Supporting Information

One-pot Sequential Asymmetric Hydrogenation of β-Aryl-β-aryloxy Acroleins†

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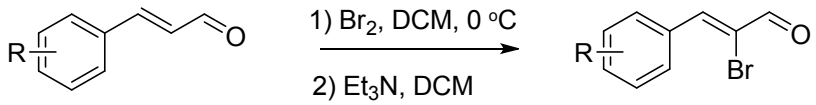
1. General Details

All reactions were performed in dry glassware or in a glove box under an atmosphere of nitrogen. The workup was carried out in air, unless otherwise noted. Solvents were dried and distilled before use by standard procedures. Commercially available reagents were used without further purification.

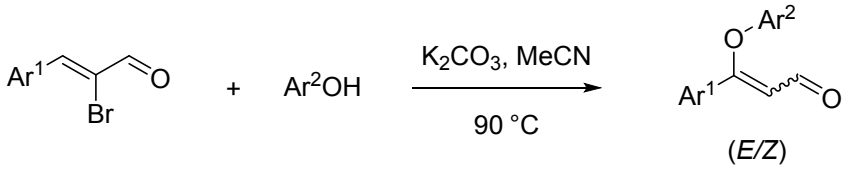
Column chromatography was performed using 100-200 mesh silica gel. Melting points were measured with SGW X-4 micro melting point apparatus. ^{1}H NMR (400 MHz), ^{13}C NMR (100 MHz), and ^{19}F NMR (376 MHz) spectra were recorded on a Varian MERCURY plus-400 spectrometer with TMS as an internal standard. HRMS was performed on a Waters Micromass Q-TOF Premier Mass Spectrometer at the Analysis Center of Shanghai Jiao Tong University. Optical rotations were measured on a Rudolph Research Analytical Autopol VI automatic polarimeter using a 50 mm path-length cell at 589 nm. IR was measured with PerkinElmer Spectrum 100 FT-IR Spectrometer. Enantioselectivity was measured by high performance liquid chromatography (HPLC) using Daicel Chiralcel OD-H or OJ-H columns with hexane/2-propanol as eluent.

2. Synthesis of β-Aryl-β-aryloxy Acroleins



General procedure:[1]

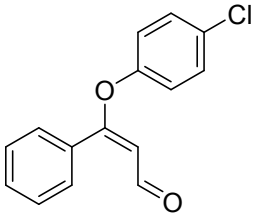
To a solution of cinnamaldehyde (20.0 g, 151 mmol) in DCM (200 mL) was added Br_2 (9.4 mL, 183 mmol, 1.2 equiv.) at 0 °C. The reaction mixture was stirred for 15 min, followed by the addition of Et_3N (36.0 mL, 258 mmol, 1.7 equiv.). After stirring for an additional 15 min, the reaction mixture was diluted with DCM and washed sequentially with a 10% $NaHSO_3$ solution, H_2O, and brine. The organic layer was separated and dried over anhydrous Na_2SO_4, filtered, and concentrated to yield orange oil. After keeping for 3 days at room temperature, the mixture crystallized completely as a bright yellow solid which was confirmed to be the desired *Z*-α-bromocinnamaldehyde (31.3 g, 98% yield).



General procedure:[2]

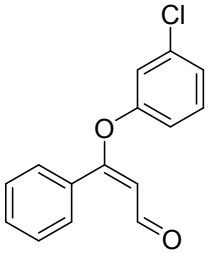
A mixture of *Z*-α-bromocinnamaldehyde (20.0 mmol), phenol (20.0 mmol), K_2CO_3 (50.0 mmol) and MeCN (20 mL) were stirred at 90 °C for 5−8 h. The mixture was cooled to RT and concentrated under reduced pressure. The residue was washed with brine and extracted with EtOAc (3 × 20 mL). The combined organic layers were dried over anhydrous $MgSO_4$, filtered and concentrated to give brownish black oil (*E/Z* = 1/1, 90% yield). The crude material was purified by flash chromatography (80/1 PE/EtOAc) to afford the *E*-isomer (20−45% yield).

(*E*)-3-(4-chlorophenoxy)-3-phenylacrylaldehyde (1a)



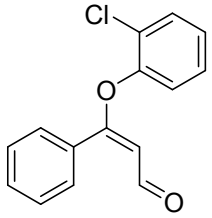
White solid, mp 92−94 °C; Yield: 45%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.08 (1H, d, J = 7.6 Hz), 7.57−7.55 (2H, m), 7.45−7.34 (3H, m), 7.22 (2H, d, J = 8.8 Hz), 6.92 (2H, d, J = 8.8 Hz), 6.26 (1H, d, J = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.4, 166.7, 155.6, 132.3, 131.6, 129.9, 129.1, 128.4, 127.4, 118.1, 116.7; HRMS (ESI-MS) Calcd. For $C_{15}H_{12}ClO_2$ $[M+H]^+$ 259.0520, Found: 259.0530; IR (v/cm^{-1}): 1668, 1623, 1590, 1485, 1448, 1388, 1320, 1302, 1281, 1212, 1164, 1139, 1091, 1010, 828, 771, 711, 692.

(*E*)-3-(3-chlorophenoxy)-3-phenylacrylaldehyde (1b)



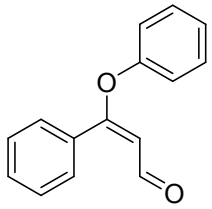
Light yellow liquid; Yield: 46%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.06 (1H, d, J = 8.0 Hz), 7.59−7.57 (2H, m), 7.44−7.38 (3H, m), 7.18−7.16 (1H, t, J = 8.0 Hz), 7.03−7.01 (2H, m), 6.87−6.85 (1H, m), 6.29 (1H, d, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.4, 166.3, 157.7, 135.4, 132.2, 131.7, 130.7, 129.1, 127.3, 123.5, 117.3, 116.8, 114.9; HRMS (ESI-MS) Calcd. For $C_{15}H_{12}ClO_2$ $[M+H]^+$ 259.0520, Found: 259.0518; IR (v/cm^{-1}): 1671, 1623, 1588, 1473, 1491, 1448, 1430, 1388, 1320, 1304, 1279, 1212, 1138, 1071, 895, 776, 717, 689.

(*E*)-3-(2-chlorophenoxy)-3-phenylacrylaldehyde (1c)



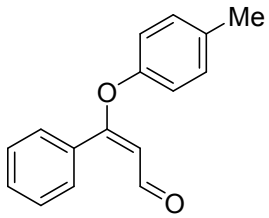
White solid, mp 95−97 °C; Yield: 40%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.1 (1H, d, J = 7.6 Hz), 7.59 (2H, d, J = 6.8 Hz), 7.44−7.37 (4H, m), 7.07−6.98 (2H, m), 6.81−6.78 (1H, m), 6.27 (1H, d, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.5, 167.0, 152.5, 132.2, 131.6, 130.8, 129.1, 127.9, 127.3, 124.3, 123.4, 117.6, 116.6; HRMS (ESI-MS) Calcd. For $C_{15}H_{12}ClO_2$ $[M+H]^+$ 259.0520, Found: 259.0526; IR (v/cm^{-1}): 1669, 1624, 1583, 1476, 1446, 1319, 1265, 1226, 1137, 1061, 1035, 1010, 770, 752, 721, 695.

(*E*)-3-phenoxy-3-phenylacrylaldehyde (1d)[3]



Light yellow liquid; Yield: 42%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.12 (1H, d, J = 8.0 Hz), 7.63 (2H, d, J = 7.6 Hz), 7.44–7.37 (3H, m), 7.31–7.27 (2H, m), 7.07–7.01 (3H, m), 6.29 (1H, d, J = 7.6 Hz).

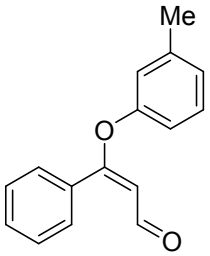
(*E*)-3-phenyl-3-(p-tolyloxy)acrylaldehyde (1e)



White solid, mp 82−83 °C; Yield: 40%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.08 (1H, d, J = 7.6 Hz), 7.60−7.58 (2H, m), 7.41−7.35 (3H, m), 7.05 (2H, d, J = 8.4 Hz), 6.87

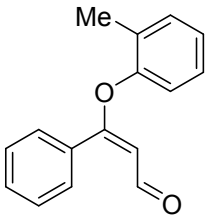
(2H, d, J = 8.4 Hz), 6.22 (1H, d, J = 8.0 Hz), 2.25 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.9, 167.5, 155.0, 132.9, 132.7, 131.3, 130.3, 128.9, 127.5, 116.7, 116.4, 20.5; HRMS (ESI-MS) Calcd. For $C_{16}H_{15}O_2$ $[M+H]^+$ 239.1067, Found: 239.1060; IR (v/cm^{-1}): 2849, 1667, 1622, 1607, 1558, 1505, 1448, 1321, 1302, 1233, 1169, 1140, 1034, 1015, 828, 737, 691.

(*E*)-3-phenyl-3-(m-tolyloxy)acrylaldehyde (1f)



Yellow liquid; Yield: 45%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.05 (1H, d, J = 7.6 Hz), 7.58 (2H, d, J = 8.0 Hz), 7.41–7.32 (3H, m), 7.13–7.09 (1H, m), 6.83–6.81 (2H,m), 6.74 (1H, d, J = 8.4 Hz), 6.24 (1H, d, J = 7.6 Hz), 2.27 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.9, 167.2, 157.3, 140.2, 132.9, 131.3, 129.6, 128.9, 127.4, 124.0, 117.4, 116.5, 113.8, 21.4; HRMS (ESI-MS) Calcd. For $C_{16}H_{15}O_2$ $[M+H]^+$ 239.1067, Found: 239.1063; IR (v/cm^{-1}): 2848, 1668, 1607, 1586, 1488, 1448, 1386, 1321, 1138, 1037, 1021, 998, 849, 775, 693.

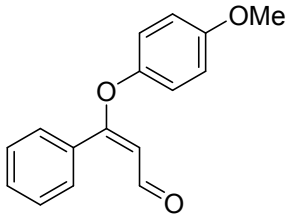
(*E*)-3-phenyl-3-(o-tolyloxy)acrylaldehyde (1g)



White solid, mp 114–115 °C; Yield: 38%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.01 (1H, d, J = 8.0 Hz), 7.54–7.52 (2H, m), 7.42–7.21 (3H, m), 7.20 (1H, d, J = 7.2 Hz), 6.98–6.90 (2H, m), 6.61 (1H, d, J = 8.0 Hz), 6.23 (1H, d, J = 7.6 Hz), 2.47 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.8, 167.7, 155.4, 132.9, 131.5, 131.4, 128.9, 127.2,

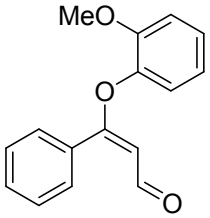
127.1, 126.8, 123.2, 116.1, 115.6, 16.2; HRMS (ESI-MS) Calcd. For $C_{16}H_{15}O_2$ $[M+1]^+$ 239.1067, Found: 239.1066; IR (v/cm^{-1}): 3060, 1667, 1621, 1585, 1574, 1558, 1507, 1488, 1465, 1226, 1160, 1146, 1012, 865, 748, 691.

(*E*)-3-(4-methoxyphenoxy)-3-phenylacrylaldehyde (1h)



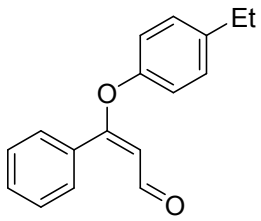
Light yellow solid, mp 52−54 °C; Yield: 41%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.09 (1H, d, J = 8.0 Hz), 7.57 (2H, d, J = 8.0 Hz), 7.40−7.33 (3H, m), 6.91 (2H, d, J = 9.2 Hz), 6.77 (2H, d, J = 8.8 Hz), 6.18 (1H, d, J = 8.0 Hz), 3.73 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.9, 167.8, 155.4, 150.9, 132.9, 131.2, 128.8, 127.6, 118.0, 116.2, 114.8, 55.5; HRMS (ESI-MS) Calcd. For $C_{16}H_{15}O_3$ $[M+H]^+$ 255.1016, Found: 255.1028; IR (v/cm^{-1}): 2836, 1667, 1622, 1558, 1504, 1448, 1387, 1298, 1281, 1246, 1202, 1140, 1036, 830, 737, 692.

(*E*)-3-(2-methoxyphenoxy)-3-phenylacrylaldehyde (1i)



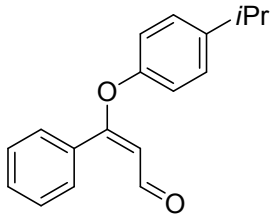
White solid, mp 122−124 °C; Yield: 37%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.13 (1H, d, J = 7.6 Hz), 7.59−7.56 (2H, m), 7.38−7.31 (3H, m), 7.02−6.74 (4H, m), 6.14 (1H, d, J = 7.6 Hz), 3.93 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.8, 168.6, 149.4, 145.8, 132.9, 131.2, 128.7, 127.4, 124.4, 120.9, 118.1, 115.5, 112.5, 55.9; HRMS (ESI-MS) Calcd. For $C_{16}H_{15}O_3$ $[M+H]^+$ 255.1016, Found: 255.1010; IR (v/cm^{-1}): 2833, 1668, 1622, 1540, 1500, 1457, 1387, 1323, 1280, 1254, 1177, 1159, 1144, 1120, 1046, 1028, 869, 753, 696.

(*E*)-3-(4-ethylphenoxy)-3-phenylacrylaldehyde (1j)



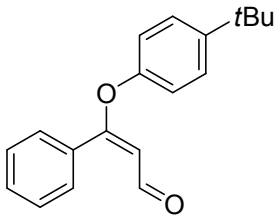
White solid, mp 69−71 °C; Yield: 38%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.07 (1H, d, J = 7.6 Hz), 7.60 (2H, d, J = 8.0 Hz), 7.41−7.36 (3H, m), 7.08 (2H, d, J = 8.4 Hz), 6.90 (2H, d, J = 8.8 Hz), 6.23 (1H, d, J = 7.6 Hz), 2.56 (2H, q, J = 7.6 Hz), 1.17 (3H, t, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.9, 167.4, 155.2, 139.1, 132.9, 131.3, 129.1, 128.9, 127.5, 116.7, 116.4, 27.9, 15.5; HRMS (ESI-MS) Calcd. For $C_{17}H_{17}O_2$ $[M+H]^+$ 253.1223, Found: 253.1229; IR (v/cm^{-1}): 2964, 1667, 1622, 1605, 1505, 1448, 1321, 1303, 1280, 1223, 1210, 1170, 1015, 833, 780, 729, 691.

(*E*)-3-(4-isopropylphenoxy)-3-phenylacrylaldehyde (1k)



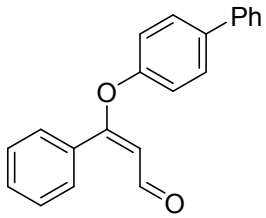
White solid, mp 70−72 °C; Yield: 43%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.07 (1H, d, J = 7.6 Hz), 7.60 (2H, d, J = 7.2 Hz), 7.43−7.36 (3H, m), 7.10 (2H, d, J = 8.4 Hz), 6.90 (2H, d, J = 8.4 Hz), 6.24 (1H, d, J = 7.6 Hz), 2.86−2.79 (1H, m), 1.18 (6H, d, J = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.0, 167.4, 155.2, 143.7, 132.9, 131.3, 128.9, 127.7, 127.5, 116.6, 116.4, 33.3, 24.0; HRMS (ESI-MS) Calcd. For $C_{18}H_{19}O_2$ $[M+H]^+$ 267.1380, Found: 267.1385; IR (v/cm^{-1}): 2960, 1667, 1622, 1603, 1505, 1448, 1386, 1320, 1302, 1209, 1171, 1139, 1033, 1014, 833, 776, 713, 691.

(*E*)-3-(4-(*tert*-butyl)phenoxy)-3-phenylacrylaldehyde (1l)



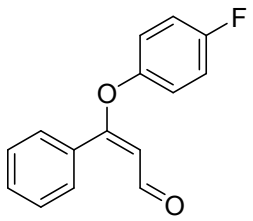
White solid, mp 99−101 °C; Yield: 45%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.09 (1H, d, J = 7.6 Hz), 7.65−7.63 (2H, m), 7.44−7.37 (3H, m), 7.29 (2H, d, J = 9.2 Hz), 6.93 (2H, d, J = 8.8 Hz), 6.28 (1H, d, J = 7.6 Hz), 1.28 (9H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.9, 167.4, 155.0, 146.0, 133.0, 131.3, 128.9, 127.5, 126.7, 116.4, 116.1, 34.2, 31.4; HRMS (ESI-MS) Calcd. For $C_{19}H_{21}O_2$ $[M+H]^+$ 281.1536, Found: 281.1534; IR (v/cm^{-1}): 2962, 1667, 1621, 1601, 1558, 1507, 1337, 1216, 1174, 1139, 1013, 831, 770, 701.

(*E*)-3-([1,1'-biphenyl]-4-yloxy)-3-phenylacrylaldehyde (1m)



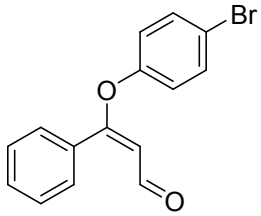
Light yellow solid, mp 99−100 °C; Yield: 46%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.11 (1H, d, J = 7.6 Hz), 7.59 (2H, d, J = 8.0 Hz), 7.45−7.42 (4H, m), 7.37−7.31 (6H, m), 7.02 (2H, d, 8.8 Hz), 6.27 (1H, d, J = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.6, 167.1, 156.8, 140.0, 136.3, 132.7, 131.5, 129.1, 128.8, 128.6, 127.5, 127.2, 126.8, 117.2, 116.7; HRMS (ESI-MS) Calcd. For $C_{21}H_{17}O_2$ $[M+H]^+$ 301.1223, Found: 301.1229; IR (v/cm^{-1}): 1667, 1622, 1603, 1513, 1485, 1448, 1386, 1320, 1279, 1214, 1185, 1170, 1138, 1005, 837, 762, 725, 693.

(*E*)-3-(4-fluorophenoxy)-3-phenylacrylaldehyde (1n)



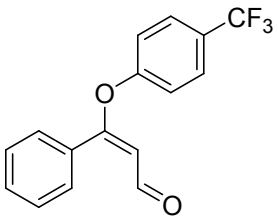
Light yellow liquid; Yield: 42%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.09 (1H, d, J = 7.6 Hz), 7.57−7.55 (2H, m), 7.42−7.34 (3H, m), 6.95−6.93 (4H, m), 6.22 (1H, d, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.6, 169.0, 155.3, 136.5, 132.9, 131.1, 130.5, 130.1, 129.5, 124.9 (d, J = 228.0 Hz), 118.5, 118.2; ^{19}F NMR (376 MHz, $CDCl_3$): δ −120.0; HRMS (ESI-MS) Calcd. For $C_{15}H_{11}O_2FNa$ $[M+Na]^+$ 265.0635, Found: 265.0641; IR (v/cm^{-1}): 1668, 1624, 1606, 1500, 1448, 1321, 1280, 1194, 1139, 1011, 834, 740.

(*E*)-3-(4-bromophenoxy)-3-phenylacrylaldehyde (1o)



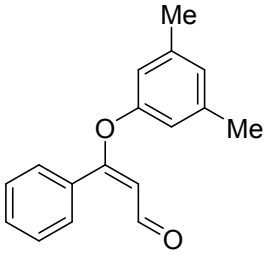
White solid, mp 100−102 °C; Yield: 47%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.07 (1H, d, J = 8.0 Hz), 7.56 (2H, d, J = 8.0 Hz), 7.45−7.35 (5H, m), 6.87 (2H, d, J = 9.2 Hz), 6.26 (1H, d, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.4, 166.6, 156.1, 132.8, 132.2, 131.6, 129.1, 127.4, 118.6, 116.7, 115.8; HRMS (ESI-MS) Calcd. For $C_{15}H_{11}O_2BrNa$ $[M+Na]^+$ 324.9835, Found: 324.9840; IR (v/cm^{-1}): 1662, 1621, 1581, 1480, 1448, 1388, 1283, 1215, 1168, 1143, 1109, 1067, 1033, 1007, 867, 825, 794, 706, 689.

(*E*)-3-phenyl-3-(4-(trifluoromethyl)phenoxy)acrylaldehyde (1p)



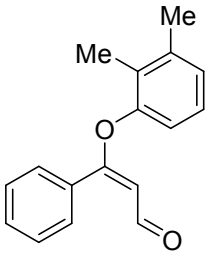
White solid, mp 145−147 °C; Yield: 41%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.07 (1H, d, J = 7.6 Hz), 7.60−7.53 (4H, m), 7.45−7.37 (3H, m), 7.08 (2H, d, J = 8.4 Hz), 6.33 (1H, d, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.1, 166.0, 159.5, 132.0, 131.8, 129.2, 127.4 (dd, J = 7.0, 4.0 Hz), 127.2, 125.5 (q, J = 33.0 Hz), 122.5, 116.9, 116.8; ^{19}F NMR (376 MHz, $CDCl_3$): δ –61.9; HRMS (ESI-MS) Calcd. For $C_{16}H_{12}O_2F_3$ $[M+H]^+$ 293.0784, Found: 293.0790; IR (v/cm^{-1}): 2852, 1668, 1625, 1609, 1512, 1449, 1325, 1278, 1222, 1165, 1122, 1107, 1066, 1009, 839, 747, 696, 594.

(*E*)-3-(3,5-dimethylphenoxy)-3-phenylacrylaldehyde (1q)



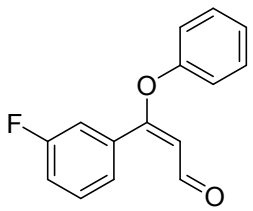
Light yellow solid, mp 59−61 °C; Yield: 44%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.04 (1H, d, J = 7.6 Hz), 7.61 (2H, d, J = 8.0 Hz), 7.42−7.35 (3H, m), 6.66−6.61 (3H, m), 6.26 (1H, d, J = 7.6 Hz), 2.23 (6H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 191.0, 167.3, 157.4, 139.8, 133.0, 131.3, 128.9, 127.4, 125.0, 116.4, 114.4, 21.3; HRMS (ESI-MS) Calcd. For $C_{17}H_{17}O_2$ $[M+H]^+$ 253.1223, Found: 253.1218; IR (v/cm^{-1}): 2919, 2848, 1668, 1612, 1592, 1491, 1472, 1448, 1386, 1321, 1280, 1138, 1051, 837, 712.

(*E*)-3-(2,3-dimethylphenoxy)-3-phenylacrylaldehyde (1r)



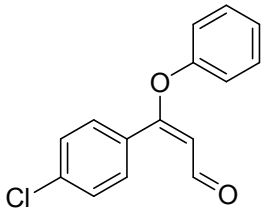
Light yellow solid, mp 55−57 °C; Yield: 45%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.00 (1H, d, J = 8.0 Hz), 7.55 (2H, d, J = 8.0 Hz), 7.42−7.33 (3H, m), 6.89−6.83 (2H, m), 6.51 (1H, d, J = 8.0 Hz), 6.23 (1H, d, J = 8.0 Hz), 2.41 (3H, s), 2.32 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.9, 167.9, 155.3, 138.9, 133.1, 131.3, 128.9, 127.2, 126.1, 125.4, 124.8, 115.9, 113.4, 20.0, 12.0; HRMS (ESI-MS) Calcd. For $C_{17}H_{17}O_2$ $[M+H]^+$ 253.1223, Found: 253.1229; IR (v/cm^{-1}): 1668, 1622, 1605, 1576, 1558, 1507, 1489, 1466, 1448, 1386, 1321, 1304, 1236, 1189, 1140, 1095, 1077, 770, 727, 689.

(*E*)-3-(3-fluorophenyl)-3-phenoxyacrylaldehyde (1s)



Light yellow liquid; Yield: 38%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.08 (1H, d, J = 7.6 Hz), 7.41−7.26 (5H, m), 7.13−6.97 (4H, m), 6.25 (1H, d, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.5, 165.5 (d, J = 3.0 Hz), 162.8 (d, J = 248.0 Hz), 156.9, 135.2 (d, J = 8.0 Hz), 130.7 (d, J = 8.0 Hz), 130.0, 123.5, 123.2 (d, J = 3.0 Hz), 118.4 (d, J = 21.0 Hz), 117.2, 116.7, 114.4 (d, J = 23.0 Hz); ^{19}F NMR (376 MHz, $CDCl_3$): δ –111.1; HRMS (ESI-MS) Calcd. For $C_{15}H_{12}FO_2$ $[M+H]^+$ 243.0816, Found: 243.0807; IR (v/cm^{-1}): 2954, 2924, 2853, 1669, 1623, 1589, 1488, 1456, 1441, 1310, 1207, 1128, 1030, 751.

(*E*)-3-(4-chlorophenyl)-3-phenoxyacrylaldehyde (1t)



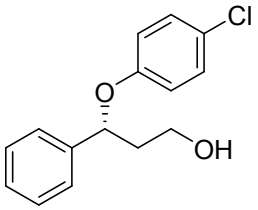
White solid, mp 74−76 °C; Yield: 20%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 10.07 (1H, d, J = 7.6 Hz), 7.52 (2H, d, J = 8.4 Hz), 7.34−7.24 (4H, m), 7.06−6.96 (3H, m), 6.23 (1H, d, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 190.5, 165.8, 156.9, 137.5, 131.2, 130.0, 129.3, 128.7, 123.5, 116.8, 116.7; HRMS (ESI-MS) Calcd. For $C_{15}H_{12}ClO_2$ $[M+H]^+$ 259.0520, Found: 259.0527; IR (v/cm^{-1}): 1669, 1621, 1592, 1488, 1406, 1386, 1311, 1296, 1207, 1165, 1140, 1093, 1027, 1011, 836, 790, 753, 690.

3. Hydrogenation of β-Aryl-β-aryloxy Acroleins

General procedure:

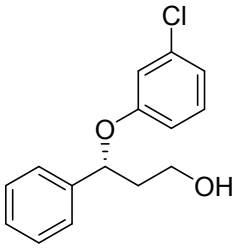
Catalyst (1 mol %) and β-aryl-β-aryloxy acrolein (0.078 mmol) were dissolved in a degassed solution of DCM (1 mL) and TFE (1 mL) under a nitrogen atmosphere. The solution was transferred to an autoclave and the H_2 pressure was adjusted to 30 bar. After stirring for several hours, the reaction mixture was concentrated under reduced pressure. The mixture was purified by flash chromatography (1/1 PE/EtOAc) to give pure product for the determination of enantioselectivity using a Daicel Chiralcel OJ-H or OD-H column with hexane/2-propanol as eluent.

(*R*)-3-(4-chlorophenoxy)-3-phenylpropan-1-ol (2a)



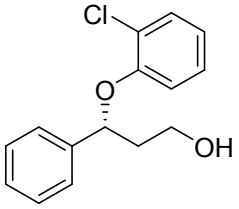
Oil; Yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32−7.22 (5H, m), 7.10 (2H, d, J = 8.8 Hz), 6.76 (2H, d, J = 8.8 Hz), 5.30 (1H, dd, J = 4.4, 8.4 Hz), 3.89−3.73 (2H, m), 2.26−2.01 (2H, m), 1.79 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.4, 140.9, 129.1, 128.7, 127.7, 125.8, 117.2, 78.3, 59.5, 41.0; HRMS (ESI-MS) Calcd. For $C_{15}H_{15}O_2ClNa$ $[M+Na]^+$ 285.0653, Found: 285.0658; IR (v/cm^{-1}): 3360, 2924, 1594, 1581, 1489, 1453, 1282, 1260, 1237, 1169, 1090, 1050, 822, 756, 701, 668; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 10.8 min (*R*), t_{R2} = 12.5 min (*S*), er = 91:9; $[\alpha]_D^{25}$ = 26 (*c* 0.26, CH_2Cl_2).

(*R*)-3-(3-chlorophenoxy)-3-phenylpropan-1-ol (2b)[4]



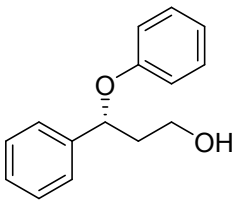
Oil; Yield: 96%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35−7.25 (5H, m), 7.07 (1H, t, J = 8.0 Hz), 6.88−6.84 (2H, m), 6.72 (1H, d, J = 8.4 Hz), 5.35 (1H, dd, J = 4,4, 8.4 Hz), 3.90−3.73 (2H, m), 2.26−2.03 (2H, m), 1.84 (1H, s); The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 10.8 min (*R*), t_{R2} = 11.7 min (*S*), er = 90:10.

(*R*)-3-(2-chlorophenoxy)-3-phenylpropan-1-ol (2c)[4]



Oil; Yield: 95%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39−7.26 (6H, m), 7.00 (1H, t, J = 7.6 Hz), 6.82 (1H, t, J = 7.6 Hz), 6.70 (1H, d, J = 7.2 Hz), 5.43 (1H, dd, J = 4.0, 8.4 Hz), 3.92−3.83 (2H, m), 2.37 (1H, s), 2.31−2.13 (2H, m); The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 16.8 min (*S*), t_{R2} = 17.6 min (*R*), er = 10:90.

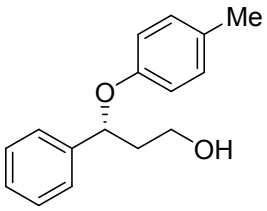
(*R*)-3-phenoxy-3-phenylpropan-1-ol (2d)[4]



Oil; Yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38−7.31 (4H, m), 7.26−7.15 (3H, m), 6.89−6.84 (3H, m), 5.37 (1H, dd, J = 4.0, 8.0 Hz), 3.89−3.80 (2H, m), 2.25−2.09

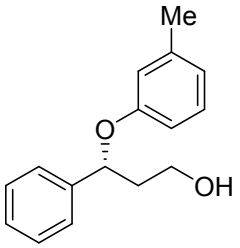
(2H, m), 1.86 (1H, s); The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 85/15, 230 nm, flow = 0.8 mL/min), t_{R1} = 13.4 min (*R*), t_{R2} = 18.2 min (*S*), er = 91:9.

(*R*)-3-phenyl-3-(p-tolyloxy)propan-1-ol (2e)



Oil; Yield: 97%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37−7.21 (5H, m), 6.97 (2H, d, J = 8.8 Hz), 6.74 (2H, d, J = 8.4 Hz), 5.32 (1H, dd, J = 4.4, 8.8 Hz), 3.90−3.76 (2H, m), 2.26−2.03 (3H, m), 2.21 (3H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.6, 141.6, 130.2, 129.8, 128.6, 127.5, 125.8, 115.8, 78.5, 60.0, 41.2, 20.4; HRMS (ESI-MS) Calcd. For $C_{16}H_{18}O_2Na$ $[M+Na]^+$ 265.1199, Found: 265.1204; IR (v/cm^{-1}): 3345, 2923, 1613, 1585, 1509, 1453, 1357, 1288, 1235, 1174, 1050, 816, 755, 700, 511; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 14.8 min (*R*), t_{R2} = 21.0 min (*S*), er = 91:9; $[\alpha]_D^{25}$ = 27 (*c* 0.38, CH_2Cl_2).

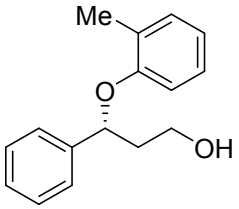
(*R*)-3-phenyl-3-(m-tolyloxy)propan-1-ol (2f)



Oil; Yield: 96%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37−7.22 (5H, m), 7.04 (1H, t, J = 8.0 Hz), 6.70−6.61 (3H, m), 5.35 (1H, dd, J = 4.4, 8.8 Hz), 3.90−3.76 (2H, m), 2.24 (3H, s), 2.22−2.03 (3H, m); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.8, 141.6, 139.4, 129.1, 128.6, 127.5, 125.8, 121.8, 116.9, 112.6, 78.1, 60.0, 41.2, 21.4; HRMS (ESI-

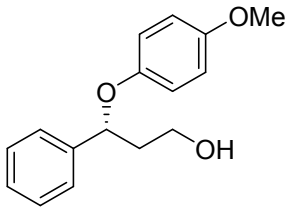
MS) Calcd. For $C_{16}H_{18}O_2Na$ $[M+Na]^+$ 265.1199, Found: 265.1204; IR (v/cm^{-1}): 3345, 2924, 1601, 1583, 1488, 1453, 1288, 1260, 1155, 1051, 769, 700; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 13.3 min (*R*), t_{R2} = 19.8 min (*S*), er = 80:20; $[\alpha]_D^{25}$ = 14 (*c* 0.36, CH_2Cl_2).

(*R*)-3-phenyl-3-(o-tolyloxy)propan-1-ol (2g)[4]



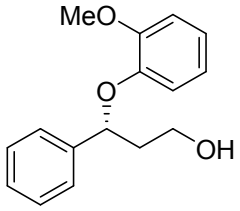
Oil; Yield: 99%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35−7.22 (5H, m), 7.12 (1H, d, *J* = 7.6 Hz), 6.96 (1H, t, *J* = 8.0 Hz), 6.78 (1H, t, *J* = 7.6 Hz), 6.62 (1H, d, *J* = 8.0 Hz), 5.40 (1H, dd, *J* = 4.0, 8.4 Hz), 3.89−3.79 (2H, m), 2.32 (3H, s), 2.26−2.11 (2H, m), 1.98 (1H, s); The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 11.8 min (*S*), t_{R2} = 15.7 min (*R*), er = 10:90.

(*R*)-3-(4-methoxyphenoxy)-3-phenylpropan-1-ol (2h)[4]



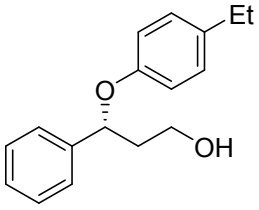
Oil; Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36−7.25 (5H, m), 6.79−6.70 (4H, m), 5.26 (1H, dd, *J* = 4.0, 8.4 Hz), 3.88−3.79 (2H, m), 3.70 (3H, s), 2.23−2.03 (2H, m); The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 34.7 min (*R*), t_{R2} = 36.8 min (*S*), er= 89:11.

(*R*)-3-(2-methoxyphenoxy)-3-phenylpropan-1-ol (2i)



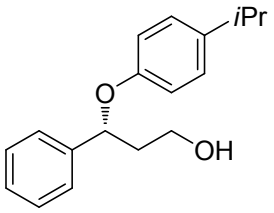
Oil; Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38−7.24 (5H, m), 6.88−6.82 (2H, m), 6.69−6.57 (2H, m), 5.23 (1H, dd, J = 3.6, 9.6 Hz), 3.92−3.83 (2H, m), 3.88 (3H, s), 3.54 (1H, s), 2.29−2.04 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.7, 147.2, 141.6, 128.6, 127.7, 125.7, 121.8, 120.6, 116.2, 111.4, 82.6, 60.9, 55.7, 41.2; HRMS (ESI-MS) Calcd. For $C_{16}H_{18}O_3Na$ $[M+Na]^+$ 281.1148, Found: 281.1154; IR (v/cm^{-1}): 3361, 2959, 2918, 2849, 1662, 1592, 1503, 1455, 1256, 1222, 1121, 1026, 797, 742, 701; The er was determined by HPLC on a Daicel Chiralcel OD-H column (hexane/2-propanol = 95/5, 230 nm, flow = 0.8 mL/min), t_{R1} = 25.2 min (*S*), t_{R2} = 36.1 min (*R*), er = 22:78; $[\alpha]_D^{25}$ = 13 (*c* 0.20, CH_2Cl_2).

