Supporting Information

for

Efficient synthesis of dihydropyrimidinones via a three-component Biginelli-type reaction of urea, alkylaldehyde and arylaldehyde

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General information

Melting points were obtained on a microscopical instrument and are uncorrected. NMR spectra were recorded on a 400 MHz spectrometer and TMS as internal standard. IR spectra were recorded on a FTIR spectrometer. HRMS data were obtained using EI ionization. All reagents and solvents used were commercially available. Column chromatography was carried out on silica gel column (300-400 mesh) with mixed solvents (hexane/ethyl acetate). Optical rotations were determined using a Perkin Elmer Model 341 polarimeter at 20 °C. The enantiomeric excesses (ee) were determined by chiral HPLC analysis on a Daicel Chiralpak AD-H column.

General procedure for the iodine-catalyzed synthesis of DHPMs 4

Mono-substituted urea **1** (2.5 mmol), aromatic aldehyde **3** (3.75 mmol) and iodine (0.25 mmol) were dissolved in 3 mL acetonitrile under nitrogen atmosphere. After the mixture was stirred at room temperature for 10 minutes, alkylaldehyde **2** (2.5 mmol) was added, and the resulting mixture was stirred under reflux for 12 hours. Then the reaction mixture was diluted with ethyl acetate, and washed with a solution of sodium thiosulfate followed by water. The organic phase was dried over anhydrous Na_2SO_4, and evaporation of the solvent followed by purification on silica gel afforded the pure desired DHPM **4**.

General procedure for the catalytic asymmetric synthesis of DHPMs 4.

Mono-substituted urea **1** (0.2 mmol), aromatic aldehyde **3** (0.3 mmol), MS 4 Å (0.1 g) and **5a** (0.02 mmol) were dissolved in 1 mL toluene under nitrogen atmosphere. After the mixture was stirred at room temperature for 30 minutes, alkyl aldehyde **2** (0.2 mmol) was added, and the resulting mixture was stirred at room temperature for 2 days. Then the reaction mixture was purified by flash chromatography on silica gel to afford the pure desired chiral DHPM **4**.

Characterization data

*1-Methyl-4-(4-nitrophenyl)-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4a**).* Yellow solid, m.p.186-188 °C; the chiral material was determined to be of 77% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / *i*-propanol = 80/20, l.0 mL/min, λ = 254 nm, t (major) = 16.60 min, t (minor) = 22.42 min]. $[\alpha]_D^{20}$ = + 5.6° (c = 0.52, $CHCl_3$). IR(neat) *v* 3250, 1682, 1651, 1519, 1497, 1455, 1403, 1270, 818, 736, 732, 700 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ =8.13 (d, *J* = 8.7 Hz, 2 H), 7.46 (d, *J* = 8.7 Hz, 2 H), 7.26-7.12 (m, 5 H), 6.55 (s, 1 H), 6.14 (s, 1 H), 5.60 (d, *J* = 2.6 Hz, 1 H), 3.20 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ= 153.24, 149.21, 147.53, 135.30, 128.78, 127.95, 127.65, 127.06, 124.86, 124.21, 112.81, 57.34, 34.79 ppm. HRMS (EI): m/z calcd for ($C_{17}H_{15}N_3O_3$): 309.1113; found: 309.1111.

*1-Methyl-4-(3-nitrophenyl)-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4b**).* Yellow solid, m.p.180-182 °C; the chiral material was determined to be of 75% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / *i*-propanol = 80/20, l.0 mL/min, λ = 254 nm, t (major) = 14.72 min, t (minor) = 21.31 min]. $[\alpha]_D^{20}$ = +8.2° (c = 0.46, $CHCl_3$). IR（neat）*v* 3239, 3084, 2928, 1681, 1599, 1497, 1456, 1404, 1349, 1270, 759, 737, 693, 596 cm^{-1}； ^1H NMR (400 MHz, $CDCl_3$) δ =8.16 (s, 1 H), 8.08 (d, *J* = 8.2 Hz, 1 H), 7.63 (d, *J* = 7.7 Hz, 1 H), 7.45 (t, *J* = 7.9 Hz, 1 H), 7.26 (s, 1 H), 7.22 (s, 1 H), 7.16 (t, *J* = 6.6 Hz, 3 H), 6.63 (s, 1 H), 6.28 (s, 1 H), 5.60 (d, *J* = 2.5 Hz, 1 H), 3.23 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ= 153.41, 148.48, 144.36, 135.27, 132.8 , 129.84, 128.76, 128.22, 126.93, 124.63, 122.98, 122.02, 112.39, 56.96, 34.79 ppm. HRMS (EI): m/z calcd for ($C_{17}H_{15}N_3O_3$): 309.1113; found: 309.1112.

*4-(1-Methyl-2-oxo-5-phenyl-1,2,3,4-tetrahydropyrimidin-4-yl)benzonitrile (**4c**).* White solid, m.p. 113-115 °C; the chiral material was determined to be of 64% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / *i*-propanol = 80/20, l.0 mL/min, λ = 254 nm, t (major) = 16.34 min, t (minor) = 26.66 min]. $[\alpha]_D^{20}$ = +4.8° (c = 0.41, $CHCl_3$); IR(neat) *v* 3249, 2925, 2228, 1679, 1603, 1498, 1456, 1404, 1327, 1269, 1108, 765, 731, 694, 587 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.2 Hz, 2 H), 7.38 (d, *J* = 7.9 Hz, 2 H), 7.26-7.19 (m, 2 H), 7.14 (dd, *J* = 17.6, 7.4 Hz, 3 H), 6.53 (s, 1 H), 6.08 (br, 1 H), 5.52 (s, 1 H), 3.17 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.26, 147.33, 135.37, 132.72, 128.72, 127.90, 127.47, 126.96, 124.81, 118.44, 112.77, 111.83, 57.50, 34.72 ppm. HRMS (EI): m/z calcd for ($C_{18}H_{15}N_3O$): 289.1215; found: 289.1225.

*4-(4-Chlorophenyl)-1-methyl-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4d**).* White solid ,m.p: 149-151℃; the chiral material was determined to be of 32% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / *i*-propanol =90/10,0.8 mL/min, λ = 254 nm, t (major) = 29.13min, t (minor) = 31.84 min]. IR (neat) *v* 3108, 1671, 1647, 1593, 1526, 1491, 1462, 1401, 1328, 726, 700 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ= 7.21 (m, 9 H), 6.53 (s, 1 H), 5.51 (s, 1 H), 5.44 (d, *J* = 2.1 Hz, 1 H), 3.19 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ=140.88, 135.67, 133.68, 129.02, 128.57, 128.13, 127.48, 126.70, 124.81, 124.81, 113.30, 57.32, 34.69 ppm. HRMS (EI): m/z calcd for ($C_{17}H_{15}ClN_2O$): 298.0873; found: 298.0870.

*4-(4-Bromophenyl)-1-methyl-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (4**e**).* White solid, m.p. 170-172 °C; the chiral material was determined to be of 53% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / *i*-propanol = 80/20, l.0 mL/min, λ = 254 nm, t (major) = 13.68 min, t (minor) = 18.87 min]. $[\alpha]_D^{20}$ = +3° (c = 0.44, $CHCl_3$). IR(neat) *v* 3265, 1673, 1485, 1455, 1405, 1325, 1266, 1003, 754, 730, 694 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ =7.41 (d, J = 8.3 Hz, 2 H), 7.25-7.19 (m, 2 H), 7.15 (t, J = 7.8 Hz, 5 H), 6.52 (s, 1 H), 5.56 (s, 1 H), 5.43 (d, J = 2.1 Hz, 1 H), 3.19 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ =153.29, 141.39, 135.71, 132.00, 128.59, 128.47, 127.51, 126.75, 124.85, 121.93, 113.30, 57.46, 34.69 ppm. HRMS (EI): m/z calcd for ($C_{17}H_{15}BrN_2O$): 342.0368; found: 340.0211.

*4-(2-Bromophenyl)-1-methyl-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4f**).* White solid, m.p: 173-175℃; the chiral material was determined to be of 20% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / *i*-propanol = 90/10, 0.8 mL/min, λ = 254 nm, t (major) = 29.30 min, t (minor) = 31.37min]. IR (neat) *v* 3242, 3080, 2923, 2238, 1678, 1597, 1496, 1470, 1402, 1325, 1262, 1185, 1111, 757, 730, 695, 599 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ =7.46 (s, 1 H), 7.39 (d, J = 7.8 Hz, 1 H), 7.23 (m, 3 H), 7.17 (m, 4 H), 6.58 (s, 1 H), 5.52 (s, 1 H), 5.43 (d, J = 2.3 Hz, 1 H), 3.23 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ=153.14, 144.57, 135.65, 131.17, 130.51, 129.87, 128.60, 127.78, 126.73, 125.41, 124.77, 122.99, 112.83, 57.58, 34.76 ppm. HRMS (EI): m/z calcd for ($C_{17}H_{15}BrN_2O$): 342.0368; found: 340.0207.

*1-Methyl-5-phenyl-4-(4-(trifluoromethyl)phenyl)-3,4-dihydropyrimidin-2(1H)-one (**4g**).* White solid, m.p. 130-132 °C; IR(neat) *v* 3318, 1663, 1598, 1496, 1451, 1406, 1324, 1167, 1125, 1067, 1009, 855, 730, 691 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ= 7.55 (d, *J* = 8.2 Hz, 2 H), 7.41 (d, *J* = 8.1 Hz, 2 H), 7.26-7.21 (m, 2 H), 7.17 (t, *J* = 6.7 Hz, 3 H), 6.56 (s, 1 H), 5.67 (s, 1 H), 5.54 (d, *J* = 2.4 Hz, 1 H), 3.21 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ =153.31, 146.15, 135.61, 130.20 (*J*=33 Hz), 128.70, 127.77, 126.88, 125.98 (*J*=229 Hz), 125.97, 125.93, 125.27, 113.14, 57.59, 34.74 ppm. HRMS (EI): m/z calcd for ($C_{18}H_{15}F_3N_2O$): 332.1136; found: 332.1138.

*1-Methyl-4,5-diphenyl-3,4-dihydropyrimidin-2(1H)-one (**4h**).* White solid, m.p. 179-181 °C; IR (neat) *v* 3319, 1681, 1651, 1599, 1495, 1454, 1403, 1326, 1269, 754, 732, 697, 595 cm^{-1}: ^1H NMR (400 MHz, $CDCl_3$) δ = 7.33-7.14 (m, 10 H), 6.55 (s, 1 H), 5.44 (d, J = 2.2 Hz, 1 H), 5.31 (s, 1 H), 3.20 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ = 153.26, 142.37, 136.05, 128.93, 128.48, 128.04, 127.38, 126.70, 126.55, 124.88, 113.60, 58.22, 34.68 ppm. HRMS (EI): m/z calcd for ($C_{17}H_{16}N_2O$): 264.1263; found: 264.1264.

*Methyl-5-phenyl-4-(p-tolyl)-3,4-dihydropyrimidin-2(1H)-one (**4i**).* White solid, m.p. 158-160 °C; IR (neat) *v* 3247, 3058, 2920, 1678, 1496, 1455, 1402, 1328, 1271, 1105, 1048, 751, 682, 593 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ =7.24-7.05 (m, 9 H), 6.53 (s, 1 H), 5.40 (d, *J* = 2.0 Hz, 1 H), 5.29 (s, 1 H), 3.19 (s, 3 H), 2.28 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ= 153.29, 139.50, 137.73, 136.13, 129.58, 128.45, 127.26, 126.60, 126.48, 124.84, 113.71, 57.89, 34.66, 21.04 ppm. HRMS (EI): m/z calcd for ($C_{17}H_{18}N_2O_2$): 278.1419; found: 278.1418.

*4-(4-Methoxyphenyl)-1-methyl-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4j**).* White solid,m.p: 168-170°C;the chiral material was determined to be of 33% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / *i*-propanol = 90/10, 0.8 mL/min, λ = 254 nm, t (major) = 43.37min, t (minor) = 48.50min]; IR (neat) *v* 3248, 1681, 1607, 1509, 1455, 1403, 1326, 1251, 1174, 752 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.25-7.19 (m, 4 H), 7.16 (m, 3 H), 6.81 (d, *J* = 8.6 Hz, 2 H), 6.53 (s, 1 H), 5.39 (d, *J* = 2.1 Hz, 1 H), 5.27 (s, 1 H), 3.76 (s, 3 H), 3.20 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.30, 153.21, 136.13, 134.71, 128.46, 127.94, 127.19, 126.51, 124.87, 114.25, 113.81, 57.68, 55.17, 34.67 ppm. HRMS(EI): m/z calcd for ($C_{18}H_{18}N_2O_2$): 294.1368; found: 294.1369.

*4-(Benzo[d][1,3]dioxol-5-yl)-1-methyl-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4k**).* White solid, m.p. 202-204 °C; IR(neat) *v* 3319, 2917, 1675, 1590, 1499, 1486, 1399, 1334, 1246, 1108, 1033, 685, 593 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.27-7.13 (m, 5 H), 6.84-6.68 (m, 3 H), 6.56 (s, 1 H), 5.92 (s, 2 H), 5.37 (d, *J* = 1.9 Hz, 1 H), 5.28 (s, 1 H), 3.22 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.13, 148.21, 147.38, 136.54, 135.95, 128.50, 127.35, 126.58, 124.79, 120.19, 113.52, 108.35, 107.11, 101.08), 58.00, 34.69 (s) ppm. HRMS (EI): m/z calcd for ($C_{18}H_{16}N_2O_3$): 308.1161; found: 308.1165.

*1-Methyl-4-(naphthalen-1-yl)-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4l**).* White solid, m.p. 162-164 °C; IR (neat) *v* 3238, 3059, 2925, 1678, 1598, 1497, 1455, 1403, 1327, 1266, 95, 780, 731, 694 $^{-1}$cm ; ^1H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.4 Hz, 1 H), 7.91 (d, *J* = 8.1 Hz, 1 H), 7.79 (d, *J* = 8.2 Hz, 1 H), 7.61 (t, *J* = 7.6 Hz, 1 H), 7.53 (m, 2H), 7.38 (t, *J* = 7.7 Hz, 1 H), 7.18 – 7.07 (m, 5 H), 6.84 (s, 1 H), 6.28 (d, *J* = 1.7 Hz, 1 H), 5.34 (s, 1 H), 3.23 (s, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.17, 135.84, 135.79, 134.41, 130.25, 129.37, 128.83, 128.49, 128.40, 126.92, 126.48, 125.78, 125.09, 124.37, 121.77, 112.11, 53.62, 34.76 ppm. HRMS (EI): m/z calcd for ($C_{21}H_{18}N_2O$): 314.1419; found: 314.1419.

*4-(Furan-2-yl)-1-methyl-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4m**).* White solid, m.p. 161-163 °C; IR (neat) *v* 3241, 2921, 1678, 1599, 1497, 1455, 1403, 1326, 1250, 1108, 1000, 780, 748, 694, 593 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.36 (s, 1 H), 7.30 (d, *J* = 7.8 Hz, 1 H), 7.26 (t, *J* = 5.9 Hz, 3 H), 7.20 (t, *J* = 6.9 Hz, 1 H), 6.61 (s, 1 H), 6.27 (s, 1 H), 6.16 (d, *J* = 2.5 Hz, 1 H), 5.48 (s, 1 H), 5.39 (s, 1 H), 3.21 (s, 3 H) ppm . ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.17, 153.66, 142.55, 135.61, 128.60, 128.09, 126.66, 124.35, 111.13, 110.38, 106.94, 51.08, 34.77 ppm. HRMS (EI): m/z calcd for ($C_{15}H_{14}N_2O_2$): 254.1055; found: 254.1051.

*4-Benzyl-1-methyl-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4n**).* White solid, m.p. 91-93 °C; IR (neat) *v* 3238, 1681, 1599, 1495, 1454, 1408, 132, 1269,752,698 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.40-7.33 (m, 4H), 7.31 (t, *J* = 7.3 Hz, 2 H), 7.25 (t, *J* = 6.9 Hz, 2 H), 7.17 (d, J = 7.3 Hz, 2 H), 6.32 (s, 1 H), 5.15 (s, 1 H), 4.66 (d, *J* = 8.4 Hz, 1 H), 3.00 (s, 3 H), 2.90 (m, 1 H), 2.77-2.66 (m, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.97, 136.63, 135.85, 129.64, 128.88, 128.44, 127.16, 126.72, 126.67, 124.50, 114.11, 55.21, 42.41, 34.37 ppm ; HRMS (EI): m/z calcd for ($C_{18}H_{18}N_2O$): 278.1419; found: 278.1418.

*5-Isopropyl-1-methyl-4-(4-nitrophenyl)-3,4-dihydropyrimidin-2(1H)-one (**4o**).* Yellow liquid, IR (neat) *v* 3249, 2940, 1678, 1521, 1462, 1341, 1262, 1113, 858, 688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.20 (d, *J* = 8.6 Hz, 2 H), 7.46 (d, *J* = 8.6 Hz, 2 H), 5.86 (s, 1 H), 5.56 (s, 1 H), 5.08 (d, *J* = 1.9 Hz, 1 H), 3.10 (s, 3 H), 1.95 (m, 1 H), 1.00 (d, *J* = 6.8 Hz, 3 H), 0.95 (d, *J* = 7.0 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.20, 150.19, 147.67, 127.87, 124.28, 124.16, 119.07, 58.06, 34.45, 28.43, 21.98, 20.73 ppm. HRMS (EI): m/z calcd for ($C_{14}H_{17}N_3O_3$): 275.1270; found: 275.1269.

*5-Butyl-1-methyl-4-(4-nitrophenyl)-3,4-dihydropyrimidin-2(1H)-one (**4p**).* Yellow solid, m.p. 121-123 °C; IR (neat) *v* 3258, 2956, 2929, 2869, 1673, 1596, 1520, 1465, 1396, 1346, 1310, 1268, 1108, 1036, 855, 754, 697 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 8.6 Hz, 2 H), 7.45 (d, *J* = 8.6 Hz, 2 H), 5.80 (s, 1 H), 5.37 (s, 1 H), 5.02 (s, 1 H), 3.07 (s, 3 H), 1.72 (d, *J* = 4.5 Hz, 2 H), 1.35-1.18 (m, 4 H), 0.83 (t, *J* = 6.9 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.20, 149.87, 147.61, 127.81, 125.17, 124.05, 112.71, 58.95, 34.27, 29.88, 29.14, 22.10, 13.74 ppm. HRMS (EI): m/z calcd for ($C_{15}H_{19}N_3O_3$): 289.1426; found: 289.1421.

*1-Methyl-4-(4-nitrophenyl)-5-pentyl-3,4-dihydropyrimidin-2(1H)-one (**4q**).* Yellow solid, m.p. 127-129 °C; IR (neat) *v* 3259, 295, 2867, 1674, 1520, 1466, 1346, 1268, 1110, 854, 753, 697 cm^{-1}; ^1H NMR (400 MHz,

$CDCl_3$) δ 8.21 (d, *J* = 8.6 Hz, 2 H), 7.47 (d, *J* = 8.6 Hz, 2 H), 5.83 (s, 1 H), 5.31 (s, 1 H), 5.04 (s, 1 H), 3.09 (s, 3 H), 1.72 (t, 2 H), 1.32-1.15 (m, 6 H), 0.86 (t, *J* = 6.8 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.13, 149.85, 147.67, 127.83, 125.19, 124.09, 112.76, 59.07, 34.31, 31.21, 30.18, 26.70, 22.34, 13.90 ppm. HRMS (EI): m/z calcd for ($C_{16}H_{21}N_3O_3$): 303.1583; found: 303.1583.

