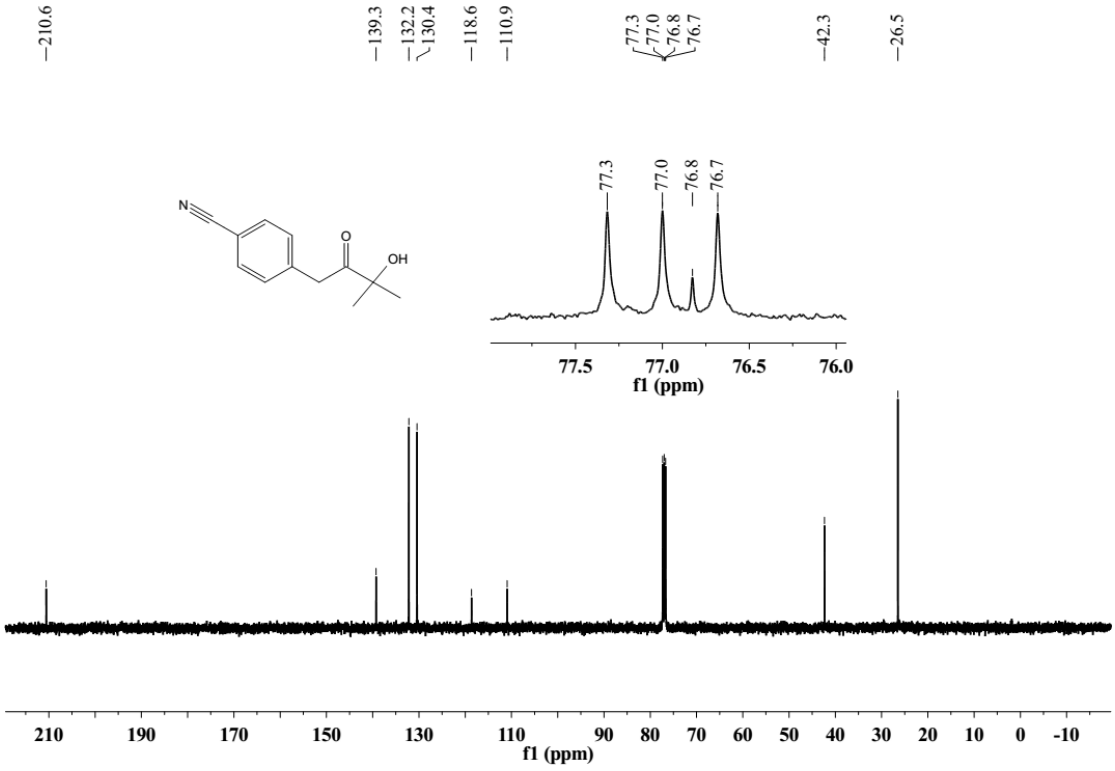
3-hydroxy-3-methyl-1-(4-nitrophenyl)butan-2-one ($2n_6$)



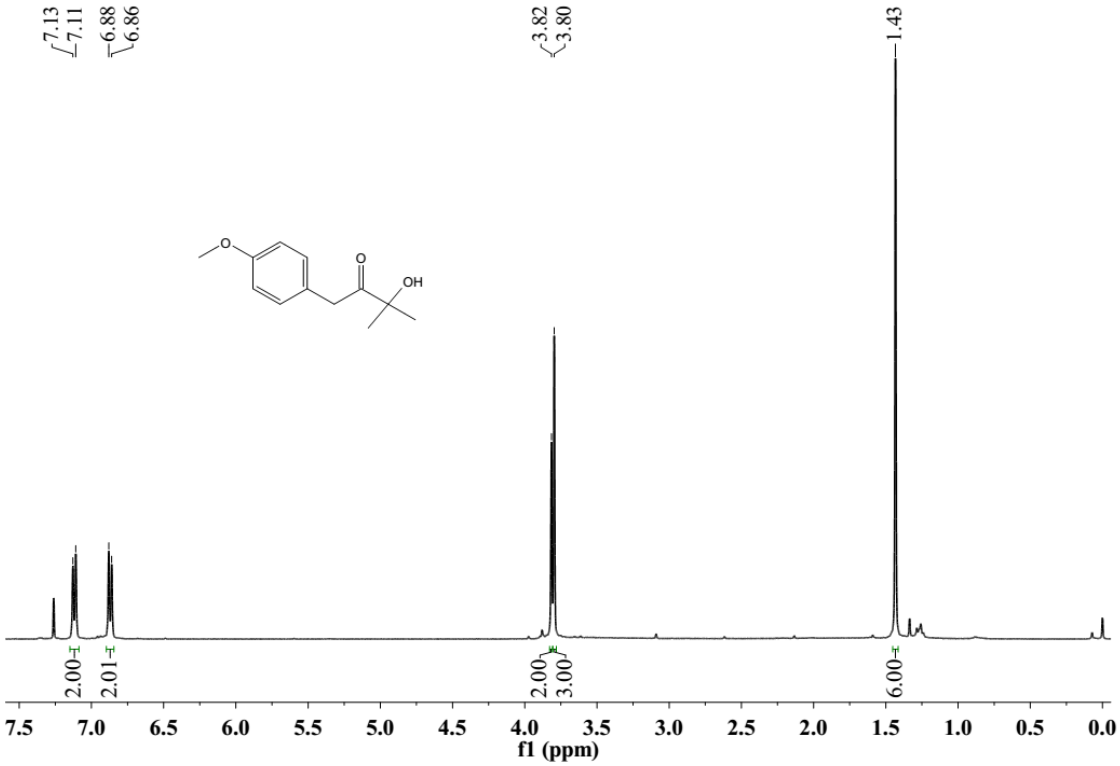


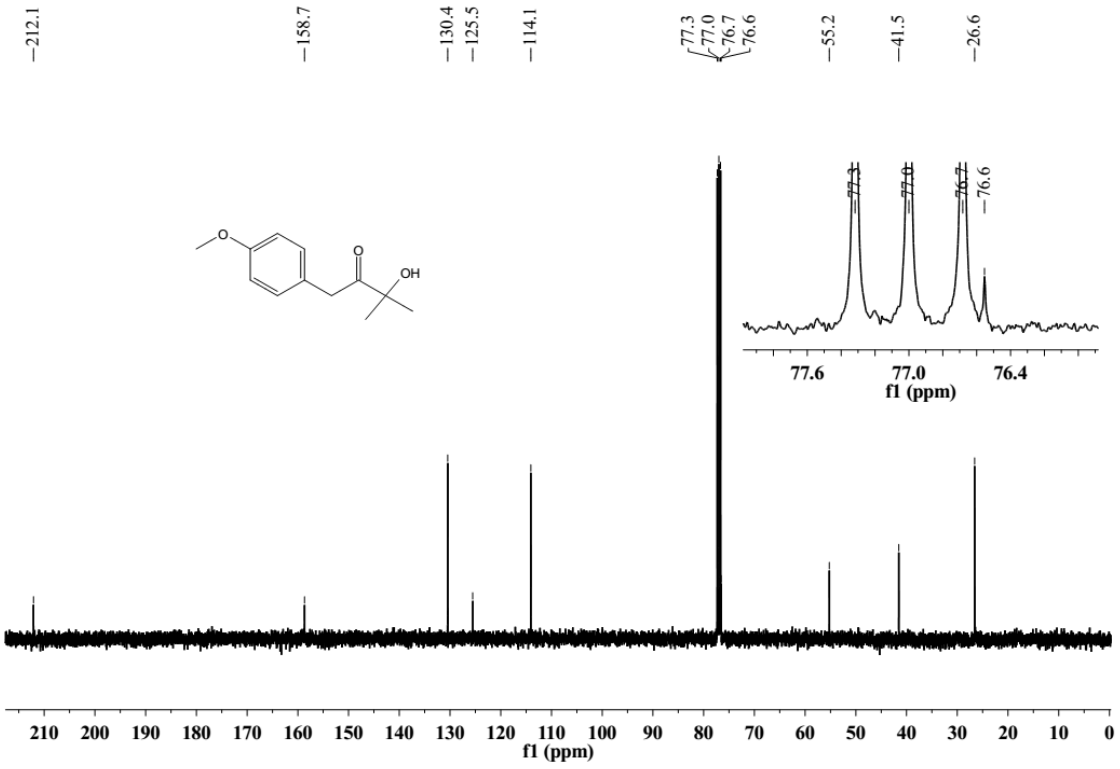
4-(3-hydroxy-3-methyl-2-oxobutyl)benzonitrile ($2n_7$)