(*R*)-3-(4-ethylphenoxy)-3-phenylpropan-1-ol (2j)



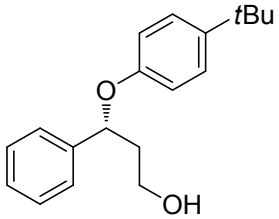
Oil; Yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37−7.23 (5H, m), 7.01 (2H, d, J = 8.4 Hz), 6.77 (2H, d, J = 8.4 Hz), 5.33 (1H, dd, J = 4.0, 8.8 Hz), 3.90−3.79 (2H, m), 2.52 (2H, q, J = 7.6 Hz), 2.22−2.03 (3H, m), 1.15 (3H, t, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.7, 141.6, 136.7, 128.6, 128.6, 127.5, 125.7, 115.7, 78.4, 60.0, 41.2, 27.9, 15.7; HRMS (ESI-MS) Calcd. For $C_{17}H_{20}O_2Na$ $[M+Na]^+$ 279.1356, Found: 279.1361; IR (v/cm^{-1}): 3355, 2961, 2926, 1611, 1509, 1453, 1295, 1234, 1175, 1053, 827, 700; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 10.7 min (*R*), t_{R2} = 12.4 min (*S*), er = 89:11; $[\alpha]_D^{25}$ = 29 (*c* 0.38, CH_2Cl_2).

(*R*)-3-(4-isopropylphenoxy)-3-phenylpropan-1-ol (2k)



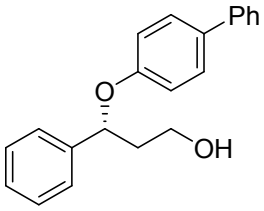
Oil; Yield: 96%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38−7.23 (5H, m), 7.03 (2H, d, J = 8.4 Hz), 6.78 (2H, d, J = 8.0 Hz), 5.32 (1H, dd, J = 4.0, 8.8 Hz), 3.87−3.78 (2H, m), 2.82−2.75 (1H, m), 2.22−2.06 (3H, m), 1.16 (6H, d, J = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.8, 141.7, 141.3, 128.6, 127.5, 127.1, 125.7, 115.6, 78.4, 60.0, 41.2, 33.1, 24.1; HRMS (ESI-MS) Calcd. For $C_{18}H_{22}O_2Na$ $[M+Na]^+$ 293.1512, Found: 293.1517; IR (v/cm^{-1}): 3355, 2924, 1609, 1509, 1454, 1382, 1362, 1282, 1237, 1177, 1050, 826, 754, 700, 574; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 7.6 min (*S*), t_{R2} = 8.3 min (*R*), er = 12:88; $[\alpha]_D^{25}$ = 27 (*c* 0.38, CH_2Cl_2).

(*R*)-3-(4-(*tert*-butyl)phenoxy)-3-phenylpropan-1-ol (2l)



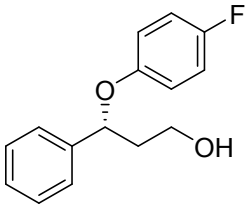
Oil; Yield: 97%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42−7.34 (4H, m), 7.31−7.27 (1H, m), 7.23 (2H, d, J = 8.8 Hz), 6.81 (2H, d, J = 8.8 Hz), 5.36 (1H, dd, J = 4.0, 8.8 Hz), 3.91−3.82 (2H, m), 2.26−2.06 (2H, m), 2.03 (1H, s), 1.27 (9H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.5, 143.6, 141.8, 128.6, 127.5, 126.1, 125.8, 115.2, 78.4, 60.1, 41.3, 34.0, 31.4; HRMS (ESI-MS) Calcd. For $C_{19}H_{24}O_2Na$ $[M+Na]^+$ 307.1669, Found: 307.1664; IR (v/cm^{-1}): 2960, 1607, 1510, 1455, 1362, 1292, 1240, 1183, 1049, 827, 802, 751. 700, 548; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 7.2 min (*S*), t_{R2} = 8.8 min (*R*), er = 12:88 ; $[\alpha]_D^{25}$ = 32 (*c* 0.20, CH_2Cl_2).

(*R*)-3-([1,1'-biphenyl]-4-yloxy)-3-phenylpropan-1-ol (2m)



Oil; Yield: 97%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48−7.23 (12H, m), 6.91 (2H, d, J = 8.4 Hz), 5.4 (1H, dd, J = 4.4, 8.8 Hz), 3.92−3.78 (2H, m), 2.28−2.06 (2H, m), 2.01 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.4, 141.4, 134.0, 128.7, 128.7, 128.1, 127.7, 126.7, 126.6, 125.8, 116.1, 78.2, 59.9, 41.2; HRMS (ESI-MS) Calcd. For $C_{21}H_{20}O_2Na$ $[M+Na]^+$ 327.1356, Found: 327.1361; IR (v/cm^{-1}): 3335, 2924, 1652, 1608, 1517, 1487, 1455, 1410, 1288, 1262, 1174, 1049, 831, 799, 762, 698; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 80/20, 230 nm, flow = 0.8 mL/min), t_{R1} = 15.0 min (*S*), t_{R2} = 22.9 min (*R*), er = 11:89; $[\alpha]_D^{25}$ = 30 (*c* 0.32, CH_2Cl_2).

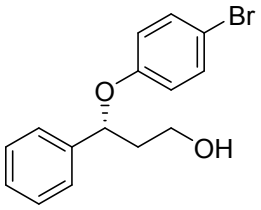
(*R*)-3-(4-fluorophenoxy)-3-phenylpropan-1-ol (2n)



Oil; Yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34−7.25 (5H, m), 6.86 (2H, t, J = 8.4 Hz), 6.79−6.76 (2H, m), 5.29 (1H, dd, J = 4.4, 8.8 Hz), 3.91−3.78 (2H, m), 2.24−2.04 (2H, m), 1.94 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.4, 155.0 (d, J = 220.0 Hz), 141.1, 128.7, 127.7, 125.8, 117.1 (d, J = 8.0 Hz), 115.7 (d, J = 23.0 Hz), 78.9, 59.7, 41.1; ^{19}F NMR (376 MHz, $CDCl_3$): δ –123.4; HRMS (ESI-MS) Calcd. For $C_{15}H_{15}O_2FNa$ $[M+Na]^+$ 269.0948, Found: 269.0954; IR (v/cm^{-1}): 3345, 2924, 1504, 1454, 1376, 1291, 1245, 1203, 1097, 1051, 982, 827, 795, 741, 700; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10,

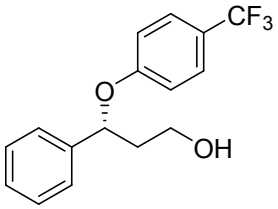
230 nm, flow = 0.8 mL/min), t_{R1} = 14.0 min (*R*), t_{R2} = 17.5 min (*S*), er = 90:10; $[\alpha]_D^{25}$ = 23 (*c* 0.38, CH_2Cl_2).

(*R*)-3-(4-bromophenoxy)-3-phenylpropan-1-ol (2o)



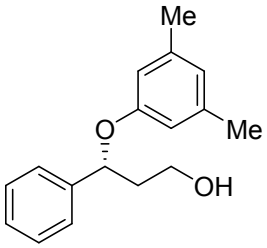
Oil; Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34−7.25 (7H, m), 6.72 (2H, d, *J* = 8.8 Hz), 5.32 (1H, dd, *J* = 4.4, 8.8 Hz), 3.88−3.77 (2H, m), 2.24−2.04 (2H, m), 1.83 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.8, 140.9, 132.1, 128.7, 127.8, 125.7, 117.7, 113.1, 78.1, 59.6, 41.1; HRMS (ESI-MS) Calcd. For $C_{15}H_{15}O_2BrNa$ $[M+Na]^+$ 329.0148, Found: 329.0153; IR (v/cm^{-1}): 3362, 2924, 1653, 1588, 1486, 1454, 1280, 1237, 1170, 1050, 820, 700; The er was determined by HPLC on a Daicel Chiralcel OD-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 13.1 min (*R*), t_{R2} = 16.9 min (*S*), er = 90:10; $[\alpha]_D^{25}$ = 27 (*c* 0.30, CH_2Cl_2).

(*R*)-3-phenyl-3-(4-(trifluoromethyl)phenoxy)propan-1-ol (2p)[4]



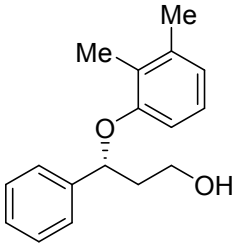
Oil; Yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (2H, d, *J* = 8.8 Hz), 7.35−7.25 (5H, m), 6.91 (2H, d, *J* = 8.4 Hz), 5.43 (1H, dd, *J* = 4.4, 8.4 Hz), 3.92−3.75 (2H, m), 2.29−2.07 (2H, m), 1.74 (1H, s); The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 7.0 min (*S*), t_{R2} = 7.7 min (*R*), er = 11:89.

(*R*)-3-(3,5-dimethylphenoxy)-3-phenylpropan-1-ol (2q)



Oil; Yield: 97%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37−7.24 (5H, m), 6.53−6.49 (3H, m), 5.35 (1H, dd, J = 4.0, 8.8 Hz), 3.86−3.79 (2H, m), 2.09−2.05 (3H, m), 2.19 (6H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.8, 141.6, 139.0, 128.6, 127.5, 125.7, 122.8, 113.5, 78.0, 60.0, 41.1, 21.4; HRMS (ESI-MS) Calcd. For $C_{17}H_{20}O_2Na$ $[M+Na]^+$ 279.1356, Found: 279.1361; IR (v/cm^{-1}): 3354, 3030, 2958, 2922, 2854, 1668, 1611, 1594, 1489, 1471, 1455, 1418, 1320, 1293, 1261, 1165, 1153, 1055, 829, 700; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 9.3 min (*R*), t_{R2} = 11.0 min (*S*), er = 91:9; $[\alpha]_D^{25}$ = 28 (*c* 0.24, CH_2Cl_2).

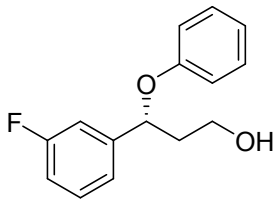
(*R*)-3-(2,3-dimethylphenoxy)-3-phenylpropan-1-ol (2r)



Oil; Yield: 98%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35−7.24 (5H, m), 6.85 (1H, t, J = 8.0 Hz), 6.70 (1H, d, J = 7.6 Hz), 6.51 (1H, d, J = 8.4 Hz), 5.37 (1H, dd, J = 4.0, 8.8 Hz), 3.89−3.78 (2H, m), 2.30−2.08 (2H, m), 2.17 (6H, d, J = 5.2 Hz), 1.98 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.4, 141.7, 137.8, 128.6, 127.5, 125.7, 125.6, 122.3, 110.6, 77.8, 60.0, 41.3, 20.1, 11.9; HRMS (ESI-MS) Calcd. For $C_{17}H_{20}O_2Na$ $[M+Na]^+$ 279.1356, Found: 279.1361; IR (v/cm^{-1}): 3334, 3030, 2923, 1583, 1471, 1453, 1302, 1256, 1101, 1049, 766, 700; The er was determined by HPLC on a Daicel Chiralcel

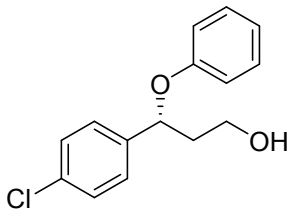
OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 9.8 min (*R*) , t_{R2} = 15.0 min (*S*), er = 87:13; $[\alpha]_D^{25}$ = −30 (*c* 0.38, CH_2Cl_2).

(*R*)-3-(3-fluorophenyl)-3-phenoxypropan-1-ol (2s)



Oil; Yield: 96%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32−7.07 (5H, m), 6.96−6.83 (4H, m), 5.36 (1H, dd, *J* = 4.0, 8.4 Hz), 3.89−3.76 (2H, m), 2.24−2.03 (2H, m), 1.90 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.1 (d, *J* = 245.0 Hz), 157.6, 144.4 (d, *J* = 7.0 Hz), 130.3 (d, *J* = 8.0 Hz), 129.4, 121.5 (d, *J* = 3.0 Hz), 121.2, 115.8, 114.6 (d, *J* = 21.0 Hz), 112.8 (d, *J* = 22.0 Hz), 77.3 (d, *J* = 1.0 Hz), 59.6, 41.0; ^{19}F NMR (376 MHz, $CDCl_3$): δ –112.3; HRMS (ESI-MS) Calcd. For $C_{15}H_{15}O_2FNa$ $[M+Na]^+$ 269.0948, Found: 269.0954; IR (v/cm^{-1}): 3361, 2925, 1615, 1597, 1493, 1351, 1289, 1236, 1172, 1137, 1051, 1028, 787, 753, 691; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 11.1 min (*R*), t_{R2} = 14.2 min (*S*), er = 87:13; $[\alpha]_D^{25}$ = 13 (*c* 0.38, CH_2Cl_2).

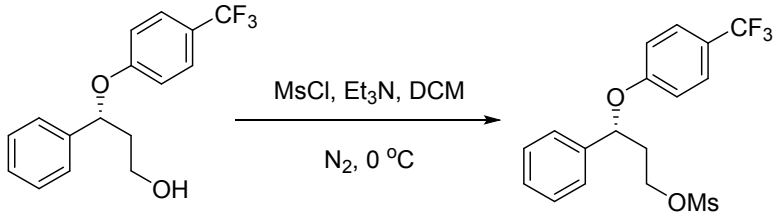
(*R*)-3-(4-chlorophenyl)-3-phenoxypropan-1-ol (2t)



Oil; Yield: 97%; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33−7.29 (4H, m), 7.19 (2H, t, *J* = 7.6 Hz), 6.89 (1H, t, *J* = 7.2 Hz), 6.82 (2H, d, *J* = 8.8 Hz), 5.35 (1H, dd, *J* = 4.4, 8.8 Hz), 3.88−3.76 (2H, m), 2.24−2.01 (2H, m), 1.87 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.8, 140.3, 133.5, 129.6, 129.1, 127.5, 121.4, 116.1, 77.5, 59.8, 41.3; HRMS (ESI-MS) Calcd. For $C_{15}H_{15}O_2ClNa$ $[M+Na]^+$ 285.0653, Found: 285.0658; IR (v/cm^{-1}): 3345, 3063, 2925, 1668, 1598, 1491, 1408, 1236, 1172, 1089, 1051, 1014,

834, 752, 691; The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 9.5 min (*R*), t_{R2} = 10.3 min (*S*), er = 83:17; $[\alpha]_D^{25}$ = 19 (*c* 0.26, CH_2Cl_2).

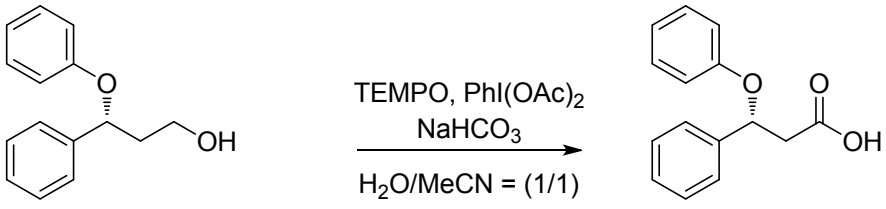
4. Further Applications



(*R*)-**2p**
89:11 er

(*R*)-**4**
82% yield, 88:12 er

The alcohol (*R*)-**2p** (21.5 mg, 0.071 mmol) was taken up in DCM (2 mL) under N_2 and cooled to 0 °C. The solution was treated sequentially with triethylamine (0.015 mL, 0.14 mmol) and methanesulfonyl chloride (0.008 mL, 0.14 mmol). After 1 hour, the mixture was stirred at RT overnight. The mixture was diluted with DCM (15 mL), washed with brine (5 mL), dried over Na_2SO_4, and concentrated to afford the crude mesylate. The crude product was purified by flash column using silica gel (10/90 PE/EtOAc) to give (*R*)-**4** as light yellow soild in 82% yield (17.4 mg).[5] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (2H, d, J = 8.4 Hz), 7.36−7.27 (5H, m), 6.89 (2H, d, J = 8.4 Hz), 5.35 (1H, dd, J = 4.4, 8.8 Hz), 4.50−4.46 (1H, m), 4.36−4.30 (1H, m), 2.95 (3H, s), 2.44−2.35 (1H, m), 2.33−2.24 (1H, m); The er was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 90/10, 230 nm, flow = 0.8 mL/min), t_{R1} = 49.2 min (*S*), t_{R2} = 59.8 min (*R*), er = 12:88.



(*R*)-**2d**
90:10 er

(R)-**5**
78% yield, 90:10 er

The alcohol (*R*)-**2d** (30 mg, 0.13 mmol), iodobenzene diacetate (93 mg, 0.28 mmol), TEMPO (4 mg, 0.03 mmol), and sodium bicarbonate (22 mg, 0.26 mmol) were combined. MeCN (1 mL) and H_2O (1 mL) were added to the vial. The vial was sealed

with a PTFE/silicone-lined septum cap, and the reaction was first stirred at 0 °C for 2 hours then stirred at RT overnight. The reaction mixture was poured into 1 M HCl (10 mL) and extracted with EtOAc (3 × 20 mL). The combined organic layers were concentrated. The crude product was purified by flash chromatography using silica gel (40/60 PE/EtOAc) to give (*R*)-**6** as colorless oil in 78% yield (24 mg).[6] ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40−7.14 (7H, m), 6.89−6.84 (3H, m), 5.61 (1H, dd, J = 4.4, 9.2 Hz), 3.07 (1H, dd, J = 9.2, 16.0 Hz), 2.81 (1H, dd, J = 4.4, 16.0 Hz). The er value was determined by HPLC on a Daicel Chiralcel OJ-H column (hexane/2-propanol = 80/20, 230 nm, flow = 0.8 mL/min), t_{R1} = 11.1 min (*S*), t_{R2} = 12.2 min (*R*), er = 10:90.

5. References

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[2] (a) P. E. Maligres, S. W. Krska and G. R. Humphrey, *Org. Lett*., 2004, **6**, 3147; (b) A. V. Mareev, I. A. Ushakov and A. Y. Rulev, *Tetrahedron*, 2015, **71**, 1971.

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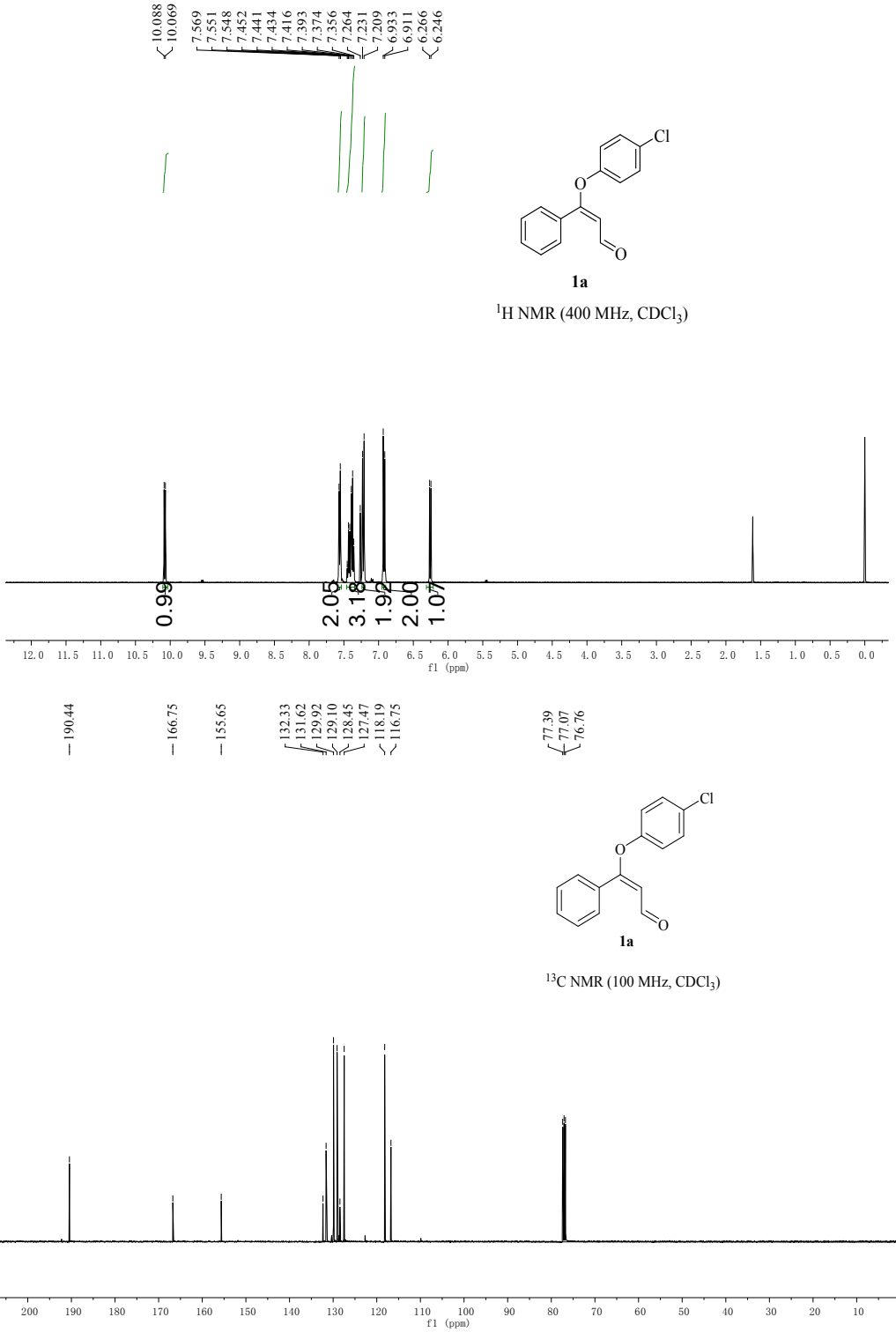
[5] (a) R. Chênevert, G. Fortier and R. B. Rhlid, *Tetrahedron,* 1992, **48**, 6769; (b) B. M. Trost, P. L. Fraisse and Z. T. Ball, *Angew. Chem. Int. Ed*., 2002, **41**, 1059.

[6] (a) L. M. Stanley and J. F. Hartwig, *Angew. Chem. Int. Ed*., 2009, **48**, 7841; (b) M. Kawasaki, H. Kakuda, M. Goto, S. Kawabata, T. Kometani, *Tetrahedron: Asymmetry*, 2003, **14**, 1529.

6. NMR Spectra

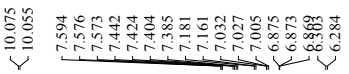


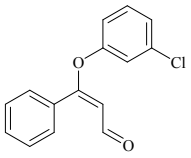
1a

^{1}H NMR (400 MHz, $CDCl_3$)

1a

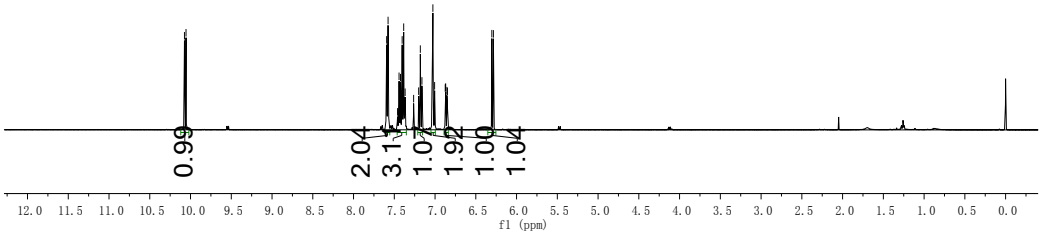
^{13}C NMR (100 MHz, $CDCl_3$)

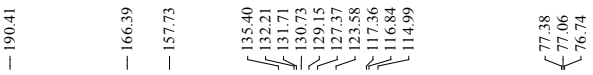


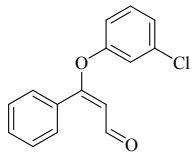


1b

^{1}H NMR (400 MHz, $CDCl_3$)

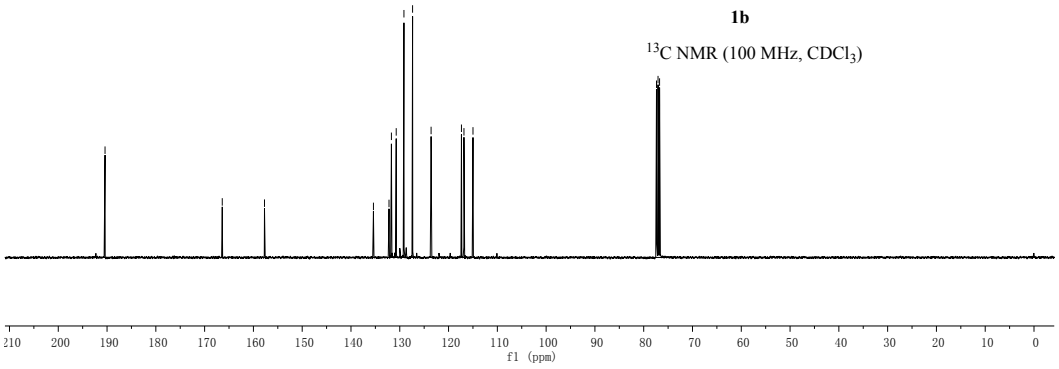


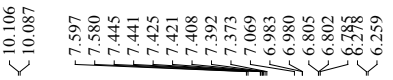


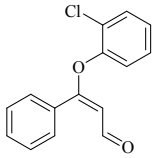


1b

^{13}C NMR (100 MHz, $CDCl_3$)

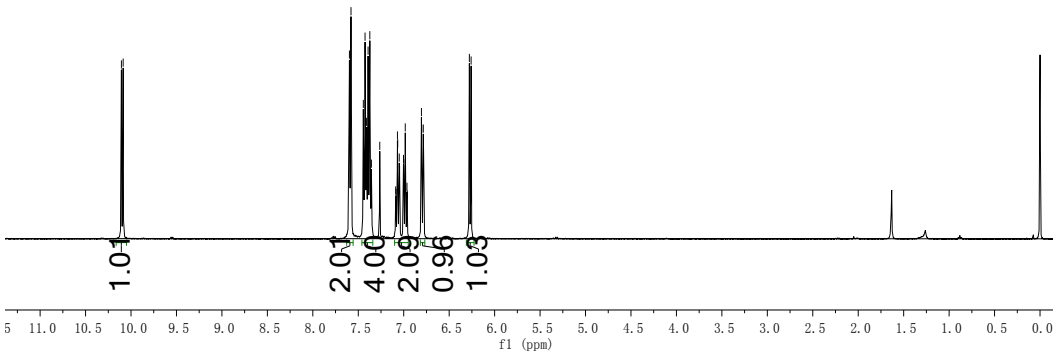


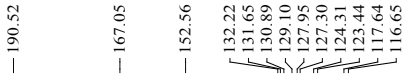


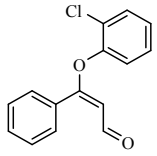


1c

^{1}H NMR (400 MHz, $CDCl_3$)

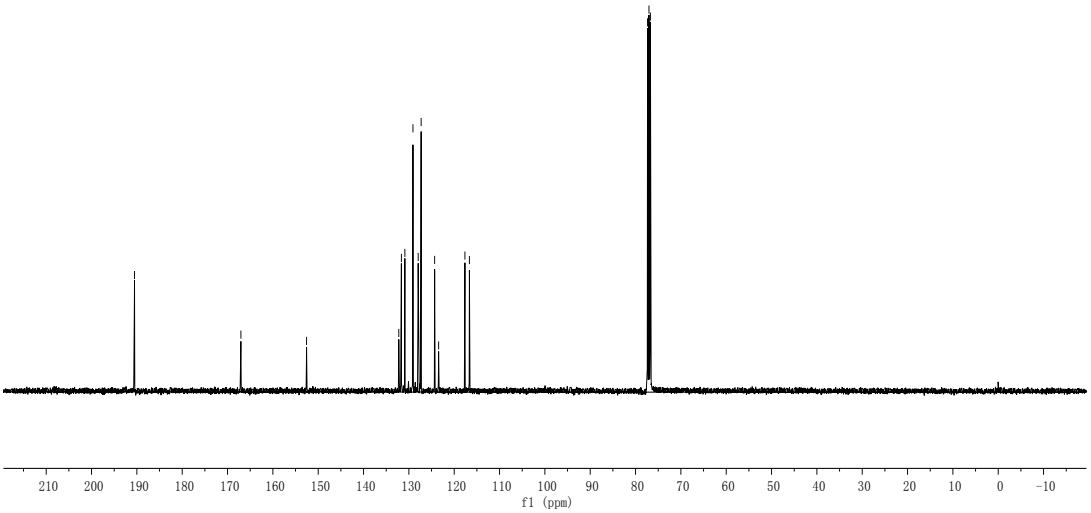


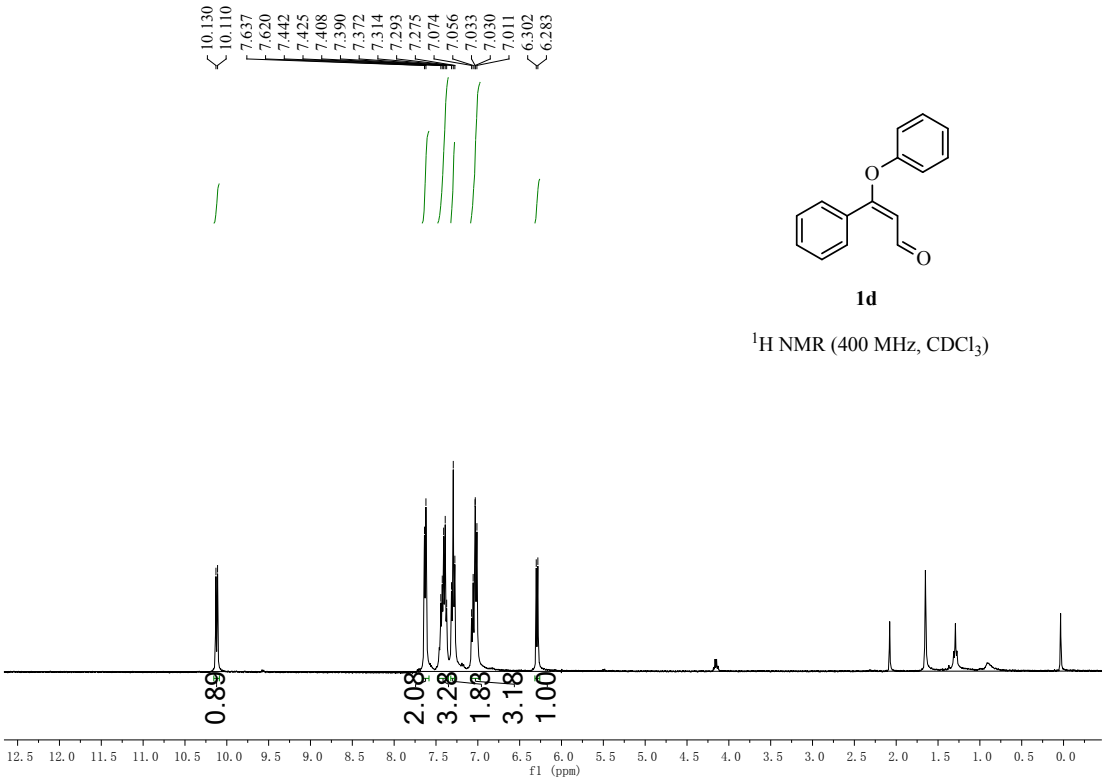




1c

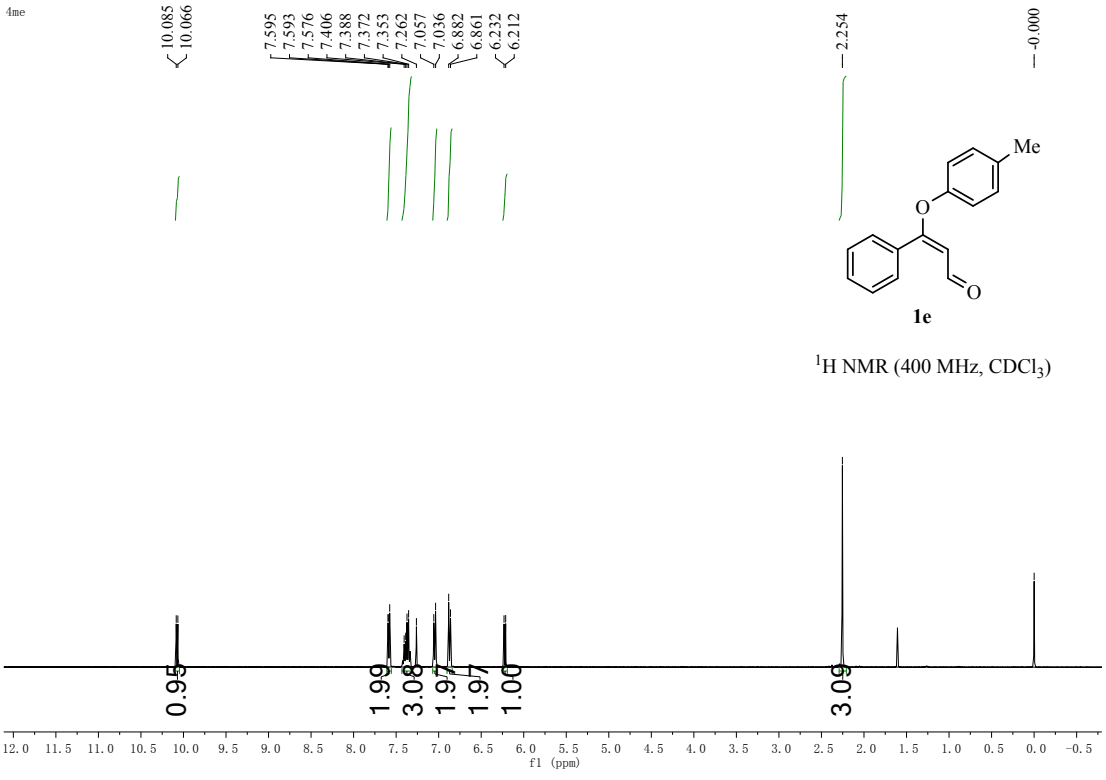
^{13}C NMR (100 MHz, $CDCl_3$)