*1-Ethyl-4-(4-nitrophenyl)-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4r**).* Yellow solid, m.p. 182-184 °C; the chiral material was determined to be of 32% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / *i*-propanol = 80/20, 0.8 mL/min, λ = 254 nm, t (major) = 28.16 min, t (minor) = 37.97 min]. $[\alpha]_D^{20}$ = +14.8°; (c = 0.36,$CHCl_3$); IR (neat) *ν* 3299, 2932, 1678, 1598, 1520, 1497, 1453, 1347, 1271, 1236, 1121, 817, 757, 731, 699 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.4 Hz, 2 H), 7.46 (d, *J* = 8.4 Hz, 2 H), 7.24 (d, *J* = 7.4 Hz, 2 H), 7.17 (m, 3 H), 6.58 (s, 1 H), 5.97 (s, 1 H), 5.58 (s, 1 H), 3.63 (m, 2 H), 1.27 (t, *J* = 7.1 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.75, 149.32, 147.49, 135.46, 128.75, 127.61, 126.99, 126.53, 124.80, 113.05, 57.12, 42.17, 14.43 ppm. HRMS (EI): m/z calcd for ($C_{18}H_{17}N_3O_3$): 323.1270; found: 323.1270.

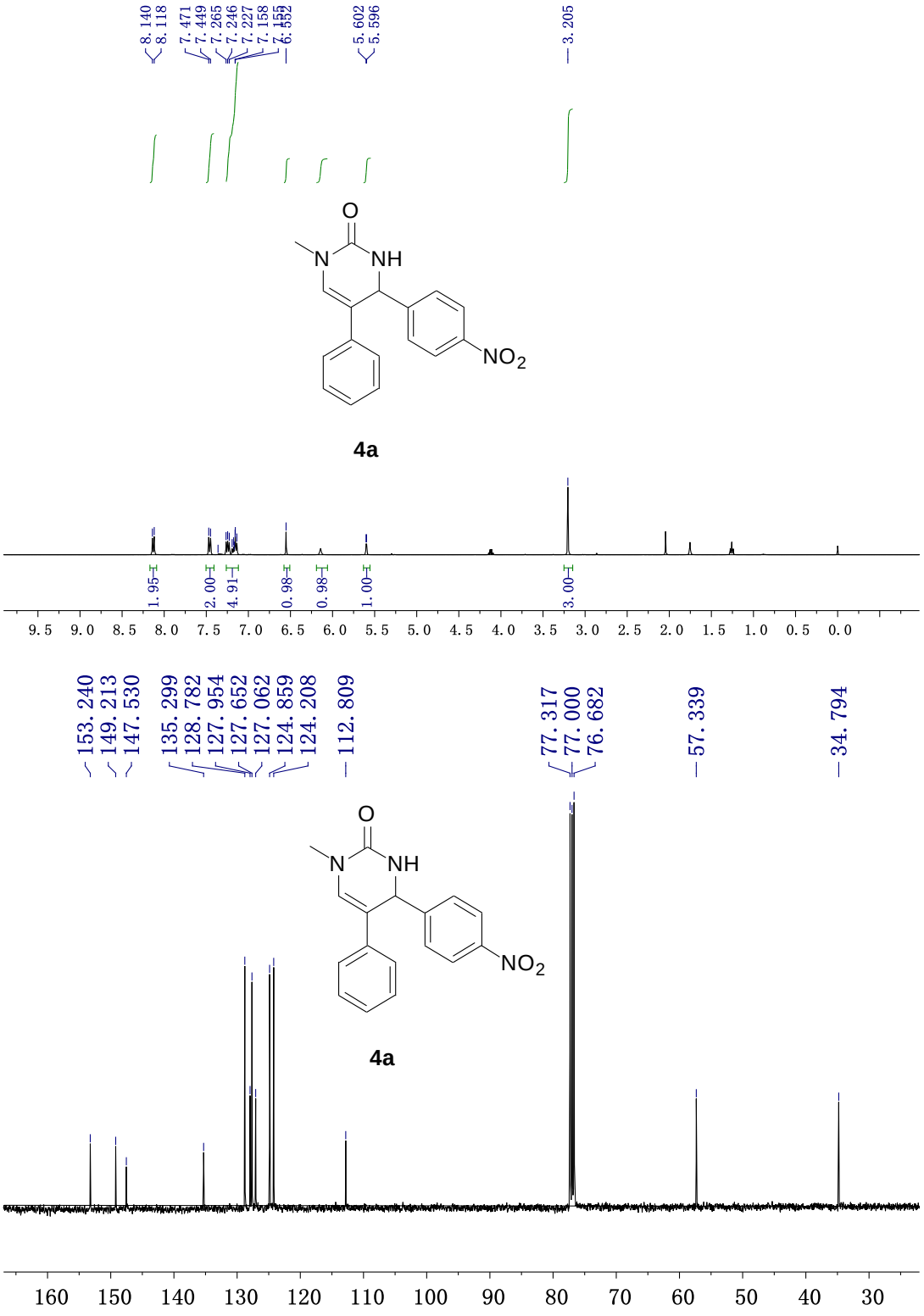
*1-Ethyl-4-(3-nitrophenyl)-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4s**).* Light yellow solid, m.p. 150-152 °C; IR (neat) *ν* 3226, 3083, 2932, 1678, 1599, 1529, 1497, 1453, 1424, 1349, 1269, 1235, 1122, 1083, 909, 803, 758, 736, 693, 593 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (s, 1 H), 8.09 (d, *J* = 8.2 Hz, 1 H), 7.63 (d, *J* = 7.7 Hz, 1 H), 7.30-7.20 (m, 1 H), 7.23 (d, *J* = 7.2 Hz, 2 H), 7.21-7.12 (m, 3 H), 6.66 (s, 1 H), 6.11 (s, 1 H), 5.57 (d, *J* = 2.5 Hz, 1 H), 3.78-3.53 (m, 2 H), 1.29 (t, *J* = 7.1 Hz, 3 H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 152.72, 148.57, 144.47, 135.41, 132.86, 129.91, 128.76, 126.93, 126.76, 124.64, 123.02, 121.91, 112.88, 77.32, 77.00, 76.68, 57.01, 42.23, 14.45 ppm. HRMS (EI): m/z calcd for ($C_{18}H_{17}N_3O_3$): 323.1270; found: 323.1273.

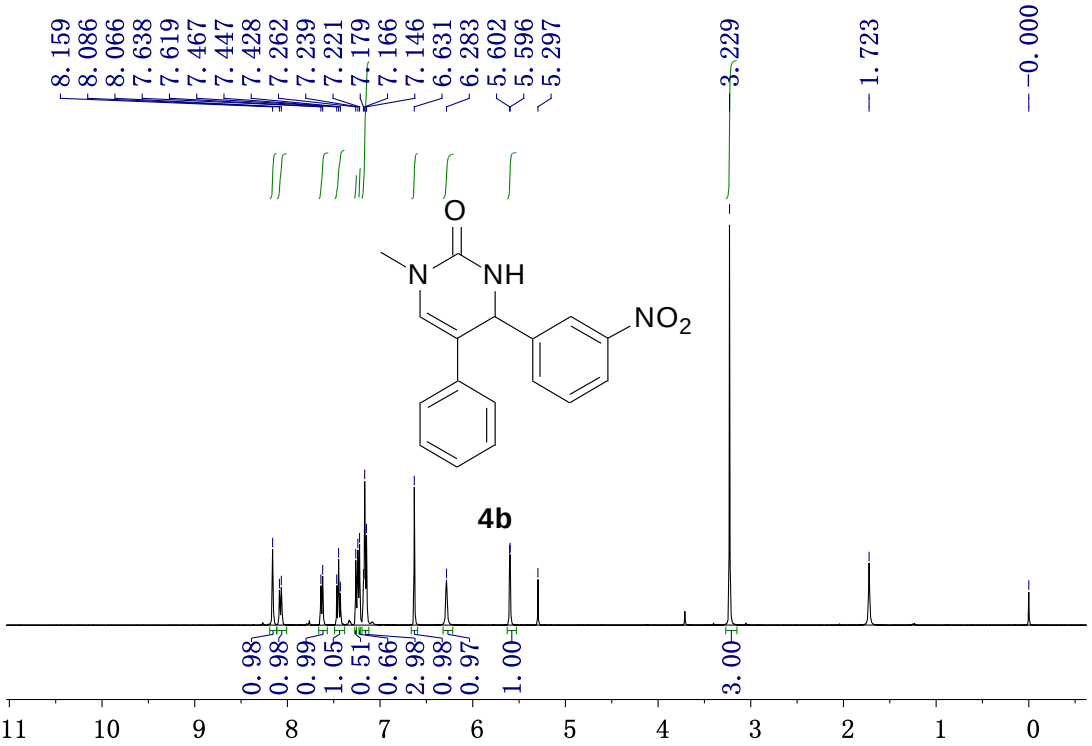
*4-(1-Ethyl-2-oxo-5-phenyl-1,2,3,4-tetrahydropyrimidin-4-yl)benzonitrile (**4t**).* White solid, m.p. 209-211 °C; IR (neat) *ν* 3241, 3083, 2976, 2933, 2228, 1681, 1603, 1498, 1454, 1372, 1270, 1236, 1180, 1122, 909, 851, 803, 765, 732, 695, 592, 554 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.8 Hz, 2 H), 7.40 (d, *J* = 7.9 Hz, 2 H), 7.24 (d, *J* = 7.7 Hz, 2 H), 7.17 (m, 3 H), 6.57 (s, 1 H), 5.52 (s, 1 H), 5.46 (s, 1 H), 3.73 – 3.54 (m, 2 H), 1.27 (t, *J* = 6.7 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.76, 147.41, 135.49, 132.70, 128.69, 127.44, 126.90, 126.46, 124.74, 118.45, 112.99, 111.75, 57.30, 42.11, 14.39 ppm. HRMS (EI): m/z calcd for ($C_{19}H_{17}N_3O$): 303.1372; found: 303.1370.

*4-(4-Chlorophenyl)-1-ethyl-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4u**).* White solid, m.p. 191-193 °C; IR (neat) *ν* 3223, 3082, 2931, 1673, 1598, 1485, 1452, 1427, 1370, 1267, 1235, 1121, 1071, 1010, 907, 801, 756, 731, 693, 593 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (m, 5 H), 7.22- 7.13 (m, 4 H), 6.56 (s, 1 H), 5.76 (s, 1 H), 5.42 (s, 1 H), 3.61 (m,, 2 H), 1.26 (t, *J* = 7.1 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.84, 140.95, 135.88, 133.74, 129.07, 128.58, 128.10, 126.72, 126.05, 124.82, 113.66, 57.26, 42.09, 14.43 ppm. HRMS (EI): m/z calcd for ($C_{18}H_{17}ClN_2O$): 312.1029; found: 312.1029.

*4-(4-Bromophenyl)-1-ethyl-5-phenyl-3,4-dihydropyrimidin-2(1H)-one (**4v**).* White solid, m.p. 189-191 °C; IR(neat) *ν* 3227, 2973, 1676, 1597, 1489, 1452, 1371, 1267, 1235, 1121, 1089, 1014, 907, 802, 757, 730, 694, 594 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.3 Hz, 2 H), 7.25-7.20 (m, 2 H), 7.15 (m,5 H), 5.63 (s, 1 H), 5.40 (d, *J* = 2.3 Hz, 1 H), 3.74 -3.51 (m, 2 H), 1.26 (t, *J* = 7.1 Hz, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.66, 141.50, 135.90, 132.02, 128.58, 128.44, 126.70, 126.13, 124.81, 121.90, 113.50, 57.37, 42.08, 14.44 ppm. HRMS (EI): m/z calcd for ($C_{18}H_{17}BrN_2O$): 356.0524; found: 356.0524.