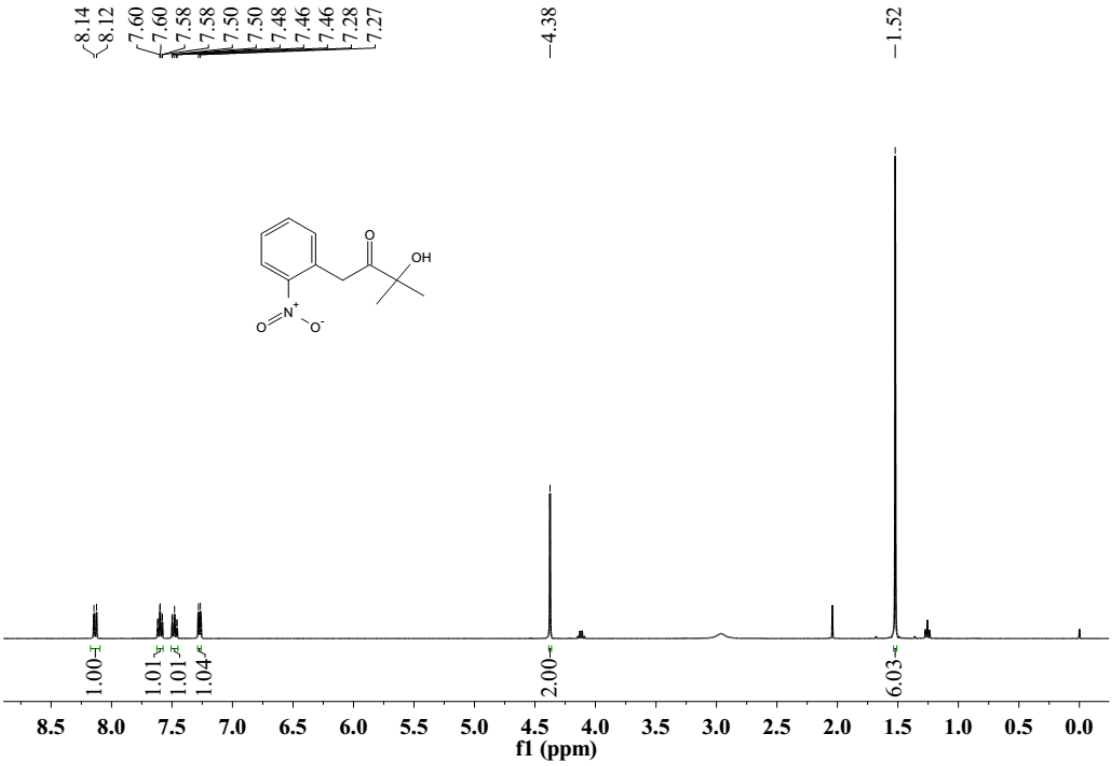


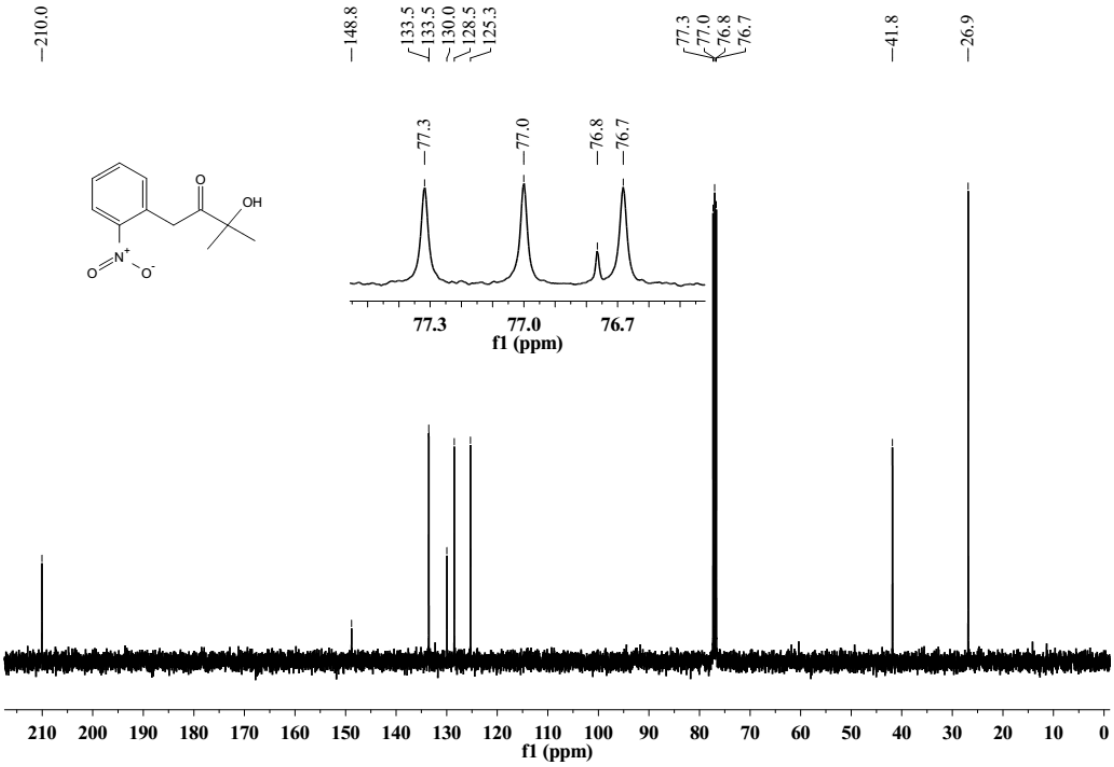
3-hydroxy-1-(4-methoxyphenyl)-3-methylbutan-2-one ($2n_8$)