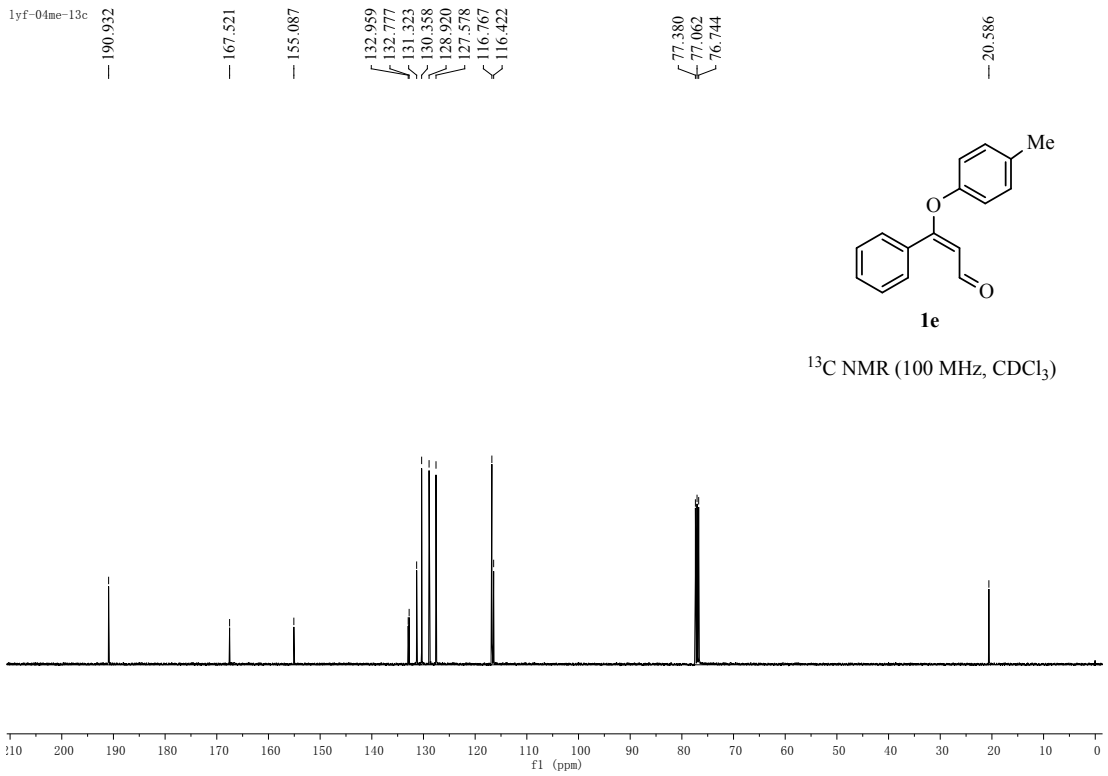
1d

^{1}H NMR (400 MHz, $CDCl_3$)



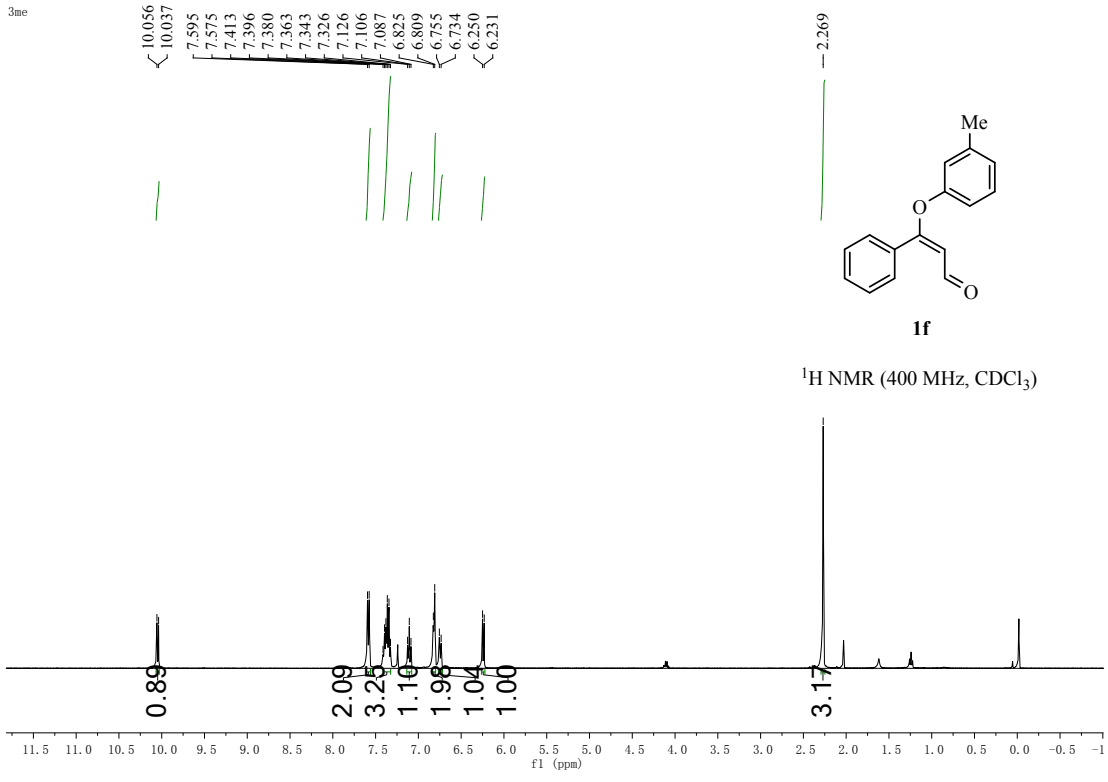
1e

^{1}H NMR (400 MHz, $CDCl_3$)



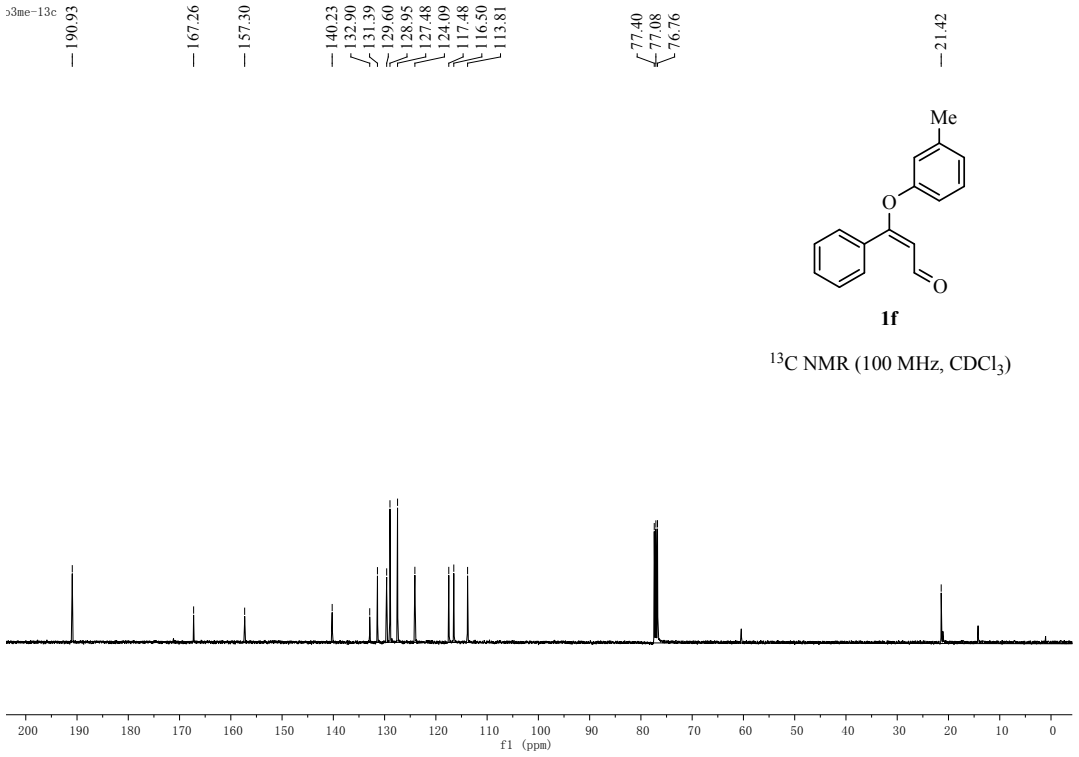
1e

^{13}C NMR (100 MHz, $CDCl_3$)



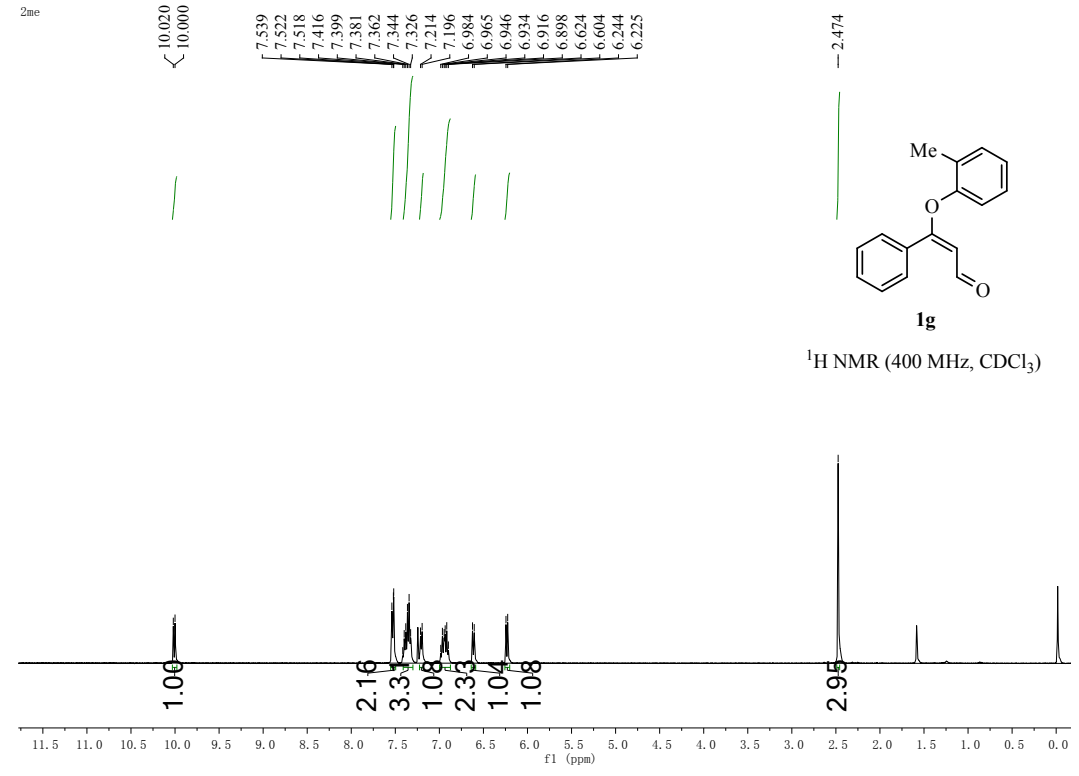
1f

^{1}H NMR (400 MHz, $CDCl_3$)



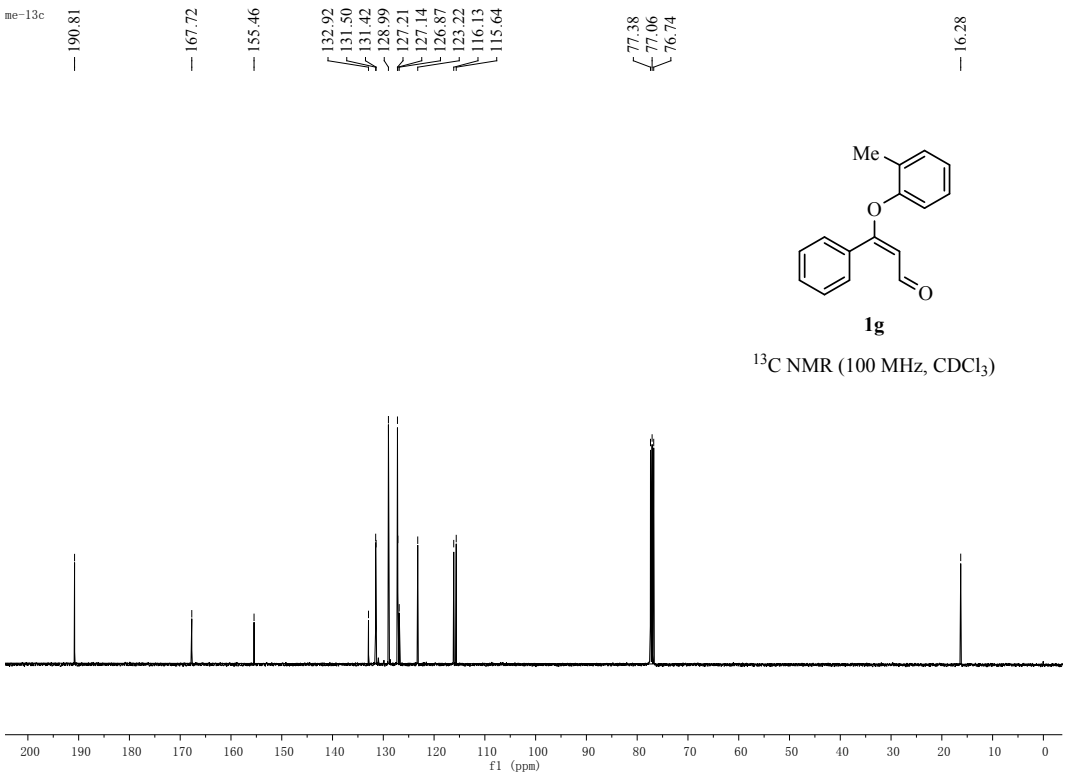
1f

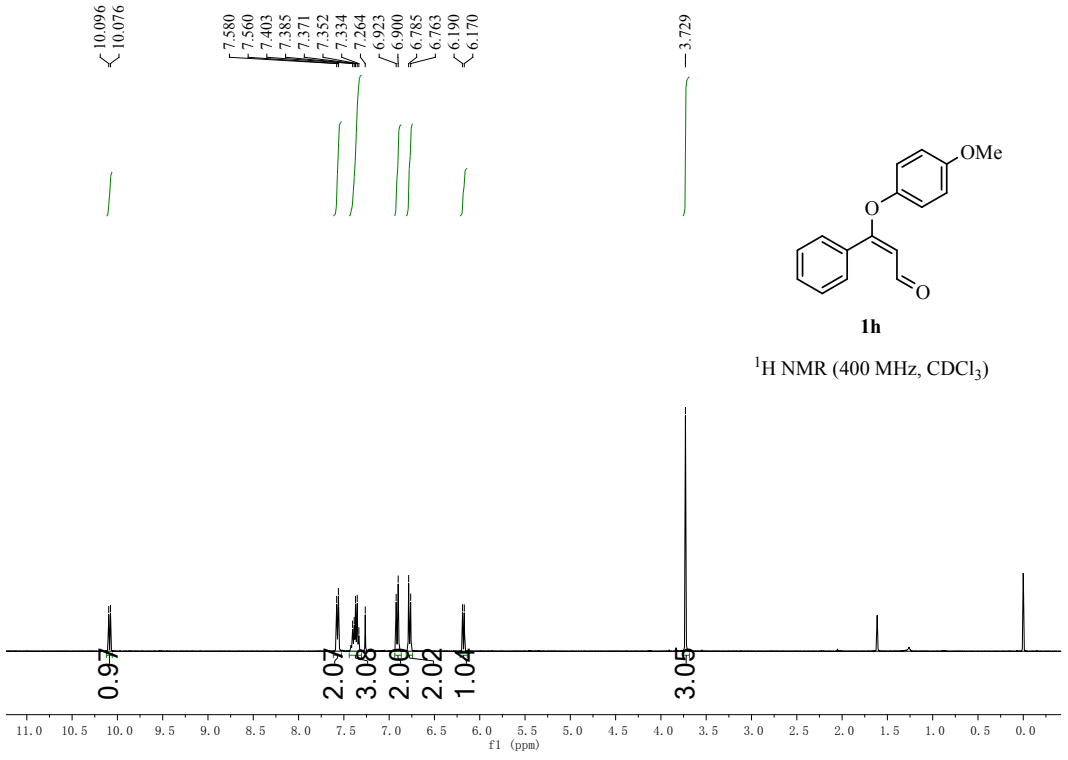
^{13}C NMR (100 MHz, $CDCl_3$)

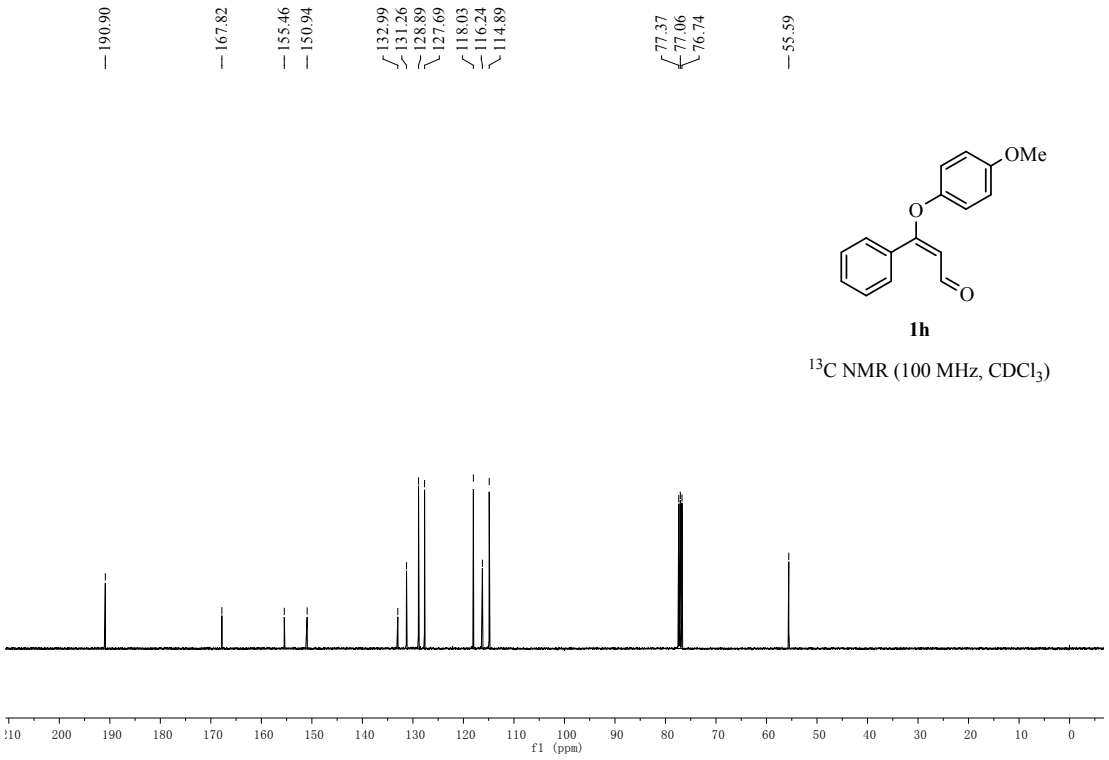


1g

^{1}H NMR (400 MHz, $CDCl_3$)

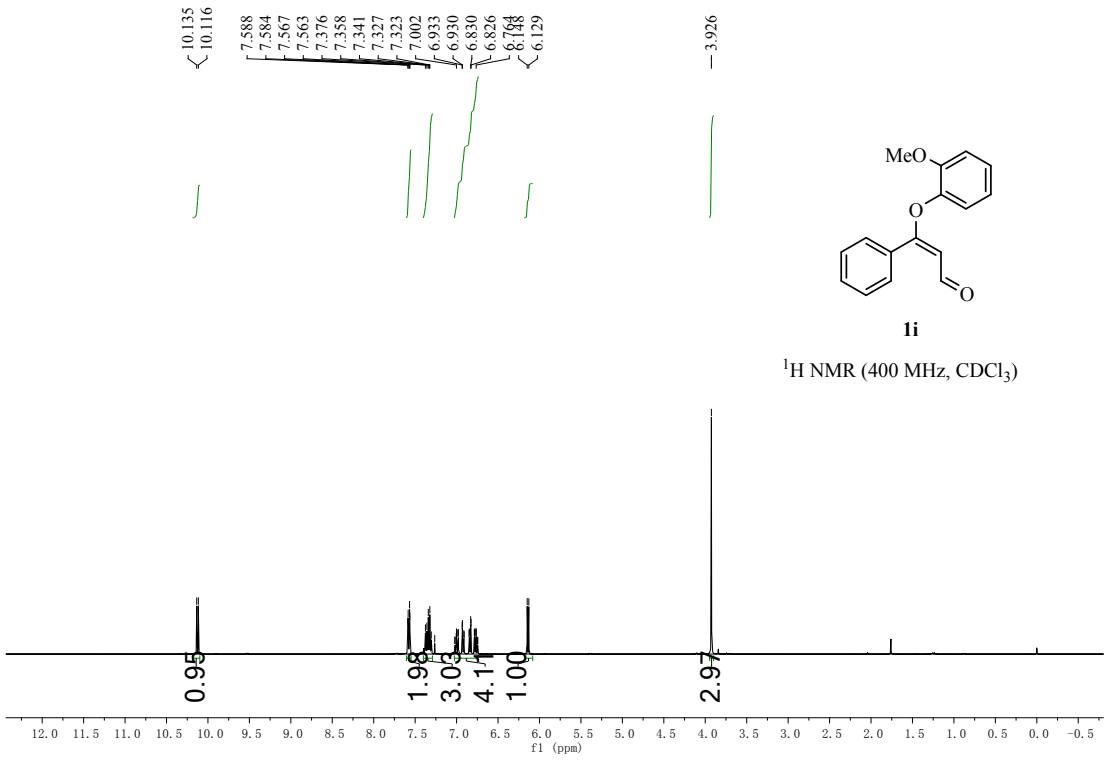

me-13c
190.81
167.72
155.46
132.92
131.50
131.42
128.99
127.21
127.14
126.87
123.22
116.13
115.64
77.38
77.06
76.74
16.28
Me
O
O
1g
^{13}C NMR (100 MHz, CDCl$_3$)
f1 (ppm)


10.096
10.076
7.580
7.560
7.403
7.385
7.371
7.352
7.334
7.264
6.923
6.900
6.785
6.763
6.190
6.170
3.729
OMe
O
O
1h
^{1}H NMR (400 MHz, CDCl$_3$)
0.97
2.07
3.08
2.00
2.02
1.04
3.05
f1 (ppm)



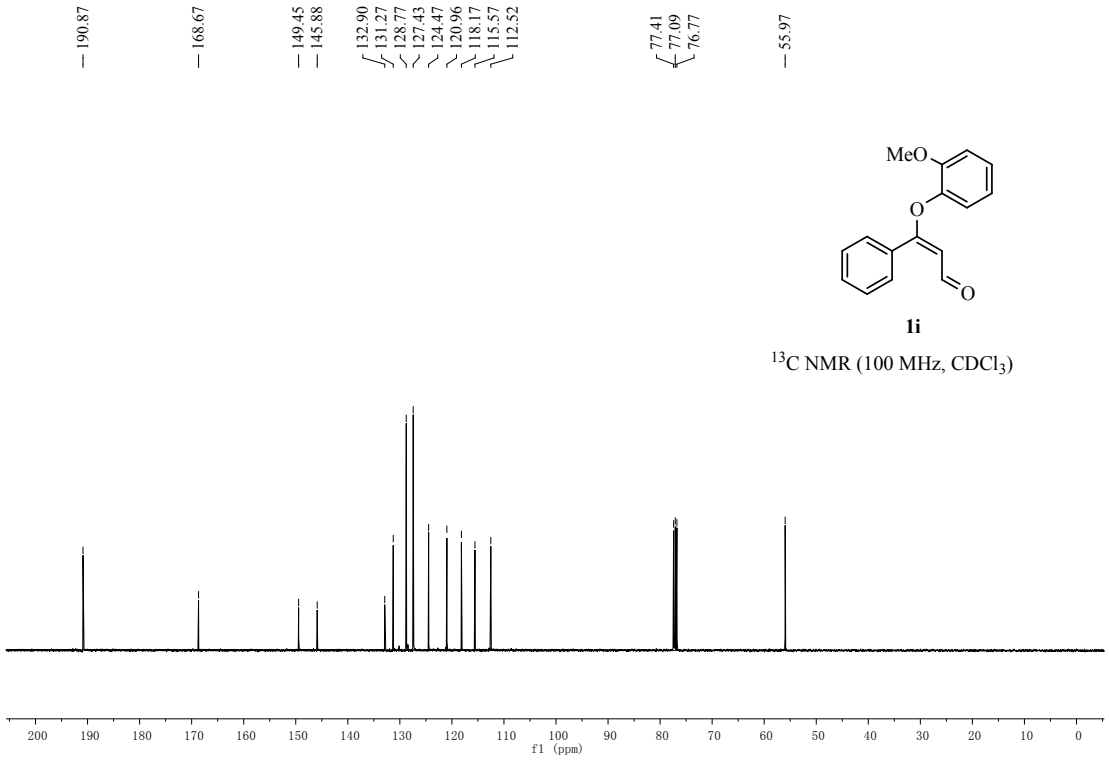
1h

^{13}C NMR (100 MHz, $CDCl_3$)



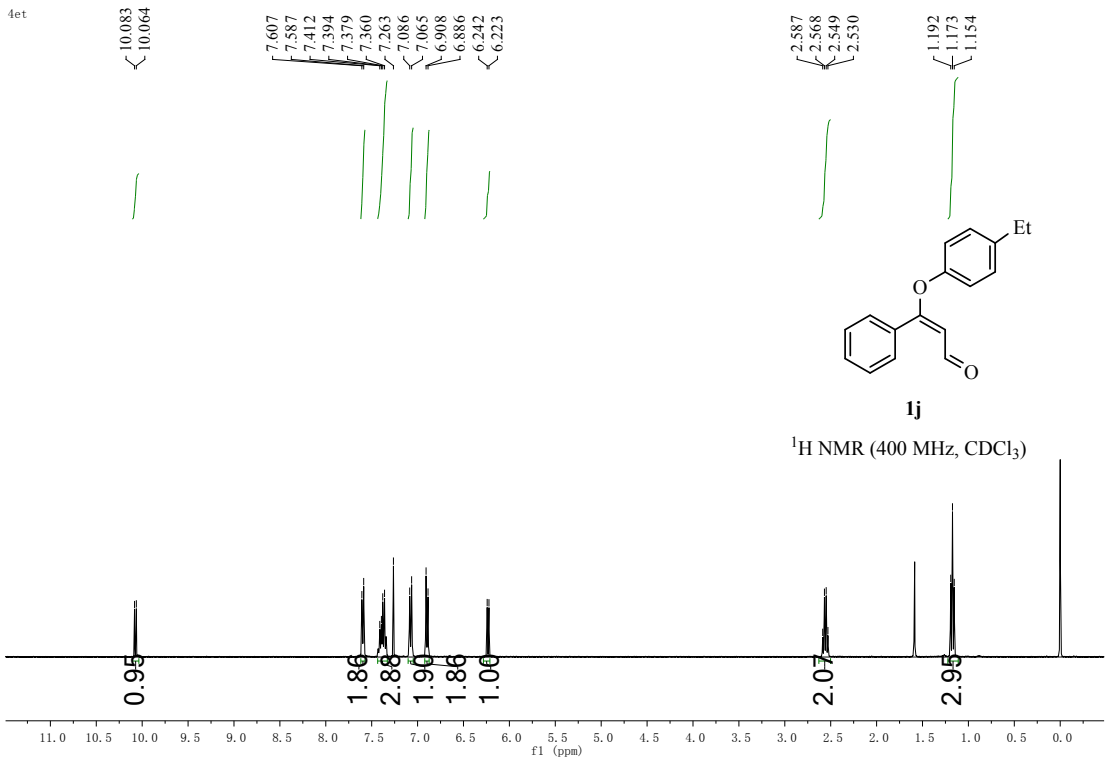
1i

^{1}H NMR (400 MHz, $CDCl_3$)



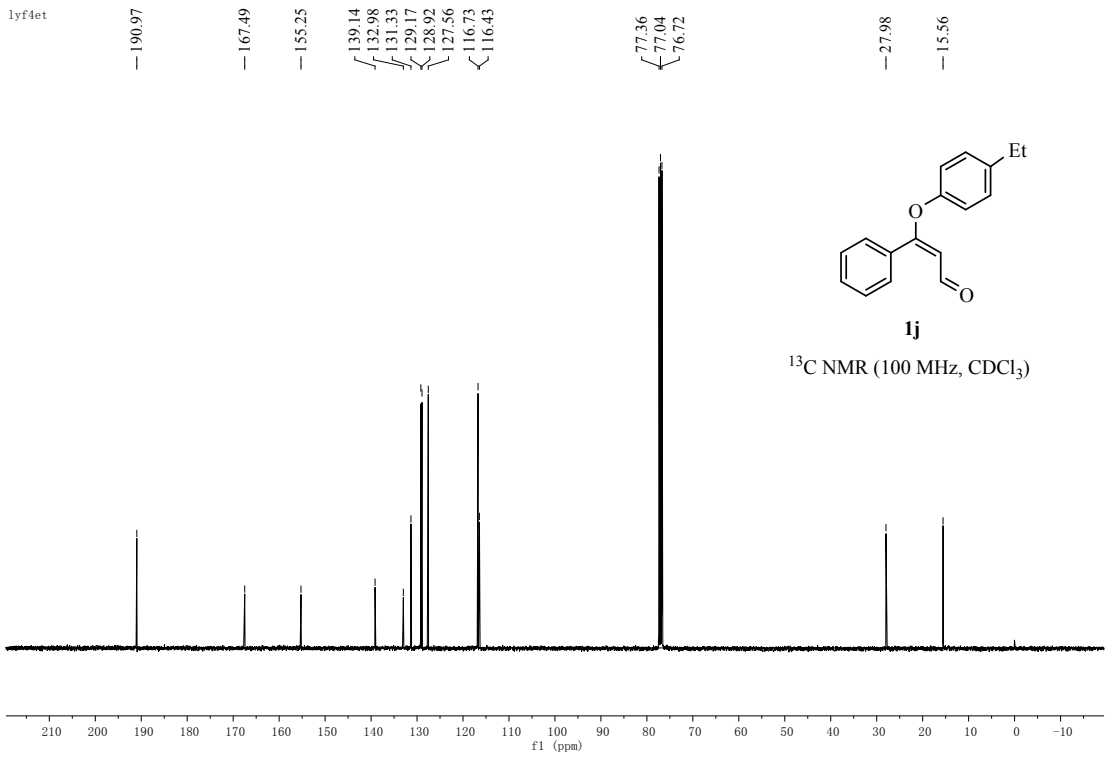
1i

^{13}C NMR (100 MHz, $CDCl_3$)



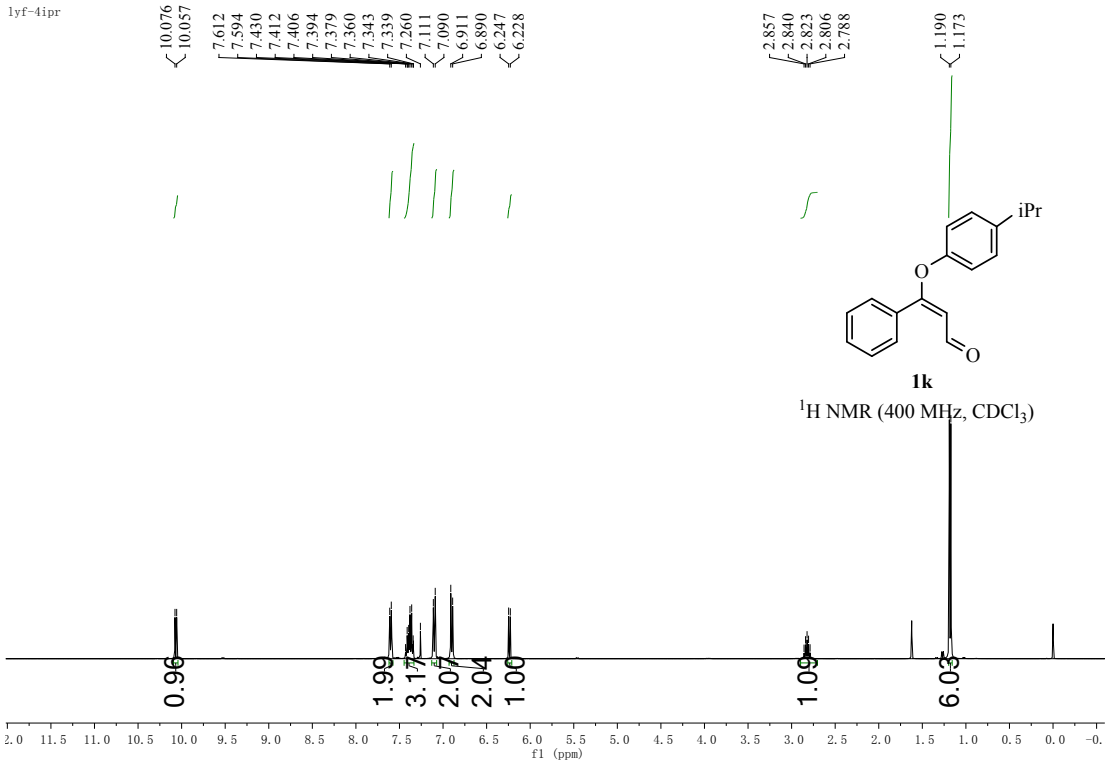
1j

^{1}H NMR (400 MHz, $CDCl_3$)