NMR spectra for compounds 4a-v

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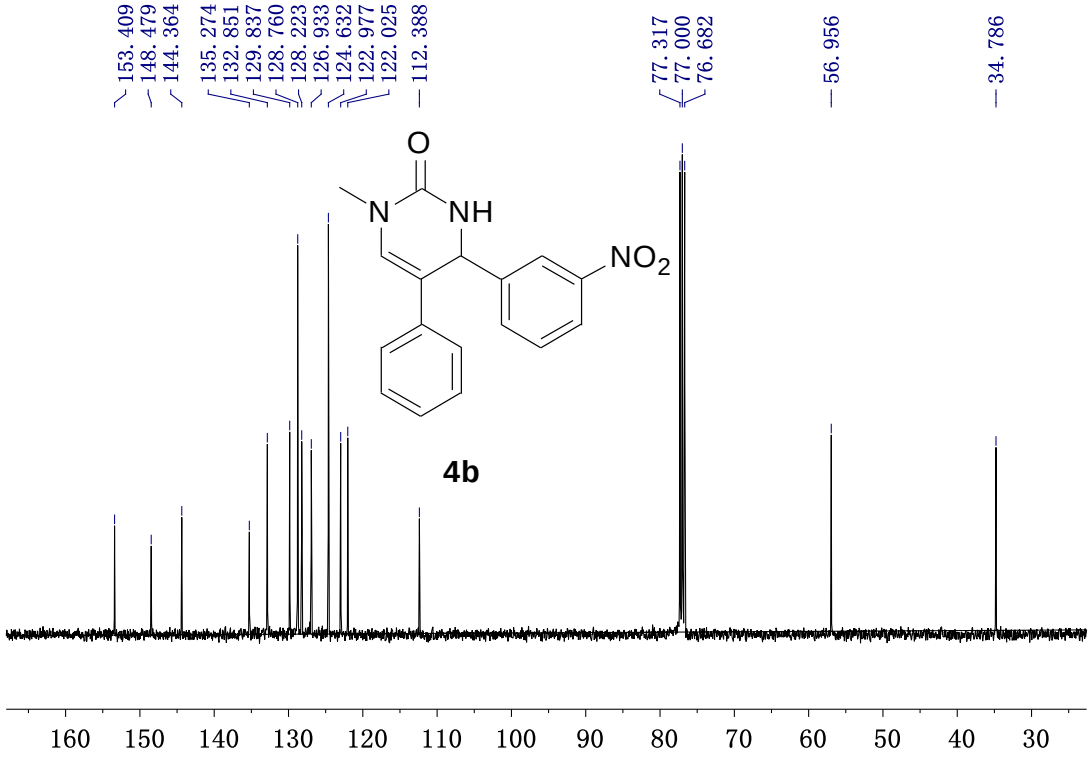
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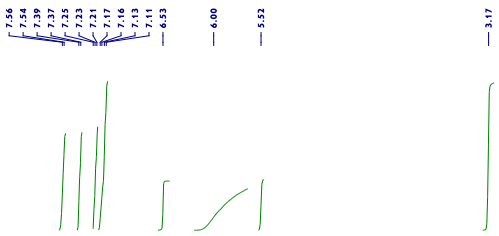
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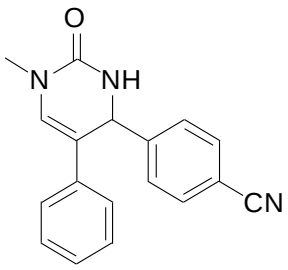
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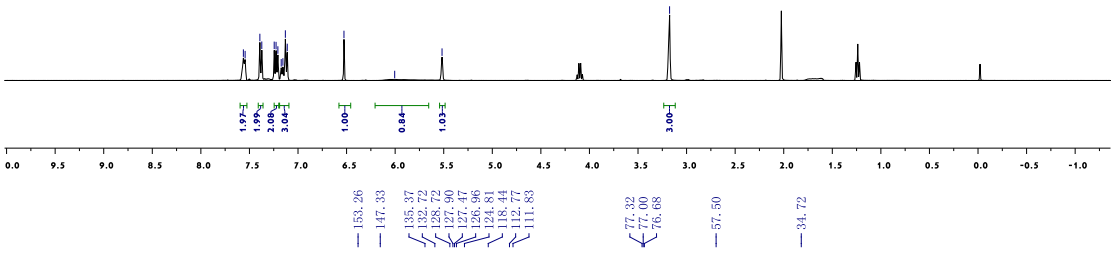
4c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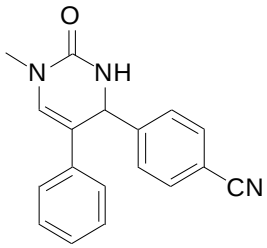
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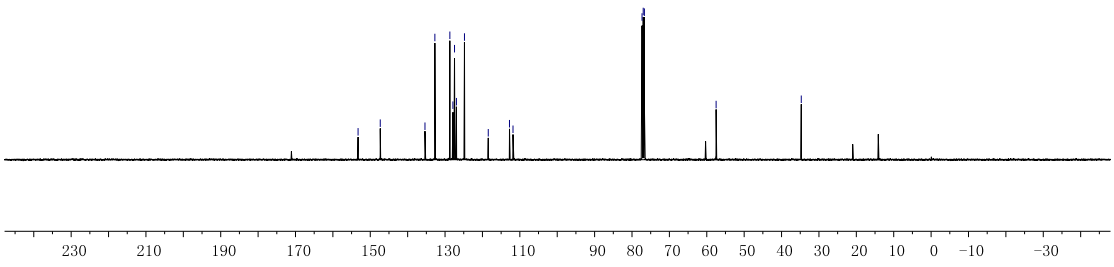
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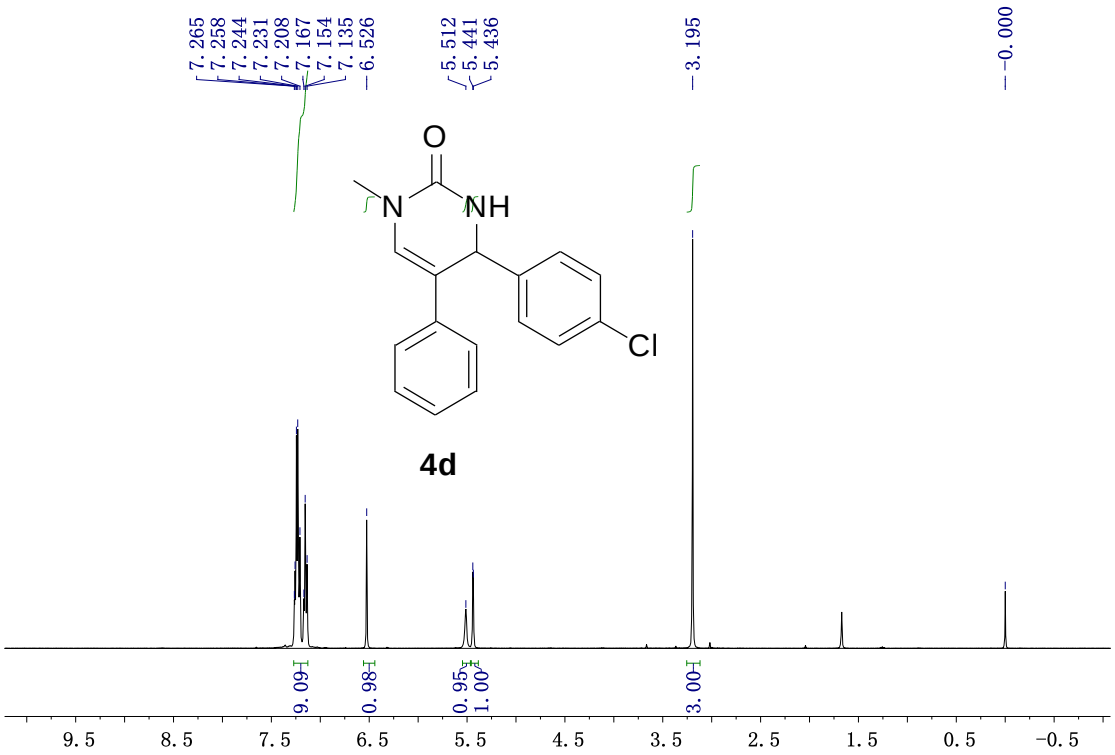
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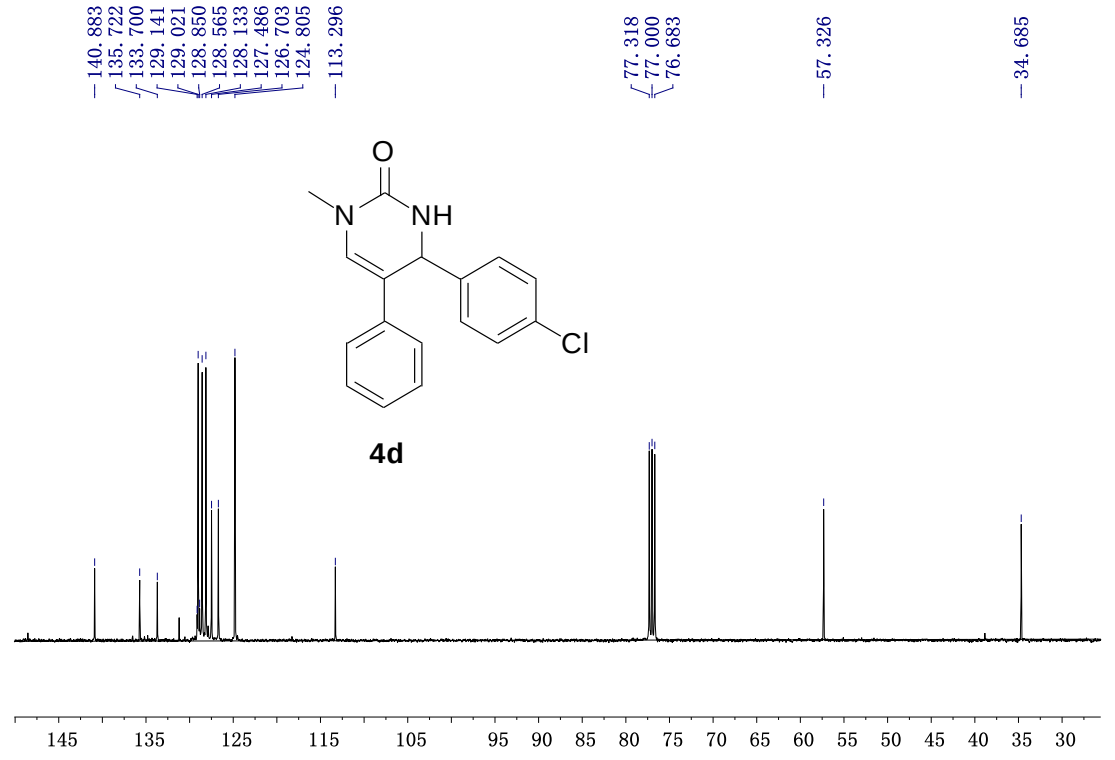
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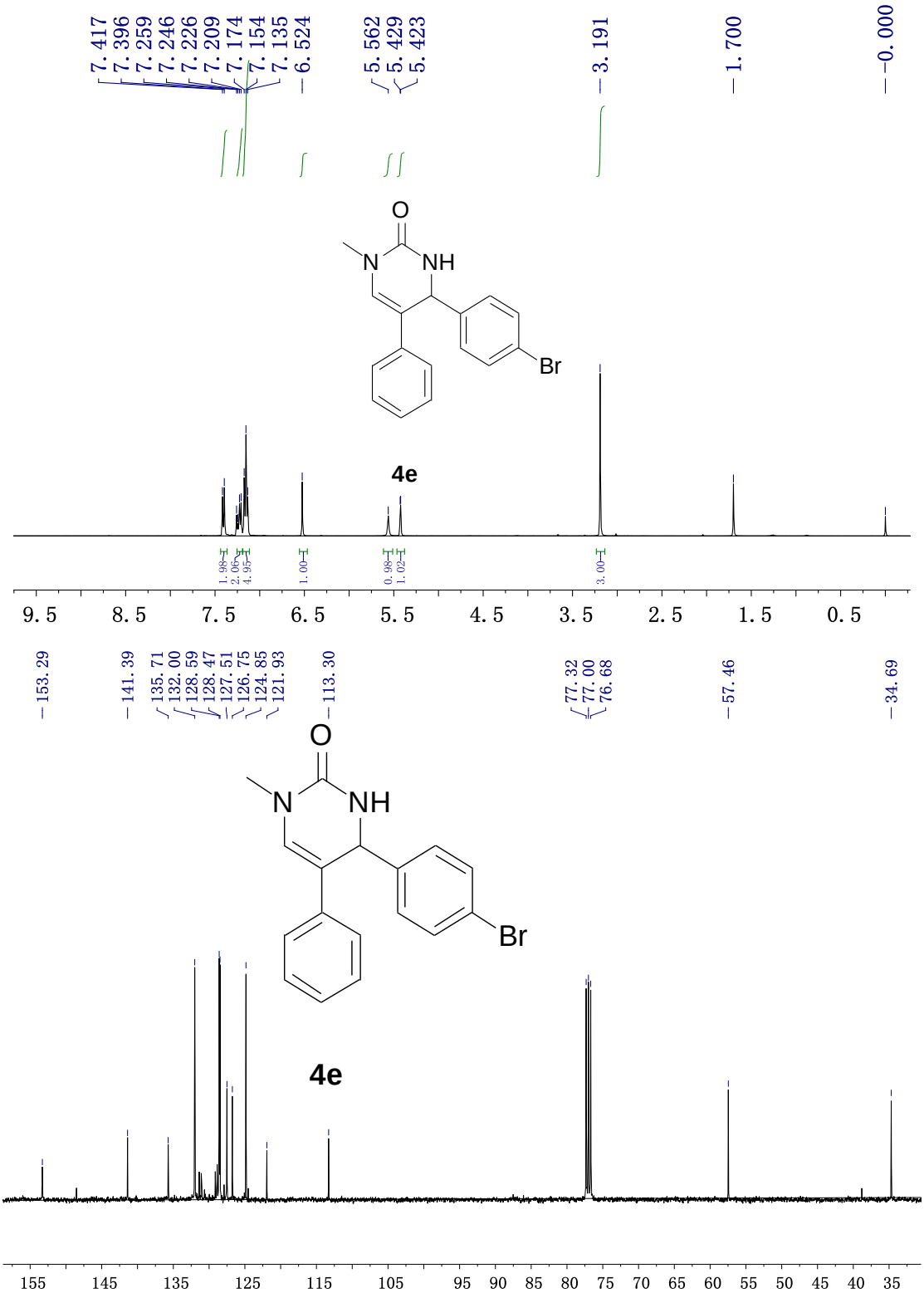
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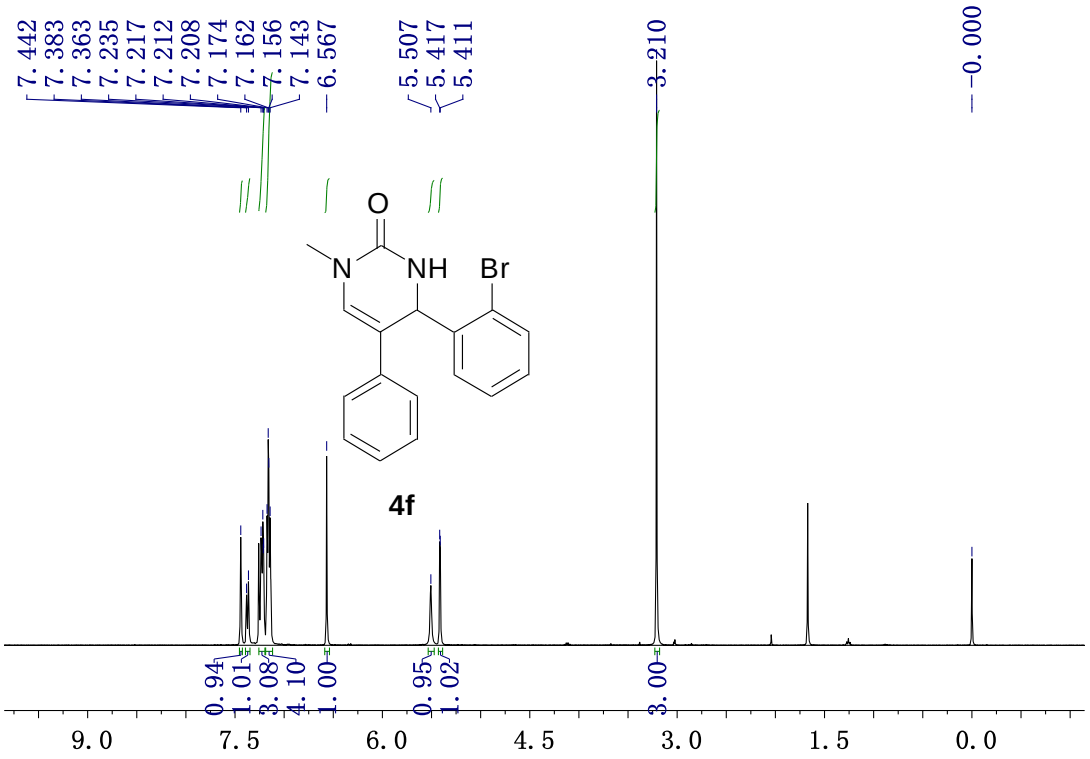
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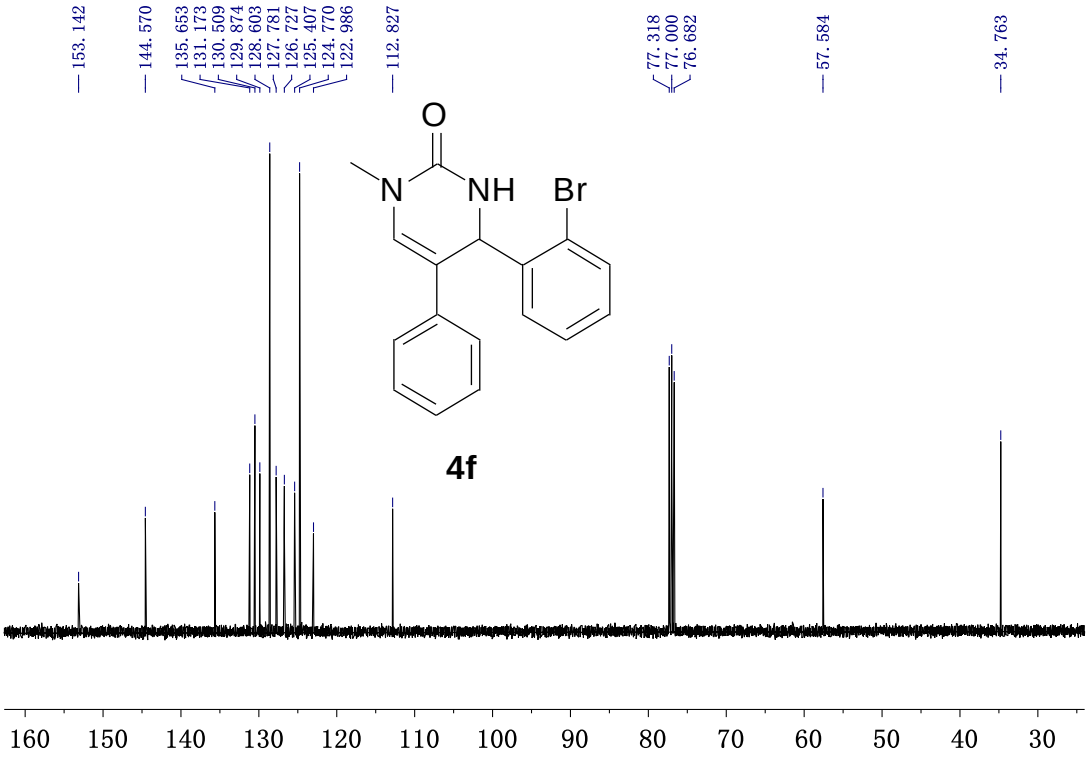
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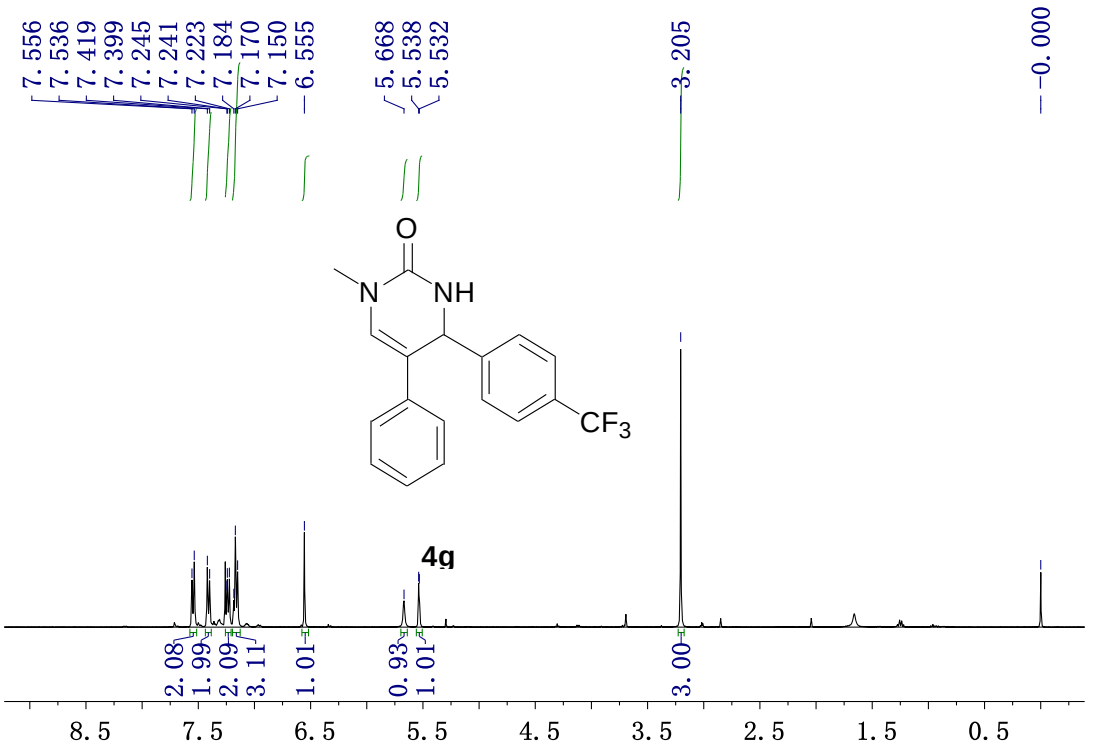
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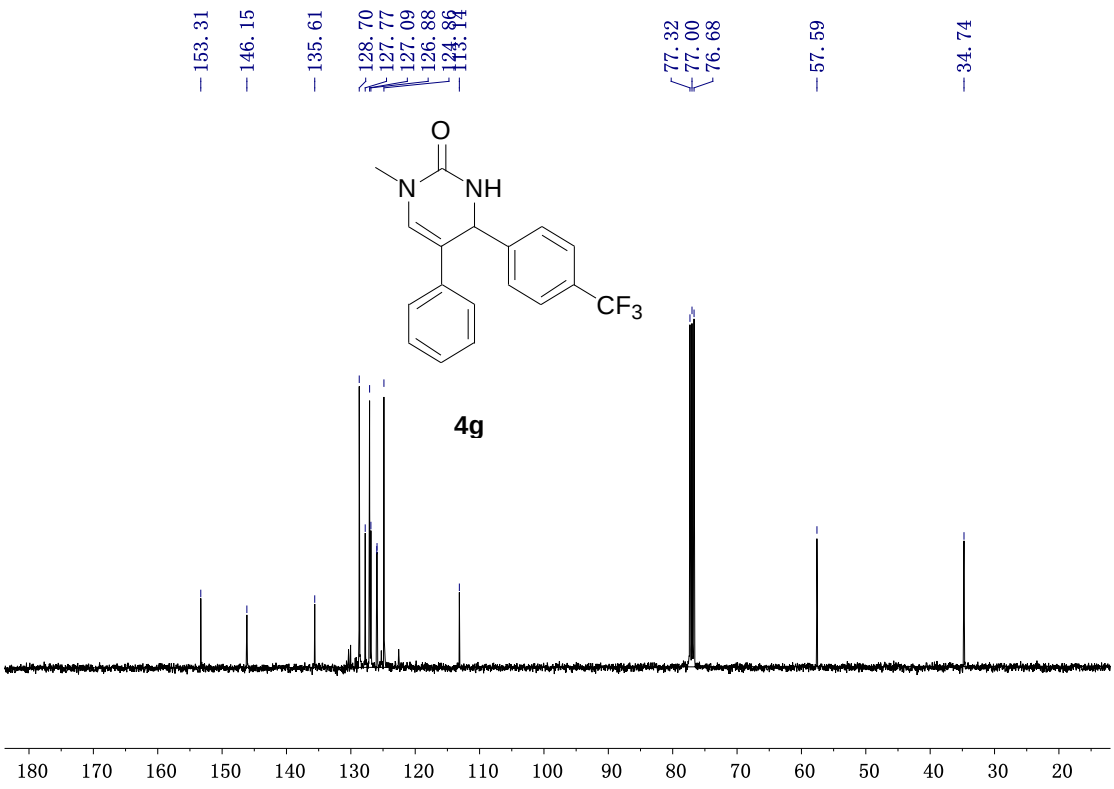
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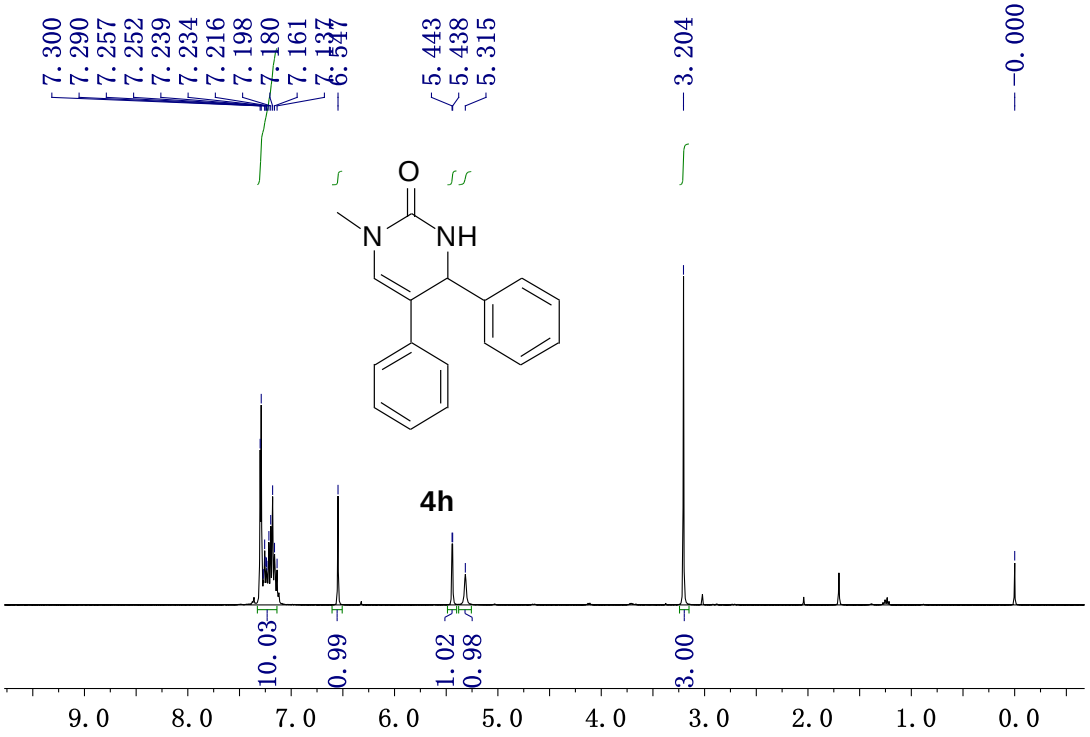
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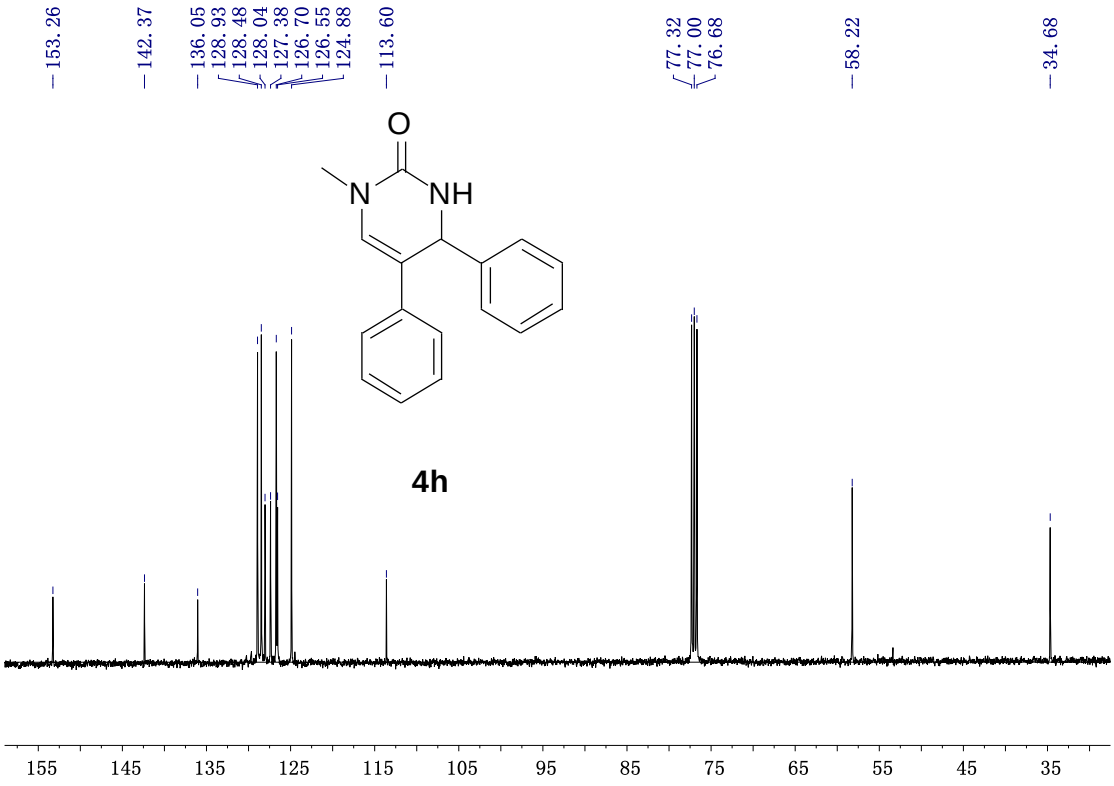
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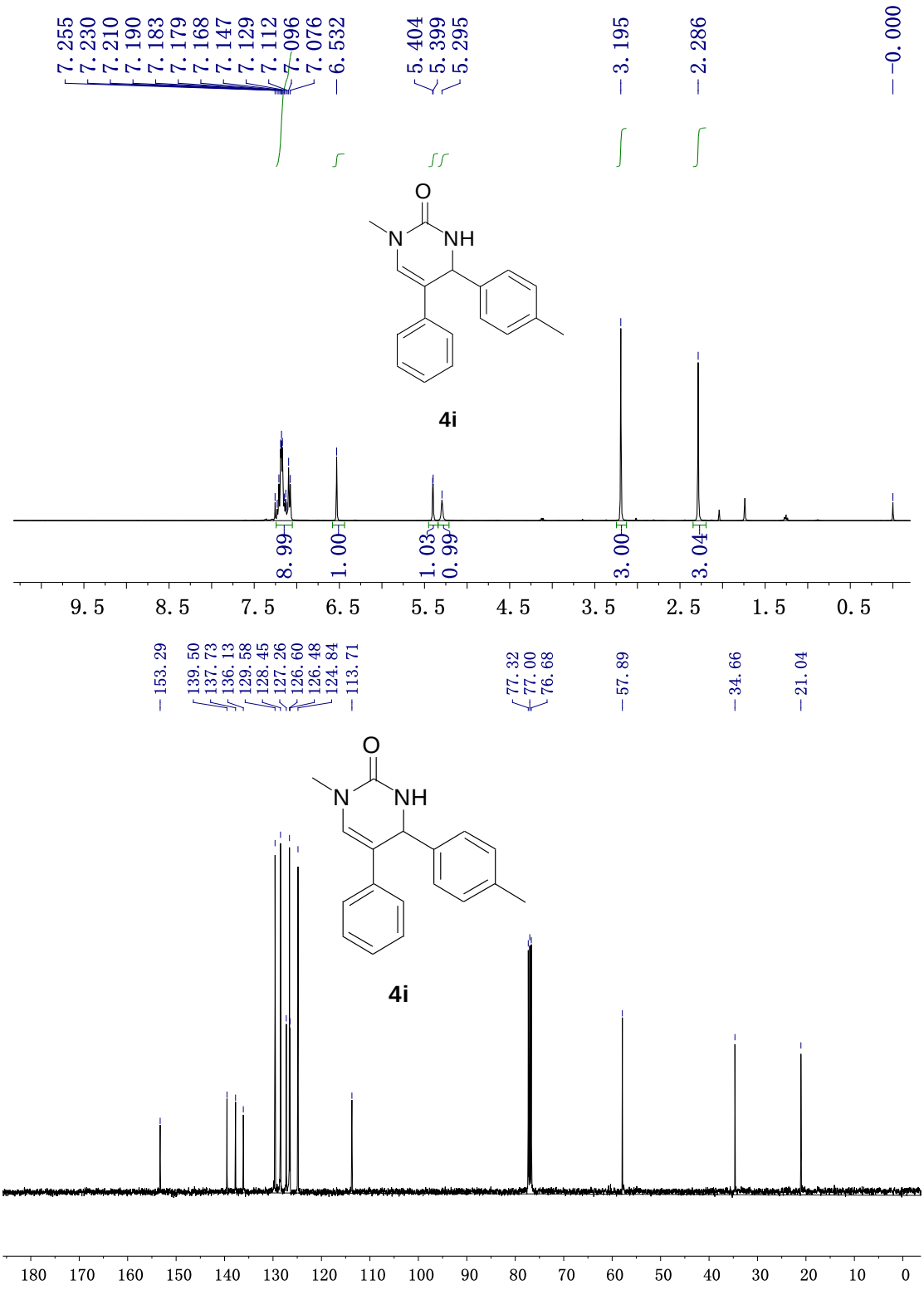