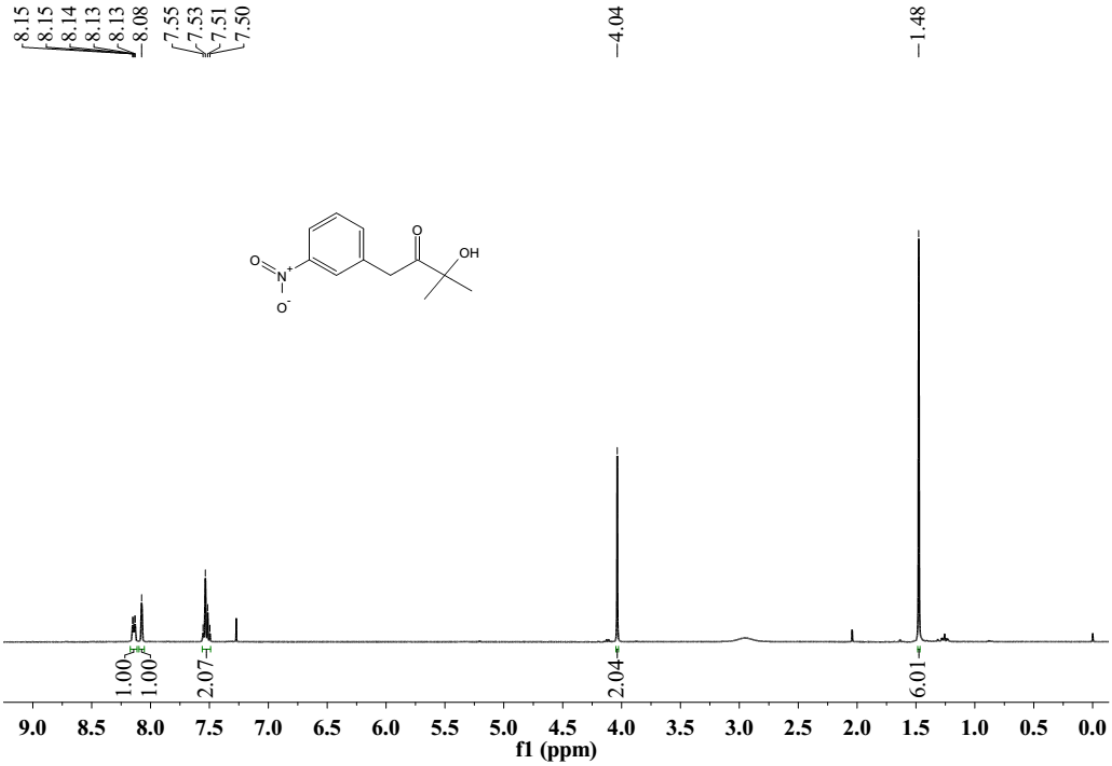


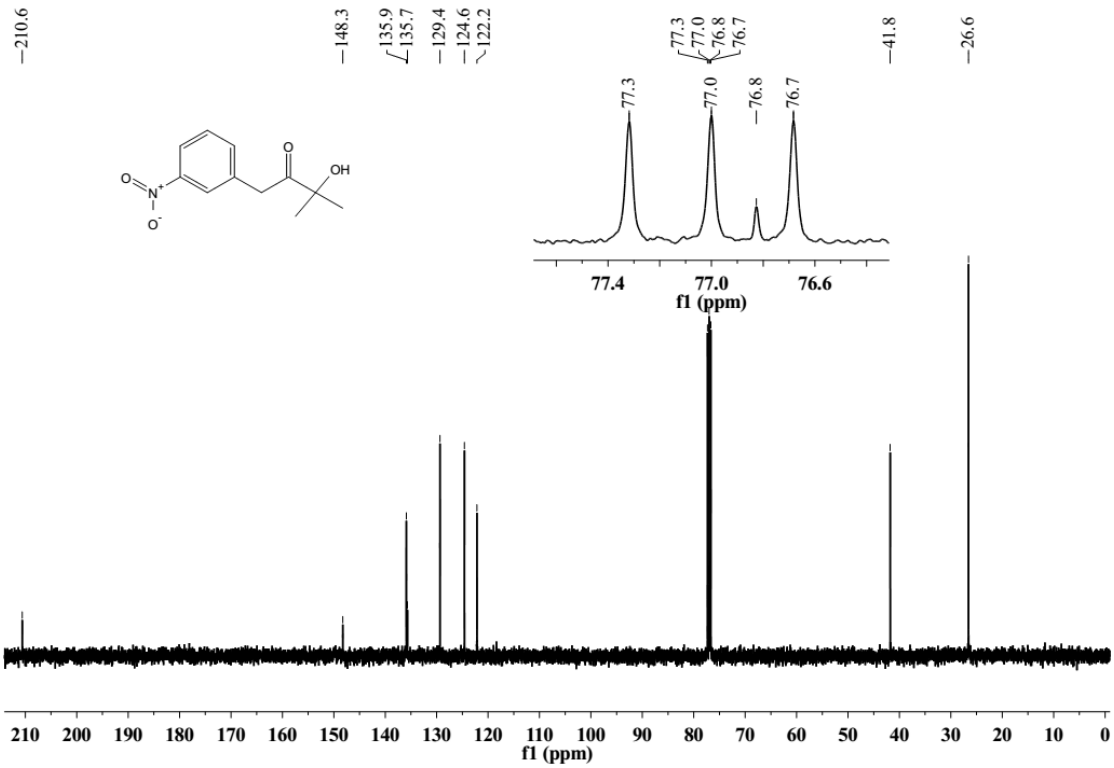
3-hydroxy-3-methyl-1-(2-nitrophenyl)butan-2-one (2o)