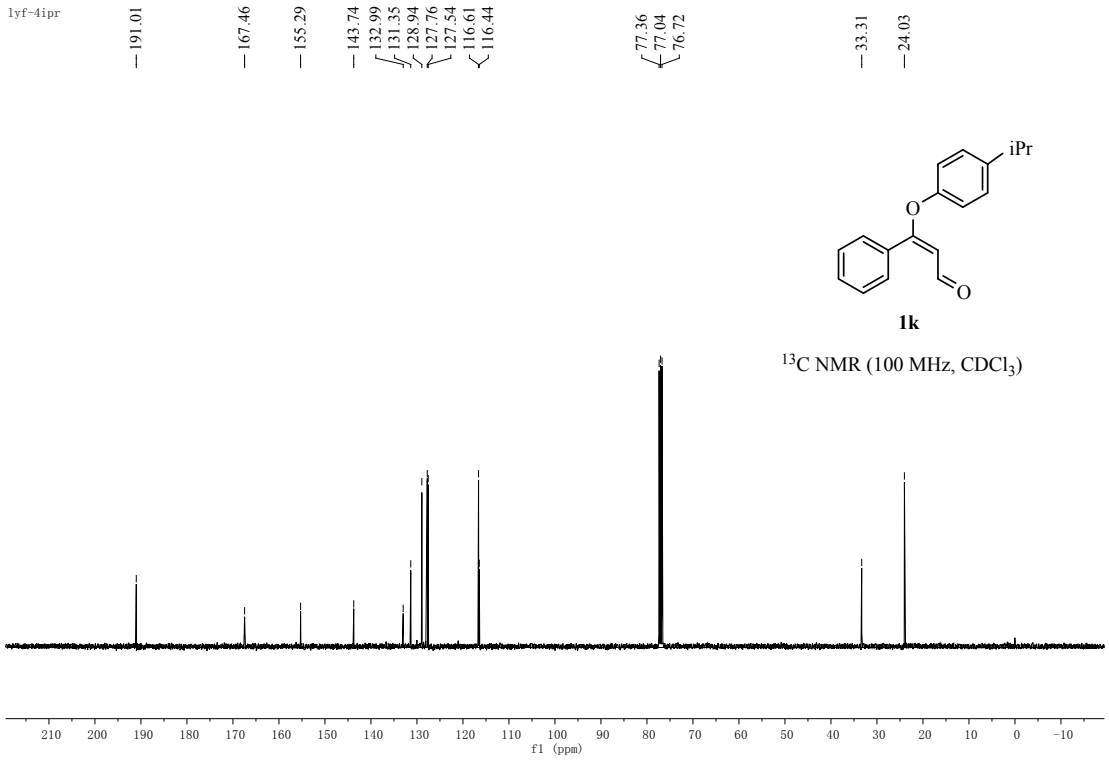
1j

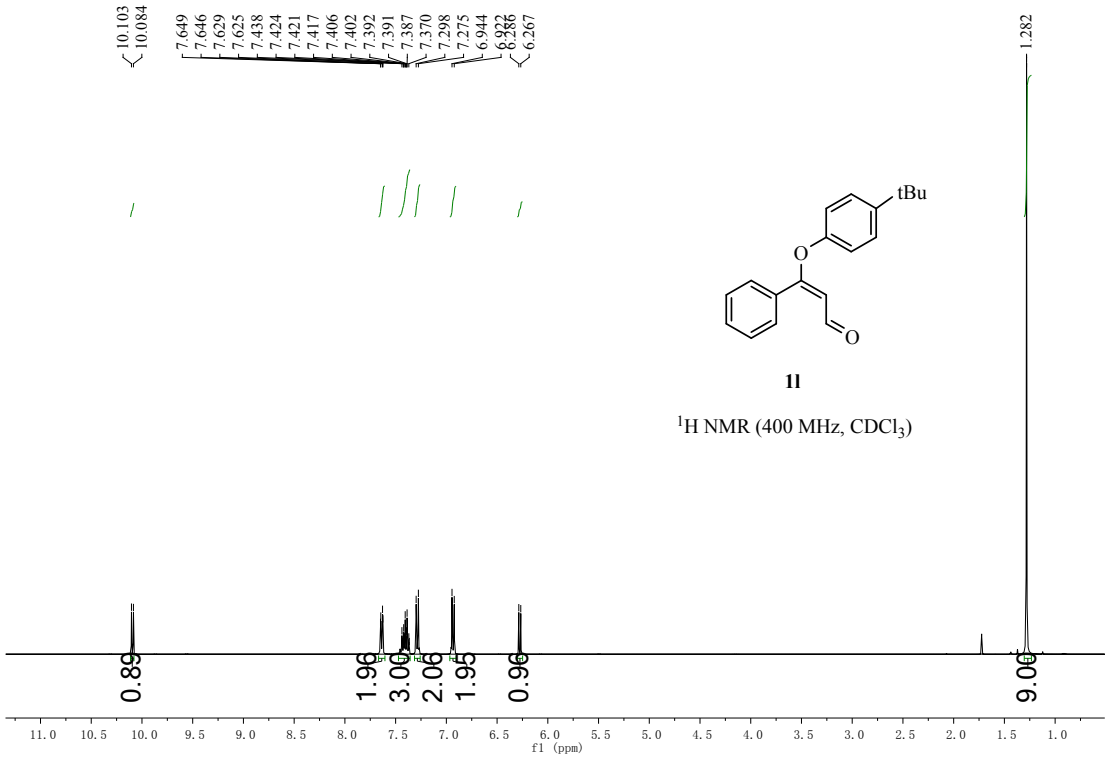
^{13}C NMR (100 MHz, $CDCl_3$)

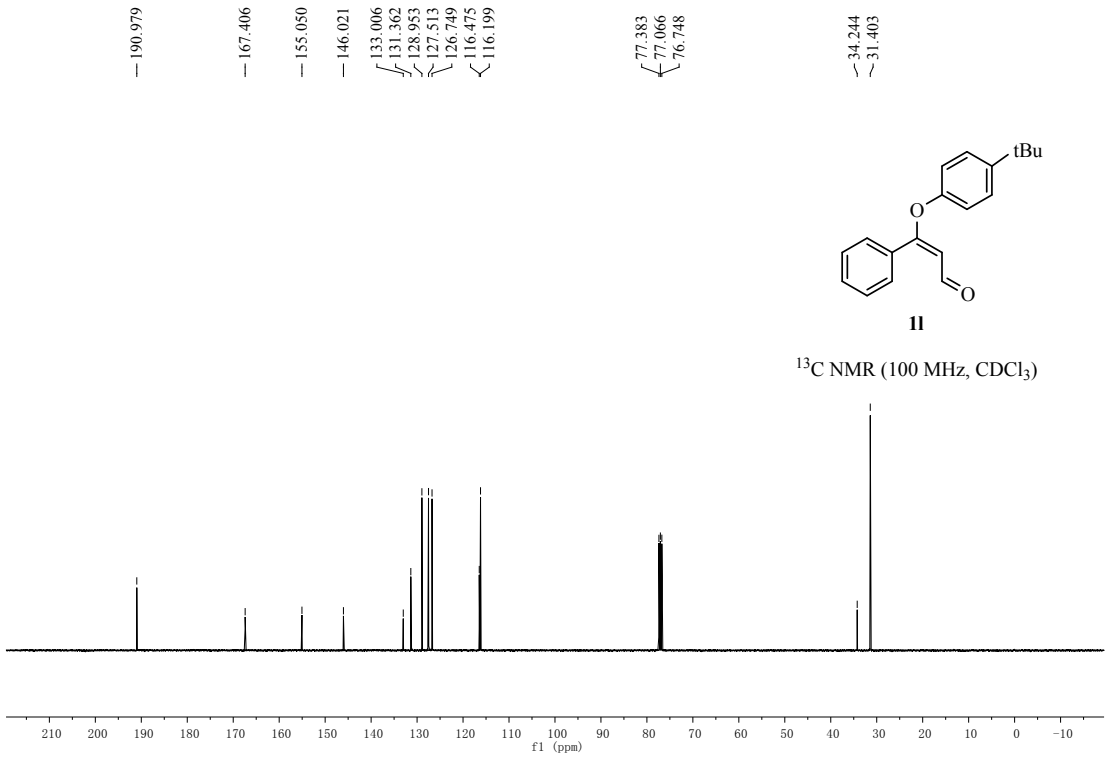


1k

^{1}H NMR (400 MHz, $CDCl_3$)

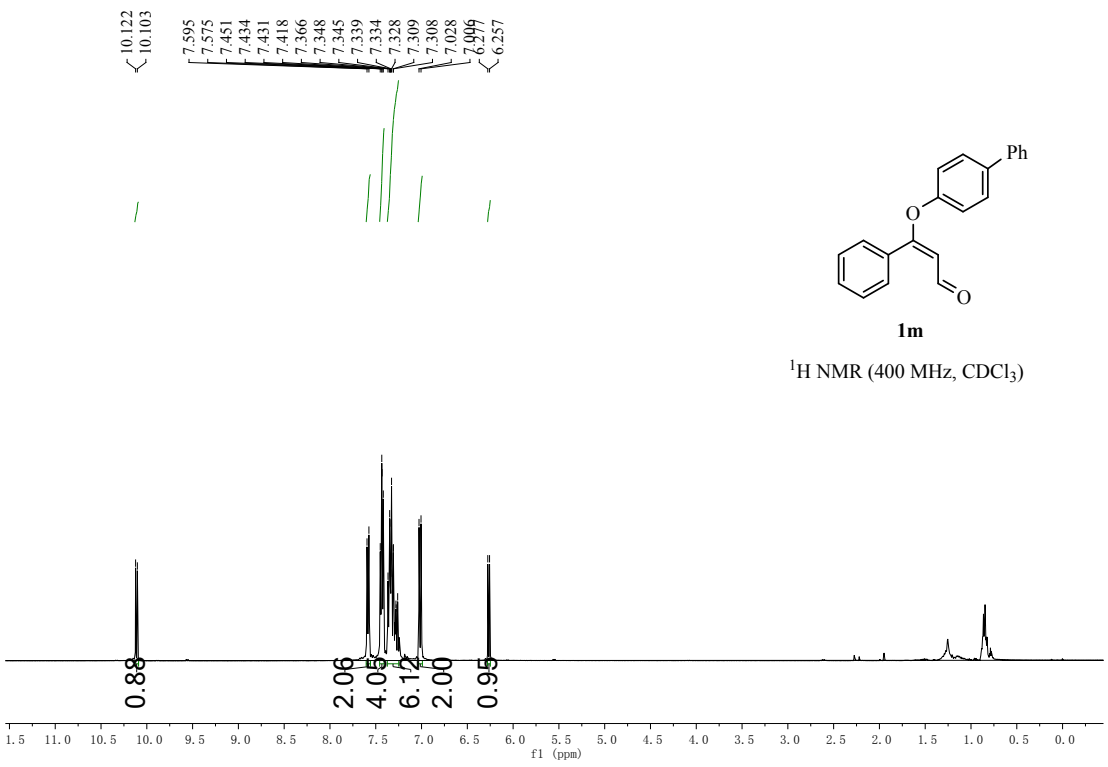

lyf-4ipr
191.01
167.46
155.29
143.74
132.99
131.35
128.94
127.76
127.54
116.61
116.44
77.36
77.04
76.72
33.31
24.03
iPr
O
O
1k
13C NMR (100 MHz, CDCl3)
f1 (ppm)


10.103
10.084
7.649
7.646
7.629
7.625
7.438
7.424
7.421
7.417
7.406
7.402
7.392
7.391
7.387
7.370
7.298
7.275
6.944
6.922
6.286
6.267
1.282
tBu
O
O
1l
1H NMR (400 MHz, CDCl3)
0.89
1.96
3.00
2.06
1.95
0.96
9.00
f1 (ppm)



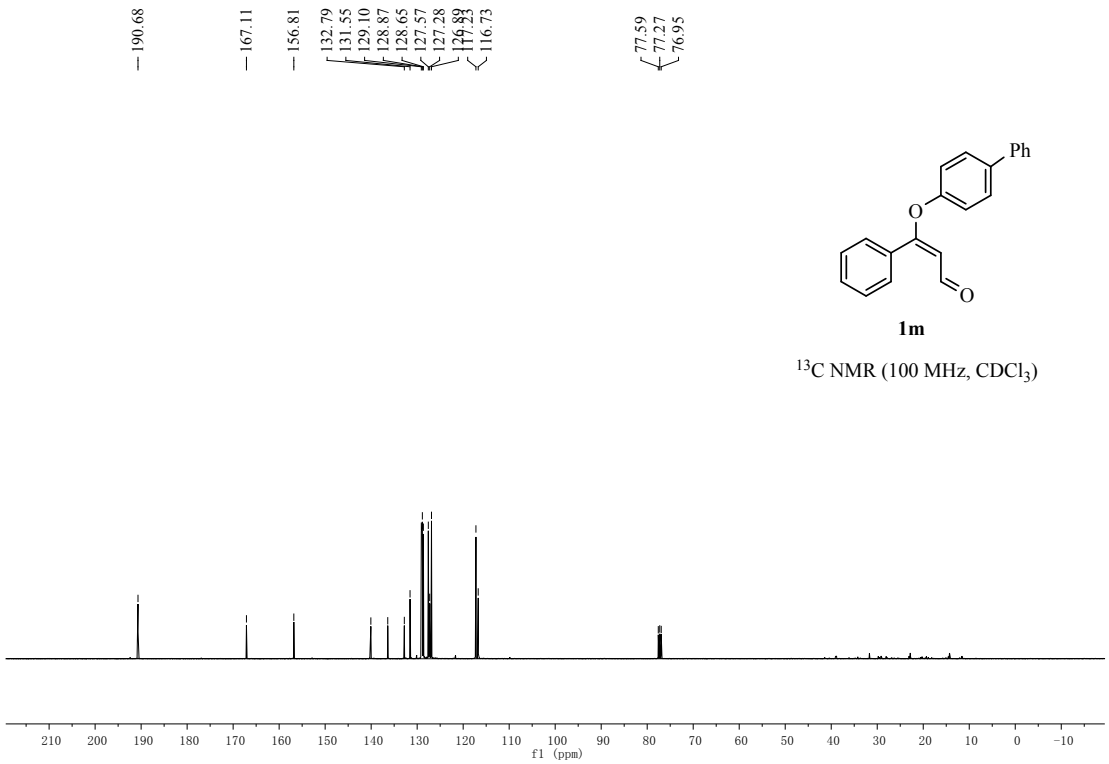
1l

^{13}C NMR (100 MHz, $CDCl_3$)



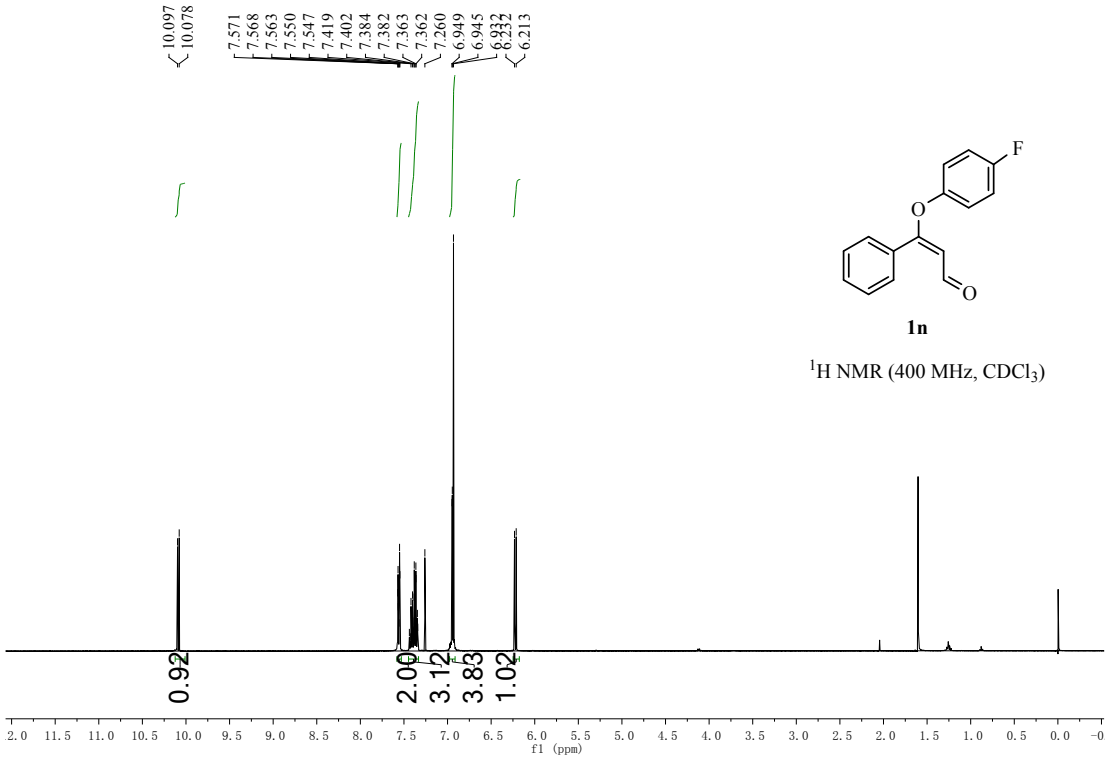
1m

^{1}H NMR (400 MHz, $CDCl_3$)



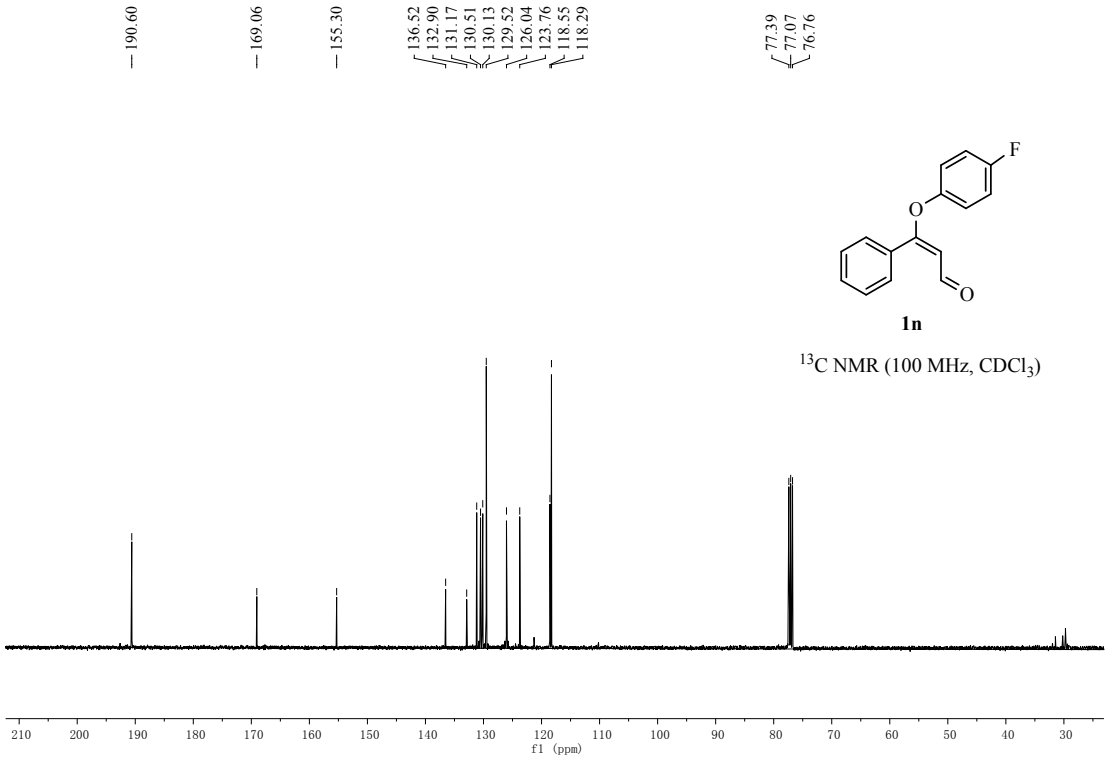
1m

^{13}C NMR (100 MHz, $CDCl_3$)



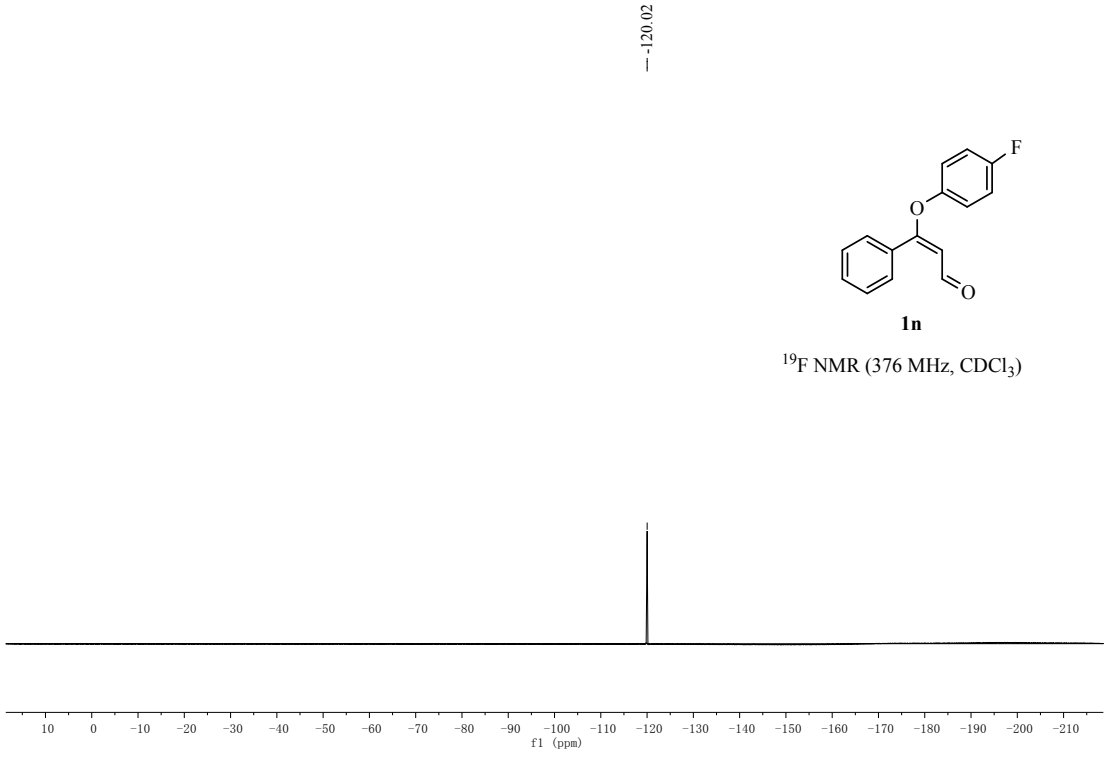
1n

^{1}H NMR (400 MHz, $CDCl_3$)



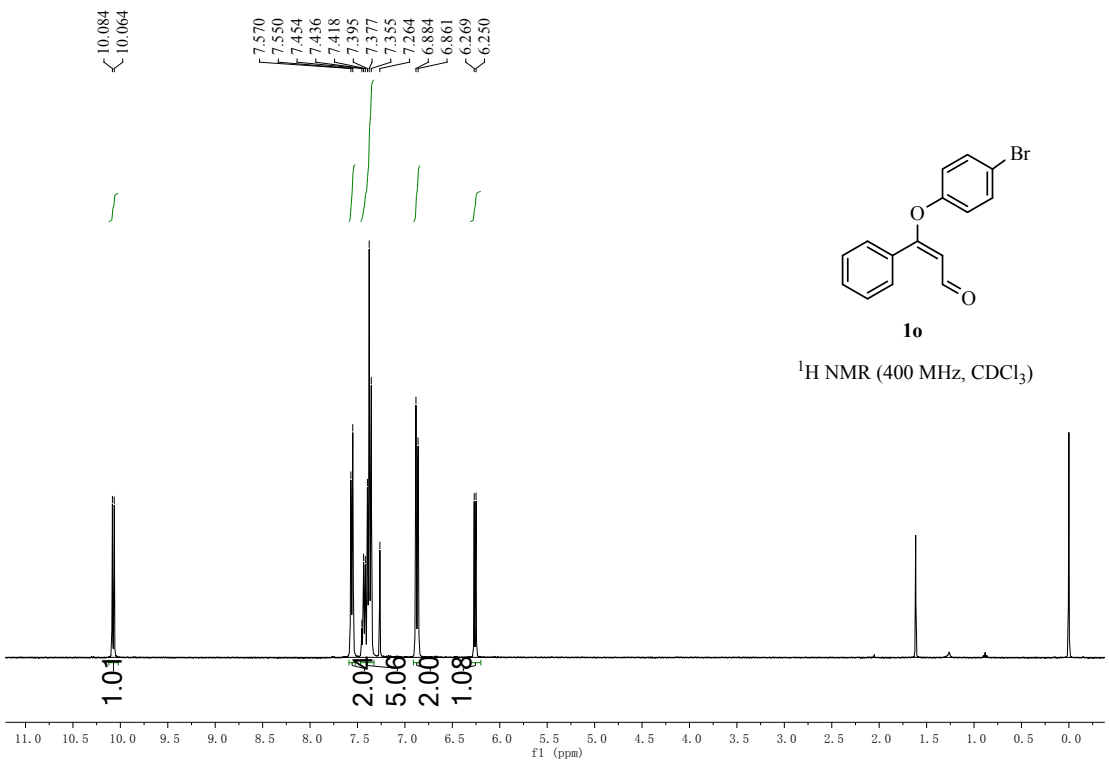
1n

^{13}C NMR (100 MHz, $CDCl_3$)



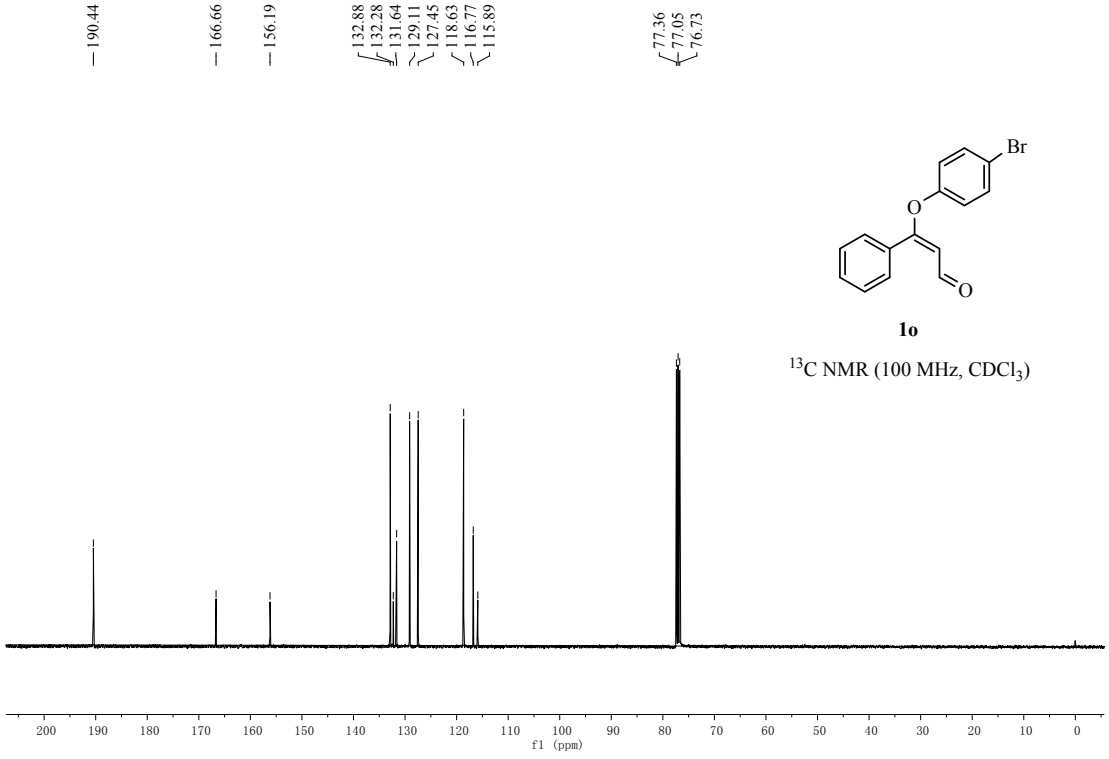
1n

^{19}F NMR (376 MHz, $CDCl_3$)



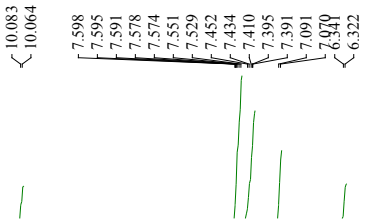
1o

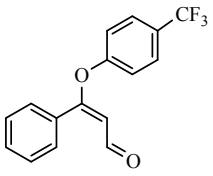
^{1}H NMR (400 MHz, $CDCl_3$)



1o

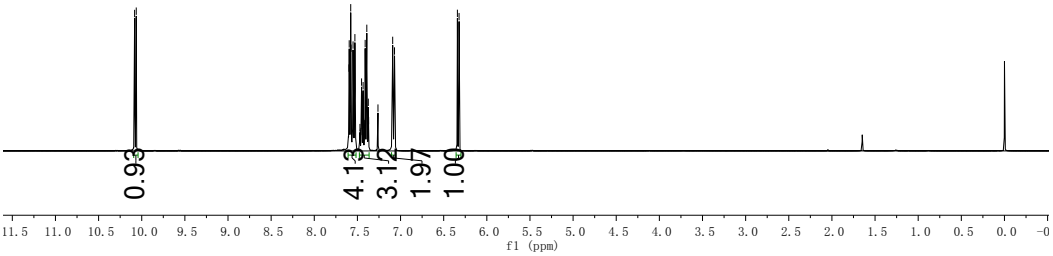
^{13}C NMR (100 MHz, $CDCl_3$)

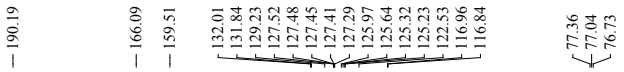


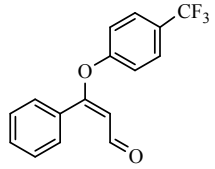


1p

^{1}H NMR (400 MHz, $CDCl_3$)

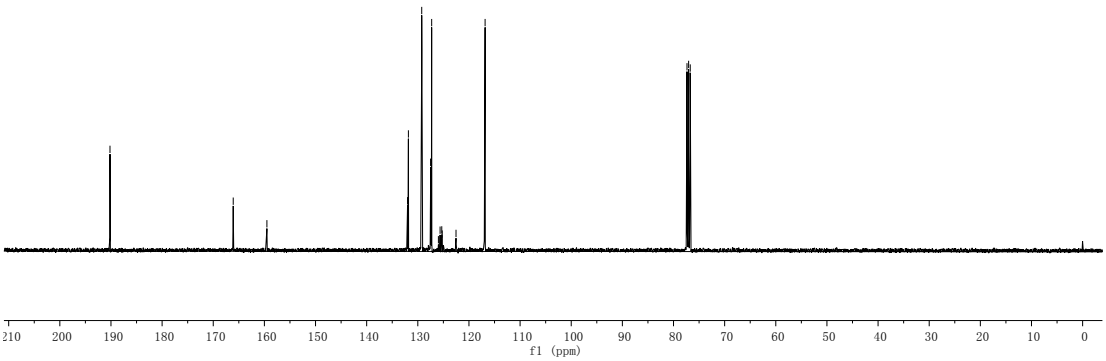


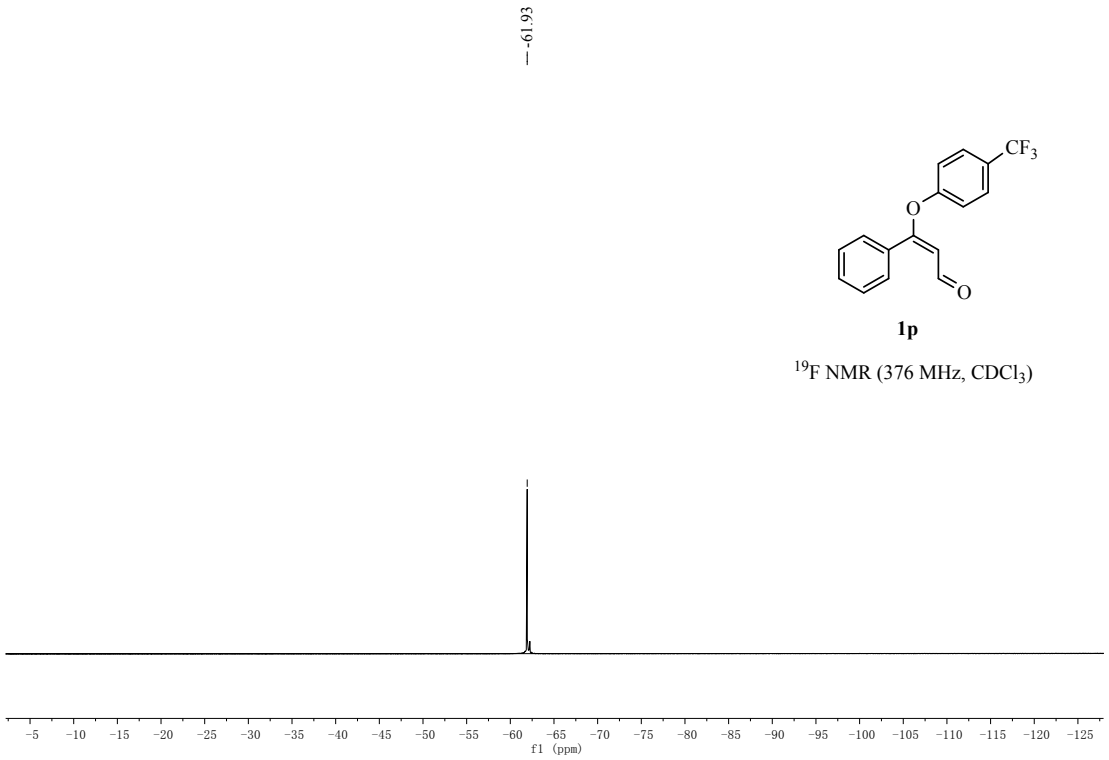




1p

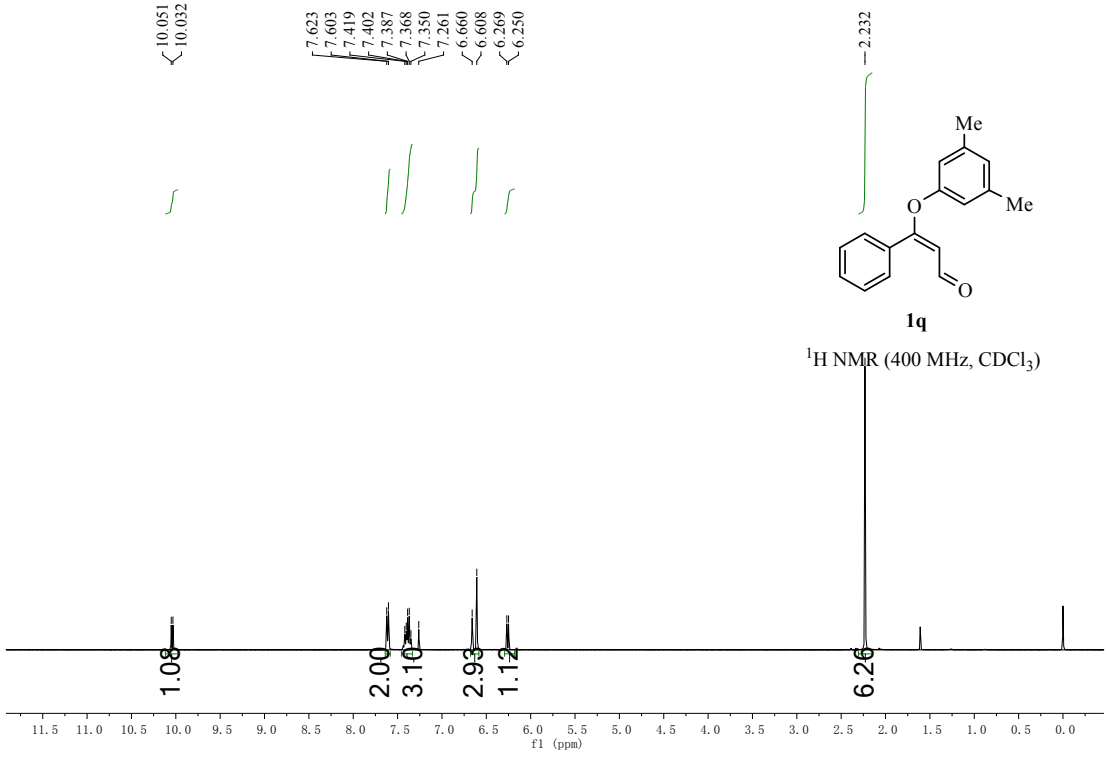
^{13}C NMR (100 MHz, $CDCl_3$)