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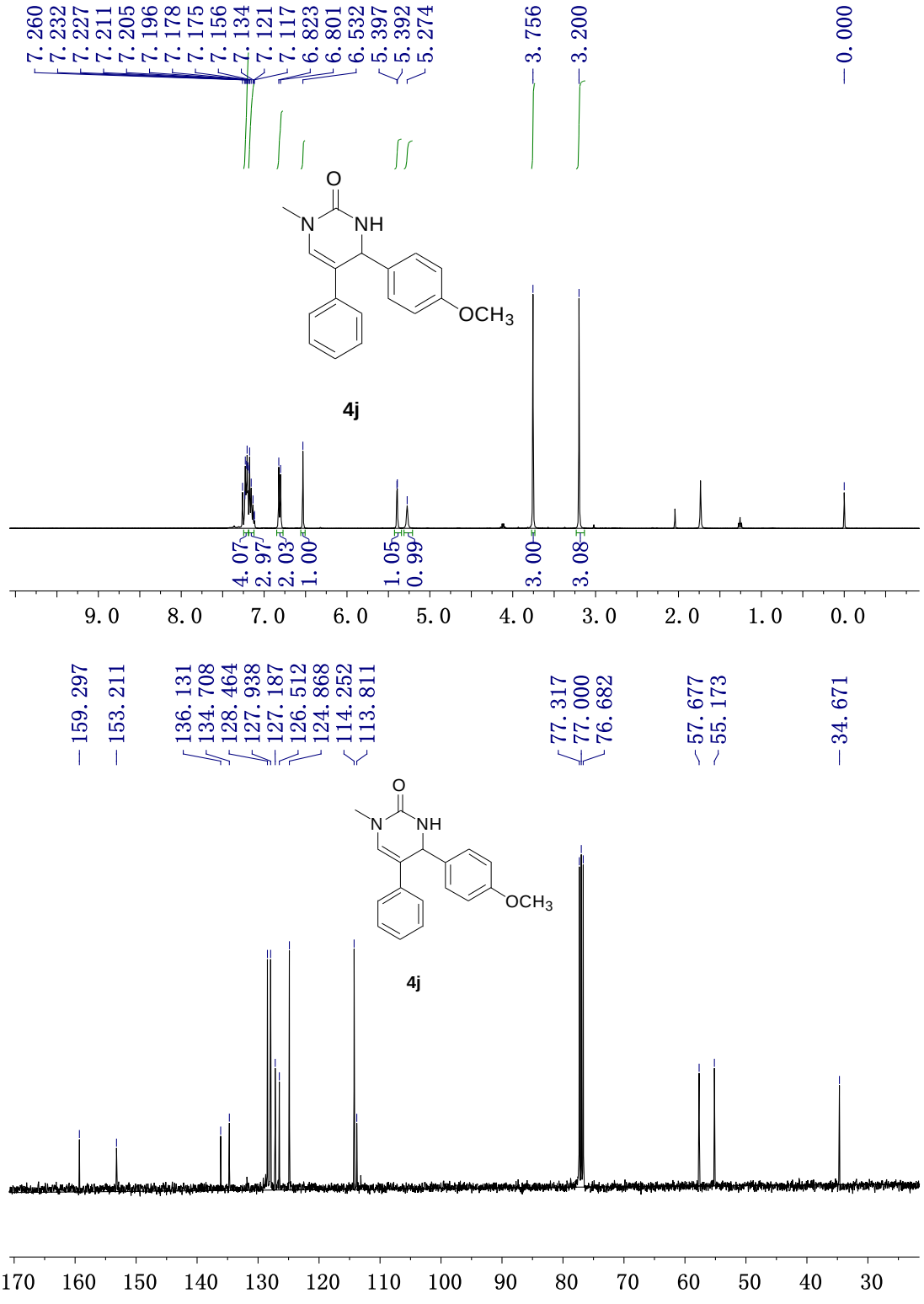
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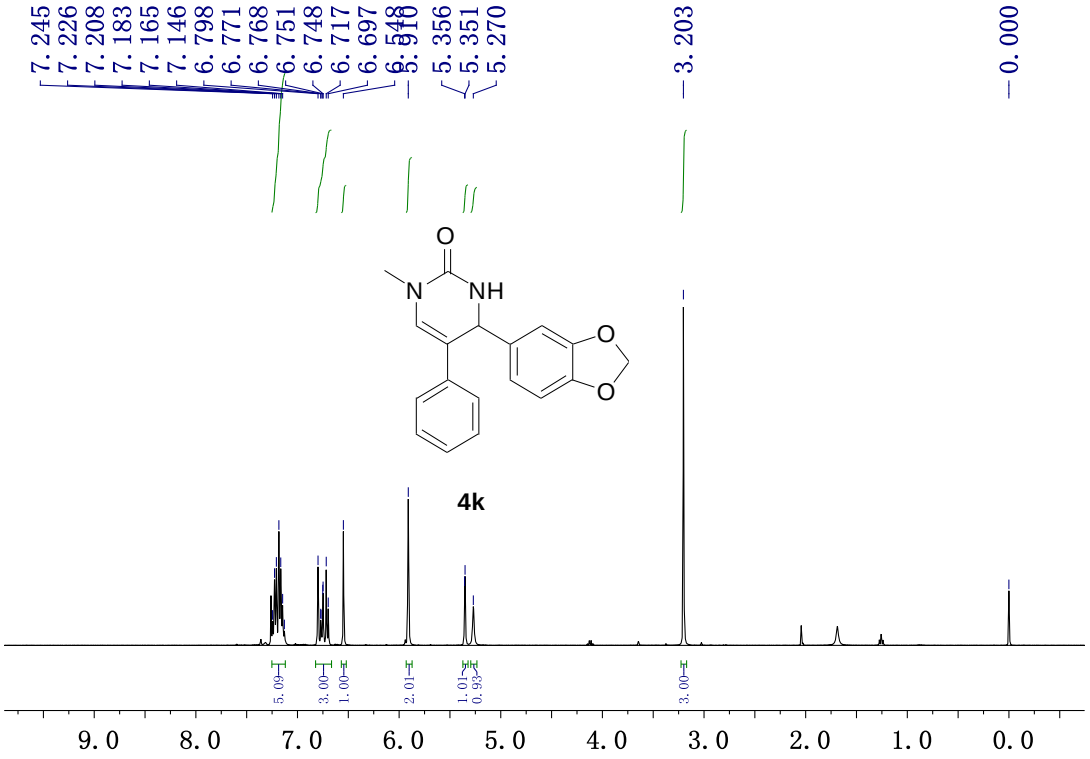
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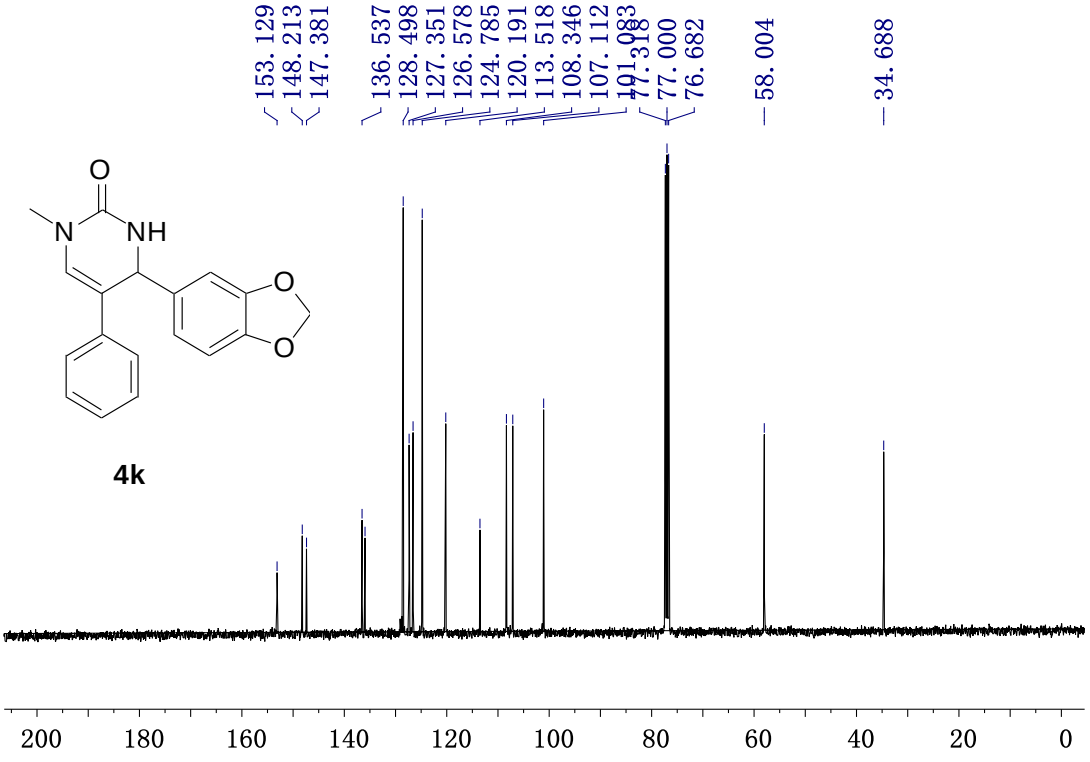
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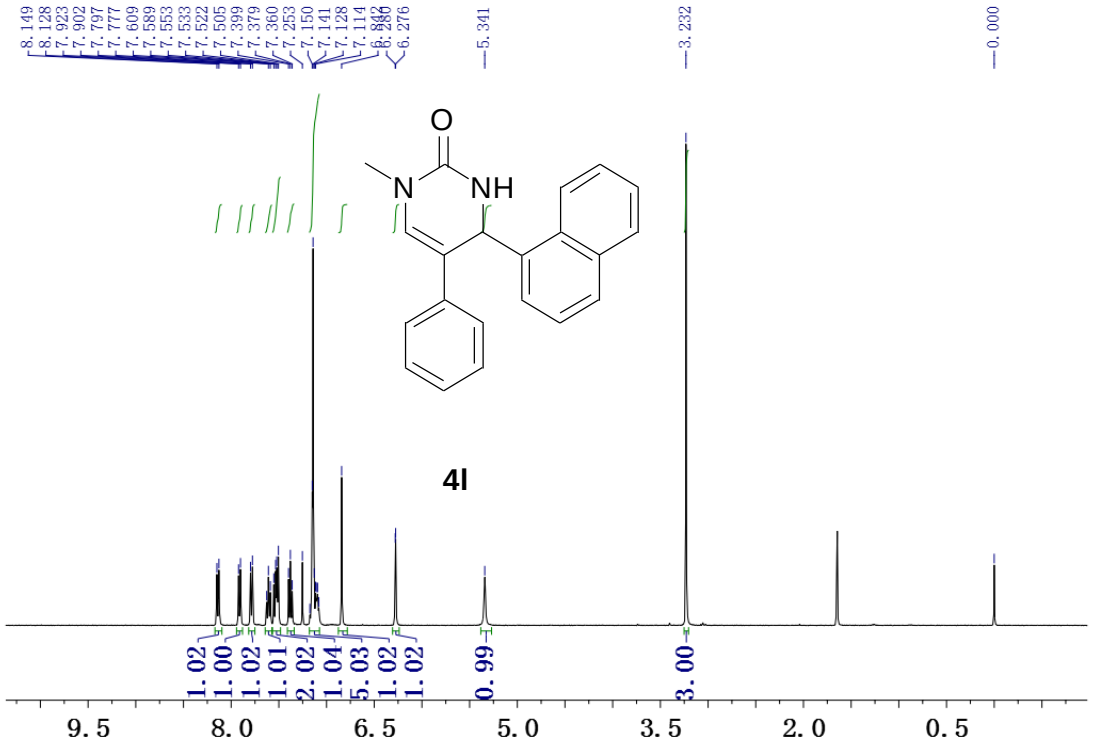
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1.01
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1.04
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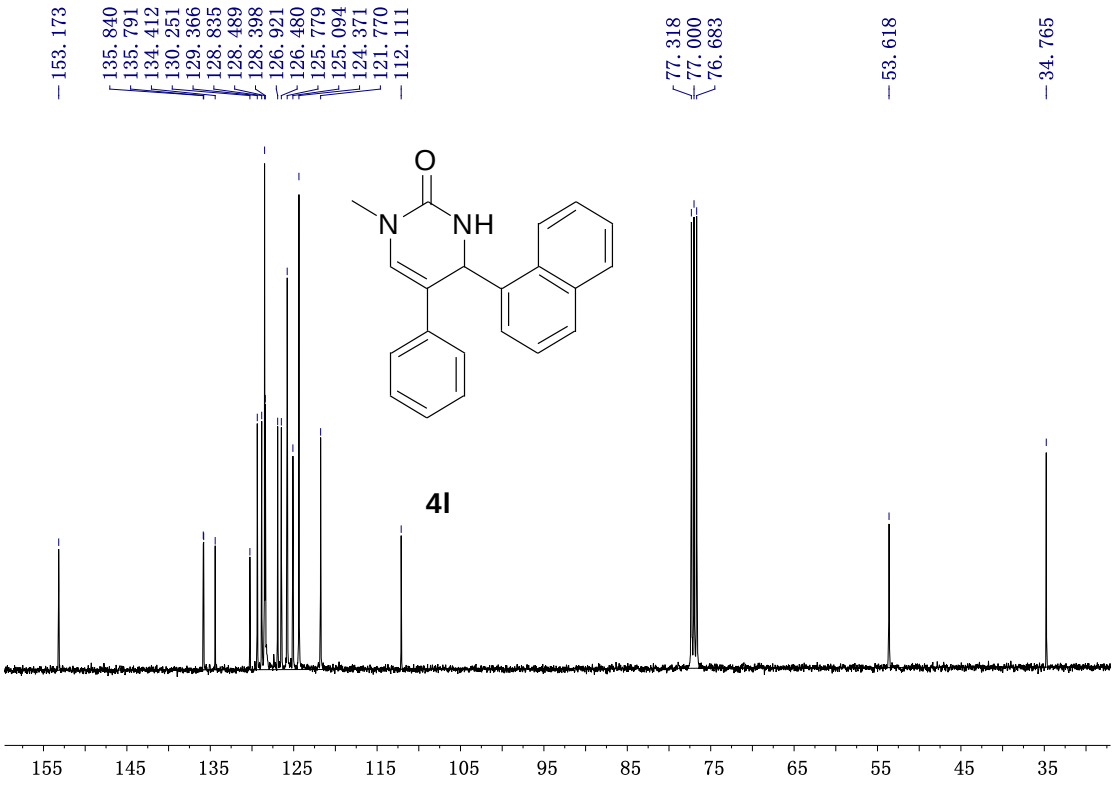
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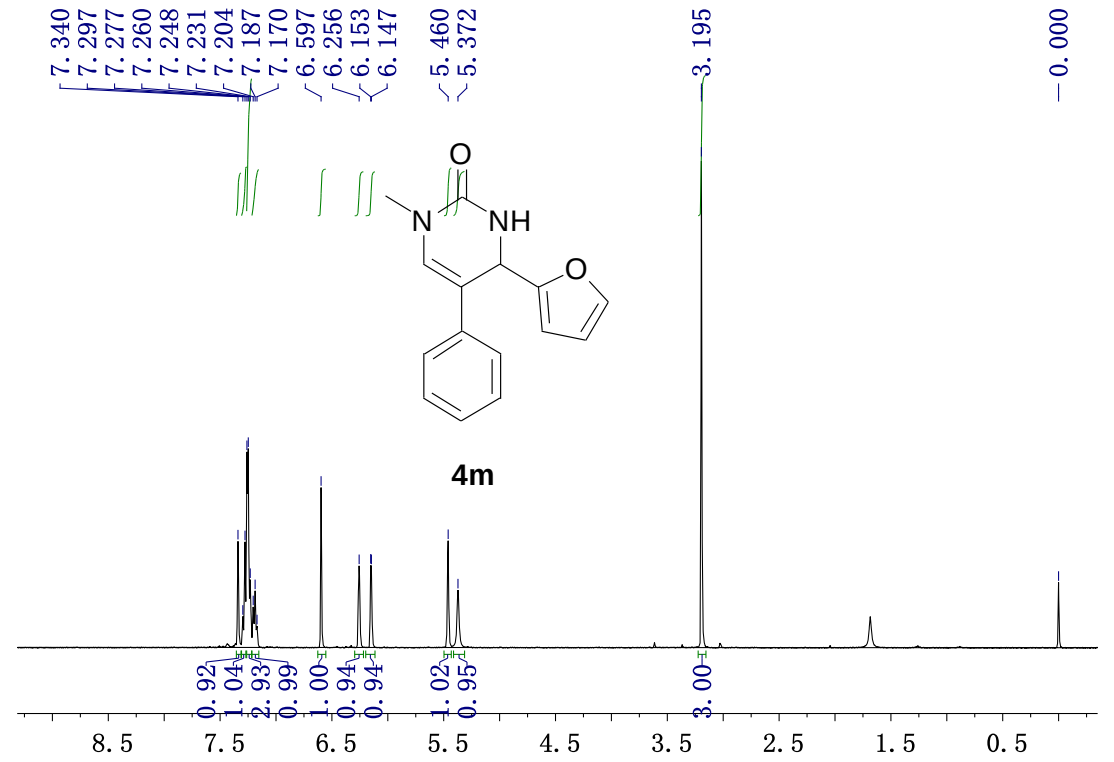
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NH
O
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1.00
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0.94
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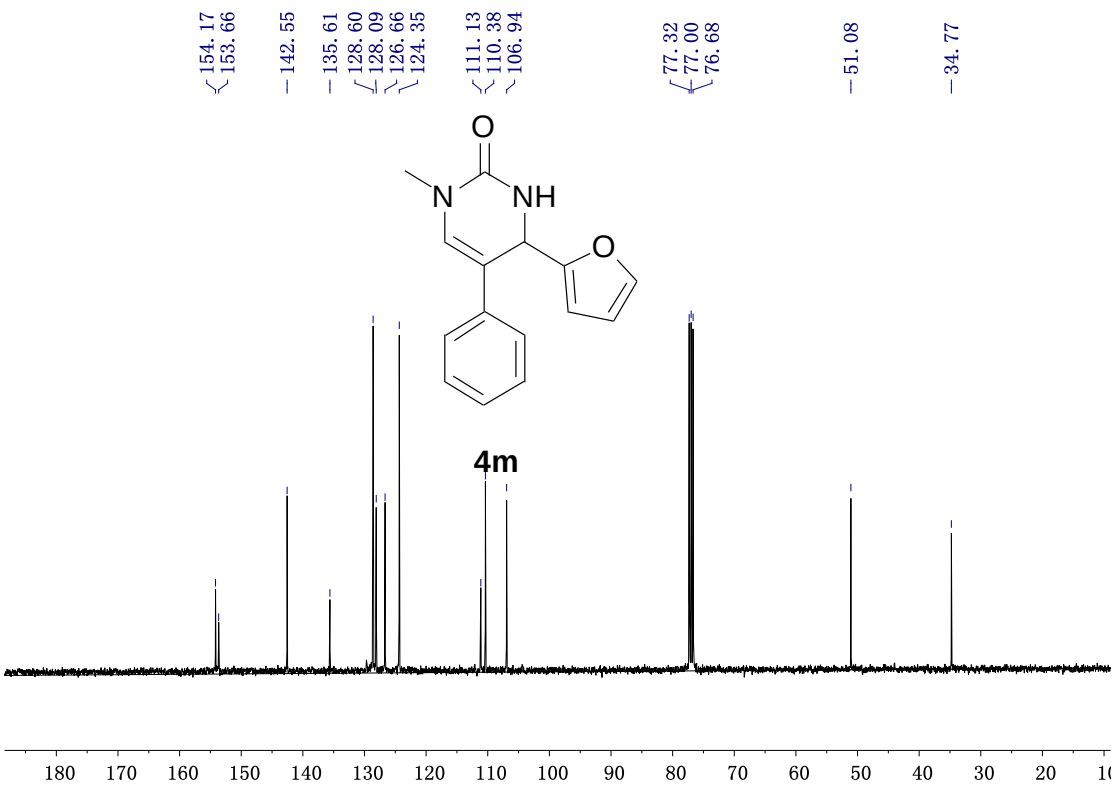
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51.08
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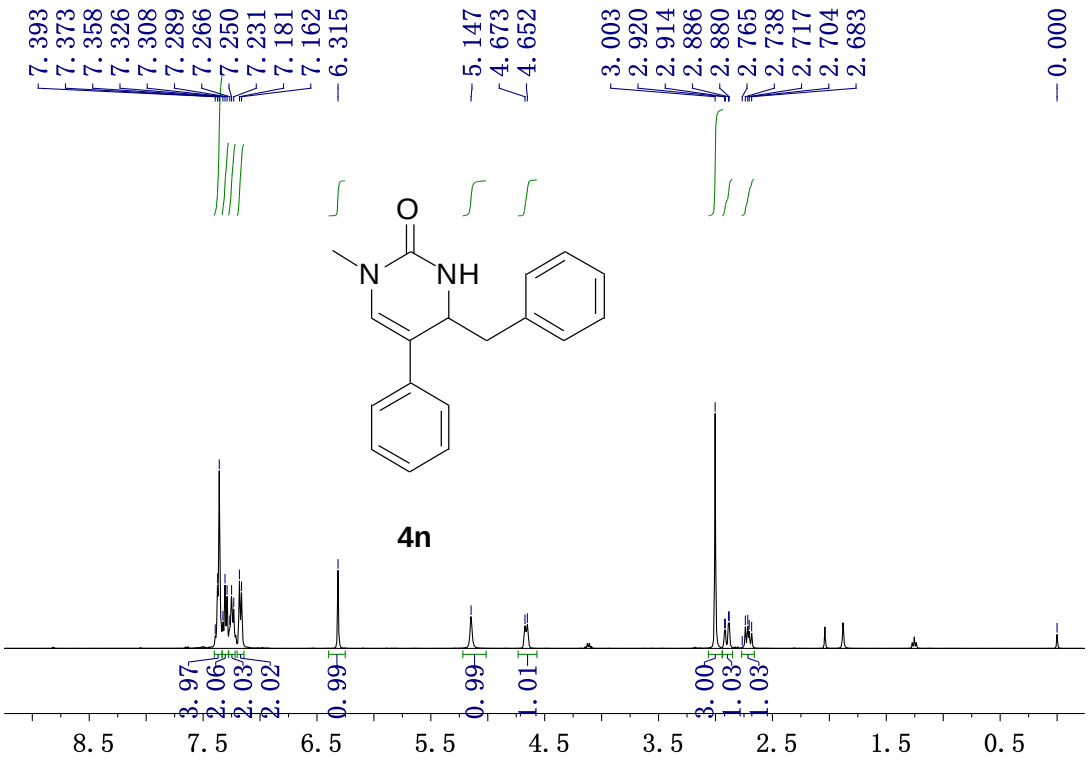
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2.914
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2.765
2.738
2.717
2.704
2.683
0.000
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NH
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2.06
2.03
2.02
0.99
0.99
1.01
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2.5
1.5