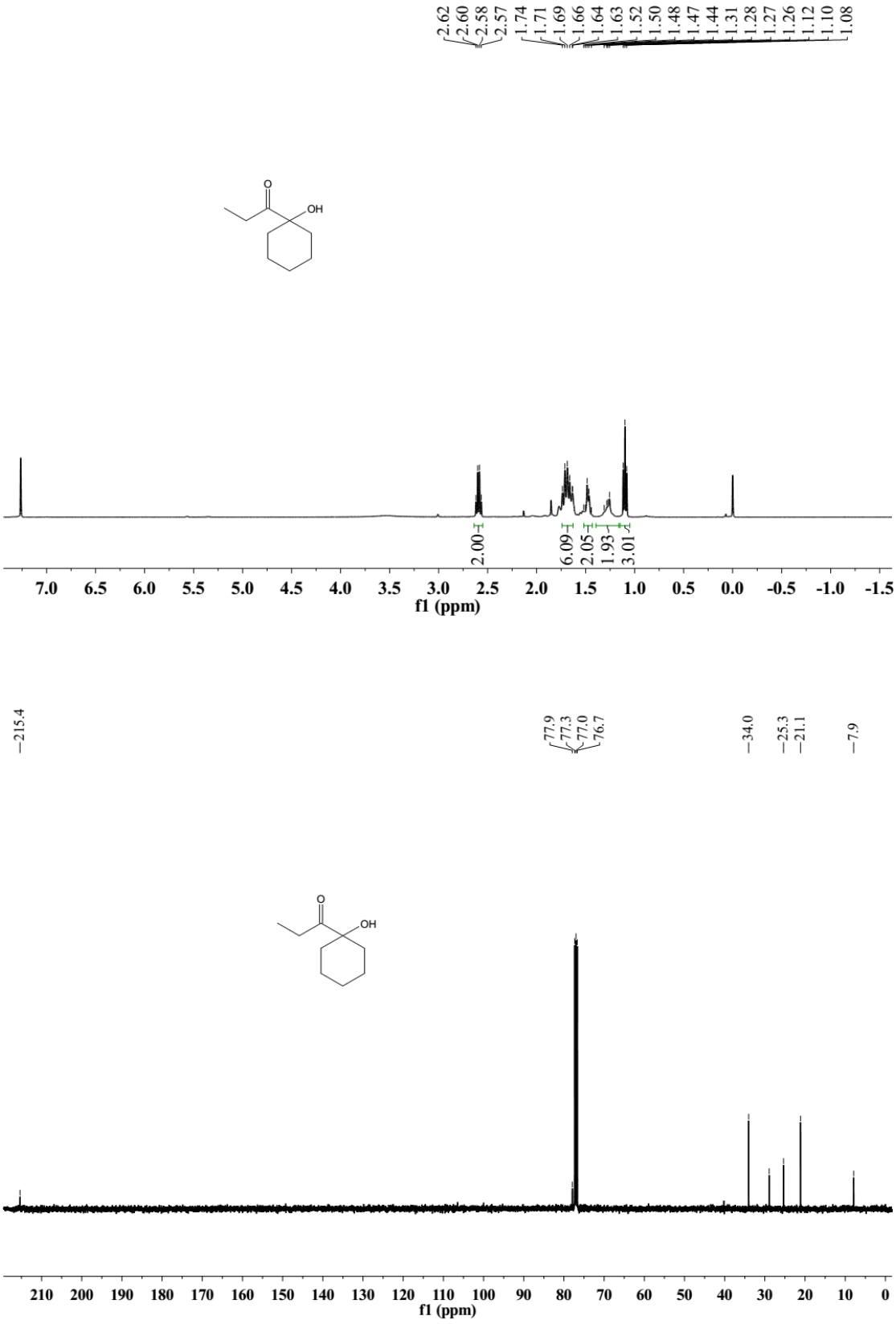


3-hydroxy-3-methyl-1-(3-nitrophenyl)butan-2-one (2p)

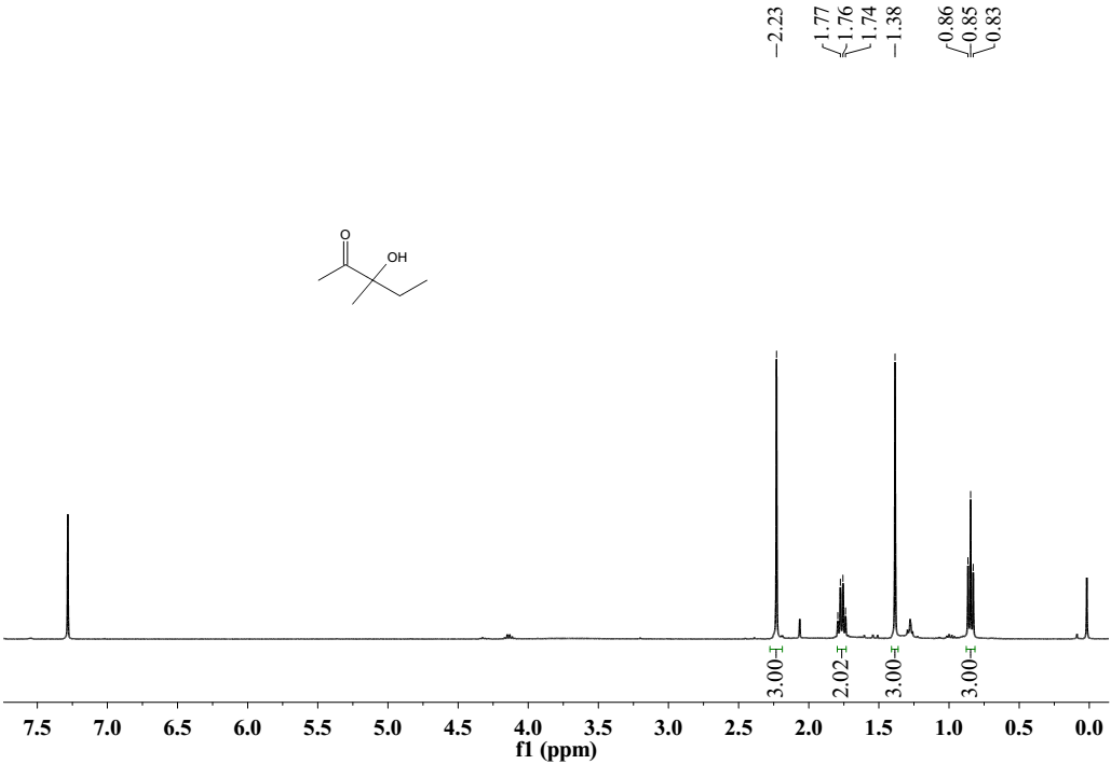


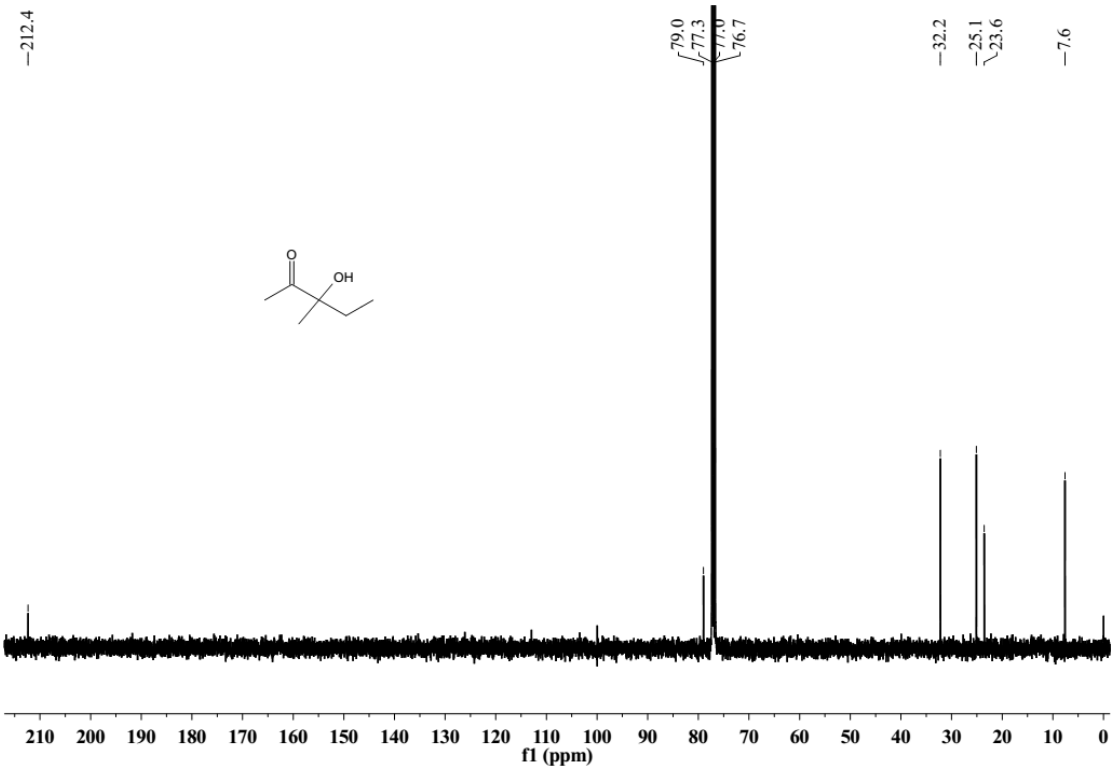


1-(1-hydroxycyclohexyl)propan-1-one (2q)