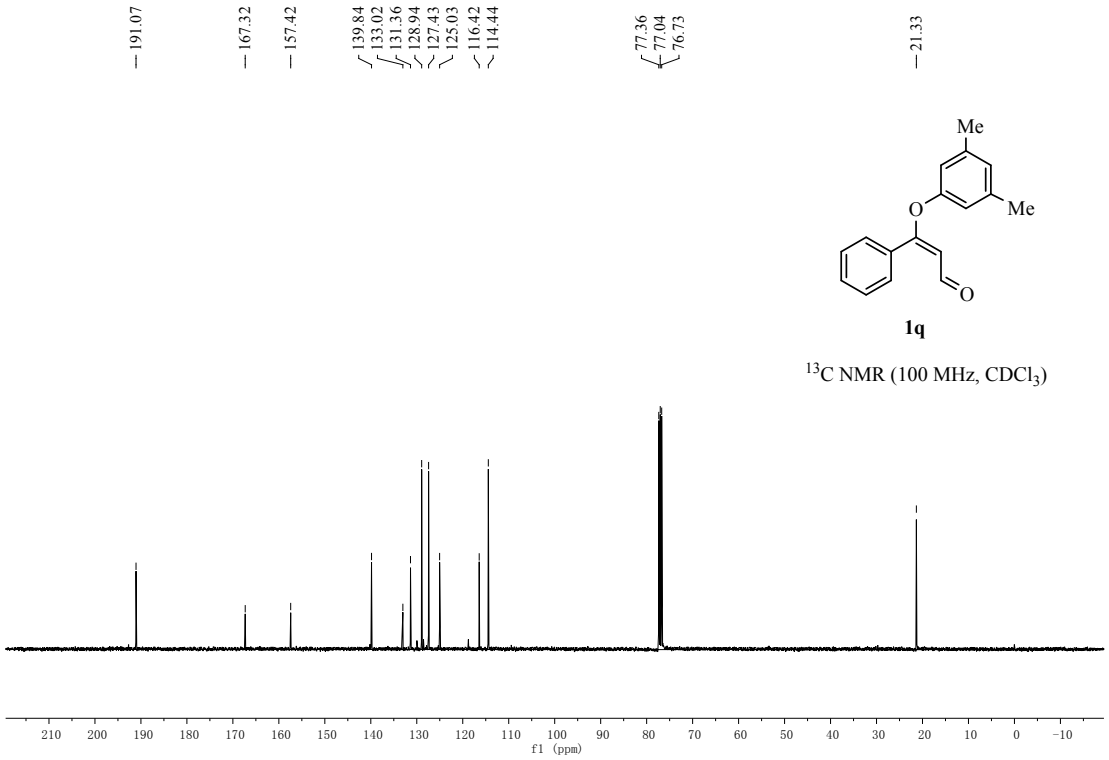
1p

^{19}F NMR (376 MHz, $CDCl_3$)



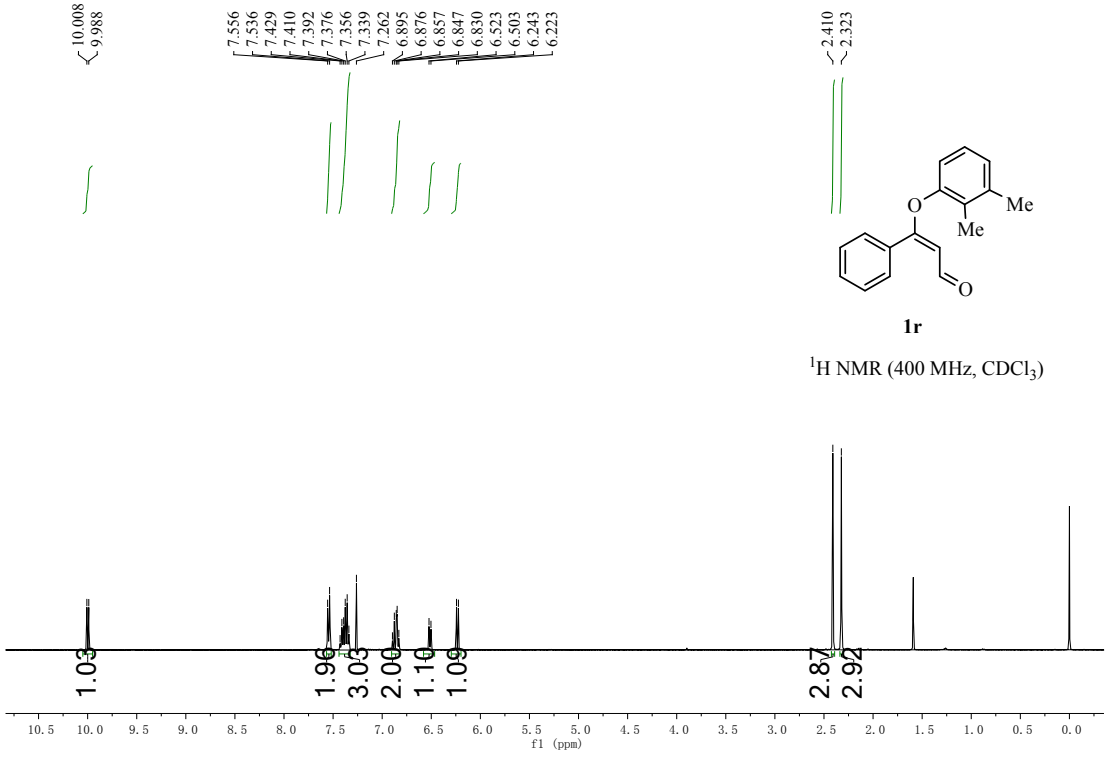
1q

^{1}H NMR (400 MHz, $CDCl_3$)



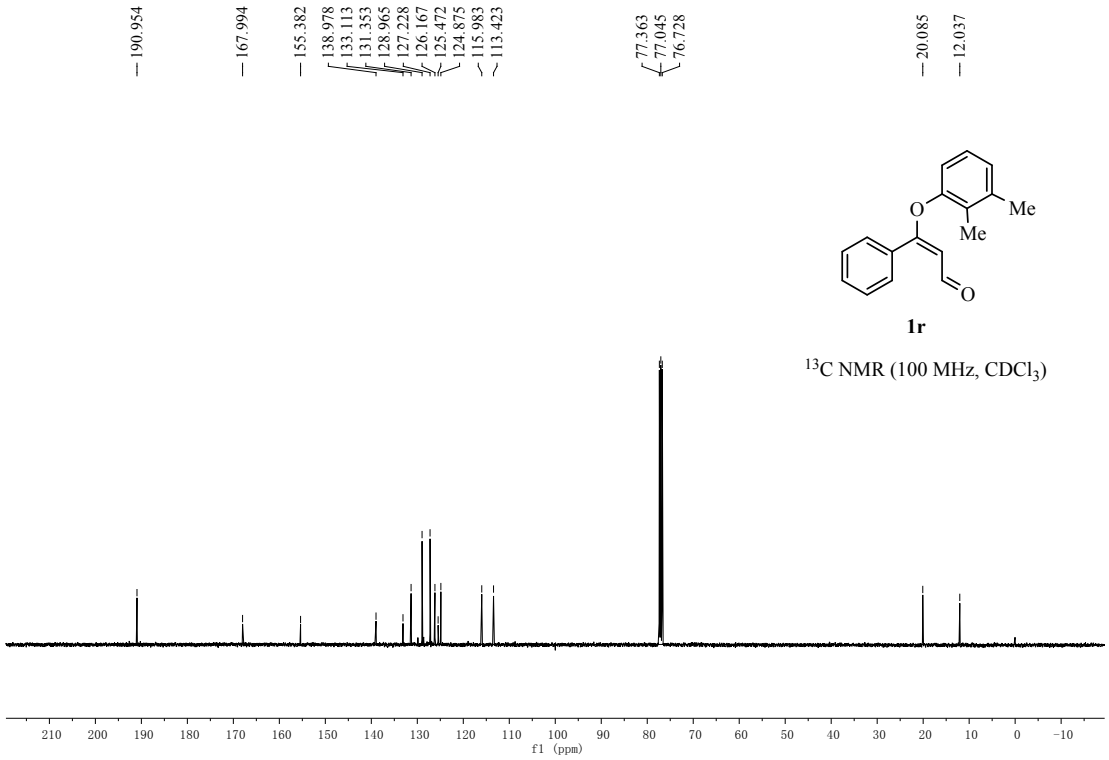
1q

^{13}C NMR (100 MHz, $CDCl_3$)



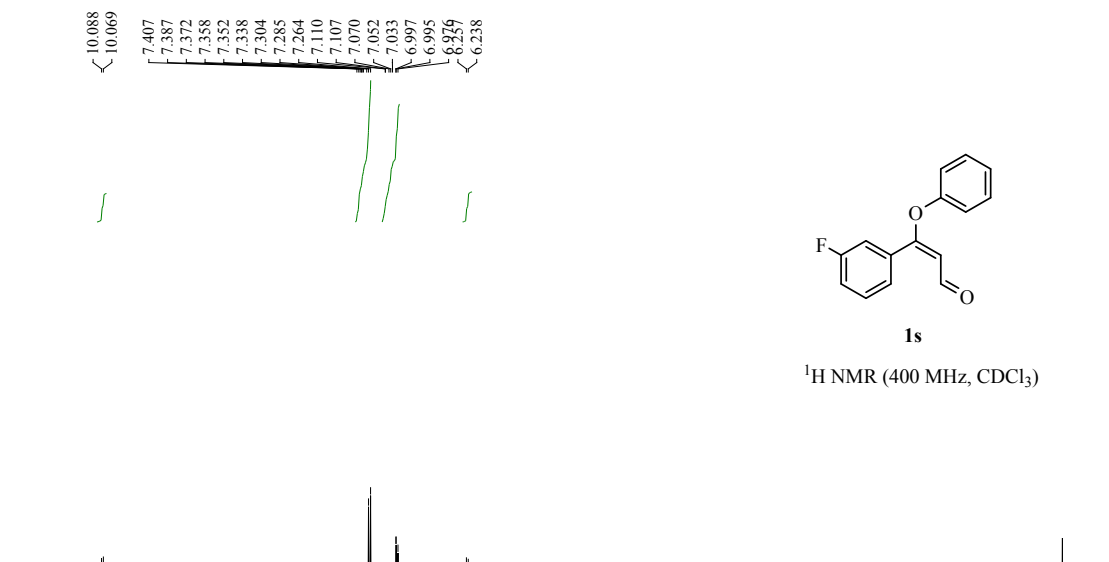
1r

^{1}H NMR (400 MHz, $CDCl_3$)



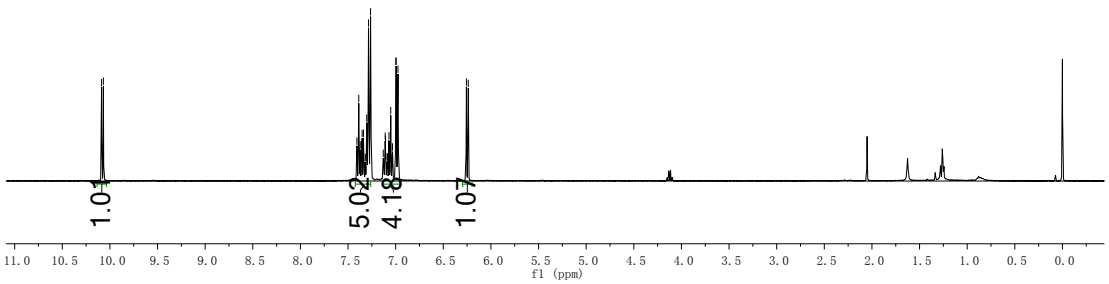
1r

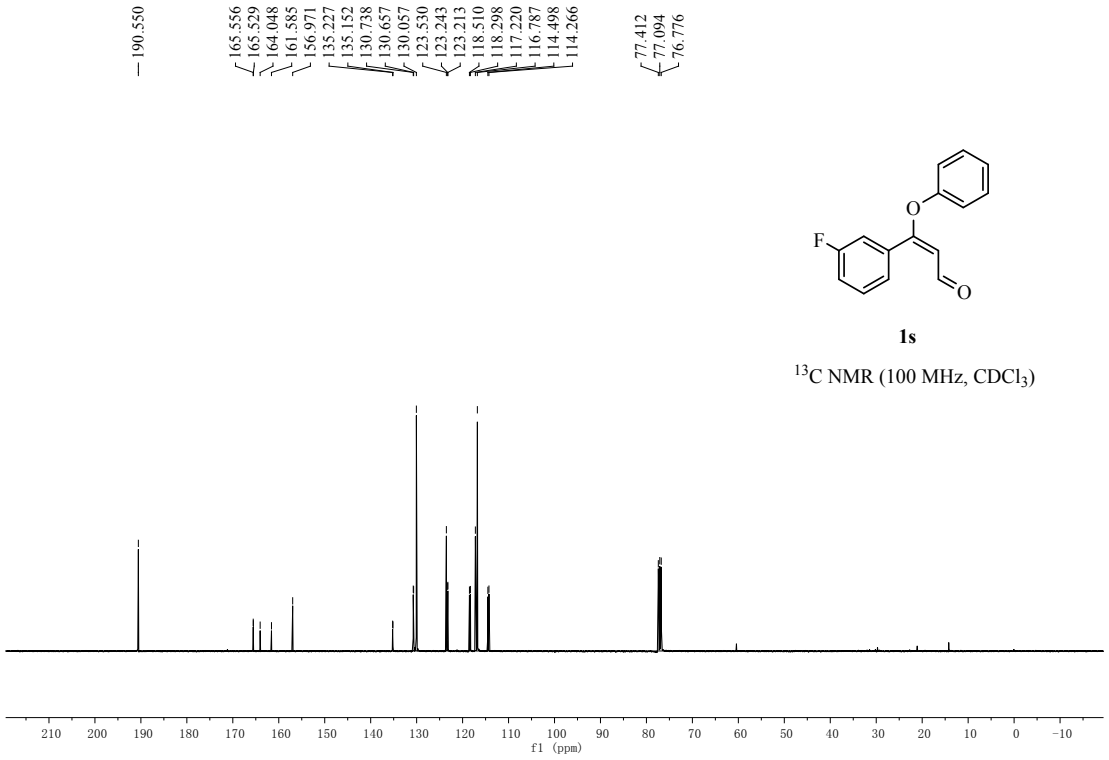
^{13}C NMR (100 MHz, $CDCl_3$)



1s

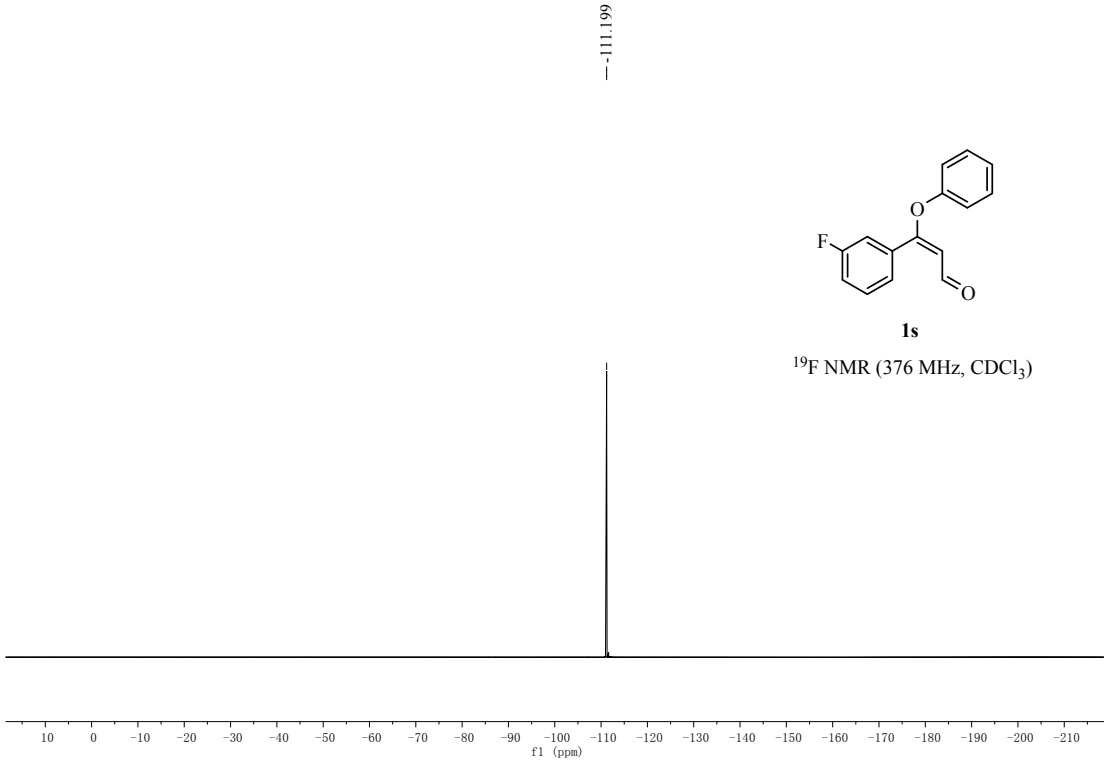
^{1}H NMR (400 MHz, $CDCl_3$)





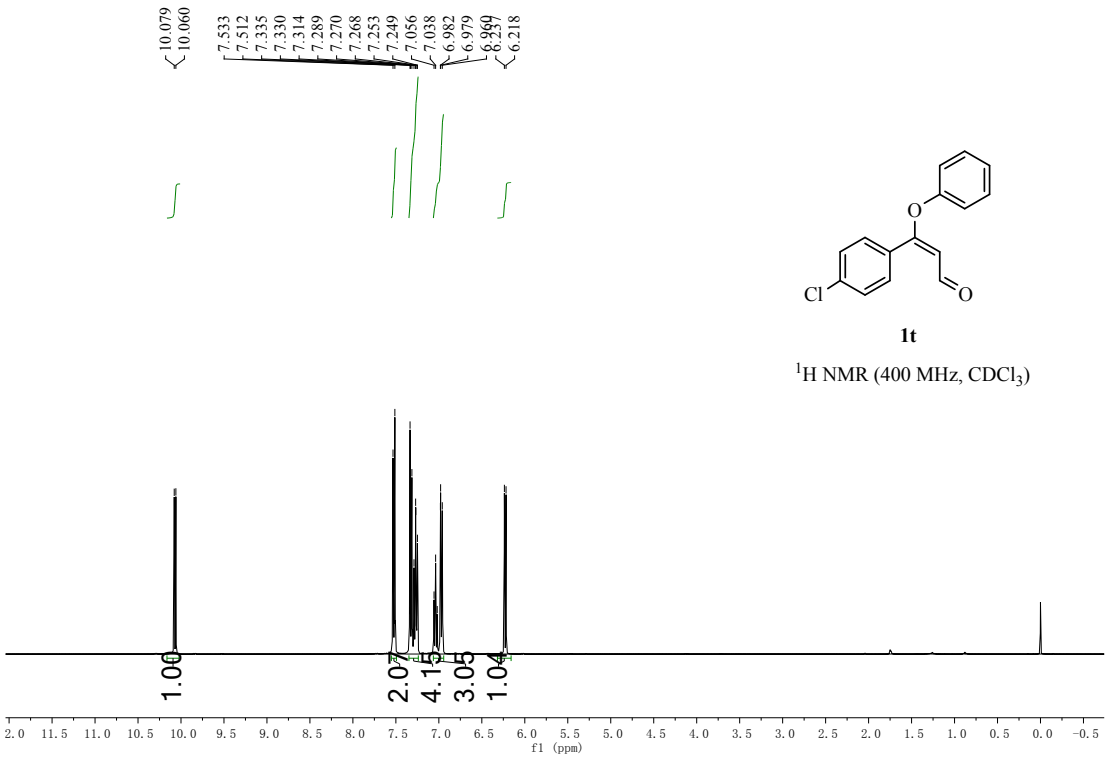
1s

^{13}C NMR (100 MHz, $CDCl_3$)



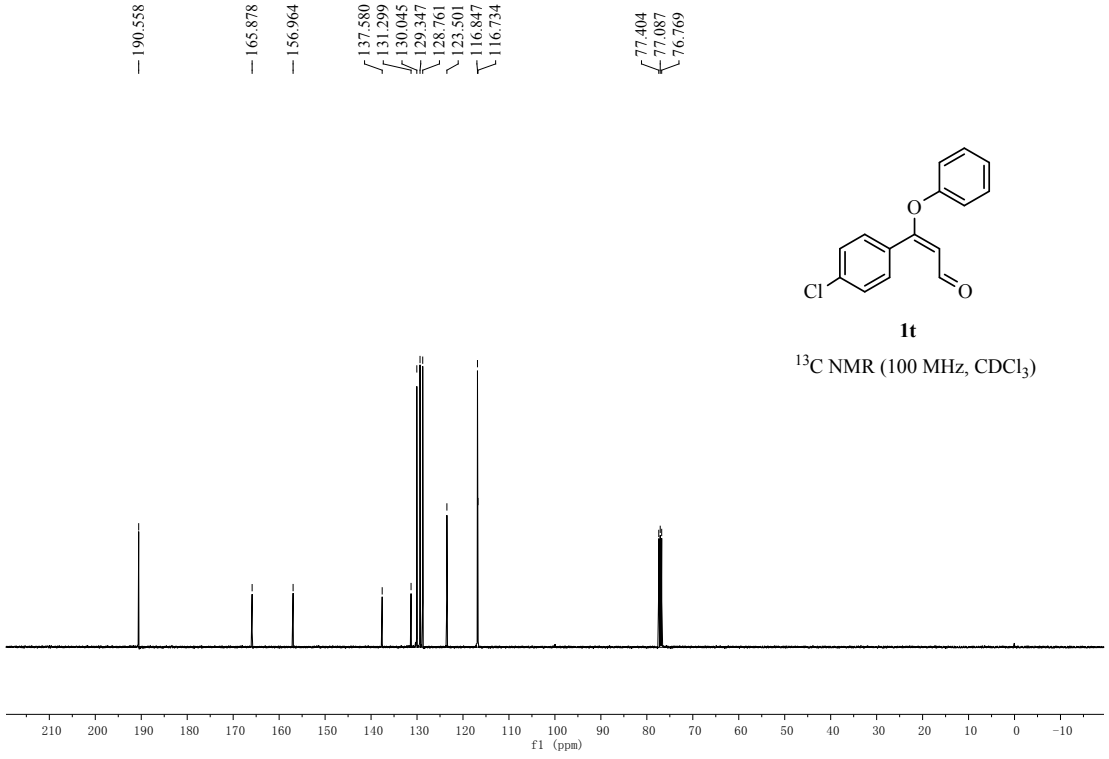
1s

^{19}F NMR (376 MHz, $CDCl_3$)



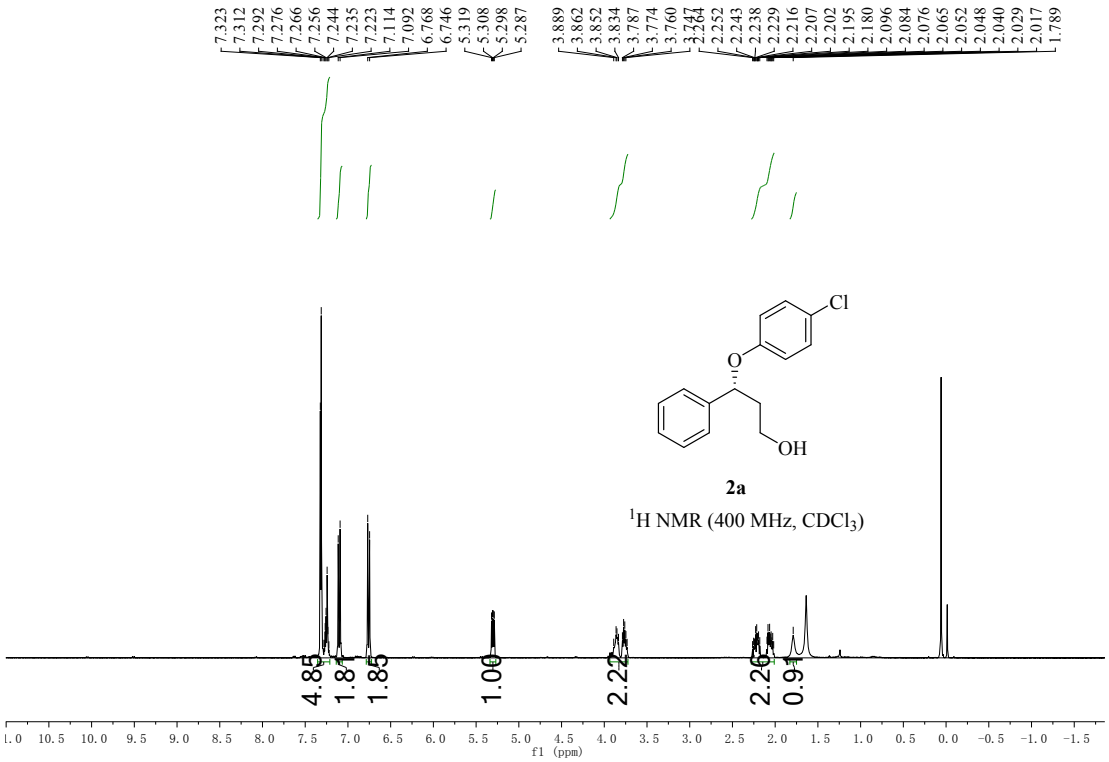
1t

^{1}H NMR (400 MHz, $CDCl_3$)



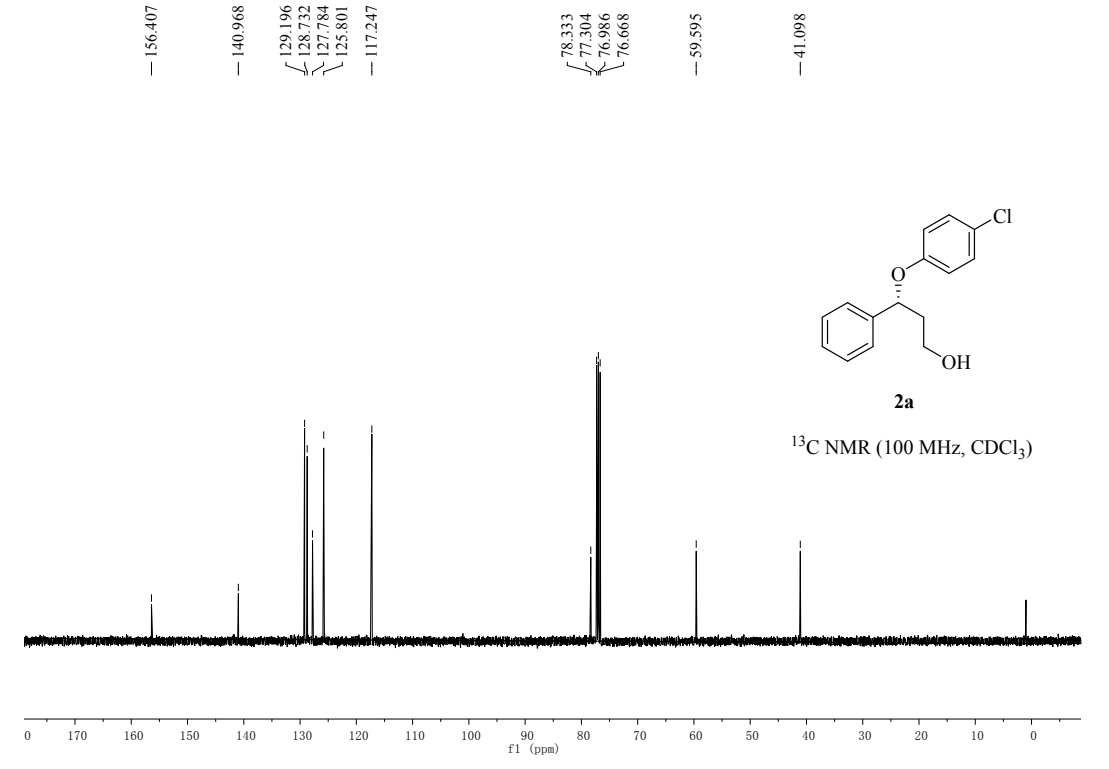
1t

^{13}C NMR (100 MHz, $CDCl_3$)



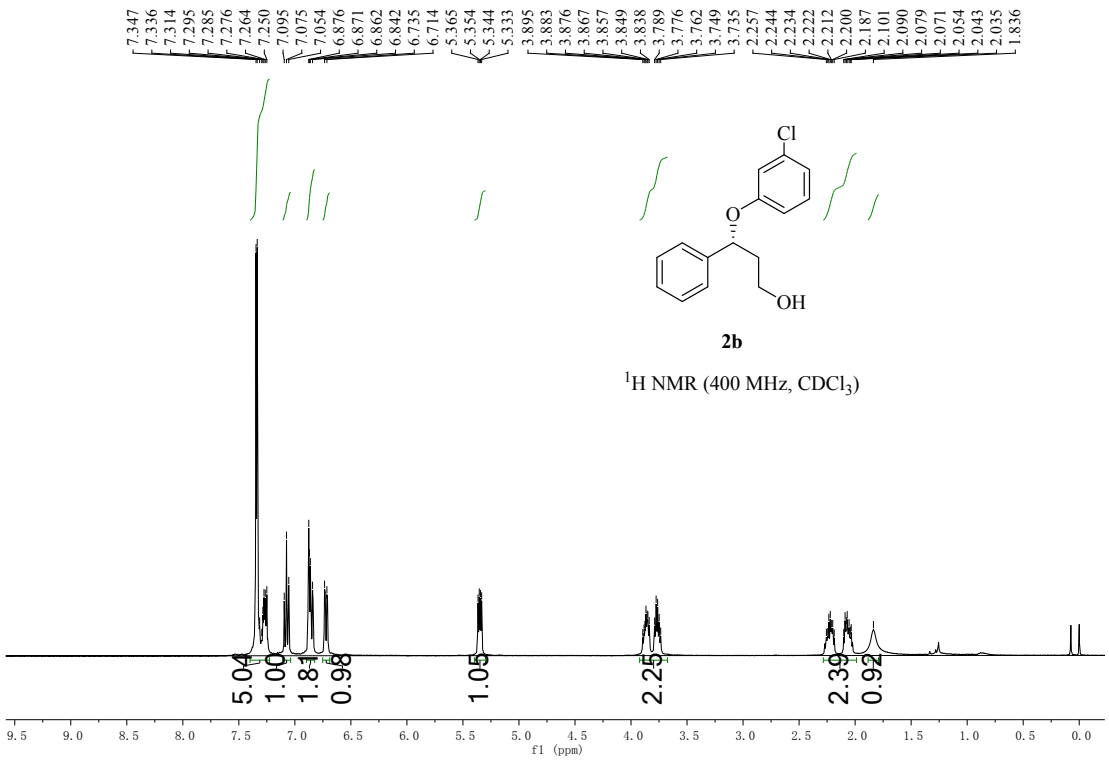
2a

^{1}H NMR (400 MHz, $CDCl_3$)



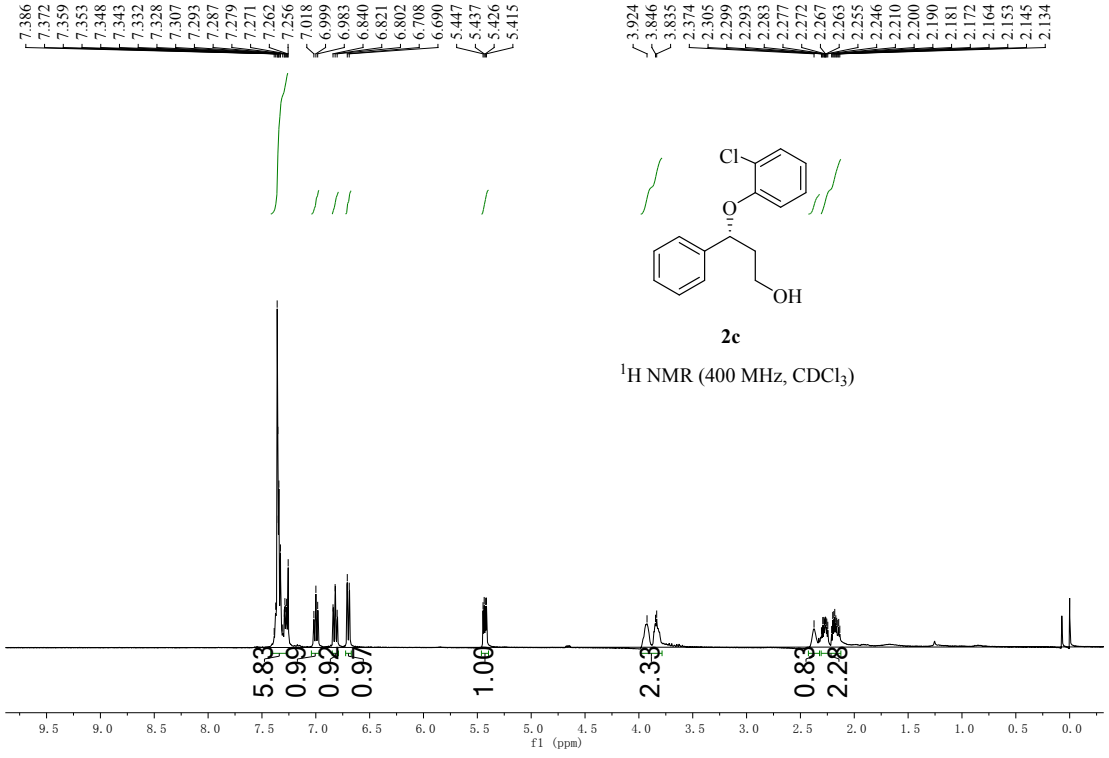
2a

^{13}C NMR (100 MHz, $CDCl_3$)



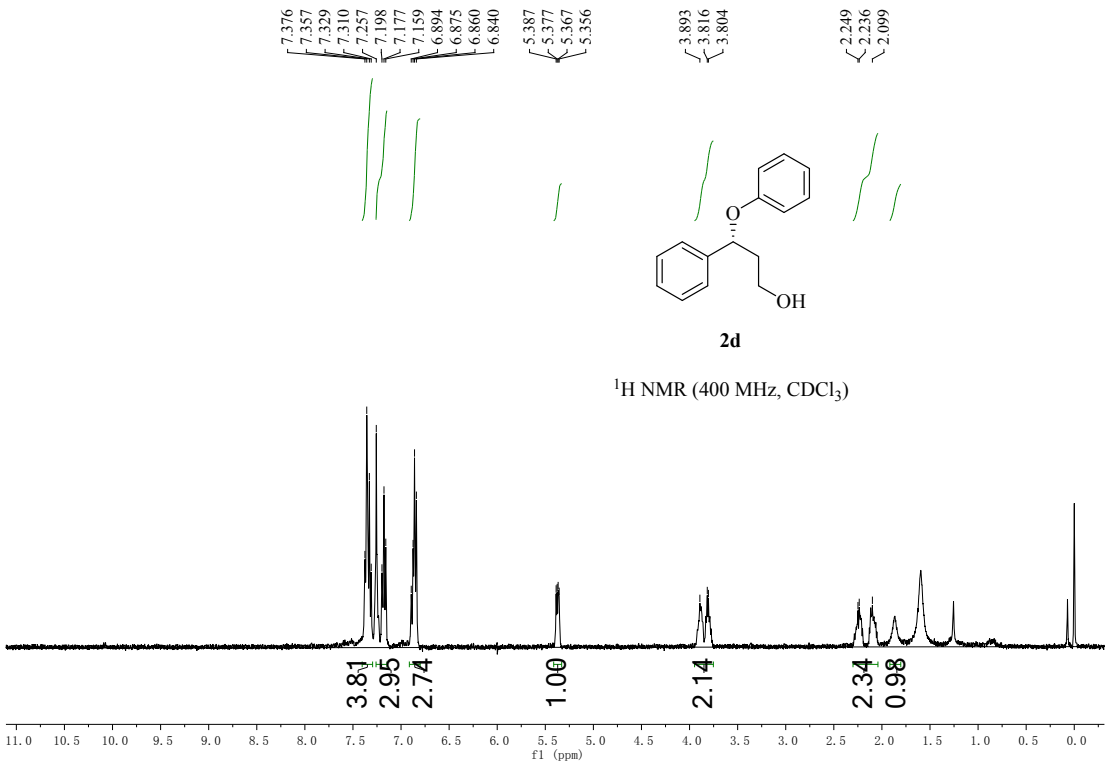
2b

^{1}H NMR (400 MHz, $CDCl_3$)