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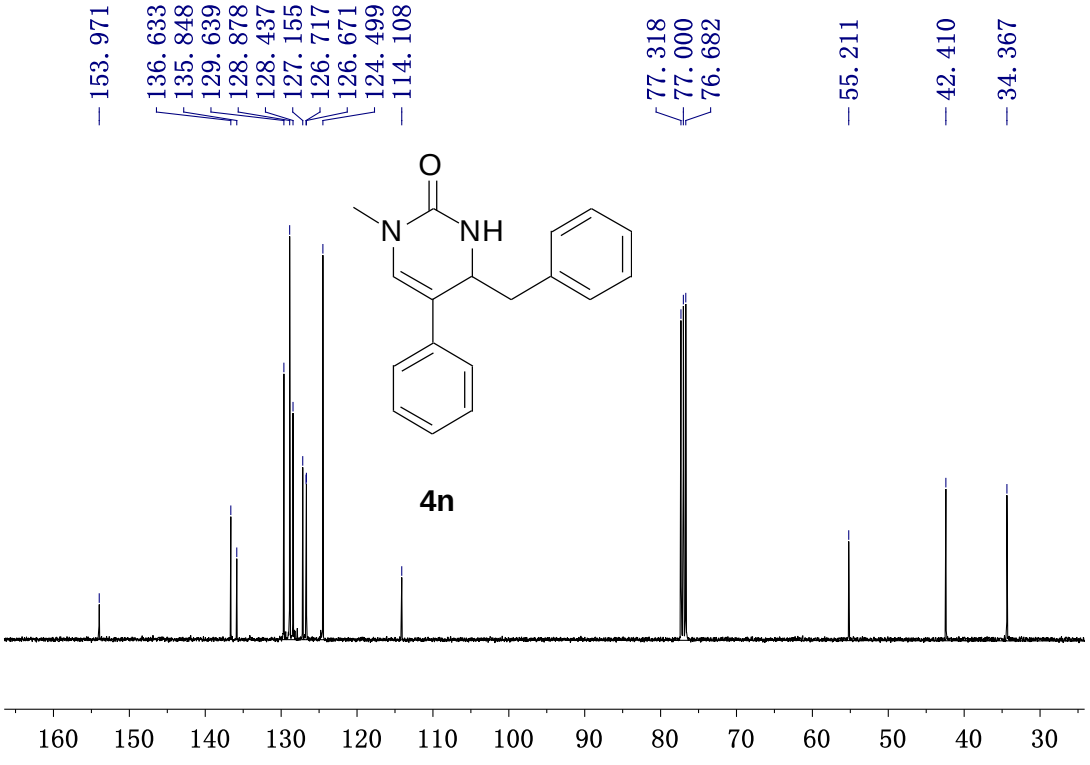
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60
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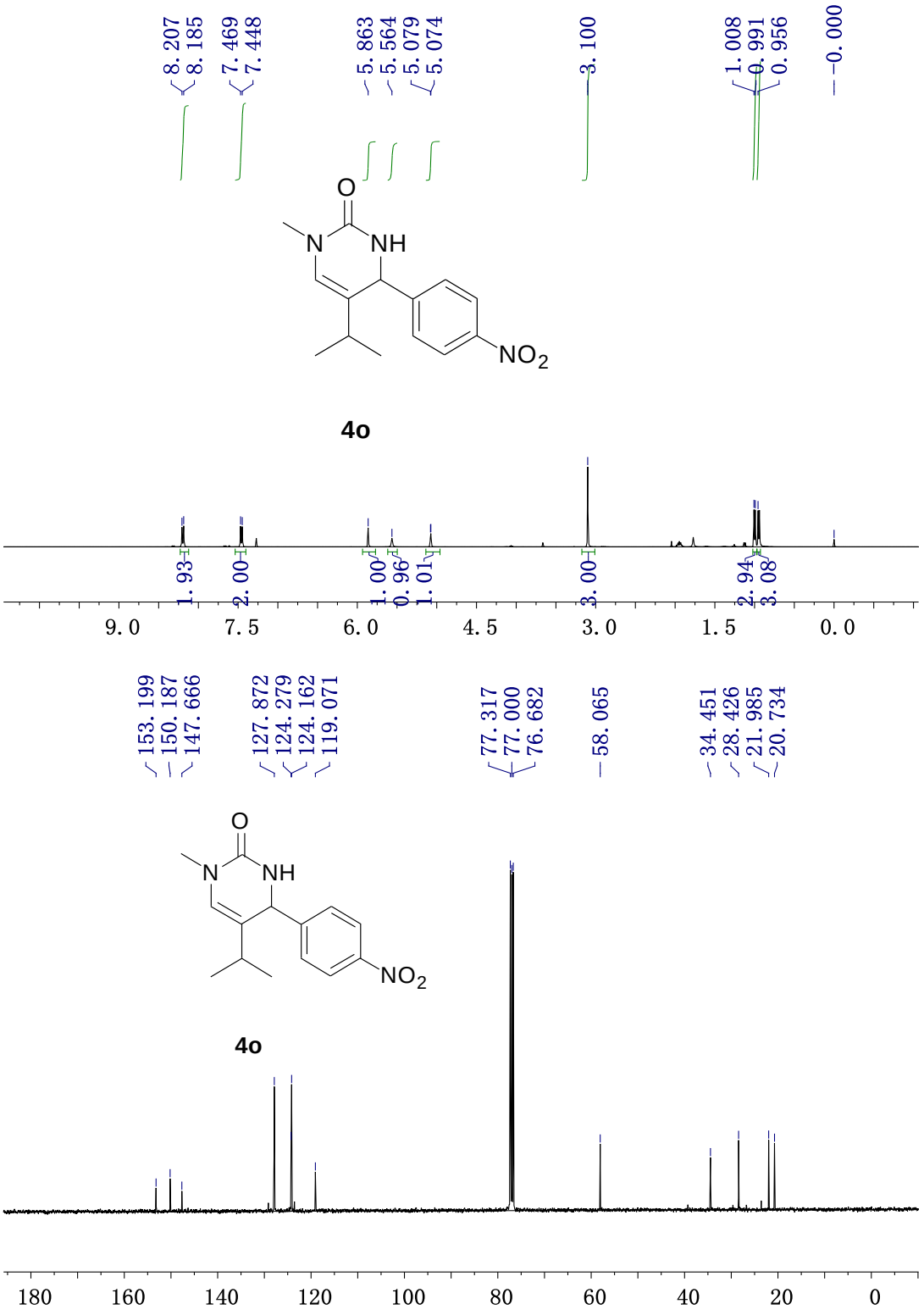
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1.00
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1.01
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2.94
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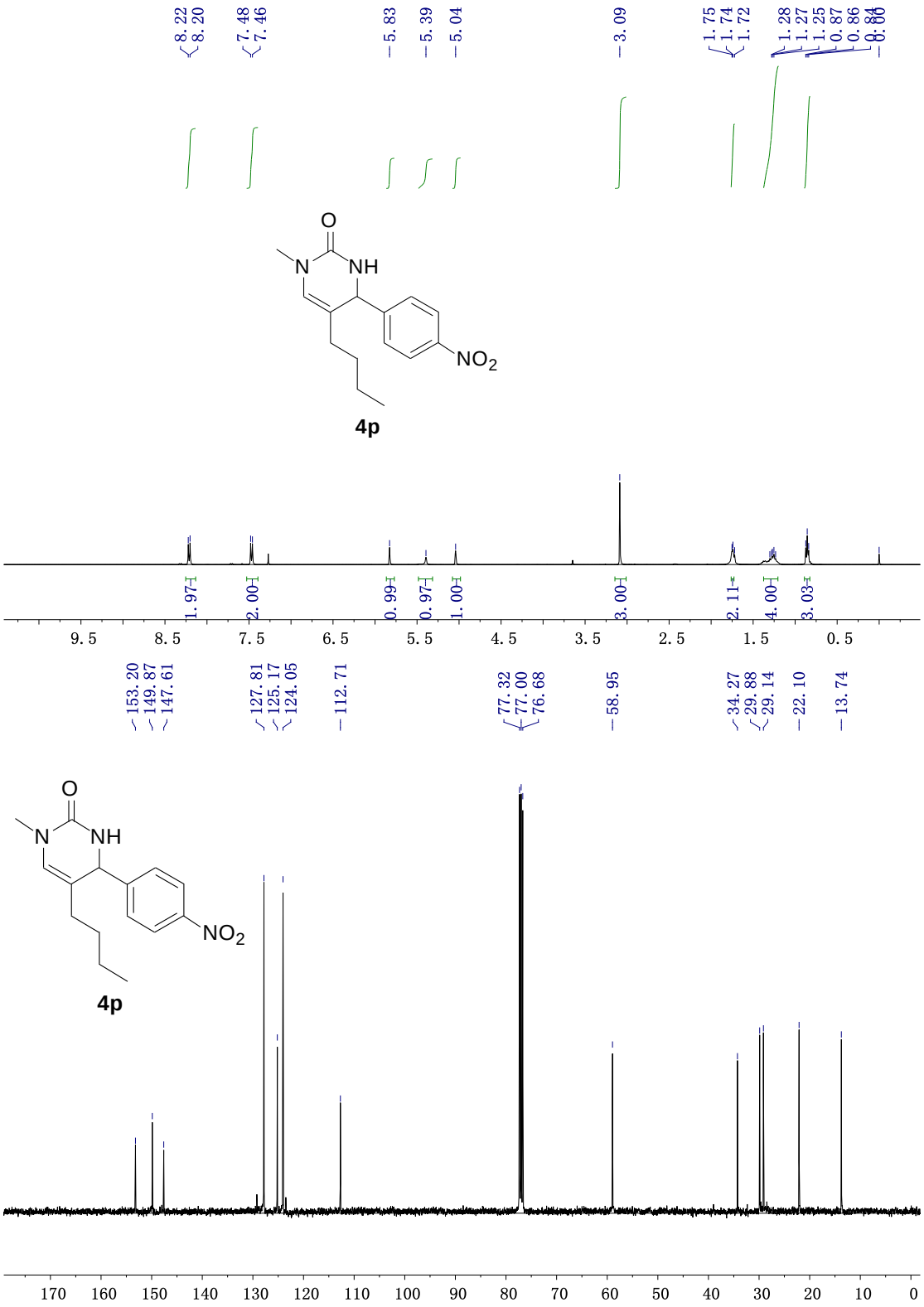
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3.00
2.11
4.00
3.03
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6.5
5.5
4.5
3.5
2.5
1.5
0.5
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127.81
125.17
124.05
112.71
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77.00
76.68
58.95
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29.88
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NH
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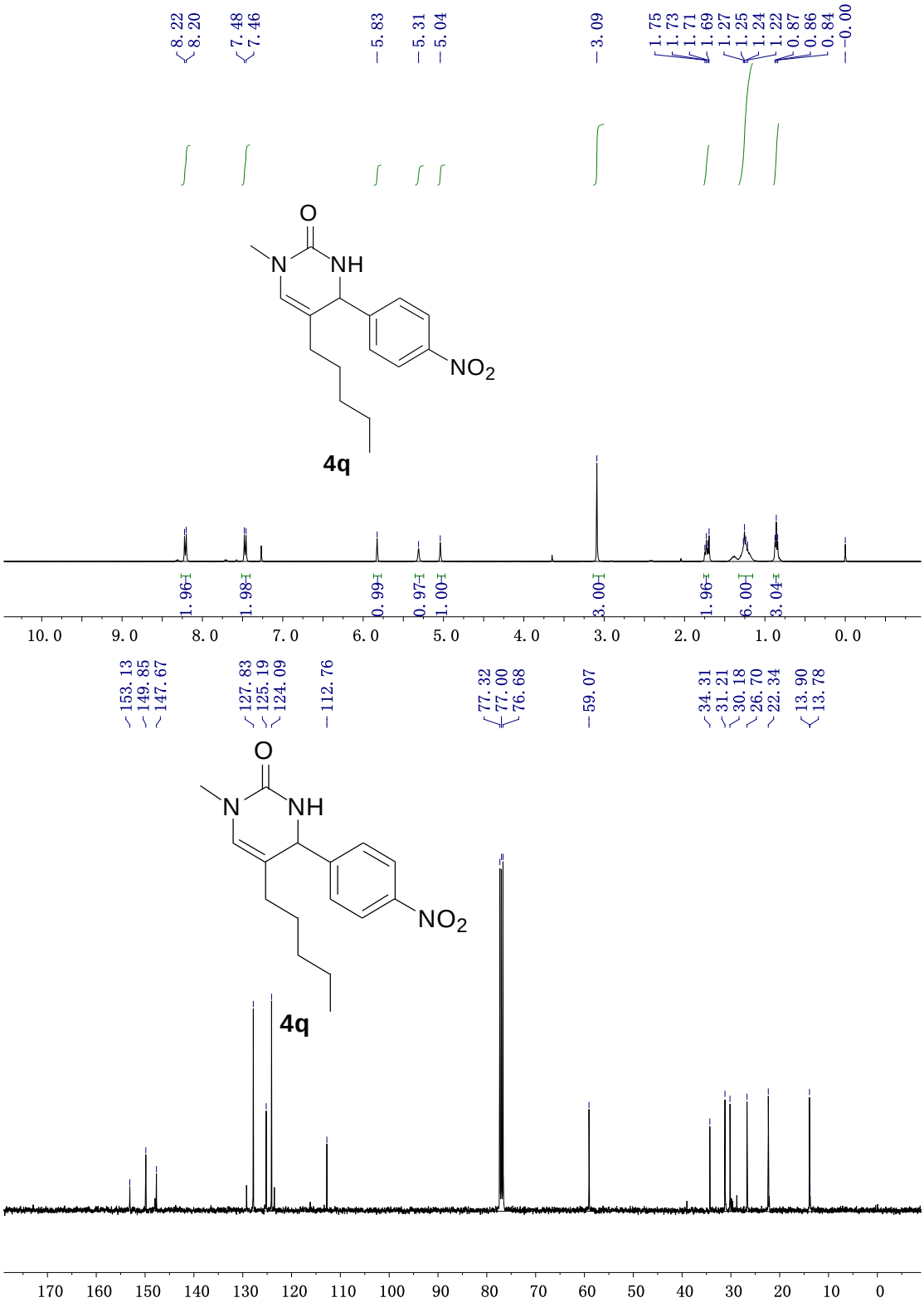
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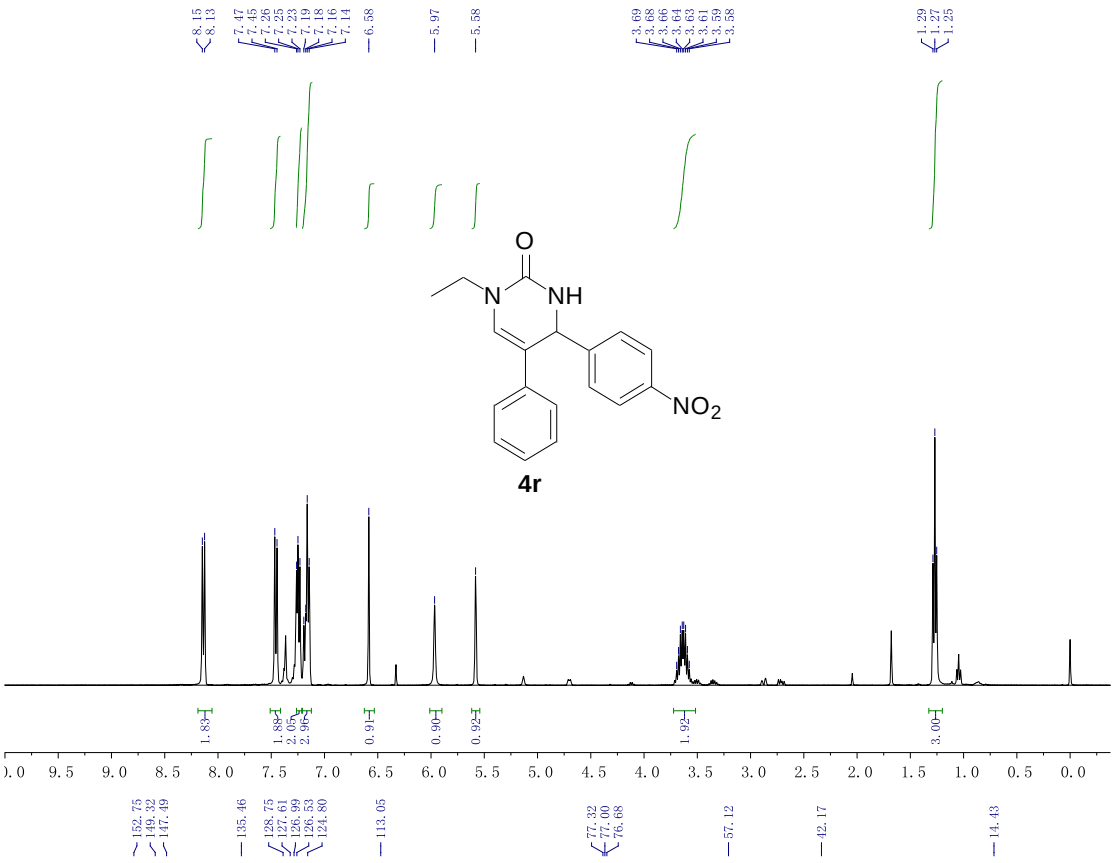
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5.58
3.69
3.68
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3.61
3.59
3.58
1.29
1.27
1.25
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6.0
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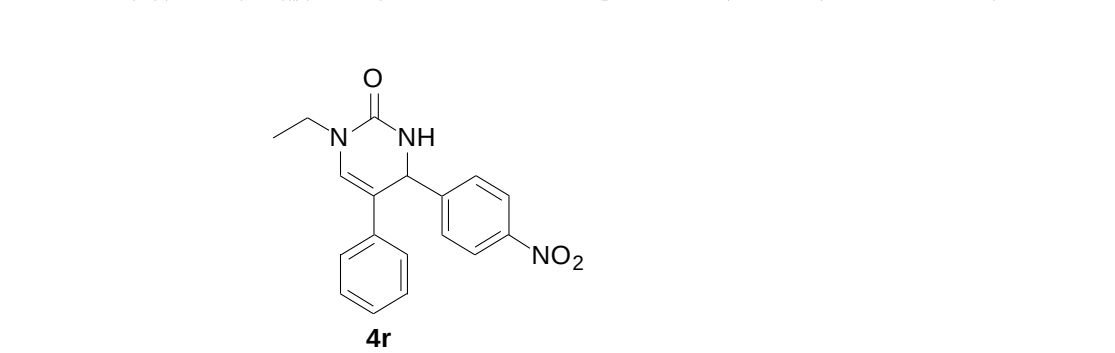
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57.12
42.17
14.43