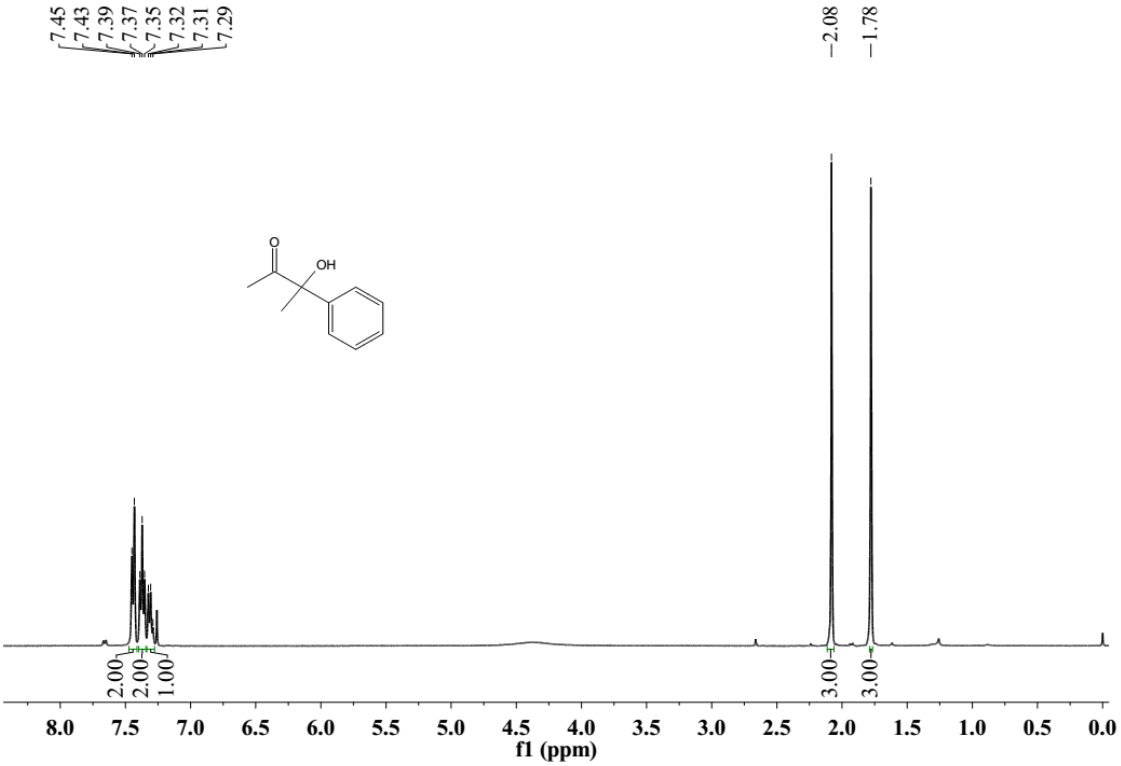


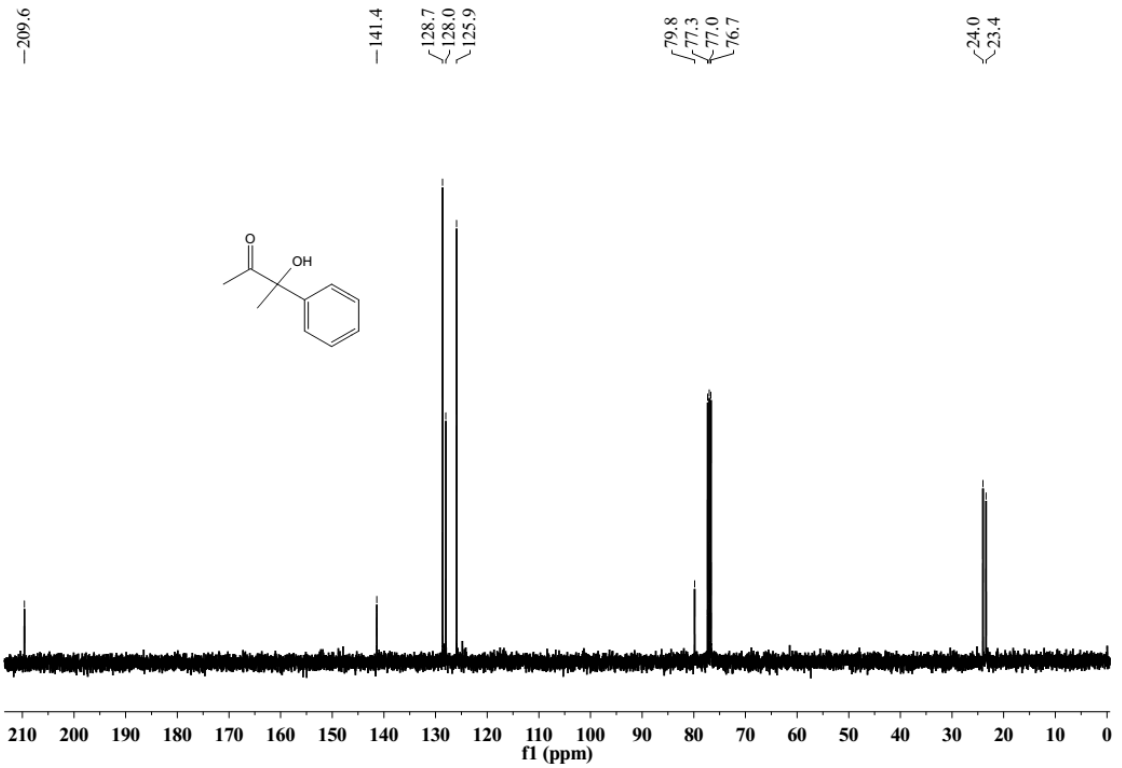
3-hydroxy-3-methylpentan-2-one (2r)