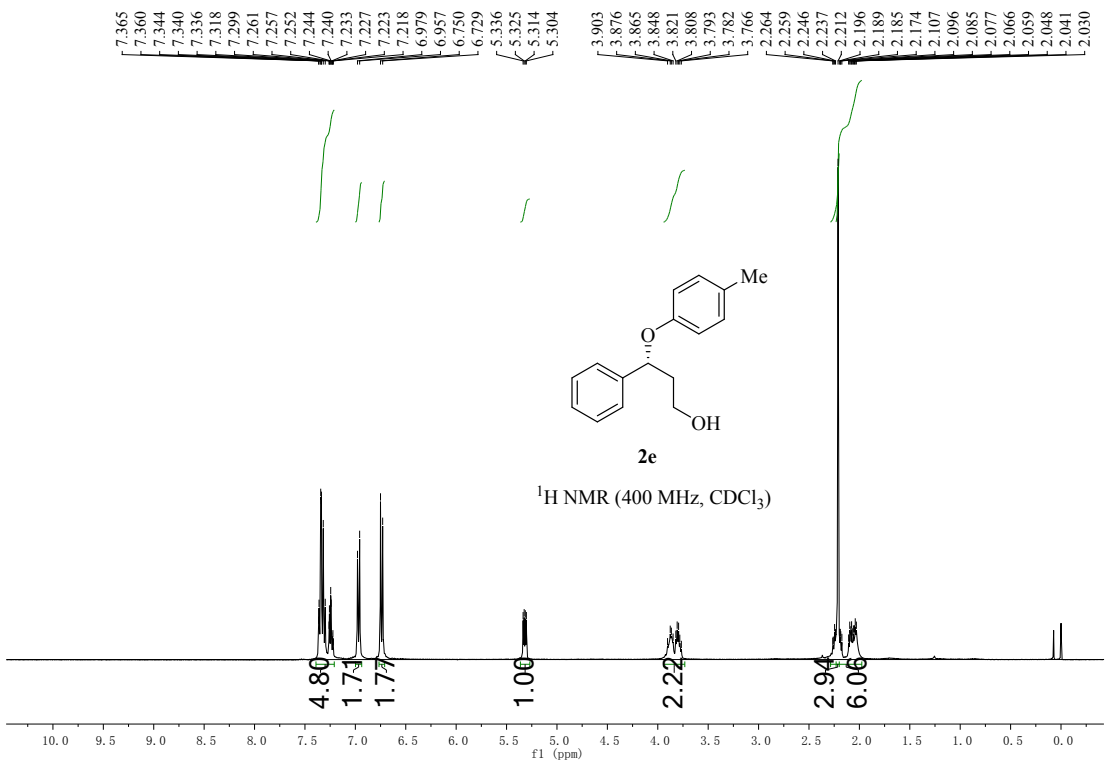
2c

^{1}H NMR (400 MHz, $CDCl_3$)



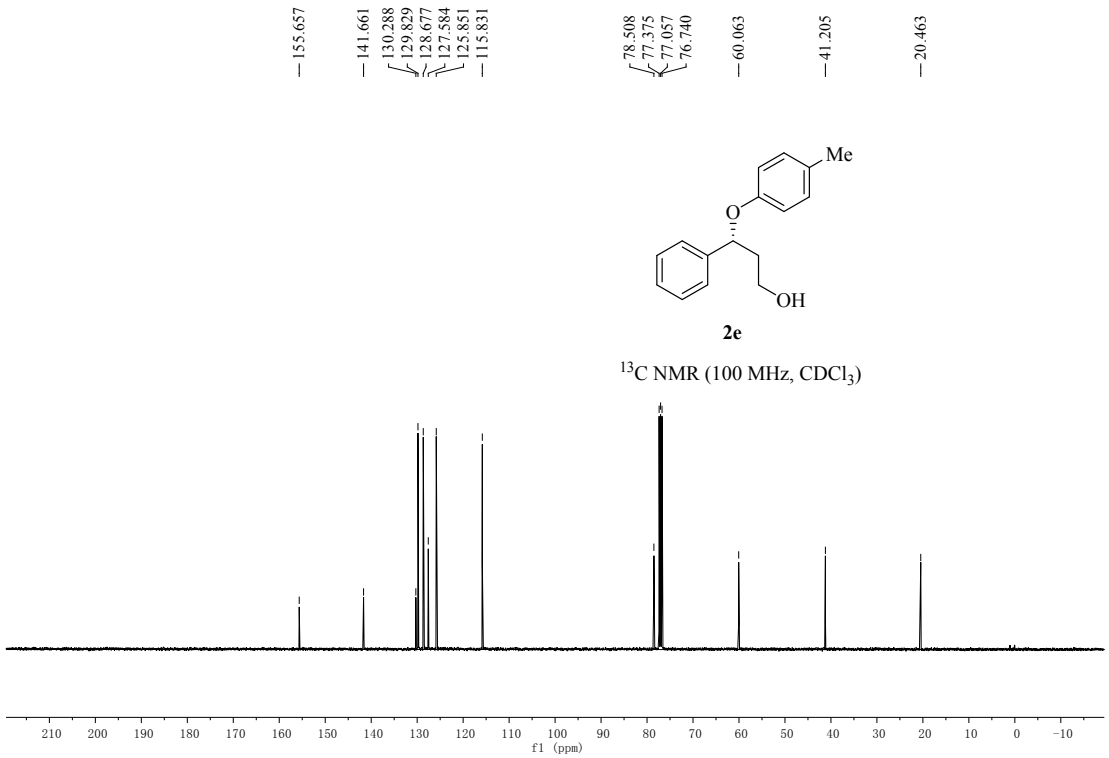
2d

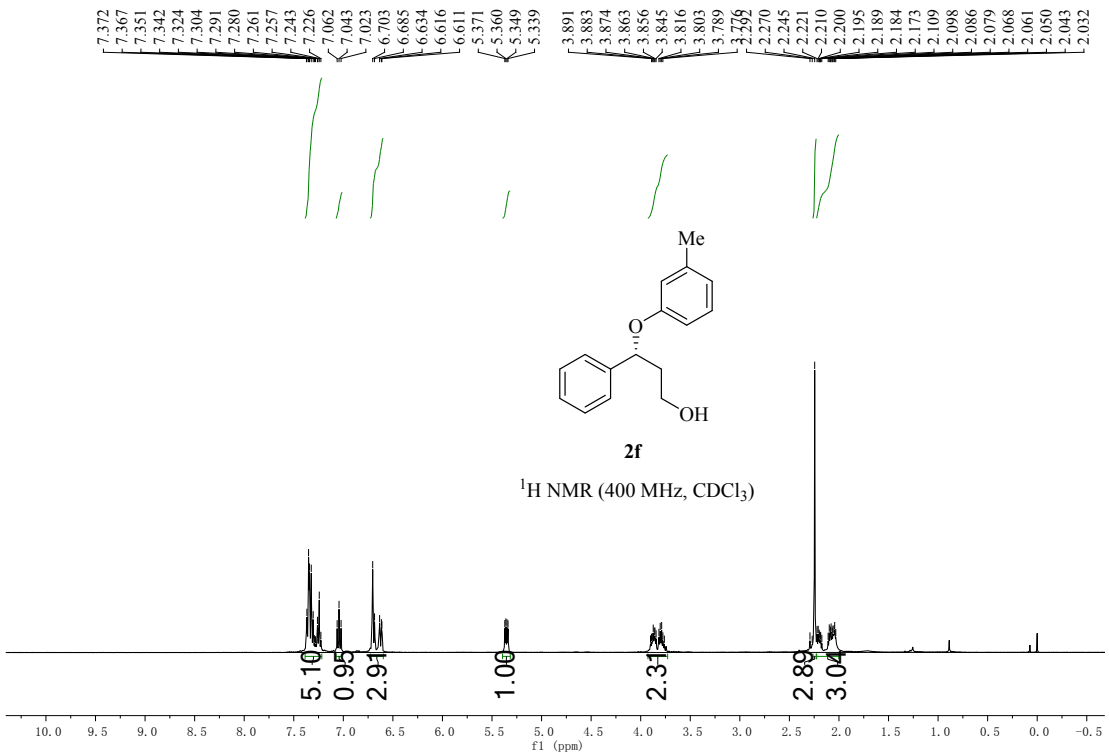
^{1}H NMR (400 MHz, $CDCl_3$)

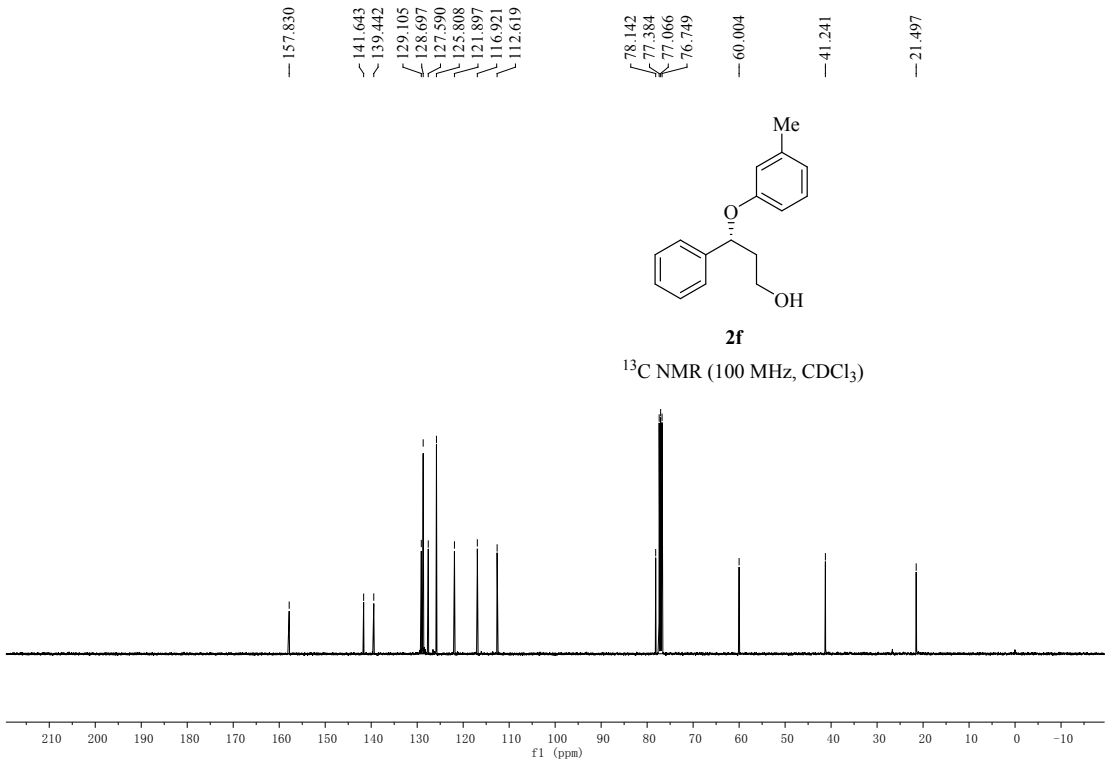


2e

^{1}H NMR (400 MHz, $CDCl_3$)

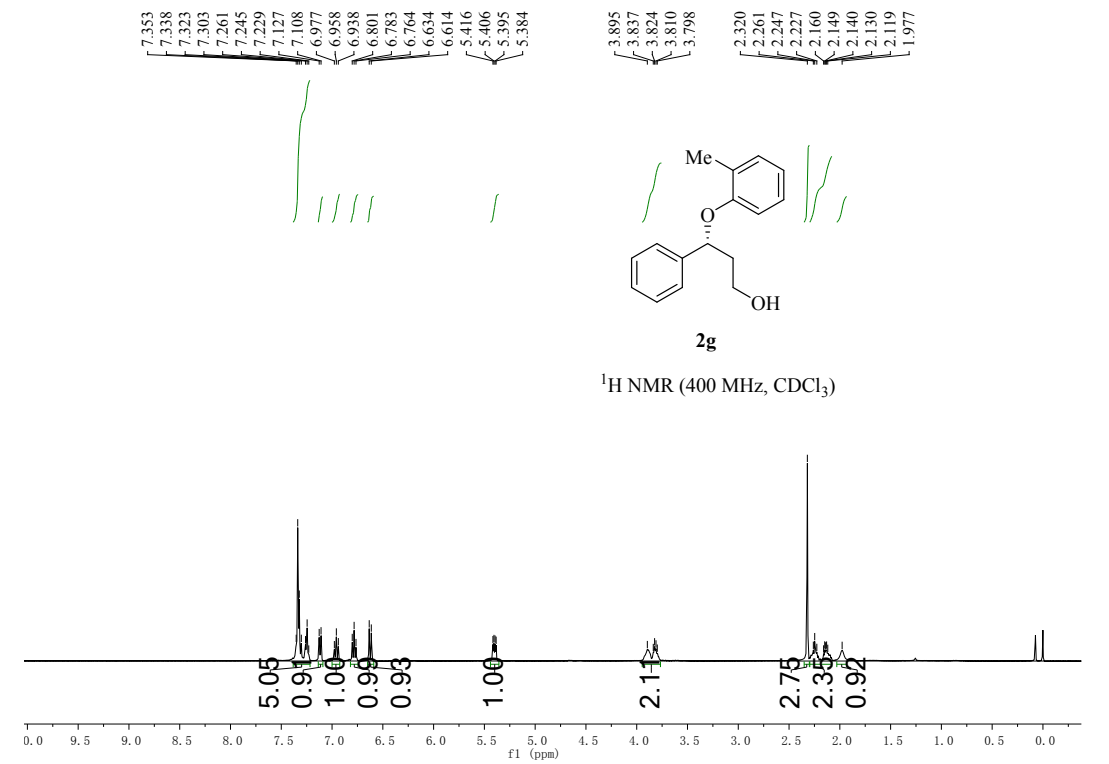

155.657
141.661
130.288
129.829
128.677
127.584
125.851
115.831
78.508
77.375
77.057
76.740
60.063
41.205
20.463
Me
O
OH
2e
13C NMR (100 MHz, CDCl3)
f1 (ppm)


7.372
7.367
7.351
7.342
7.324
7.304
7.291
7.280
7.261
7.257
7.243
7.226
7.062
7.043
7.023
6.703
6.685
6.634
6.616
6.611
5.371
5.360
5.349
5.339
3.891
3.883
3.874
3.863
3.856
3.845
3.816
3.803
3.789
2.270
2.245
2.221
2.210
2.200
2.195
2.189
2.184
2.173
2.109
2.098
2.086
2.079
2.068
2.061
2.050
2.043
2.032
Me
O
OH
2f
1H NMR (400 MHz, CDCl3)
5.10
0.95
2.91
1.00
2.31
2.89
3.04
f1 (ppm)



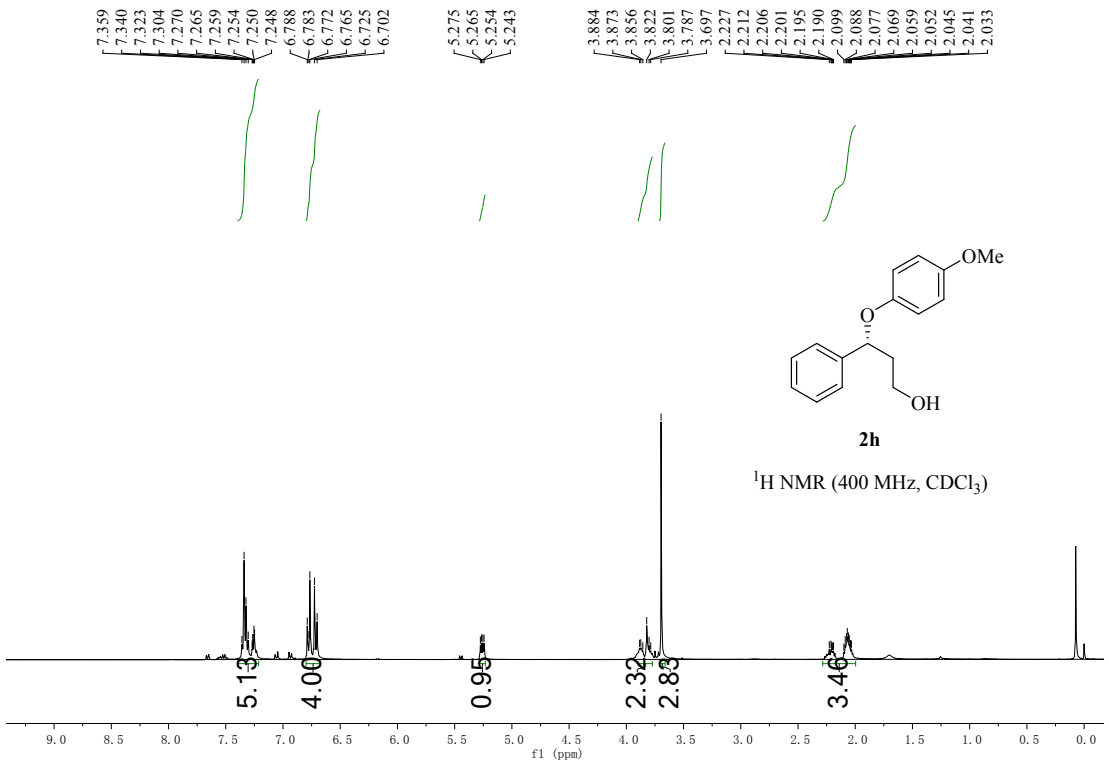
2f

^{13}C NMR (100 MHz, $CDCl_3$)



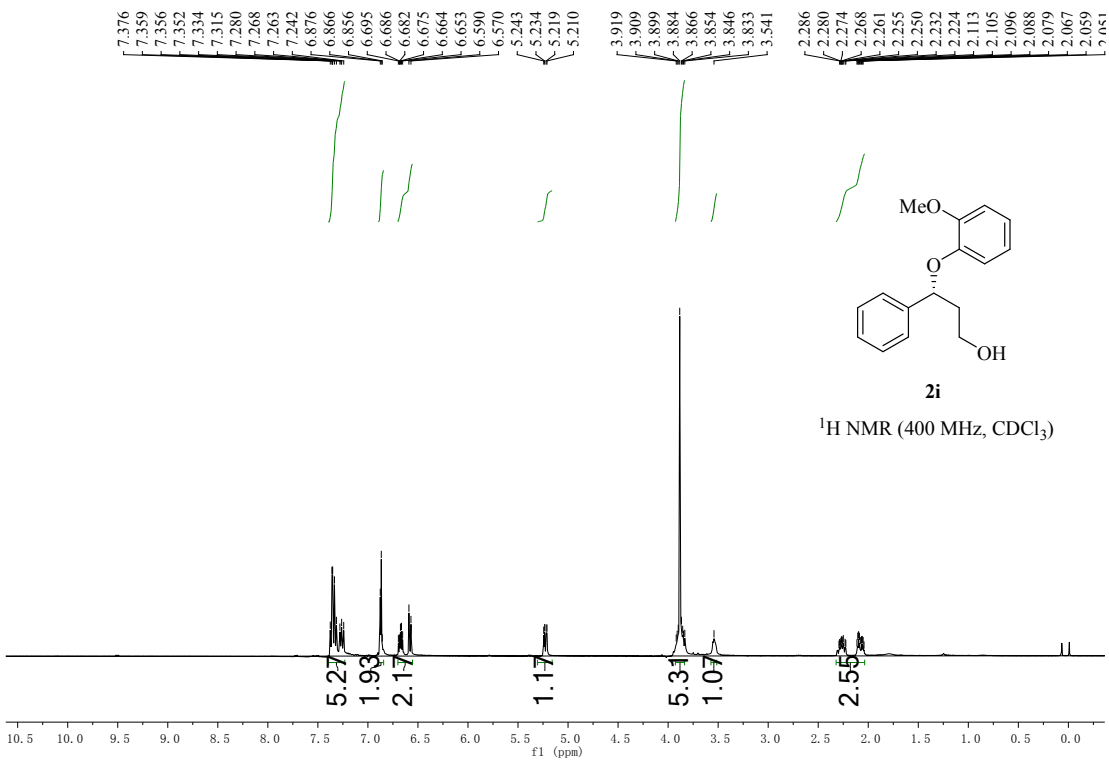
2g

^{1}H NMR (400 MHz, $CDCl_3$)



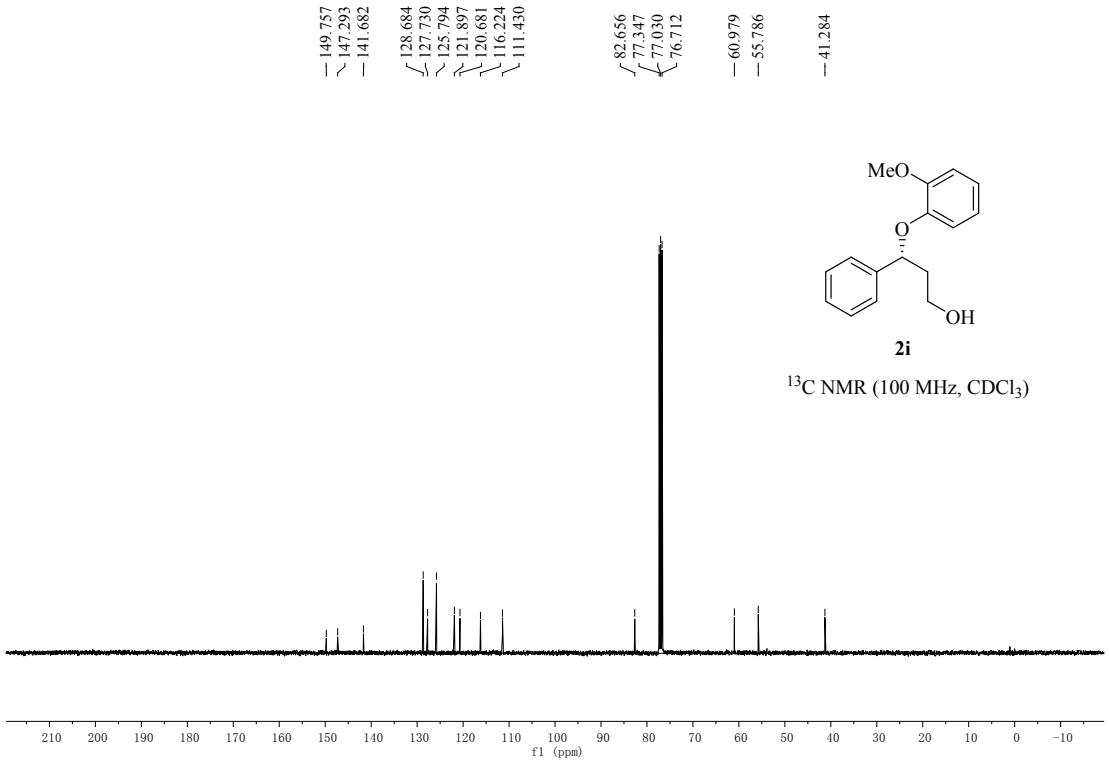
2h

^{1}H NMR (400 MHz, $CDCl_3$)



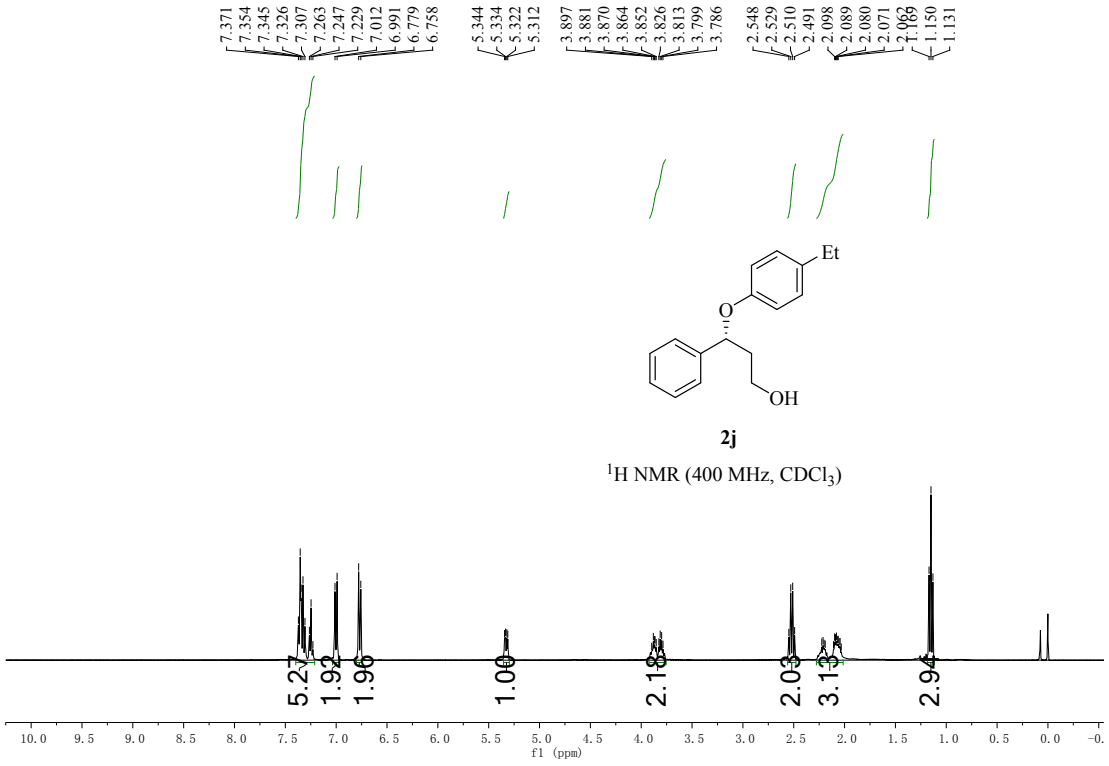
2i

^{1}H NMR (400 MHz, $CDCl_3$)



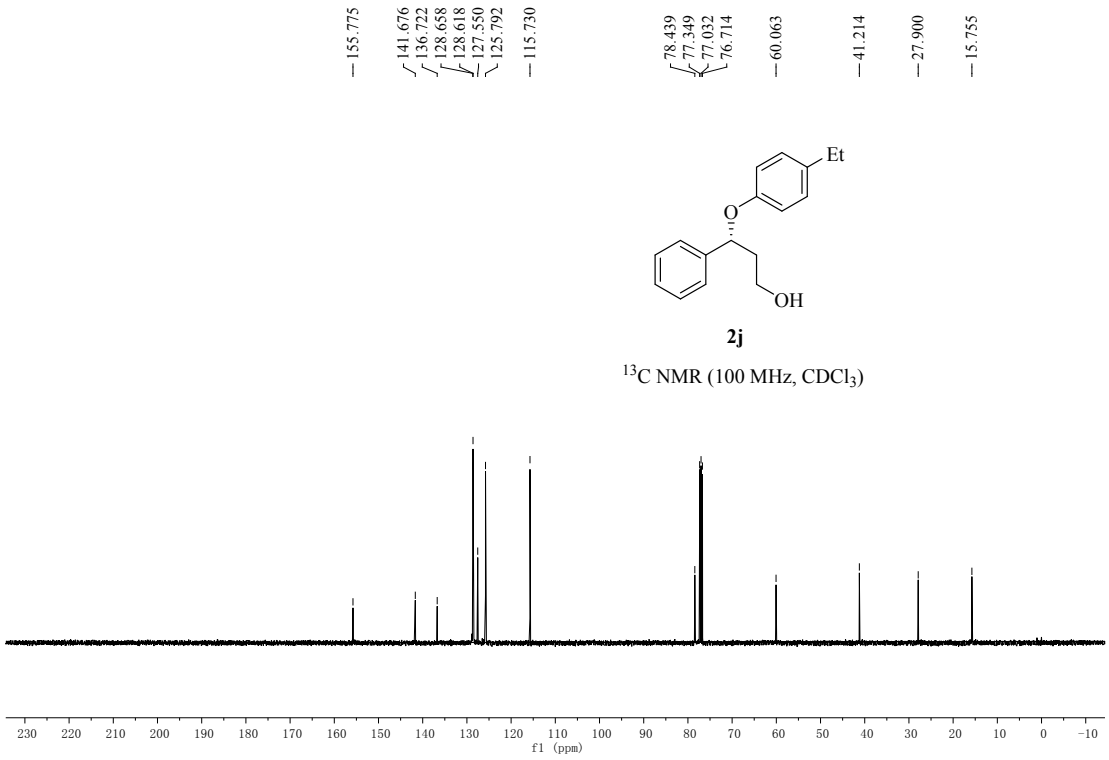
2i

^{13}C NMR (100 MHz, $CDCl_3$)



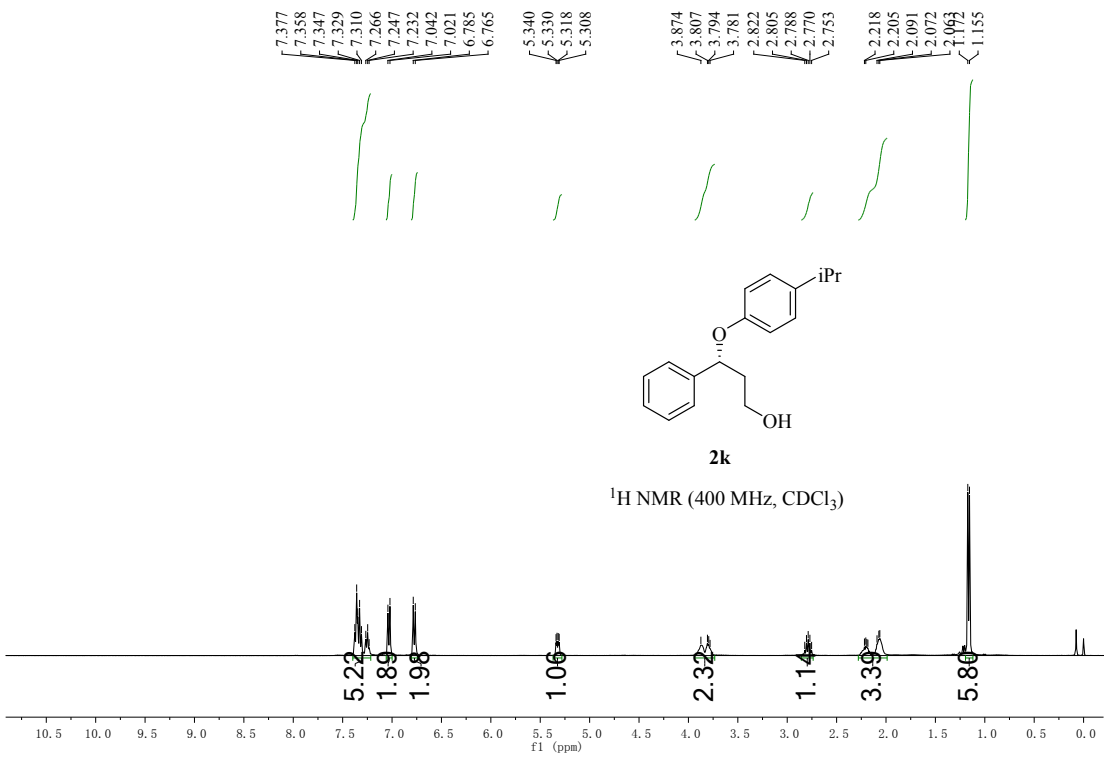
2j

^{1}H NMR (400 MHz, $CDCl_3$)



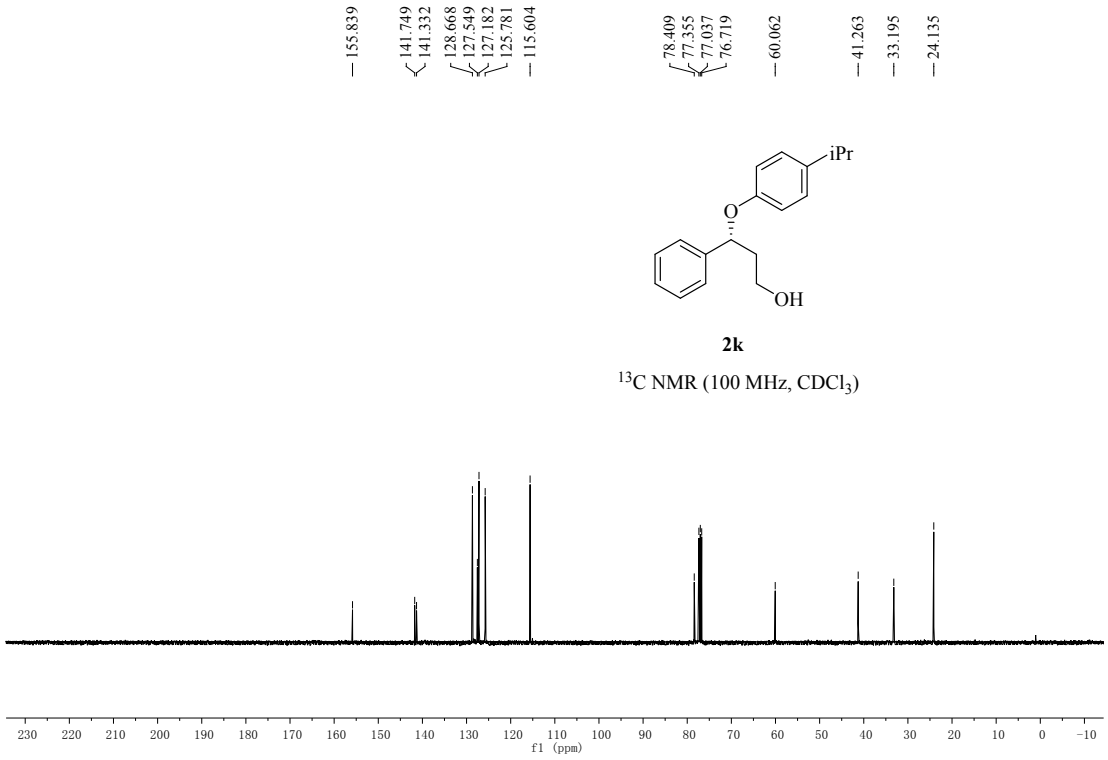
2j

^{13}C NMR (100 MHz, $CDCl_3$)