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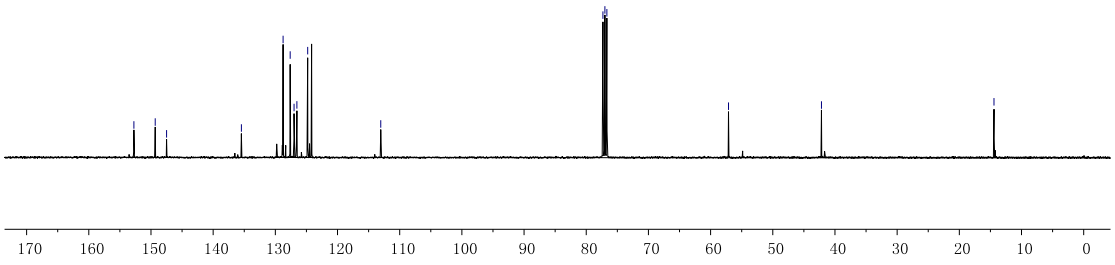
NH
NO2

4r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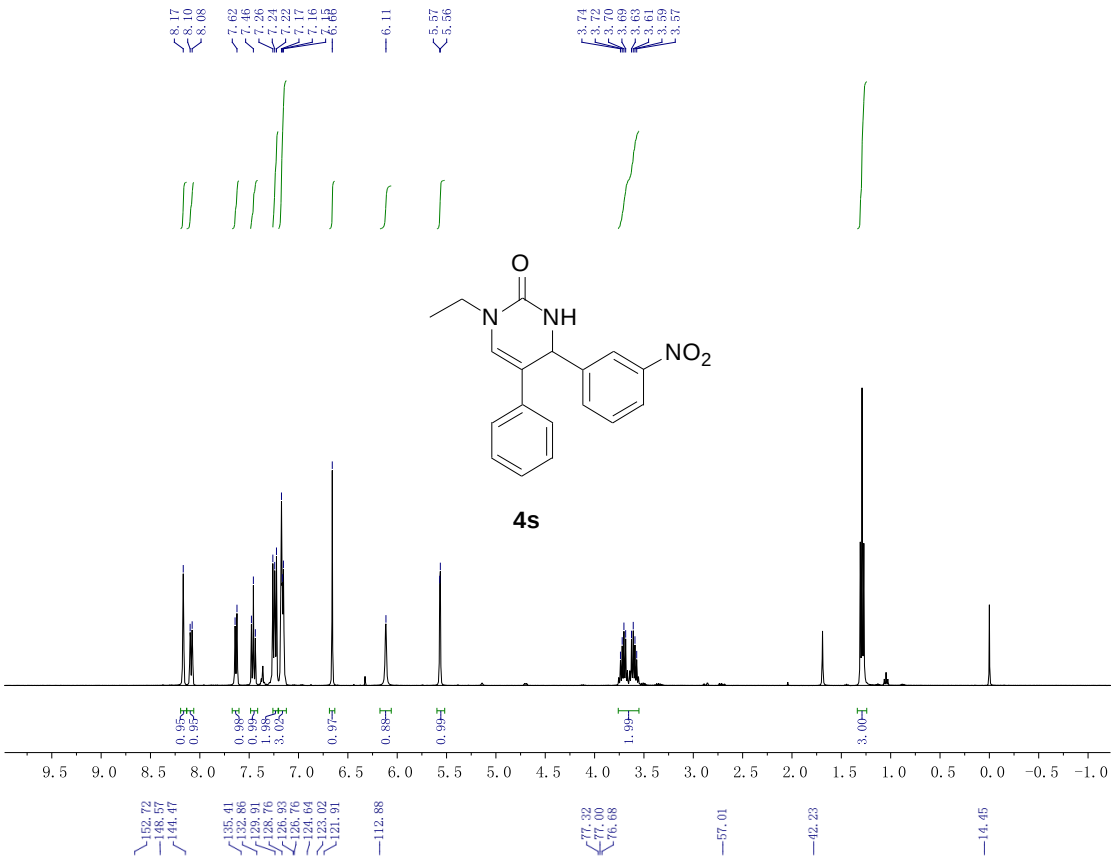
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5.56
3.74
3.72
3.70
3.69
3.63
3.61
3.59
3.57
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NO2
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0.95
0.99
0.99
1.98
3.02
0.97
0.88
0.99
1.99
3.00
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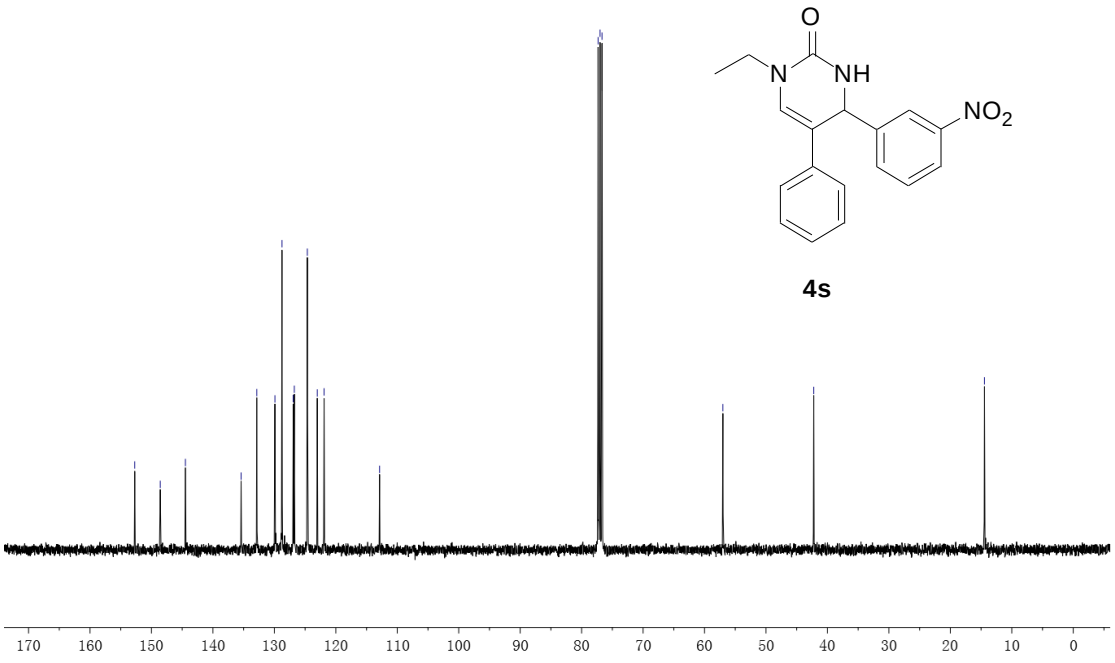
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77.32
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NO2
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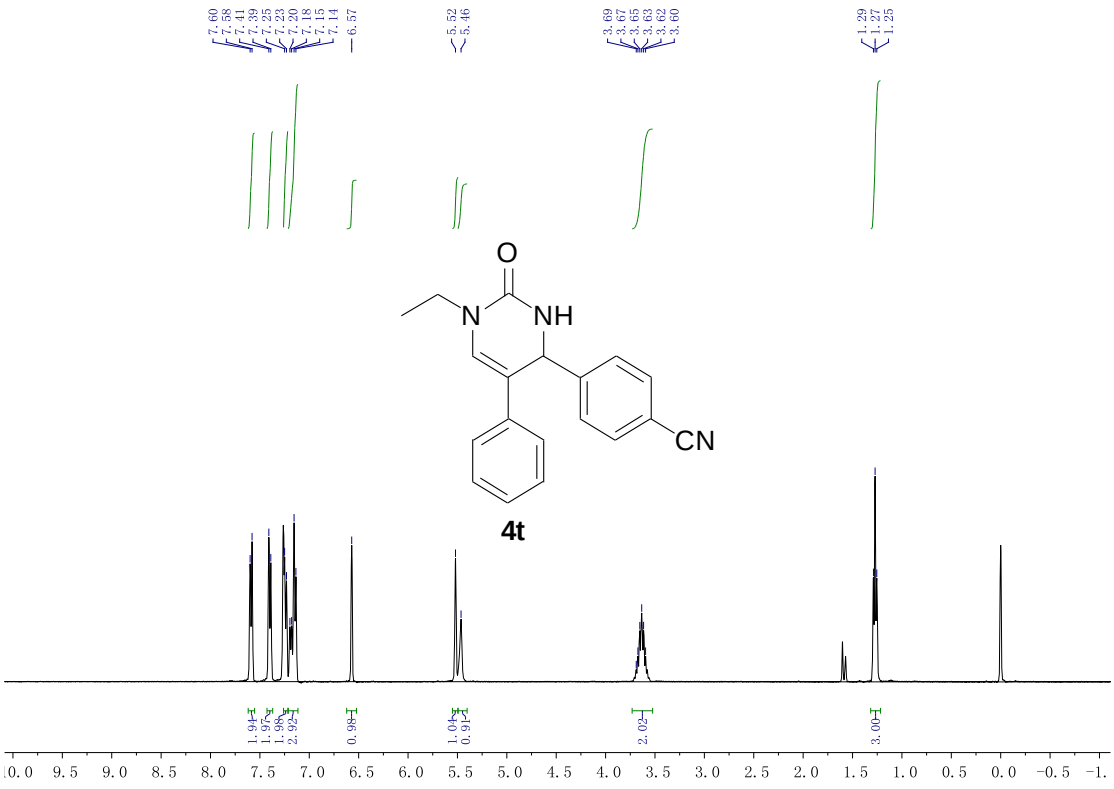
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3.62
3.60
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1.27
1.25
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1.04
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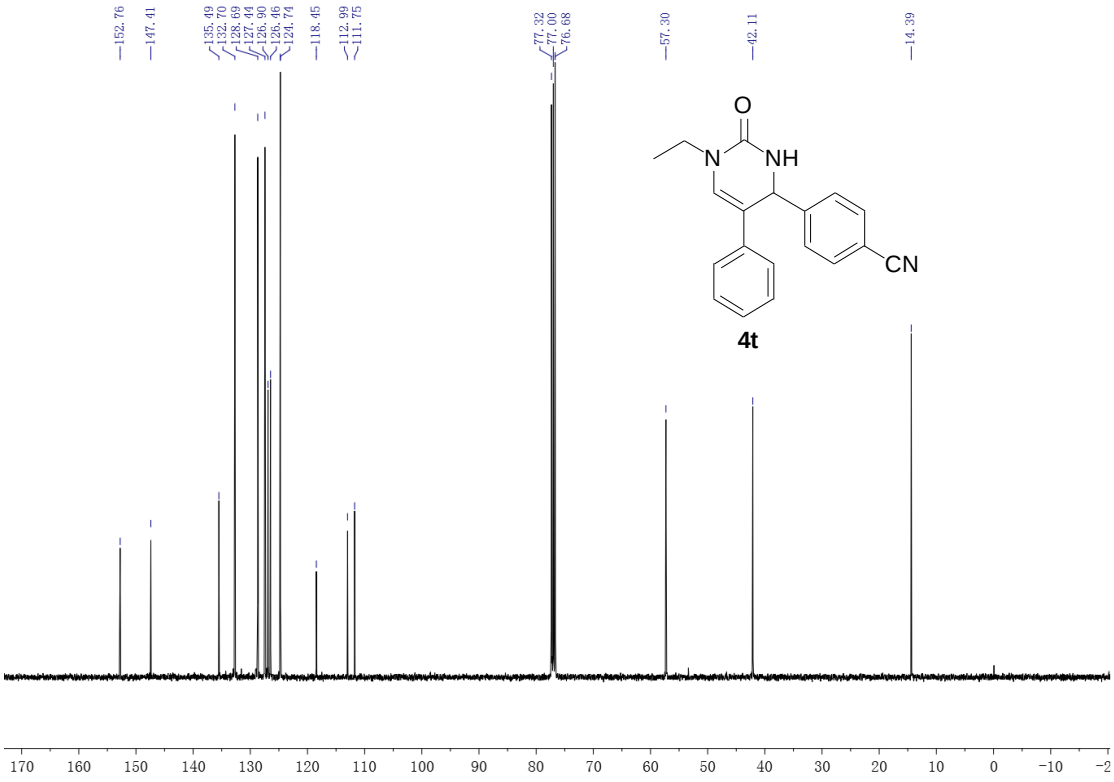
2.02
3.00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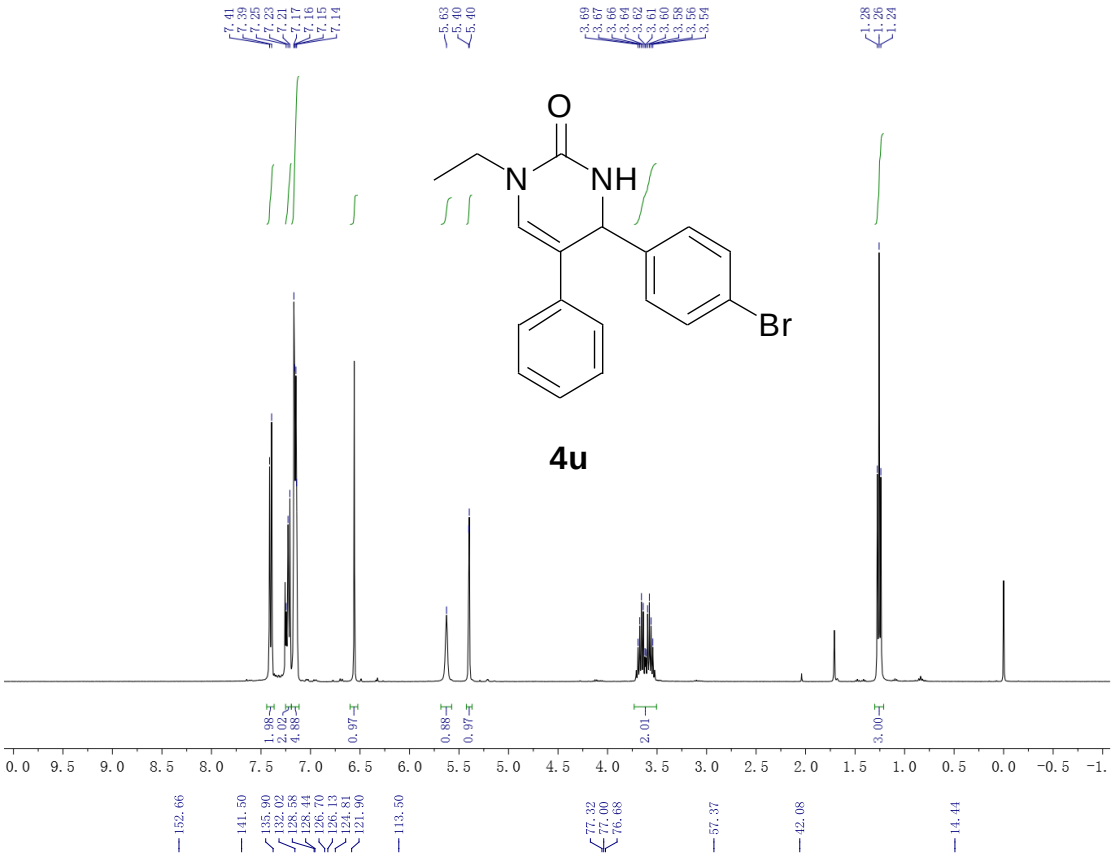
CN
4t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3.67
3.66
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3.61
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3.54
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1.26
1.24
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NH
Br
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1.02
2.02
4.88
0.97
0.88
0.97
2.01
3.00
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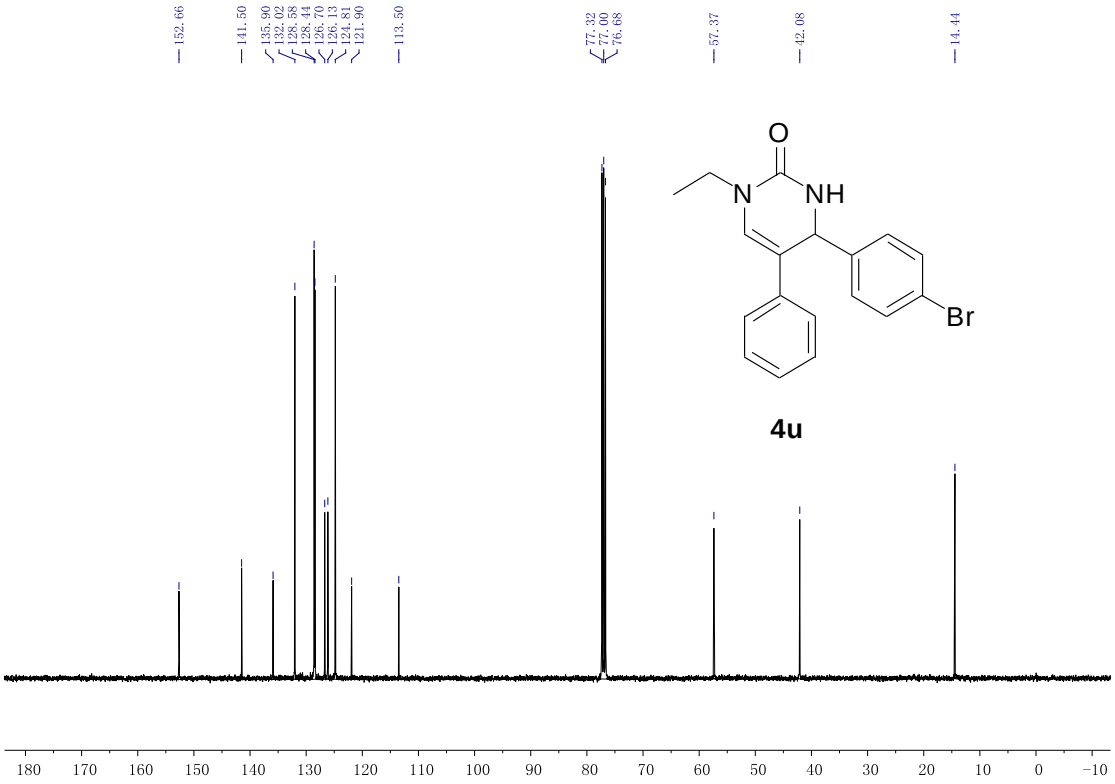
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Br
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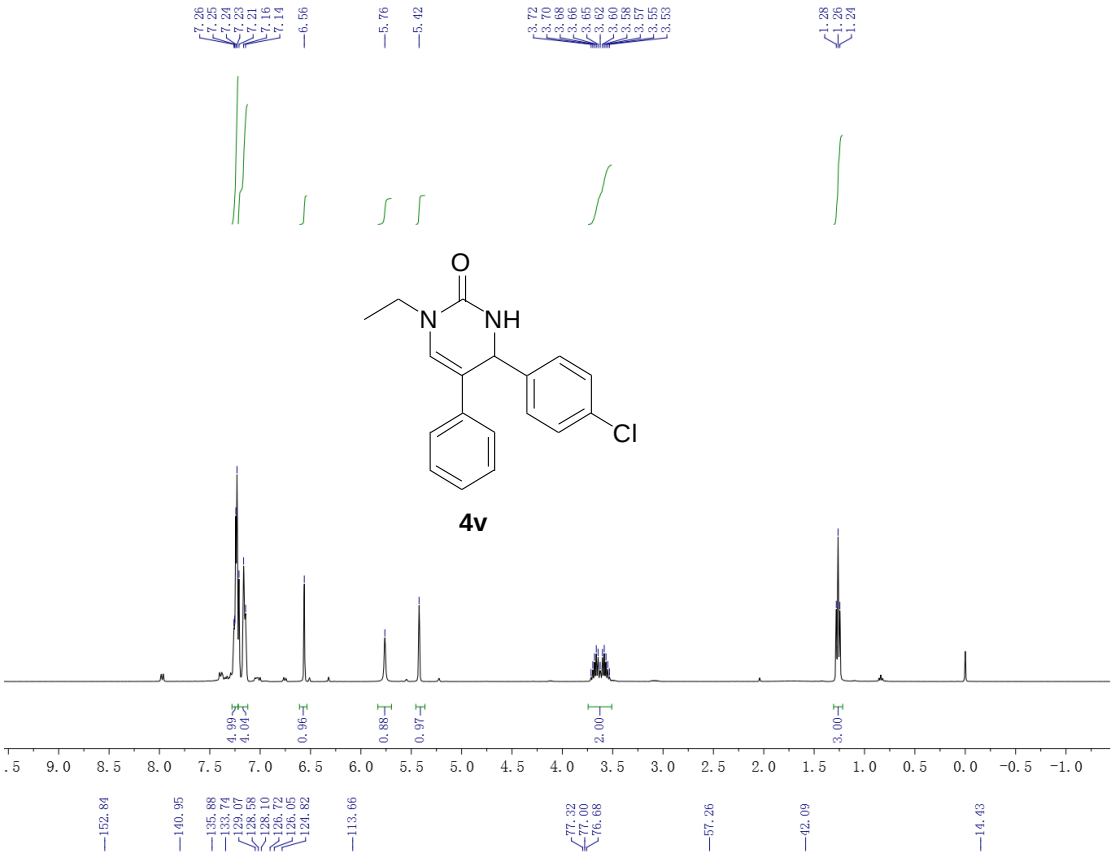
4u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3.58
3.57
3.55
3.53
1.28
1.26
1.24
O
N
NH
Cl
4.99
4.04
0.96
0.88
0.97
2.00
3.00
9.0
8.5
8.0
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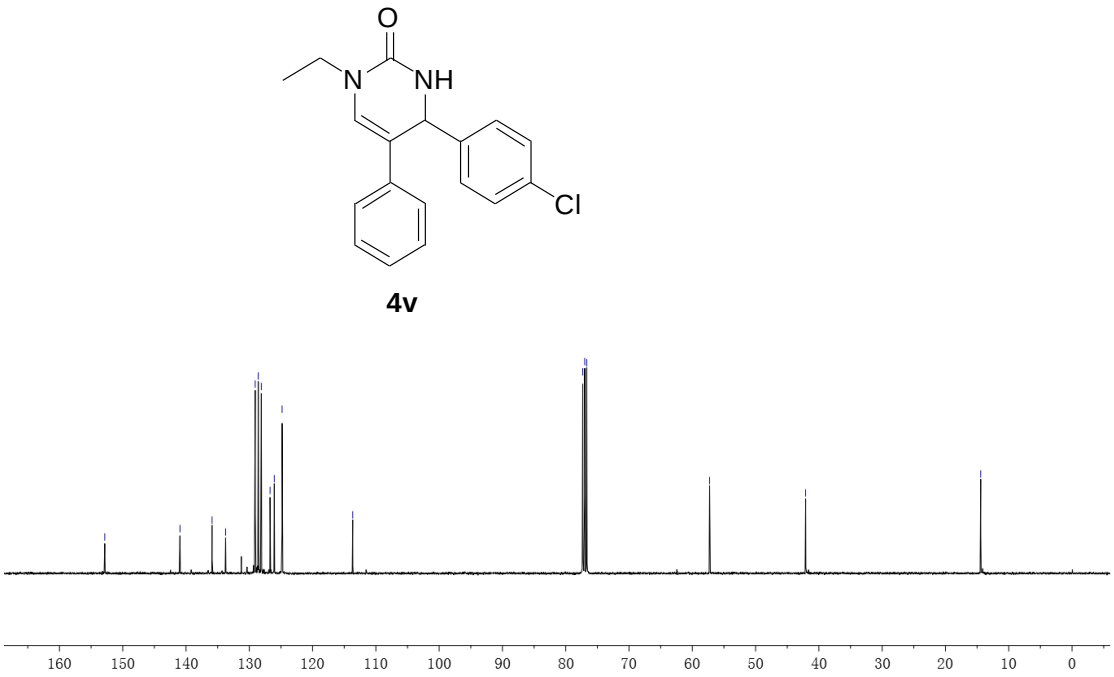
4v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14.43
O
N
NH
Cl
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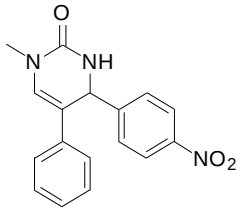
0