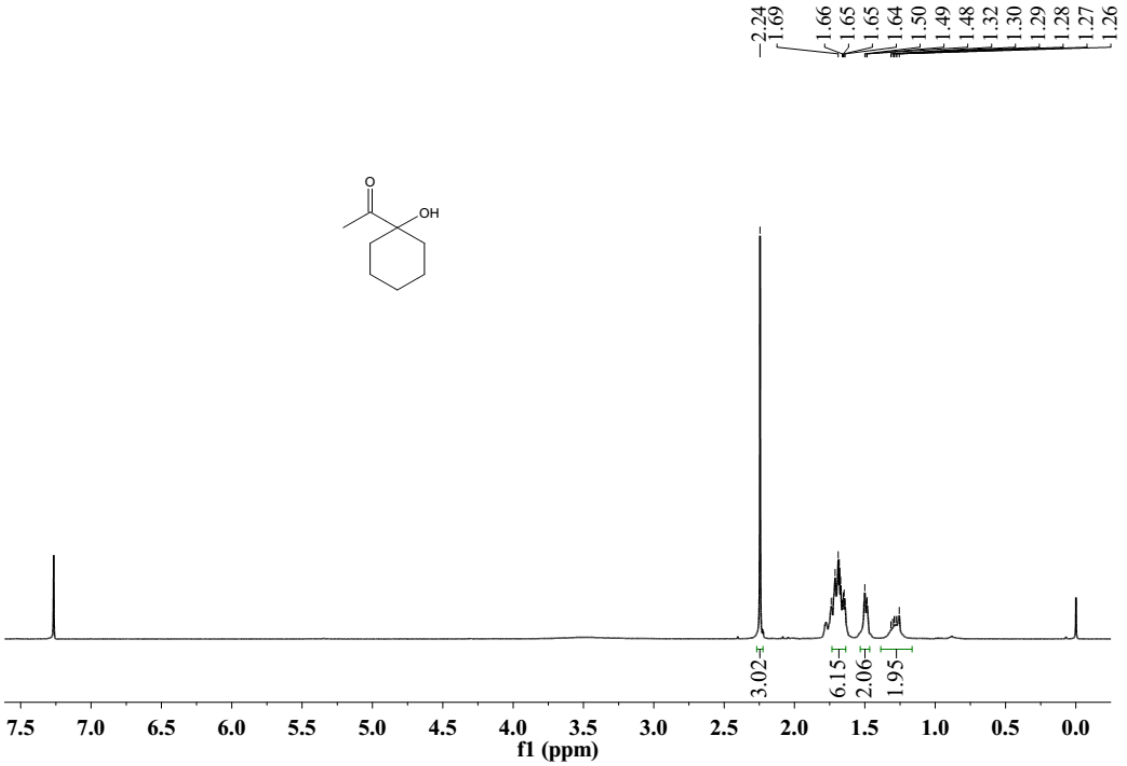


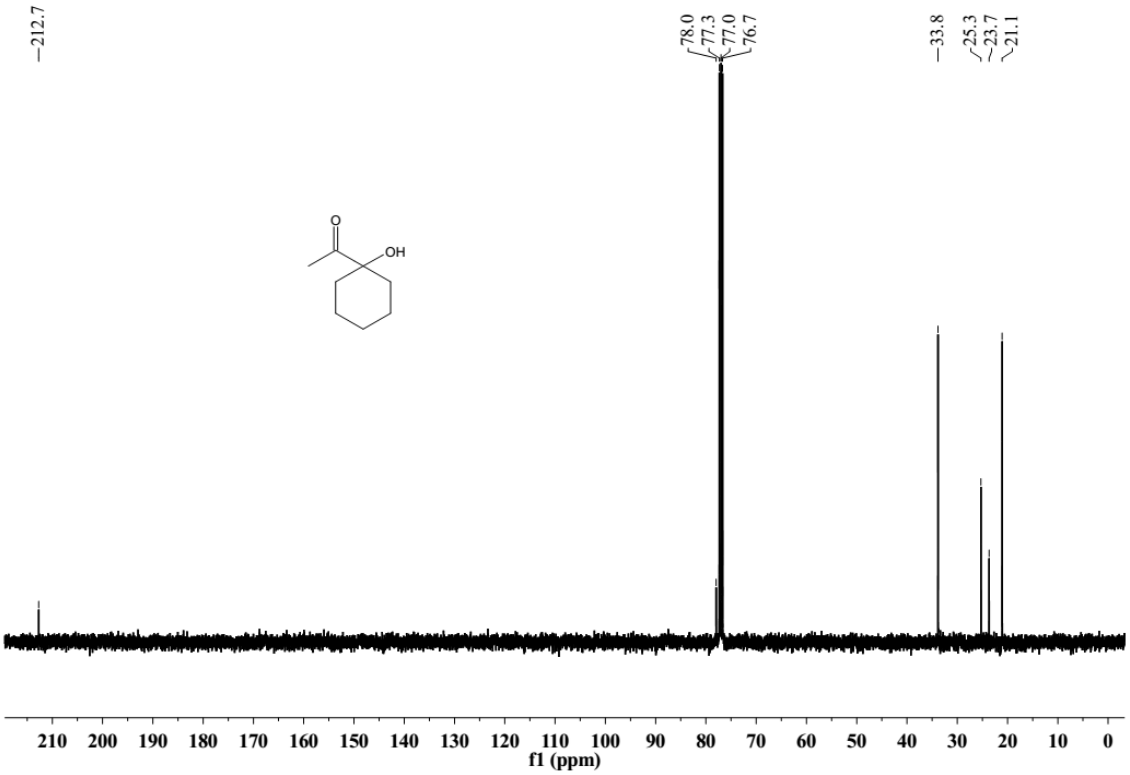
3-hydroxy-3-phenylbutan-2-one (2s)