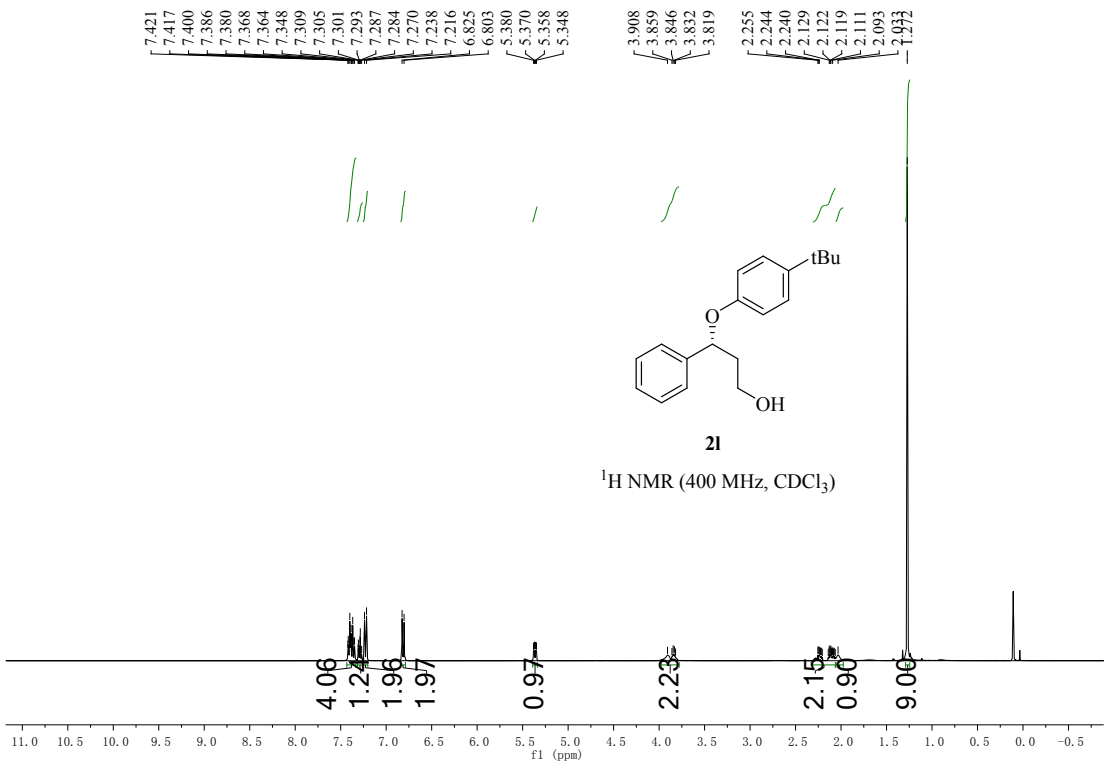
2k

^{1}H NMR (400 MHz, $CDCl_3$)



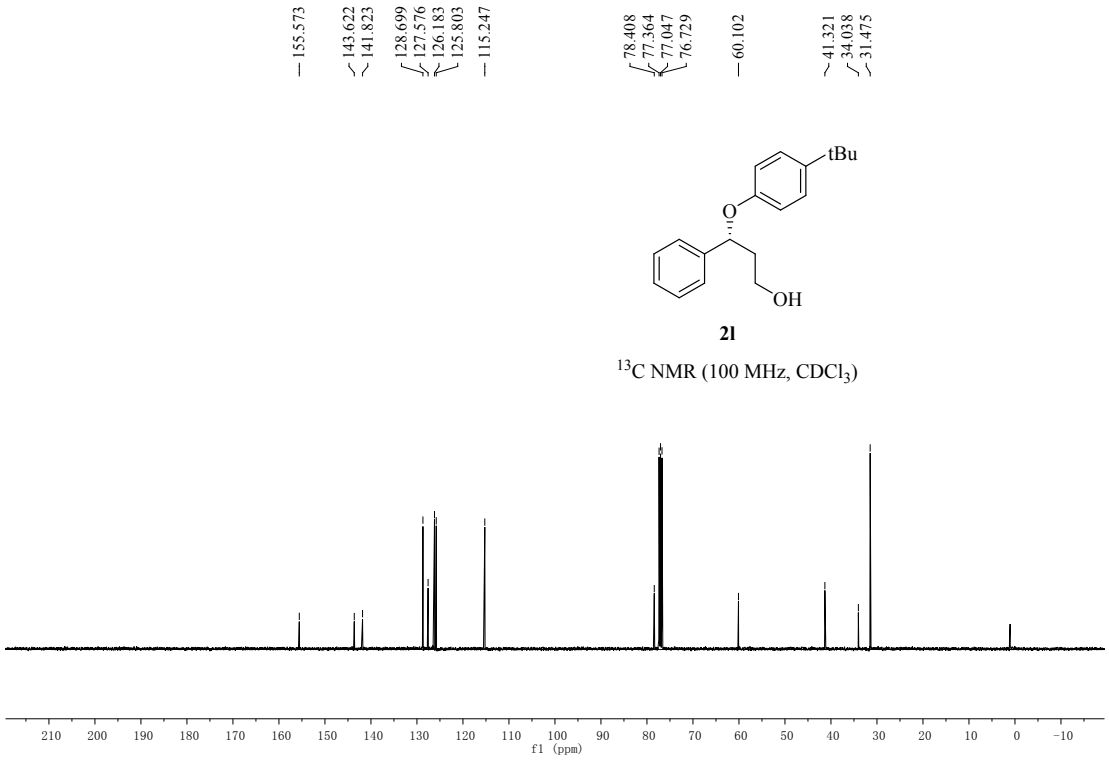
2k

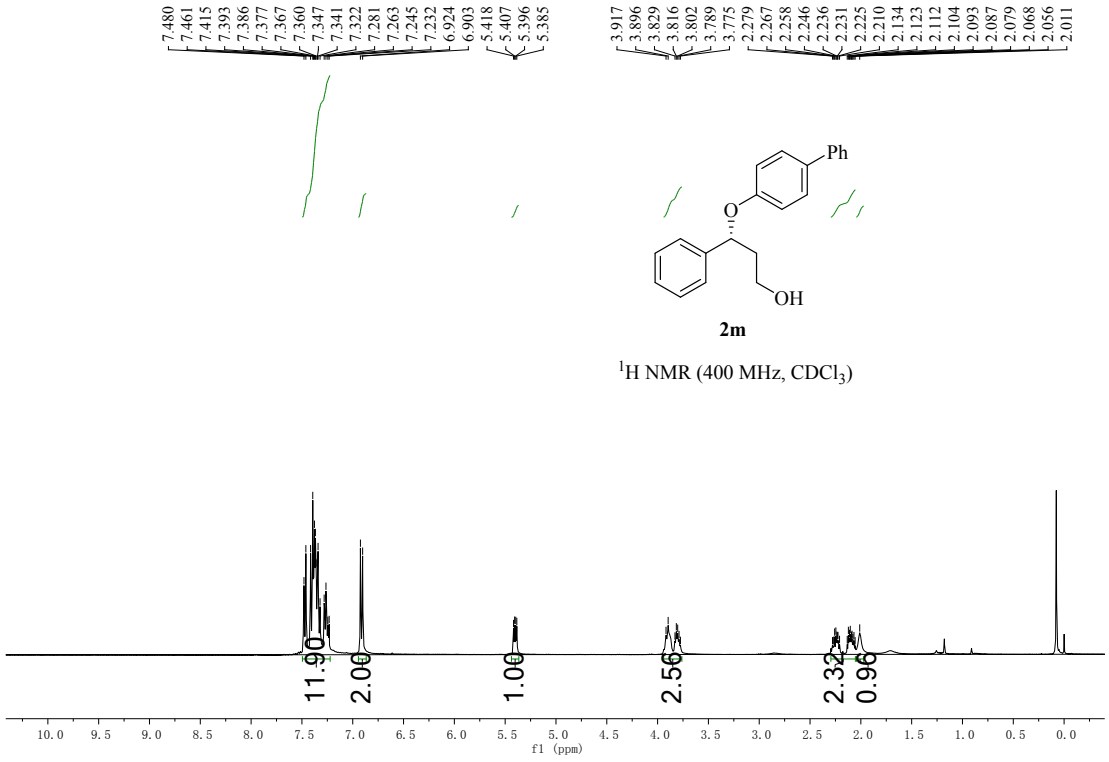
^{13}C NMR (100 MHz, $CDCl_3$)

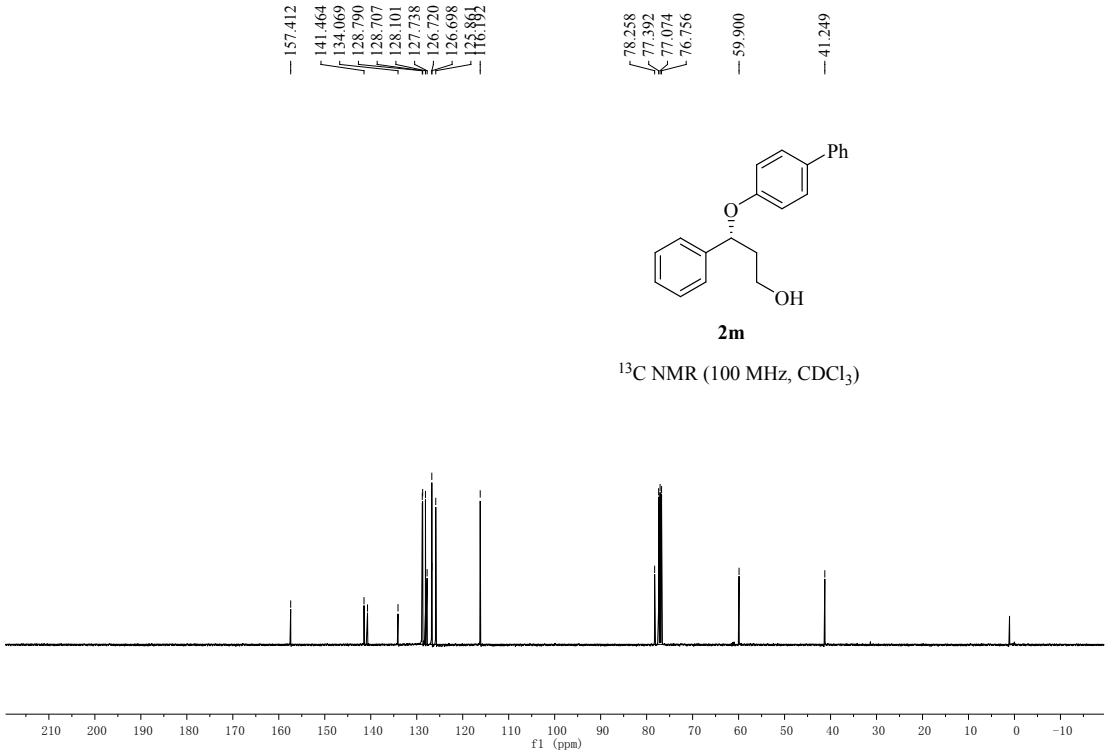


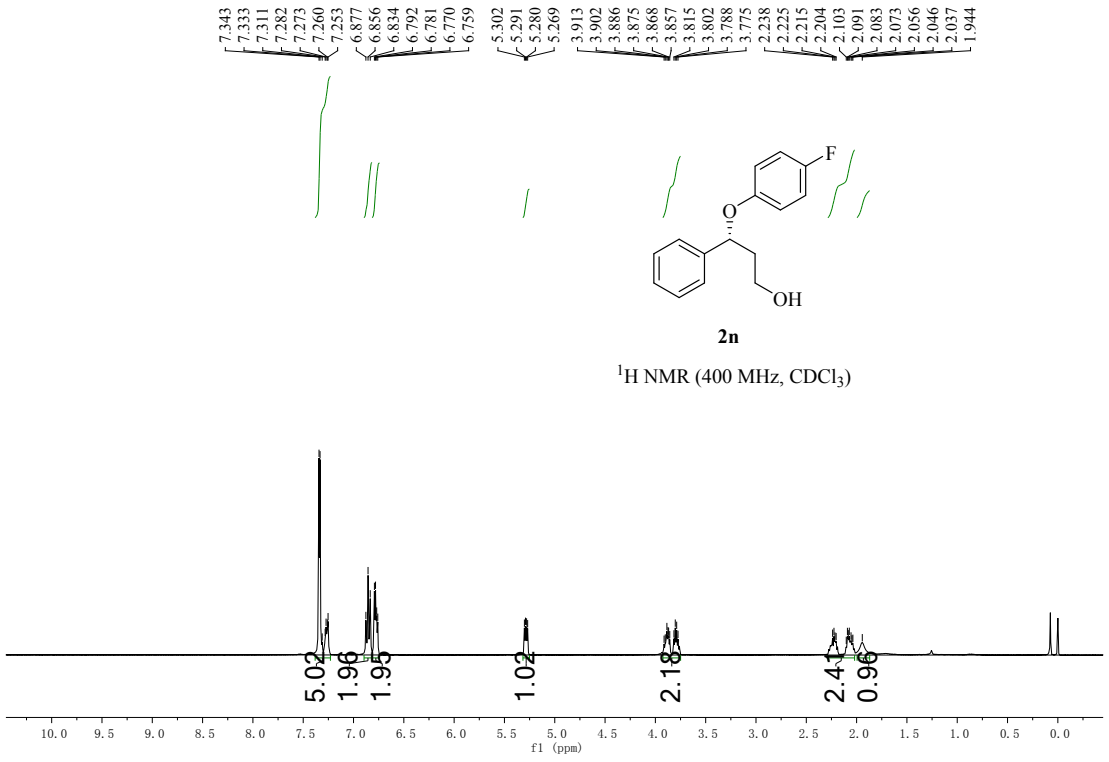
2l

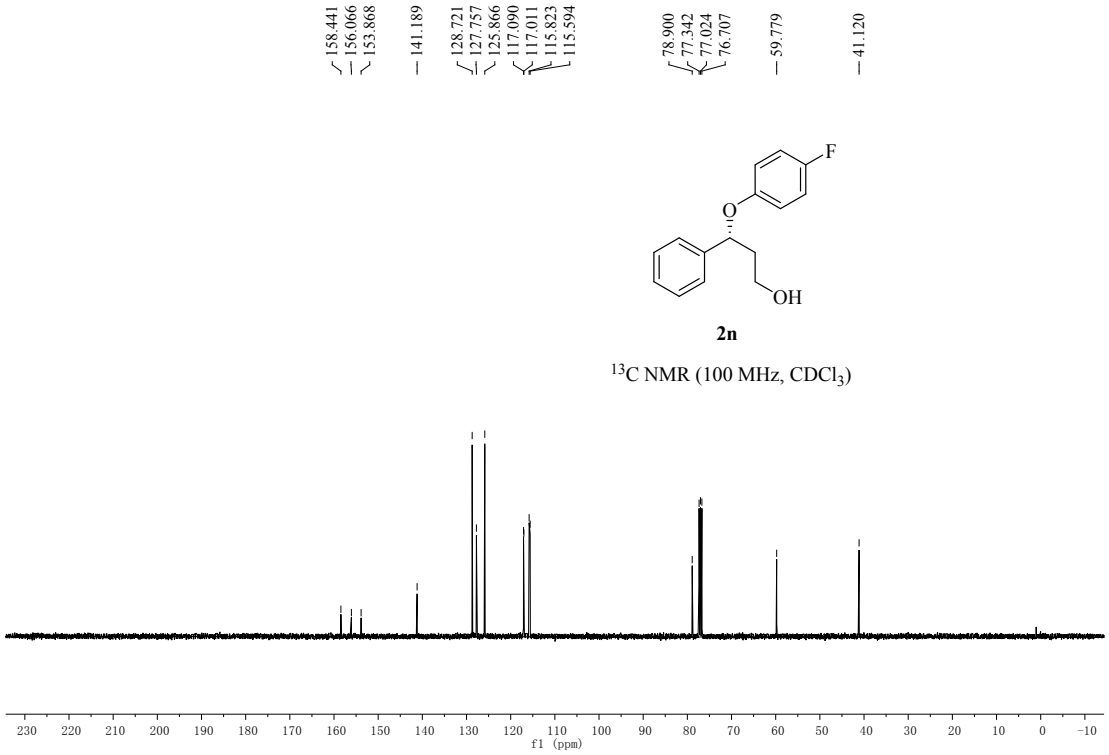
^{1}H NMR (400 MHz, $CDCl_3$)


155.573
143.622
141.823
128.699
127.576
126.183
125.803
115.247
78.408
77.364
77.047
76.729
60.102
41.321
34.038
31.475
tBu
O
OH
2l
13C NMR (100 MHz, CDCl3)
f1 (ppm)


7.480
7.461
7.415
7.393
7.386
7.377
7.367
7.360
7.347
7.341
7.322
7.281
7.263
7.245
7.232
6.924
6.903
5.418
5.407
5.396
5.385
3.917
3.896
3.829
3.816
3.802
3.789
3.775
2.279
2.267
2.258
2.246
2.236
2.231
2.225
2.210
2.134
2.123
2.112
2.104
2.093
2.087
2.079
2.068
2.056
2.011
Ph
O
OH
2m
1H NMR (400 MHz, CDCl3)
11.90
2.00
1.00
2.56
2.32
0.96
f1 (ppm)

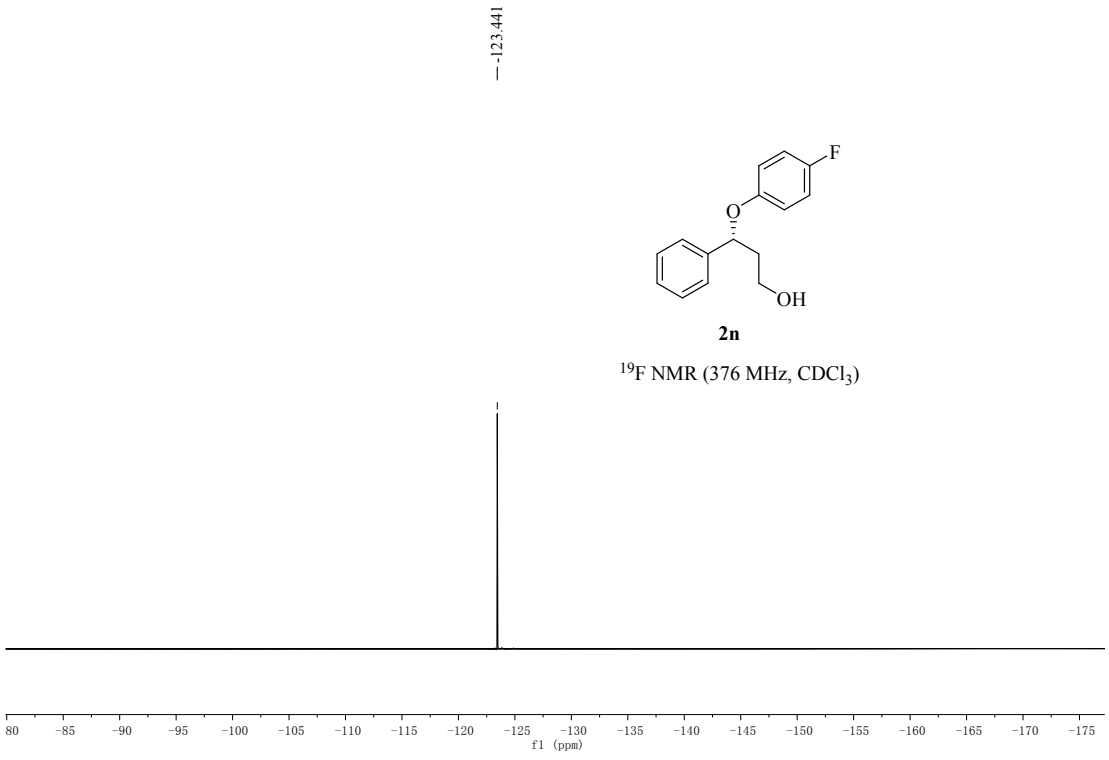

157.412
141.464
134.069
128.790
128.707
128.101
127.738
126.720
126.698
125.861
116.192
78.258
77.392
77.074
76.756
59.900
41.249
Ph
O
OH
2m
13C NMR (100 MHz, CDCl3)
f1 (ppm)


7.343
7.333
7.311
7.282
7.273
7.260
7.253
6.877
6.856
6.834
6.792
6.781
6.770
6.759
5.302
5.291
5.280
5.269
3.913
3.902
3.886
3.875
3.868
3.857
3.815
3.802
3.788
3.775
2.238
2.225
2.215
2.204
2.103
2.091
2.083
2.073
2.056
2.046
2.037
1.944
F
O
OH
2n
1H NMR (400 MHz, CDCl3)
5.02
1.96
1.95
1.02
2.18
2.41
0.96
f1 (ppm)



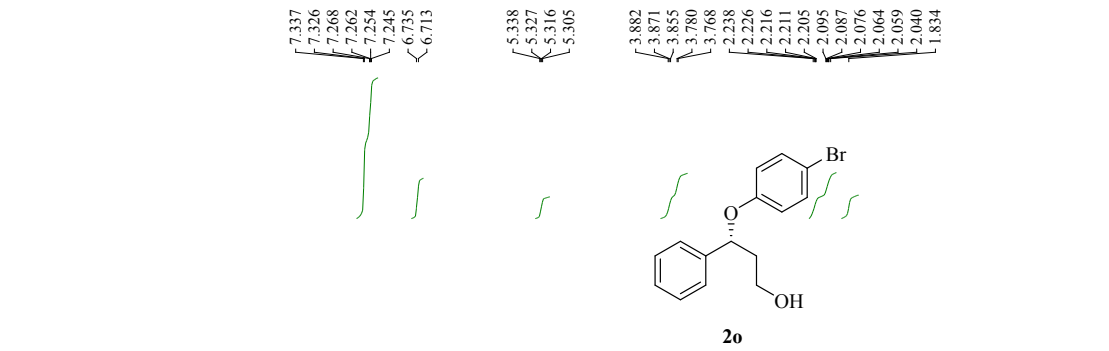
2n

^{13}C NMR (100 MHz, $CDCl_3$)



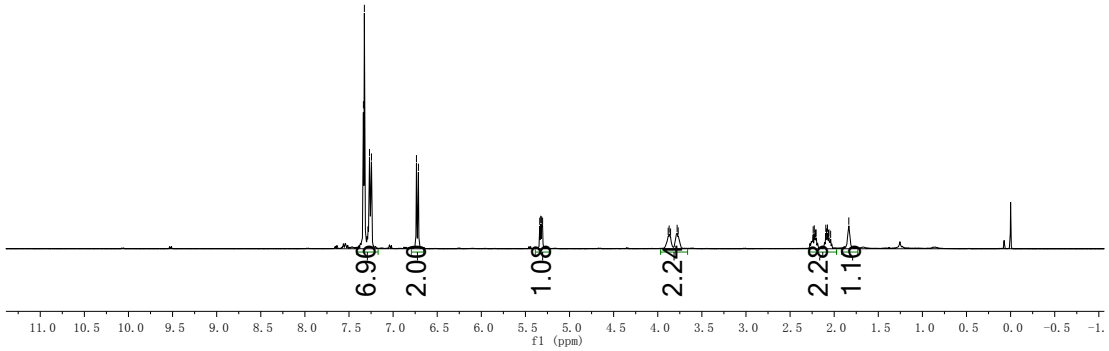
2n

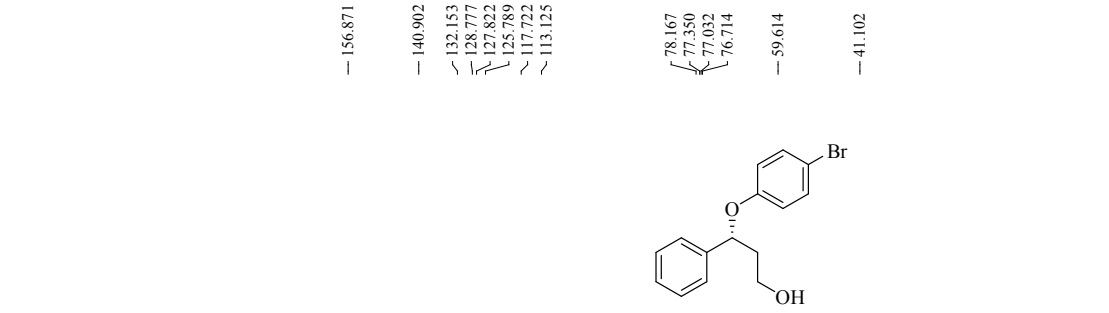
^{19}F NMR (376 MHz, $CDCl_3$)



2o

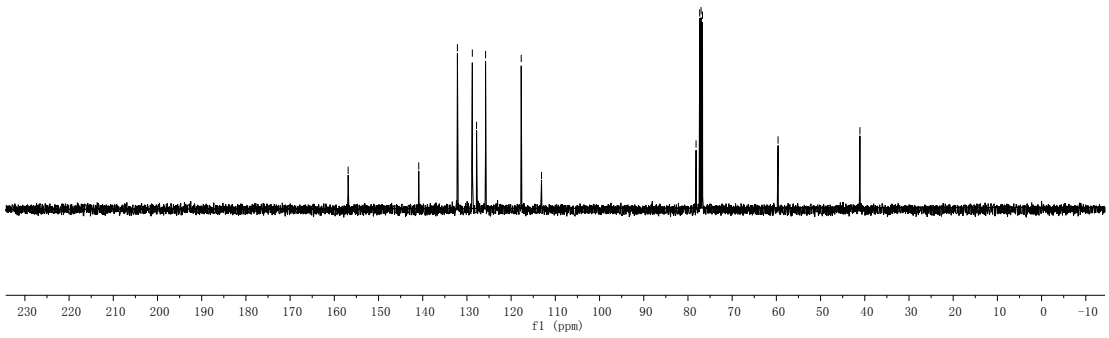
^{1}H NMR (400 MHz, $CDCl_3$)

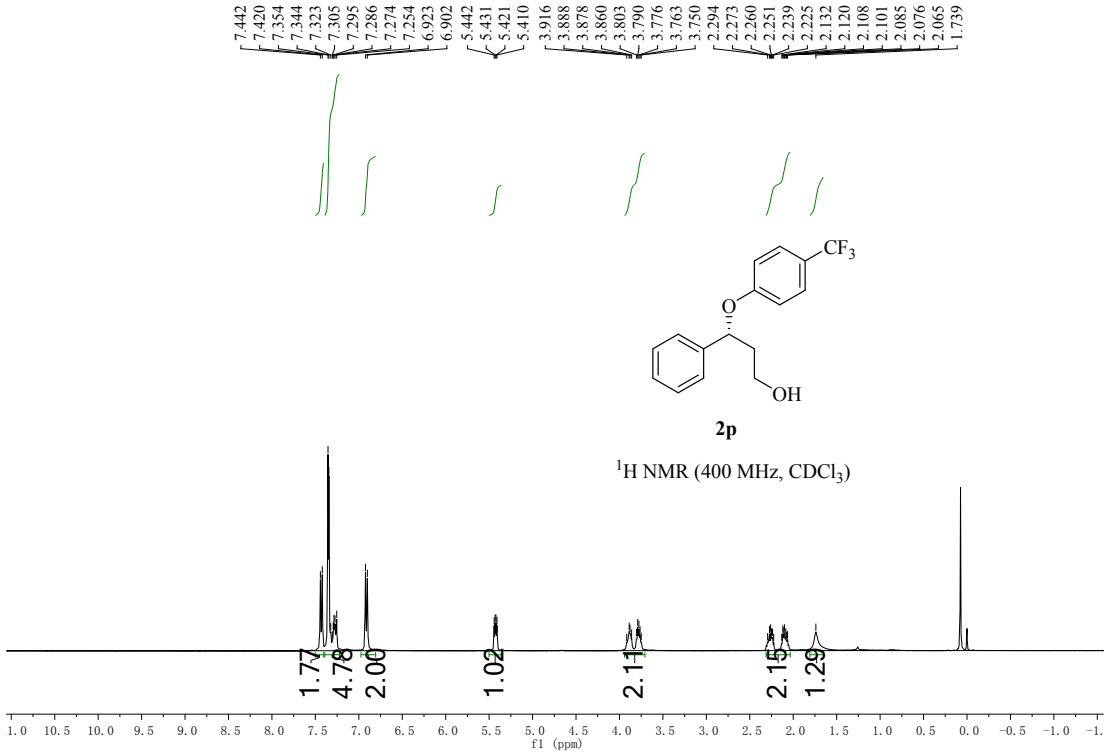




2o

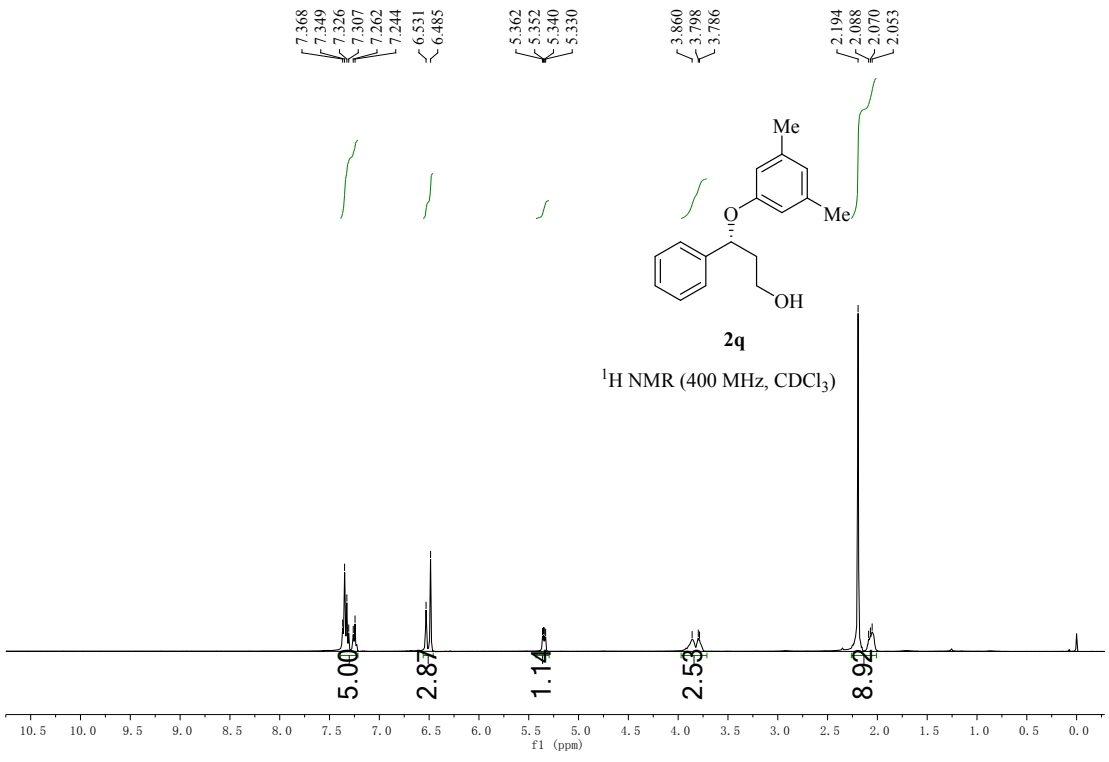
^{13}C NMR (100 MHz, $CDCl_3$)





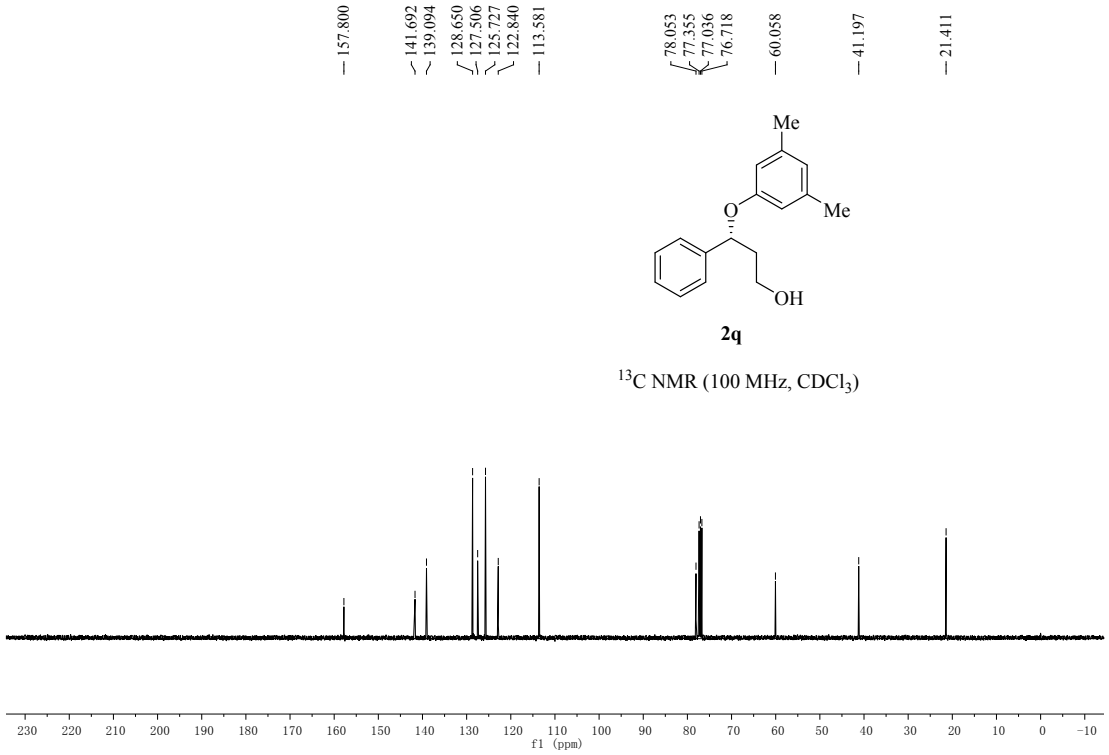
2p

^{1}H NMR (400 MHz, $CDCl_3$)