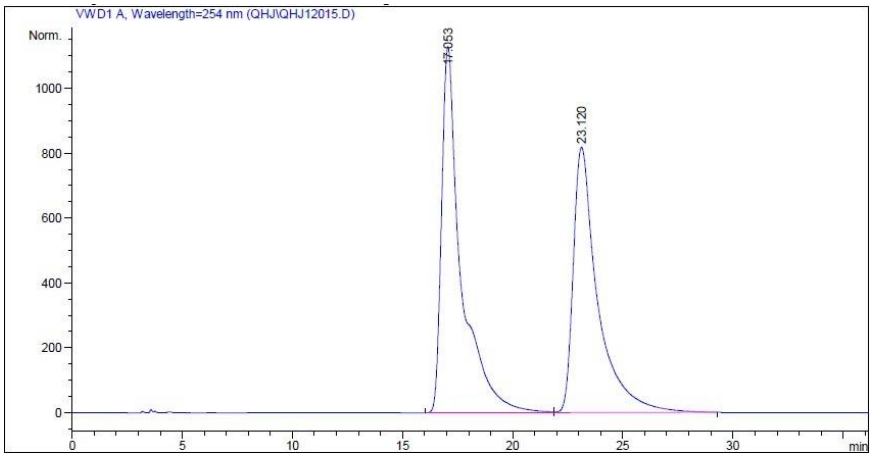
4v

HPLC spectra

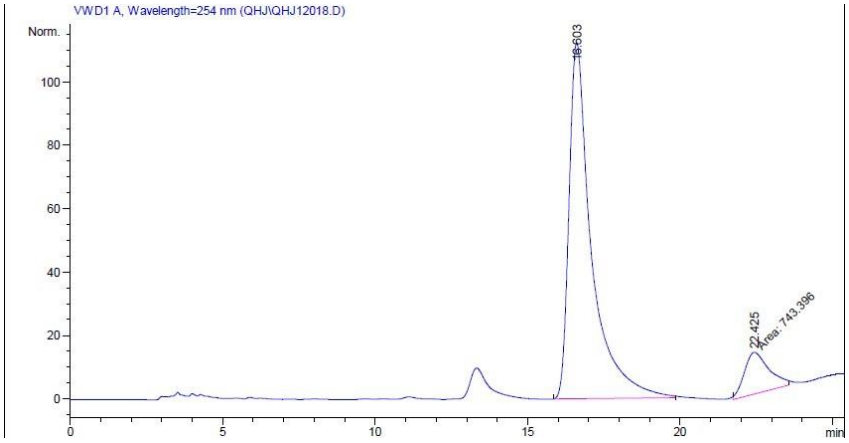


4a

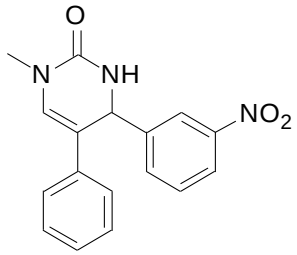
77% ee. [Daicel Chiralpak AD-H, *n*-hexane / isopropanol = 80 / 20, 1.0 mL/min, λ = 254 nm]



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.053	BV	0.8698	6.94847e4	1128.44250	52.3359
2	23.120	VB	1.1035	6.32821e4	818.01239	47.6641
Totals :				1.32767e5	1946.45490	

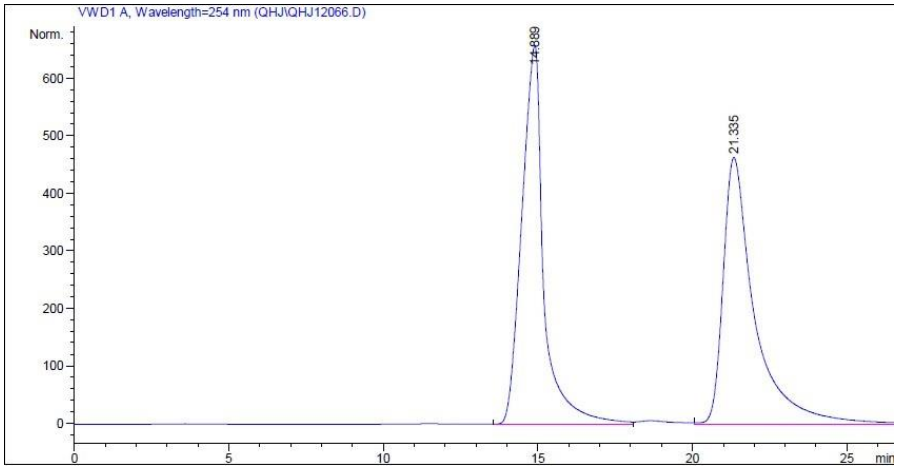


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.603	BB	0.7377	5761.65234	112.42514	88.5720
2	22.425	MM	0.9375	743.39648	13.21661	11.4280
Totals :				6505.04883	125.64175	