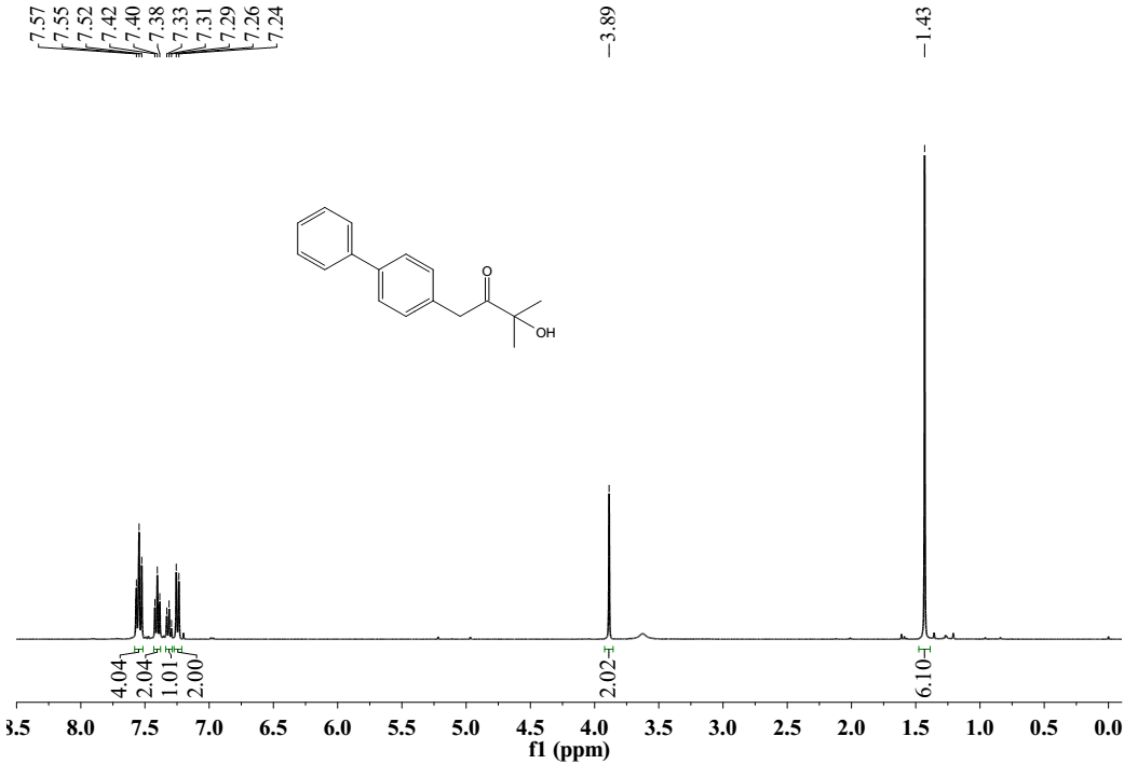


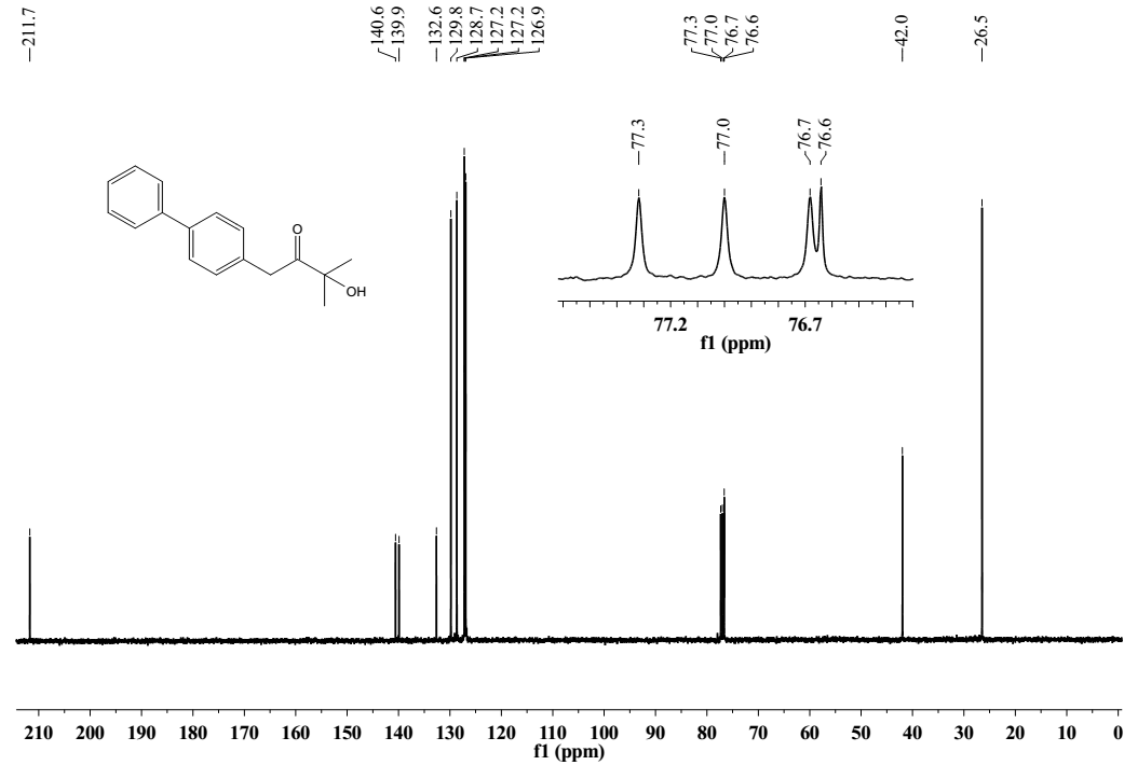
1-(1-hydroxycyclohexyl)ethanone (2t)



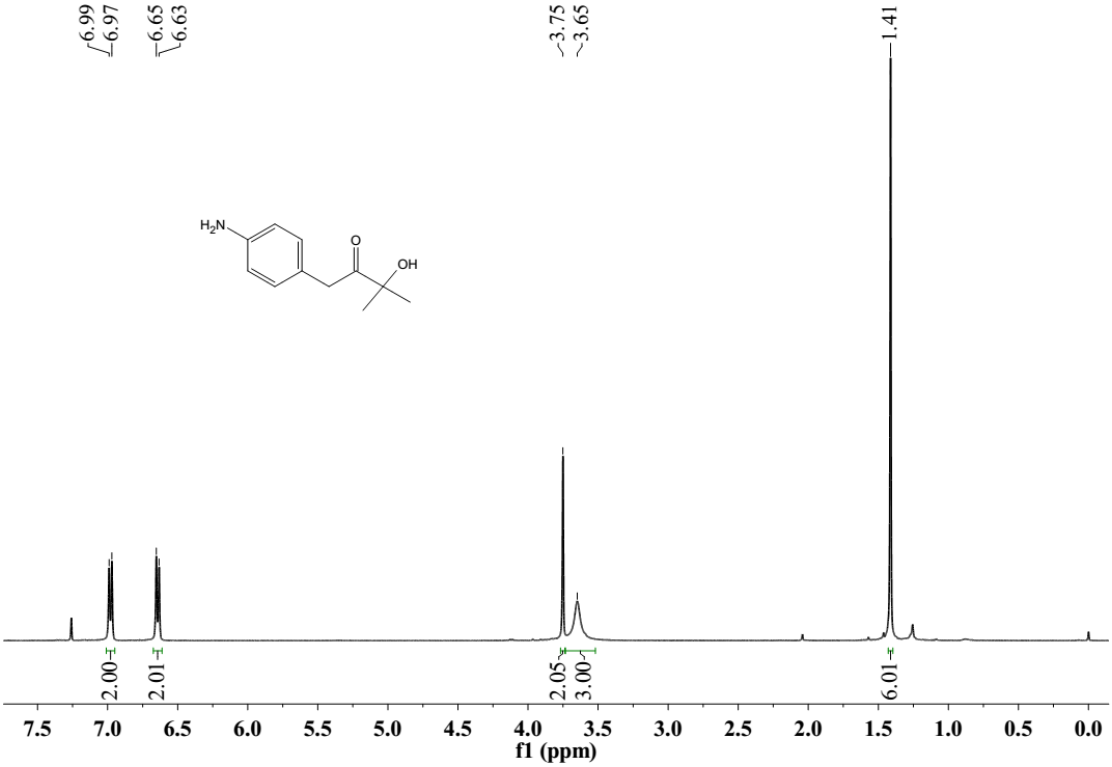


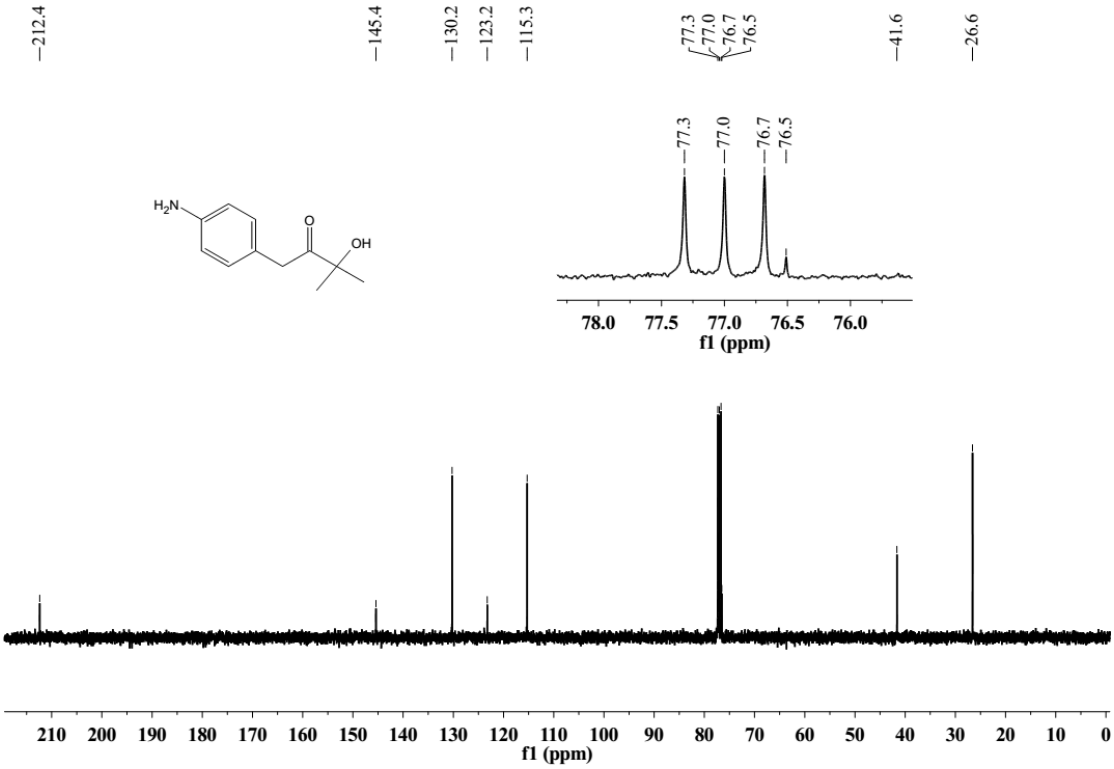
1-(biphenyl-4-yl)-3-hydroxy-3-methylbutan-2-one (3)