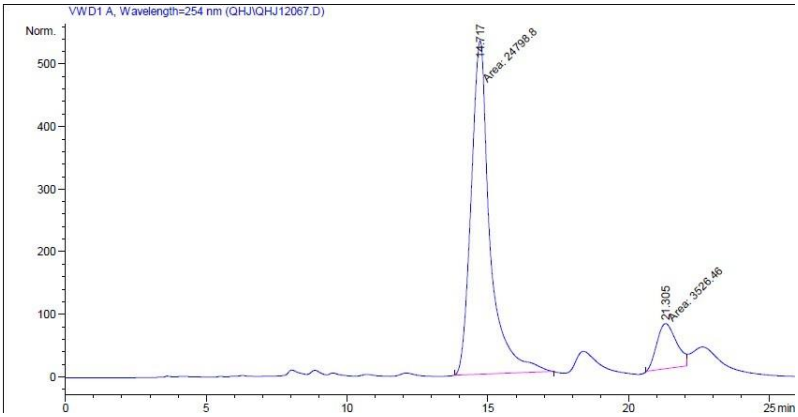


4b

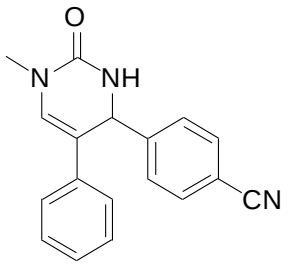
75% ee. [Daicel Chiralpak AD-H, *n*-hexane / isopropanol = 80 / 20, 1.0 mL/min, λ = 254 nm]



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.889	BV	0.7669	3.26258e4	658.93701	49.7644
2	21.335	VBA	1.0233	3.29348e4	463.92462	50.2356
Totals :				6.55606e4	1122.86163	

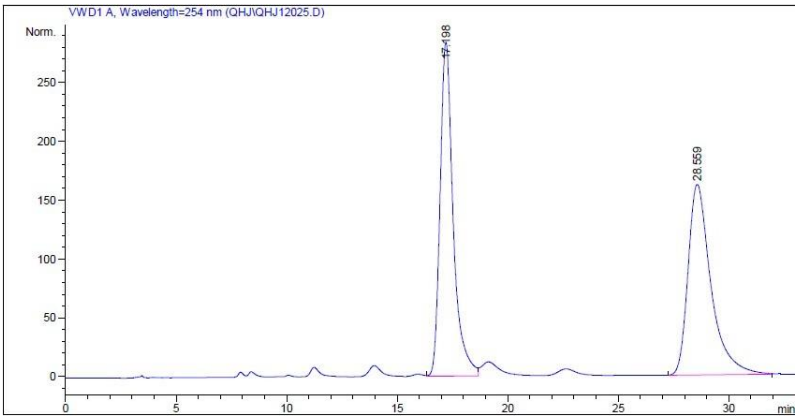


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.717	MM	0.7747	2.47988e4	533.51208	87.5501
2	21.305	MM	0.8116	3526.46167	72.41758	12.4499
Totals :				2.83252e4	605.92966	

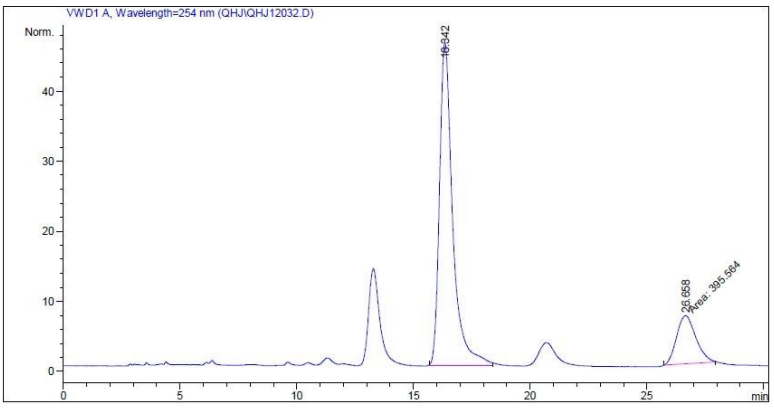


4c

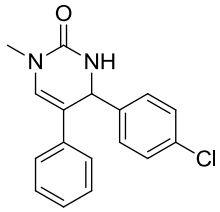
64% ee. [Daicel Chiralpak AD-H, *n*-hexane / isopropanol = 80 / 20, 1.0 mL/min, λ = 254 nm]



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.198	VV	0.6143	1.16938e4	284.00729	49.9626
2	28.559	BB	1.0813	1.17113e4	162.17480	50.0374
Totals :				2.34051e4	446.18210	

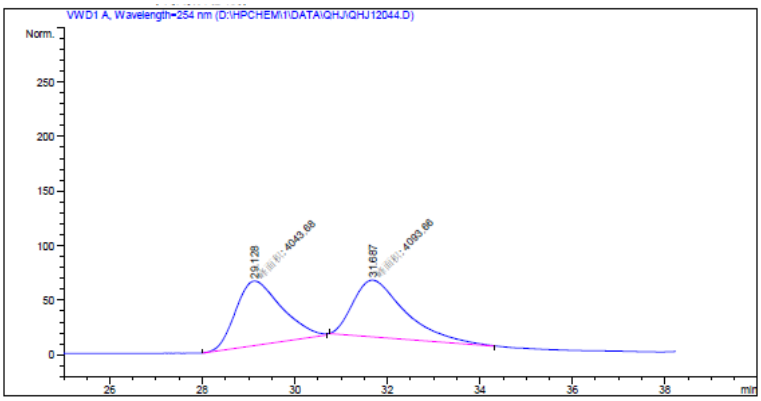


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.342	BB	0.5914	1809.20642	46.14186	82.0587
2	26.658	MM	0.9567	395.56396	6.89142	17.9413
Totals :				2204.77039	53.03328	



4d

32% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / isopropanol = 90/10,0.8 mL/min, λ = 254 nm, *t* (major) = 29.13min, *t* (minor) = 31.84 min].



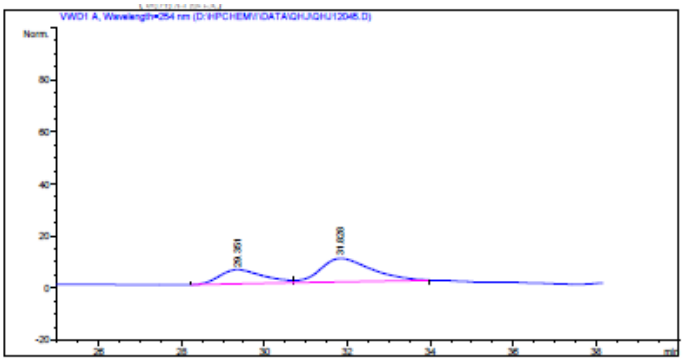
==========
面积百分比报告
==========

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	29.128	MM	1.1421	4043.67993	59.01037	49.6929
2	31.687	MM	1.3107	4093.66016	52.05369	50.3071

总量 : 8137.34009 111.06406



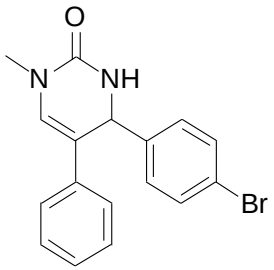
==========
面积百分比报告
==========

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=254 nm

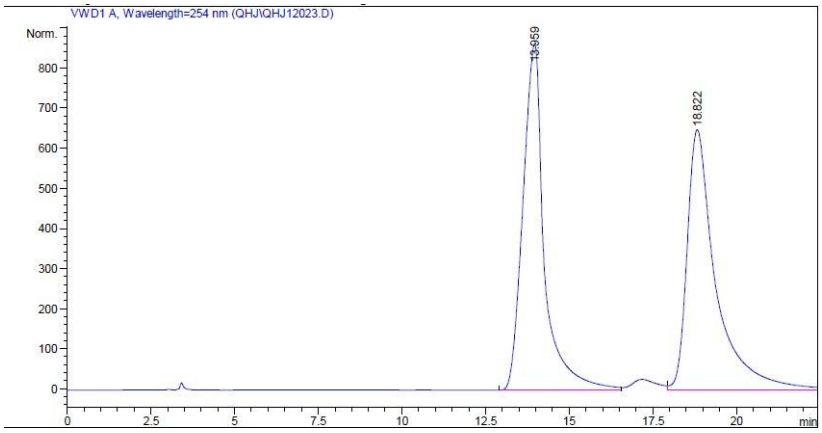
峰 #	保留时间 (min)	类型	峰宽 (min)	峰面积 mAU *s	峰高 (mAU)	峰面积 %
1	29.351	BV	0.8643	397.15692	5.43445	33.8463
2	31.828	VB	1.0489	776.25714	9.08826	66.1537

总量 : 1173.41406 14.52271

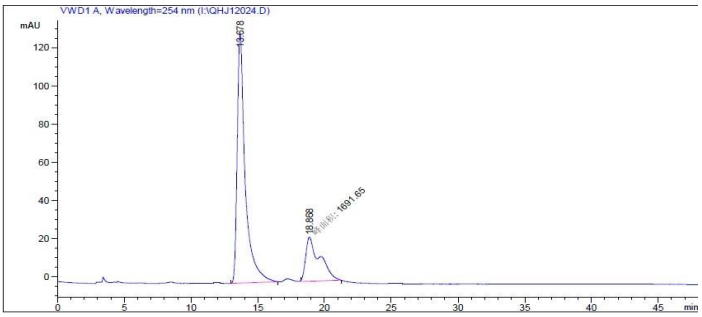


4e

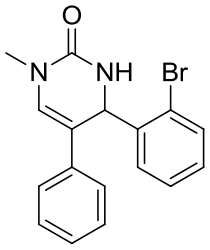
53% ee. [Daicel Chiralpak AD-H, *n*-hexane / isopropanol = 80 / 20, 1.0 mL/min, λ = 254 nm]



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.959	BV	0.6734	3.84118e4	863.40009	49.6912
2	18.822	VBA	0.8672	3.88892e4	649.72180	50.3088
Totals :				7.73011e4	1513.12189	

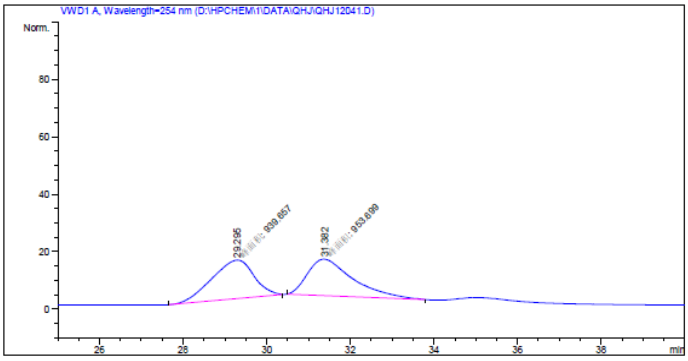


峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	13.678	BB	0.6000	5445.21436	130.98067	76.2971
2	18.868	MM	1.2166	1691.64575	23.17445	23.7029



4f

20% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / isopropanol = 90/10, 0.8 mL/min, λ = 254 nm, *t* (major) = 29.30 min, *t* (minor) = 31.37 min].

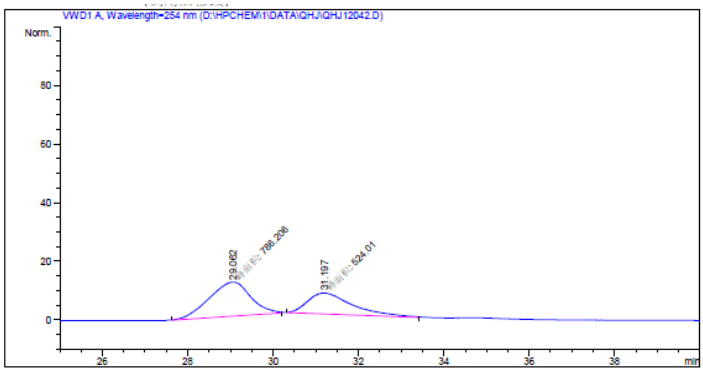


==========
面积百分比报告
==========

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	29.295	MM	1.1731	939.65710	13.34975	49.6292
2	31.382	MM	1.2570	953.69946	12.64469	50.3708
总量 :				1893.35657	25.99444	

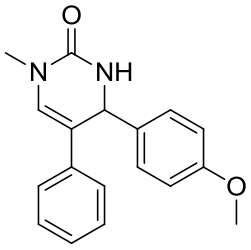


==========
面积百分比报告
==========

排序 : 信号
乘积因子 : 1.0000
稀释因子 : 1.0000
内标使用乘积因子和稀释因子

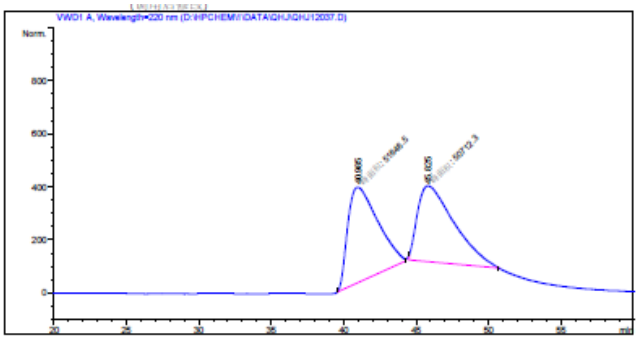
信号 1: VWD1 A, Wavelength=254 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	29.062	MM	1.1321	786.20575	11.57461	60.0058
2	31.197	MM	1.2169	524.00977	7.17682	39.9942
总量 :				1310.21552	18.75143	



4j

33% ee by chiral HPLC analysis [Daicel Chiralpak AD-H, *n*-hexane / isopropanol = 90/10, 0.8 mL/min, λ = 254 nm, *t* (major) = 43.37 min, *t* (minor) = 48.50 min]



面积百分比报告

排序	:	信号
乘积因子	:	1.0000
稀释因子	:	1.0000

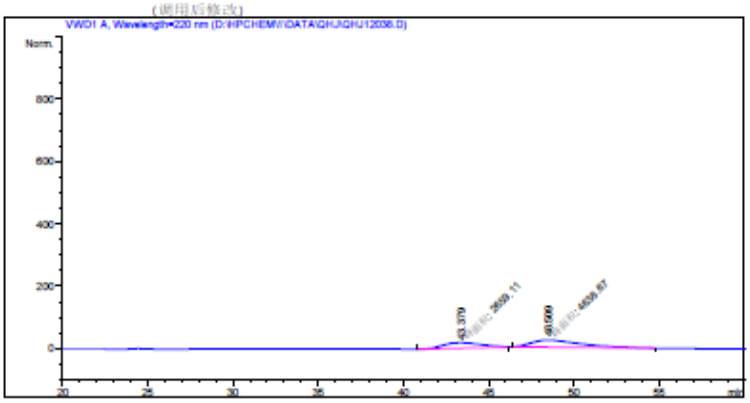
内标使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	40.985	MM	2.3898	5.16465e4	360.18765	50.4564
2	45.825	MM	2.9569	5.07123e4	285.83899	49.5436

总量 : 1.02359e5 646.02664

(调用后修改)



面积百分比报告

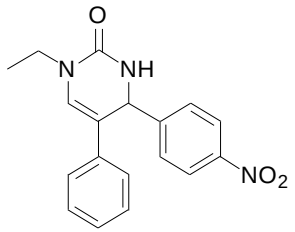
排序	:	信号
乘积因子	:	1.0000
稀释因子	:	1.0000

内标使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

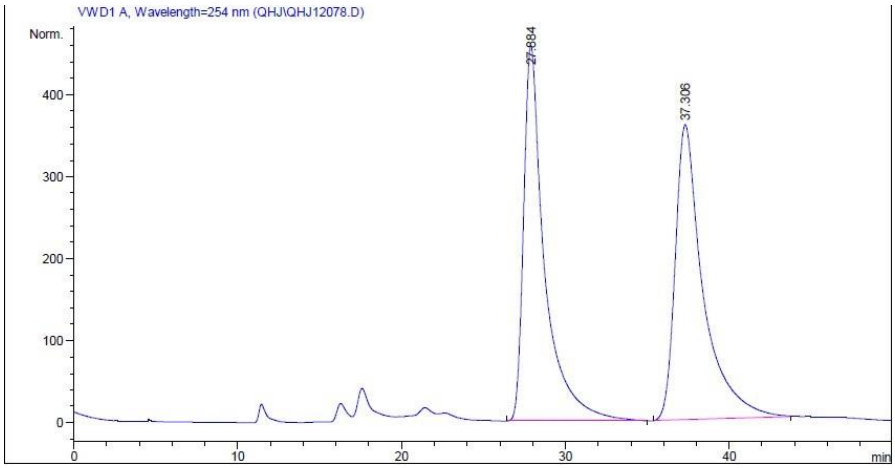
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	43.379	MM	2.5333	2659.10962	17.49437	36.4372
2	48.509	MM	3.3599	4638.67139	23.01027	63.5628

总量 : 7297.78101 40.50464

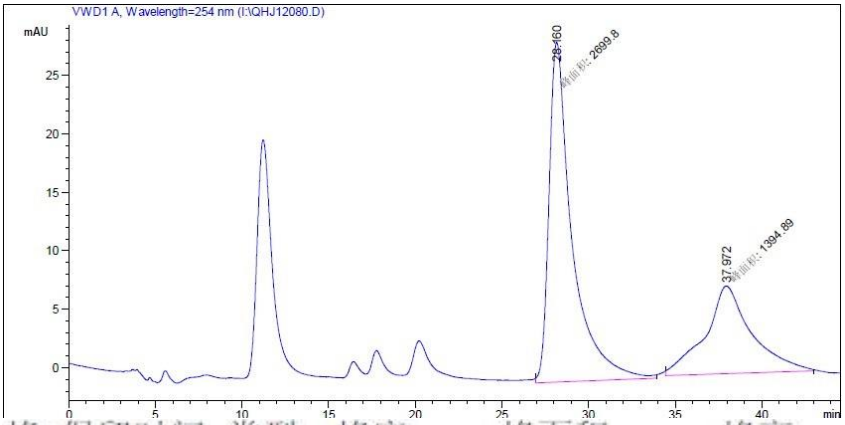


4r

32% ee. [Daicel Chiralpak AS-H, *n*-hexane / isopropanol = 80 / 20, 0.8 mL/min, λ = 254 nm]



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	27.884	PP	1.2738	4.08288e4	457.63019	48.8943
2	37.306	PB	1.7210	4.26754e4	359.94171	51.1057
Totals :				8.35043e4	817.57190	



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 mAU *s	峰高 [mAU]	峰面积 %
1	28.160	MM	1.5473	2699.80127	29.08039	65.9342
2	37.972	MM	3.1005	1394.88831	7.49815	34.0658