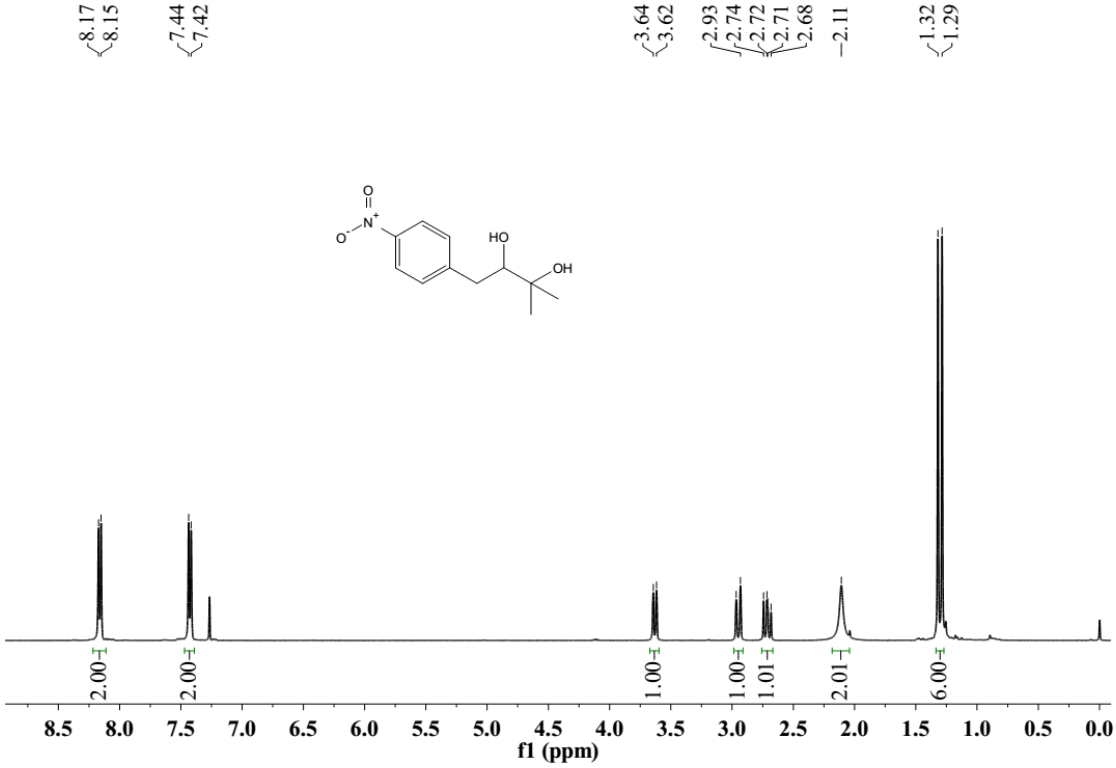


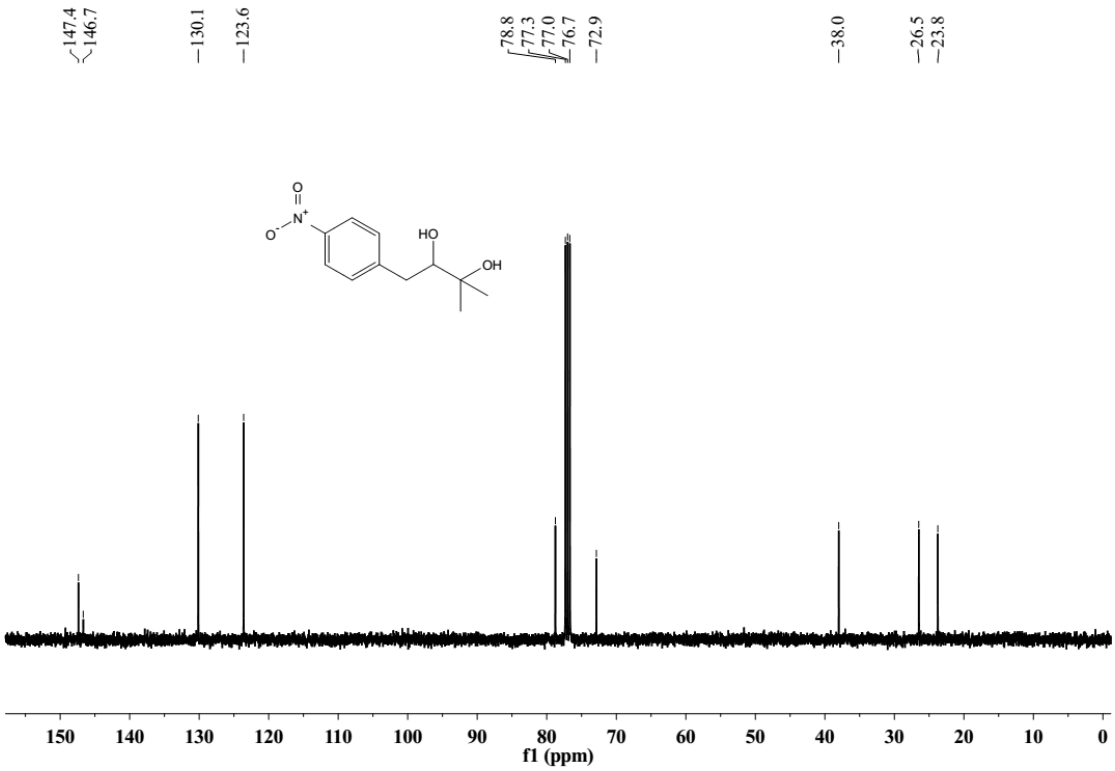
1-(4-aminophenyl)-3-hydroxy-3-methylbutan-2-one (4)





3-methyl-1-(4-nitrophenyl)butane-2,3-diol (5)





(Z)-4,4-dimethyl-5-(pyridin-2-ylmethylene)-1,3-dioxolan-2-one (6a)