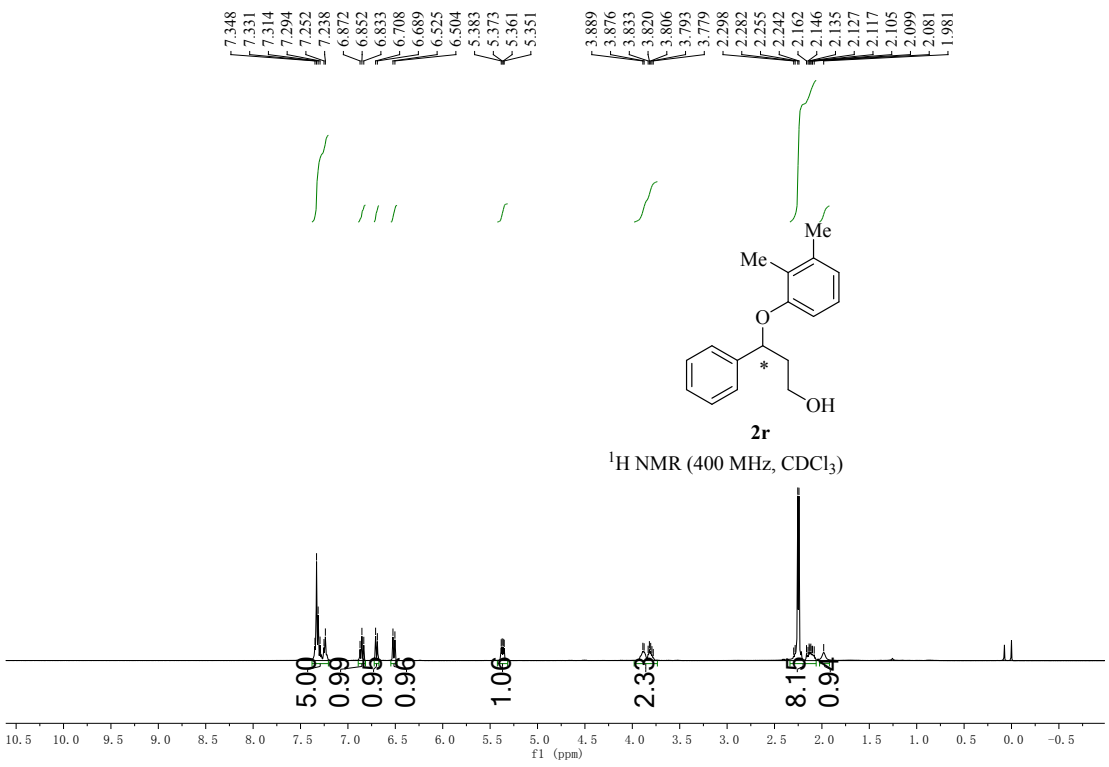
2q

^{1}H NMR (400 MHz, $CDCl_3$)



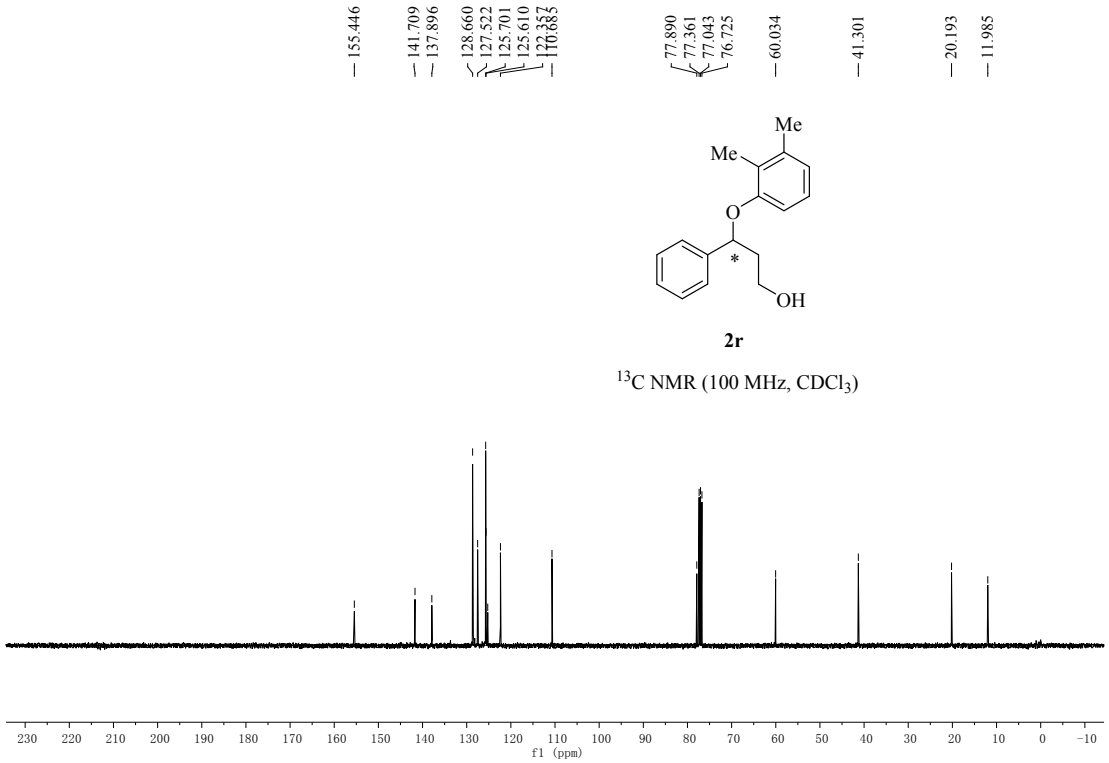
2q

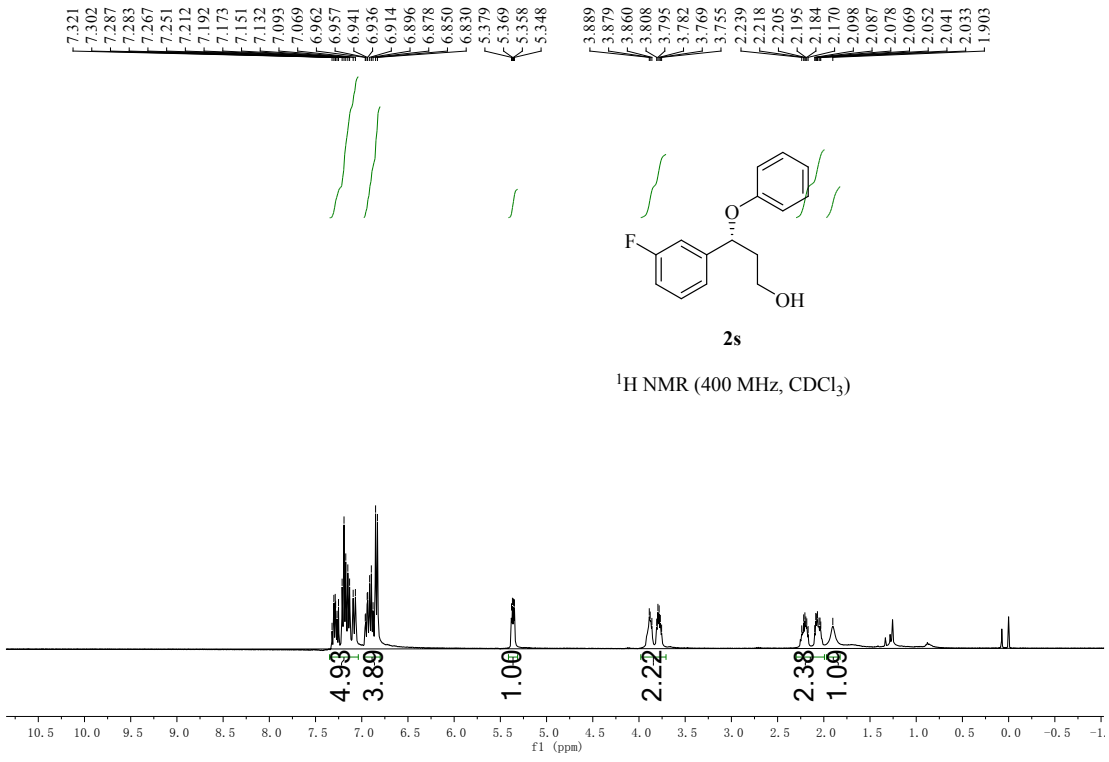
^{13}C NMR (100 MHz, $CDCl_3$)

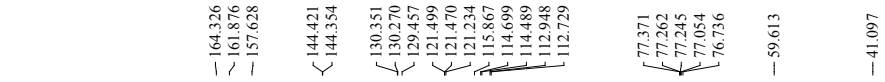


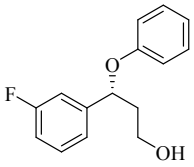
2r

^{1}H NMR (400 MHz, $CDCl_3$)


155.446
141.709
137.896
128.660
127.522
125.701
125.610
122.357
110.685
77.890
77.361
77.043
76.725
60.034
41.301
20.193
11.985
Me
Me
O
*
OH
2r
13C NMR (100 MHz, CDCl3)
f1 (ppm)

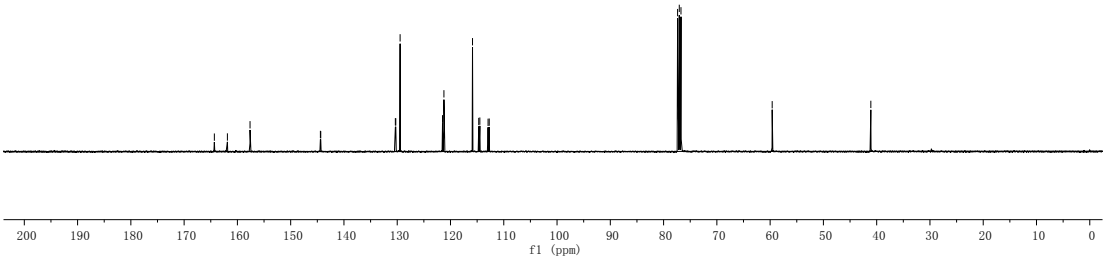

7.321
7.302
7.287
7.283
7.267
7.251
7.212
7.192
7.173
7.151
7.132
7.093
7.069
6.962
6.957
6.941
6.936
6.914
6.896
6.878
6.850
6.830
5.379
5.369
5.358
5.348
3.889
3.879
3.860
3.808
3.795
3.782
3.769
3.755
2.239
2.218
2.205
2.195
2.184
2.170
2.098
2.087
2.078
2.069
2.052
2.041
2.033
1.903
O
F
OH
2s
1H NMR (400 MHz, CDCl3)
4.93
3.89
1.00
2.22
2.38
1.09
f1 (ppm)

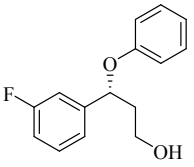




2s

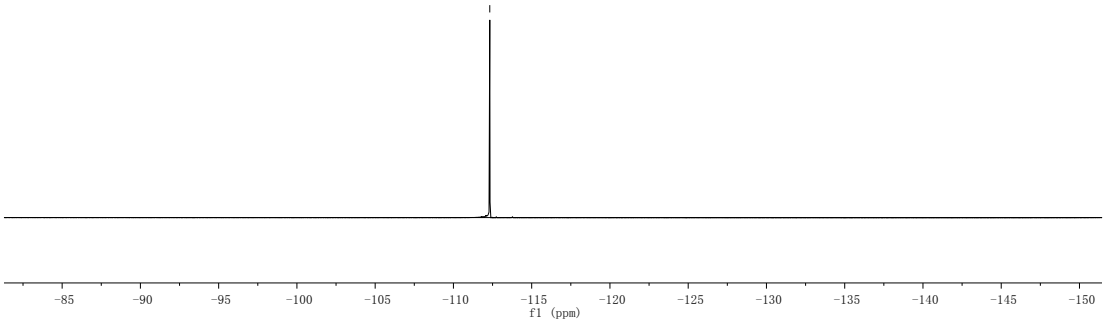
^{13}C NMR (100 MHz, $CDCl_3$)

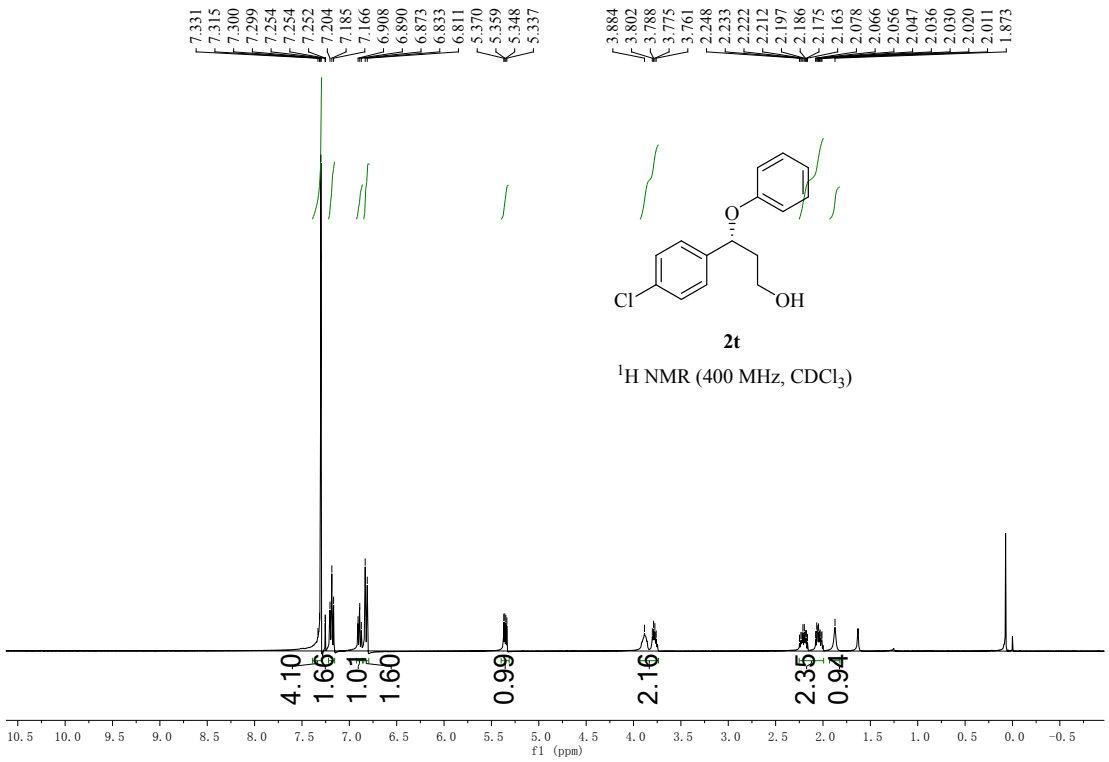




2s

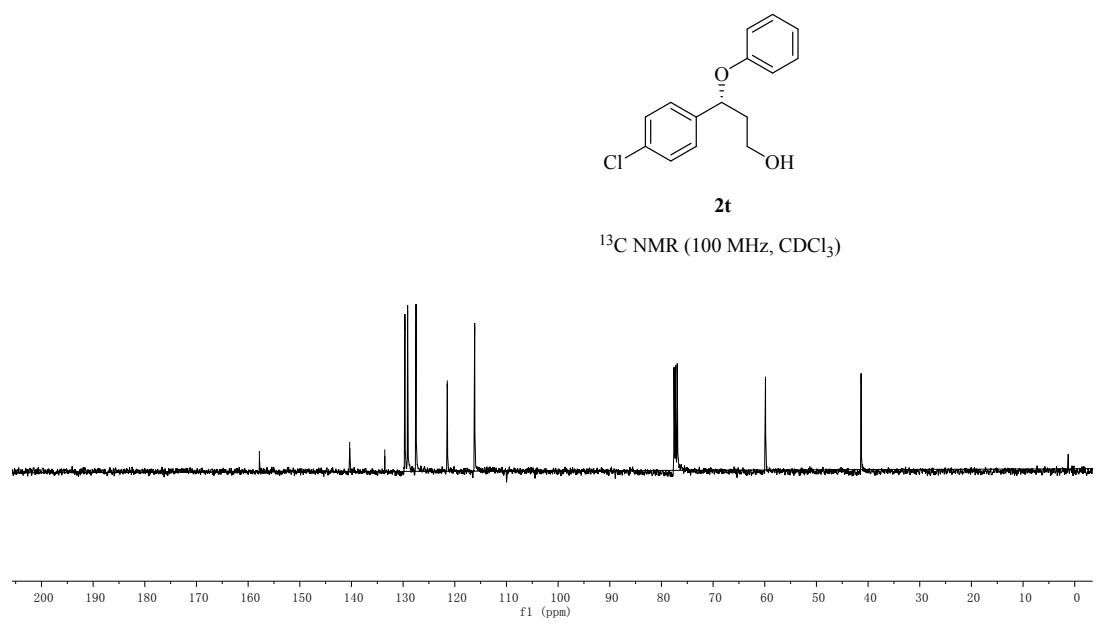
^{19}F NMR (376 MHz, $CDCl_3$)





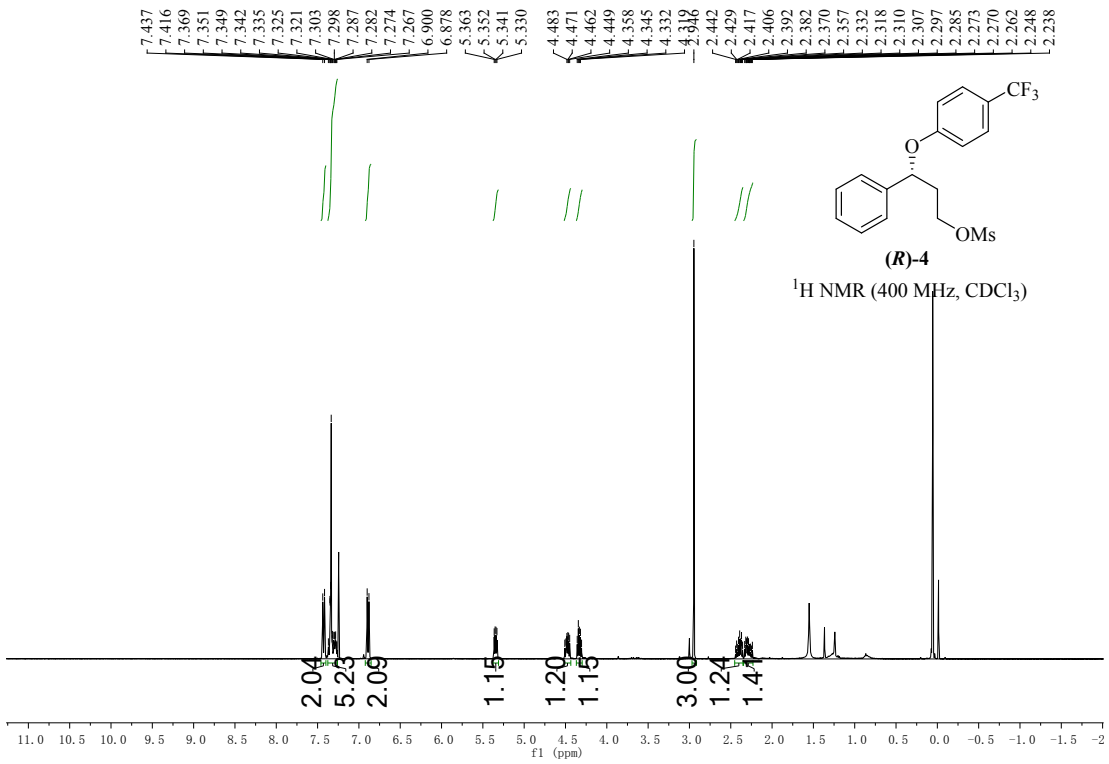
2t

^{1}H NMR (400 MHz, $CDCl_3$)



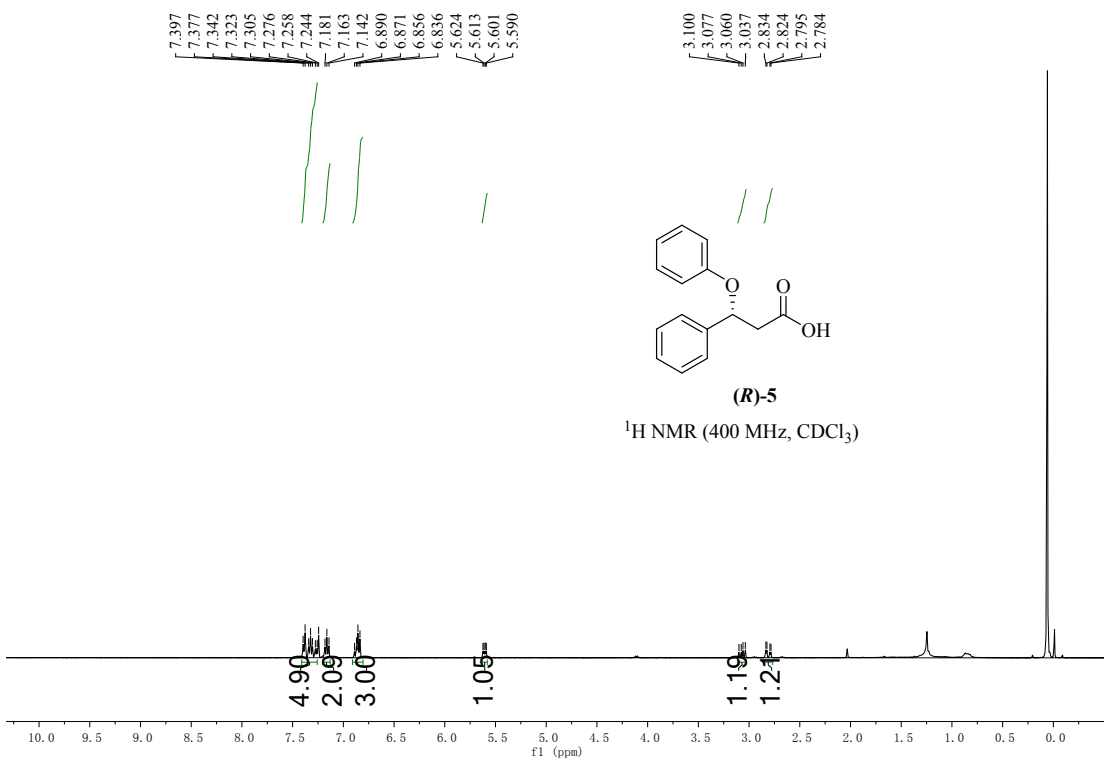
2t

^{13}C NMR (100 MHz, $CDCl_3$)



(*R*)-4

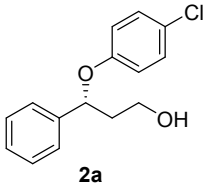
^{1}H NMR (400 MHz, $CDCl_3$)



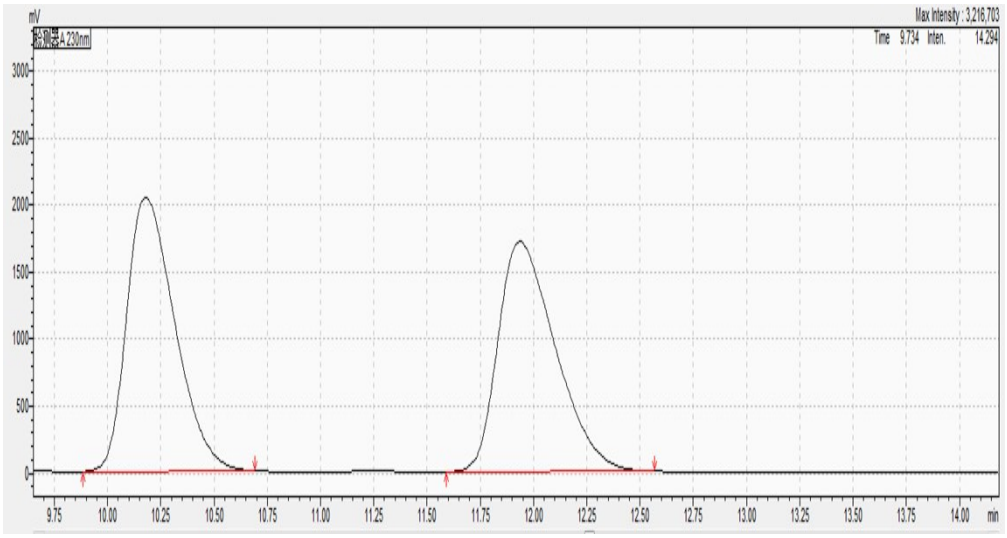
(*R*)-5

^{1}H NMR (400 MHz, $CDCl_3$)

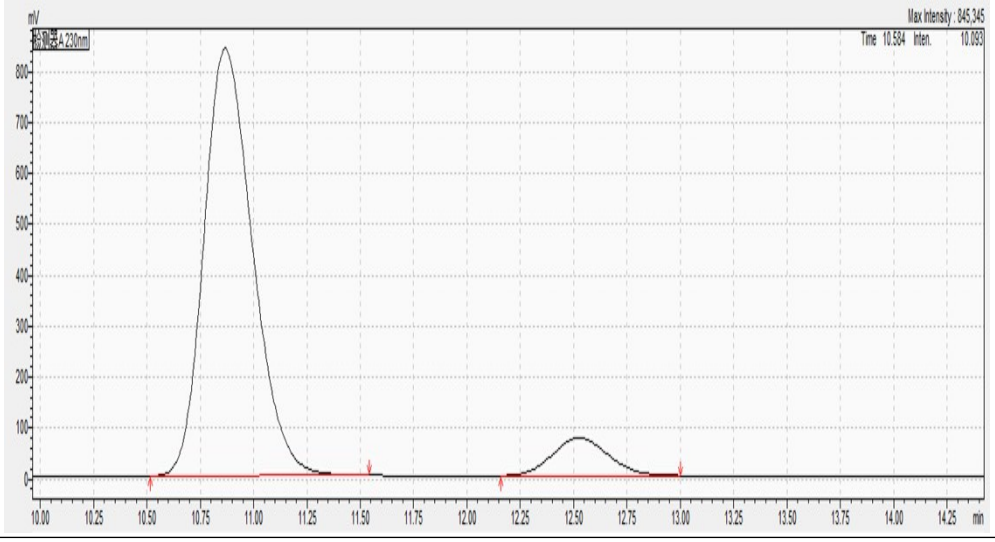
7. HPLC Spectra



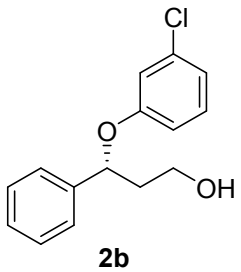
2a



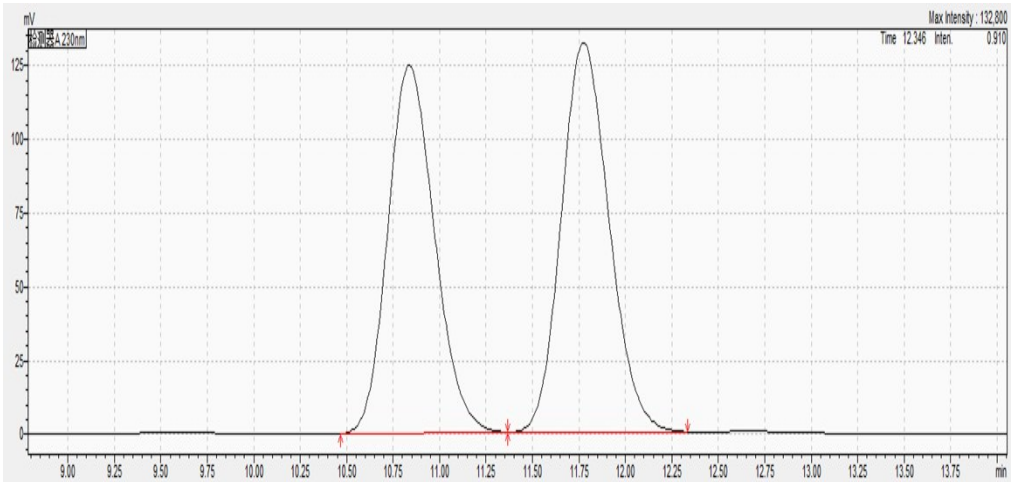
Peak	Rentention Time/min	Area %
1	10.179	50.059
2	11.935	49.941



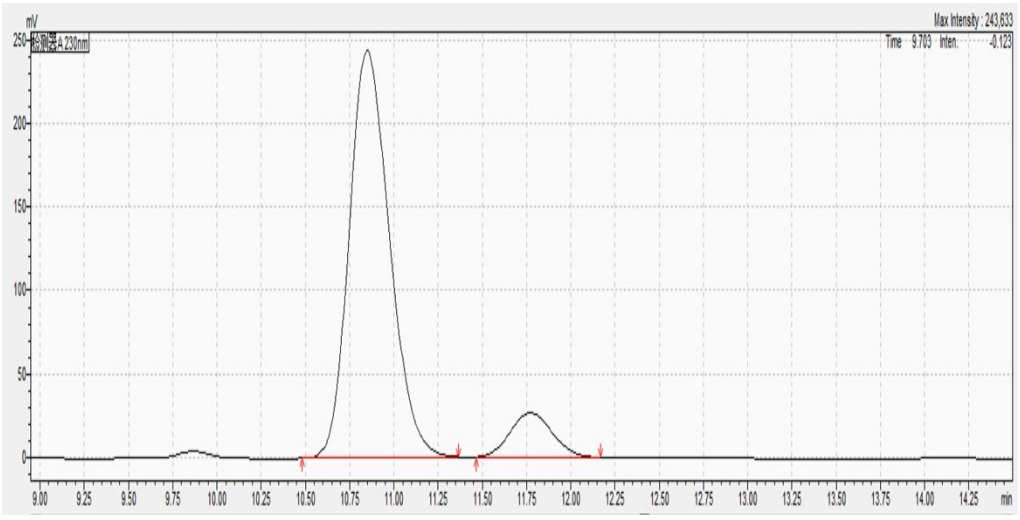
Peak	Rentention Time/min	Area %
1	10.867	90.616
2	12.525	9.384



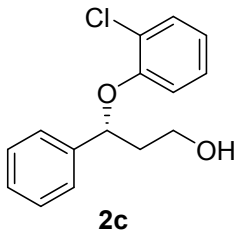
2b



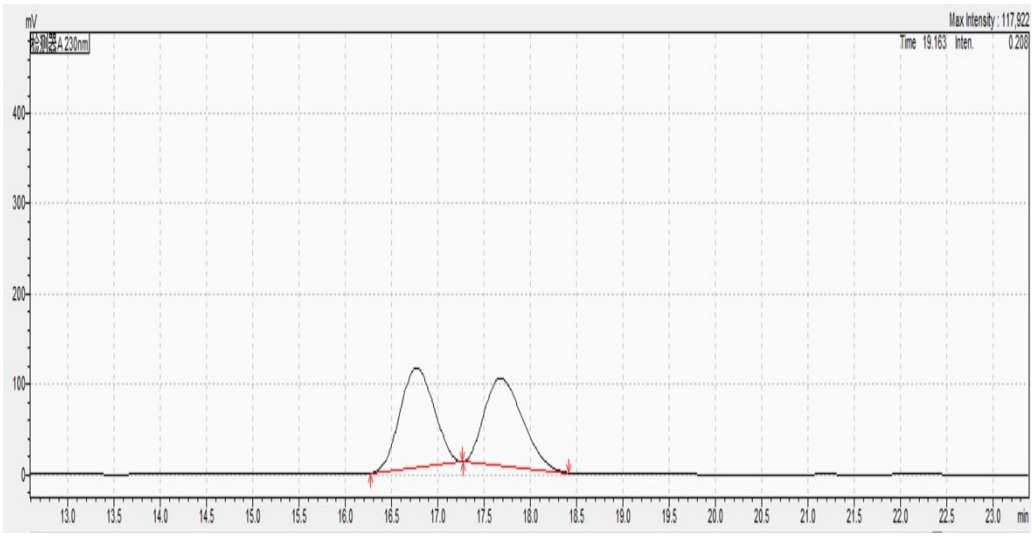
Peak	Rentention Time/min	Area %
1	10.837	49.703
2	11.774	50.297



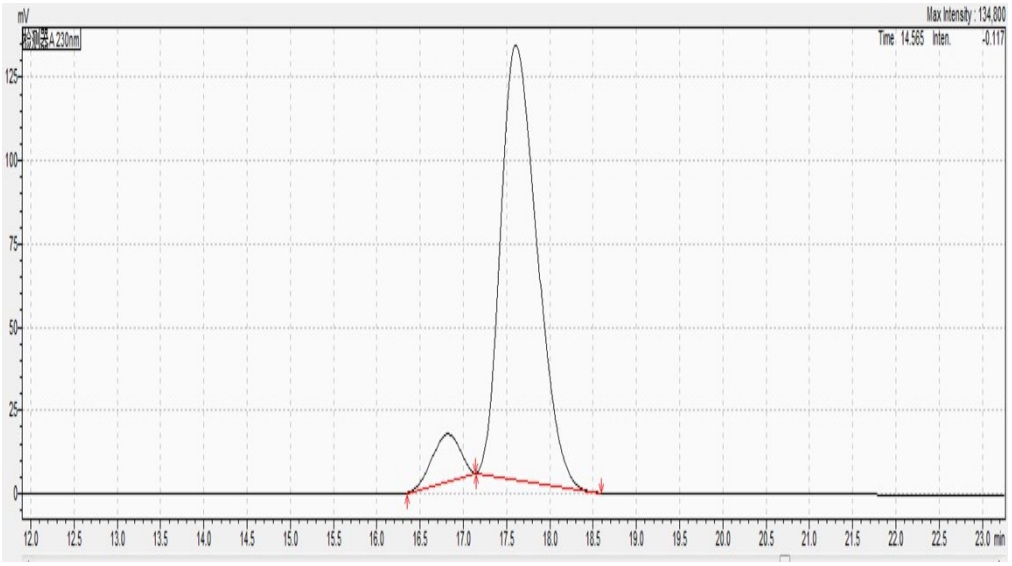
Peak	Rentention Time/min	Area %
1	10.849	90.452
2	11.772	9.548



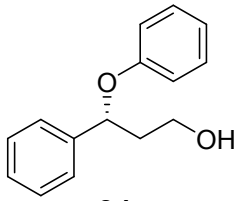
2c



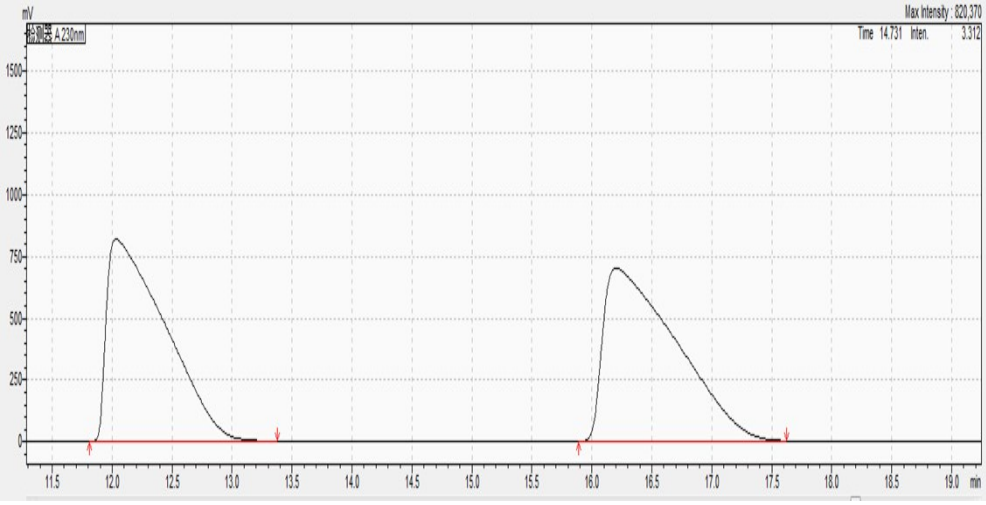
Peak	Rentention Time/min	Area %
1	16.768	50.565
2	17.676	49.435



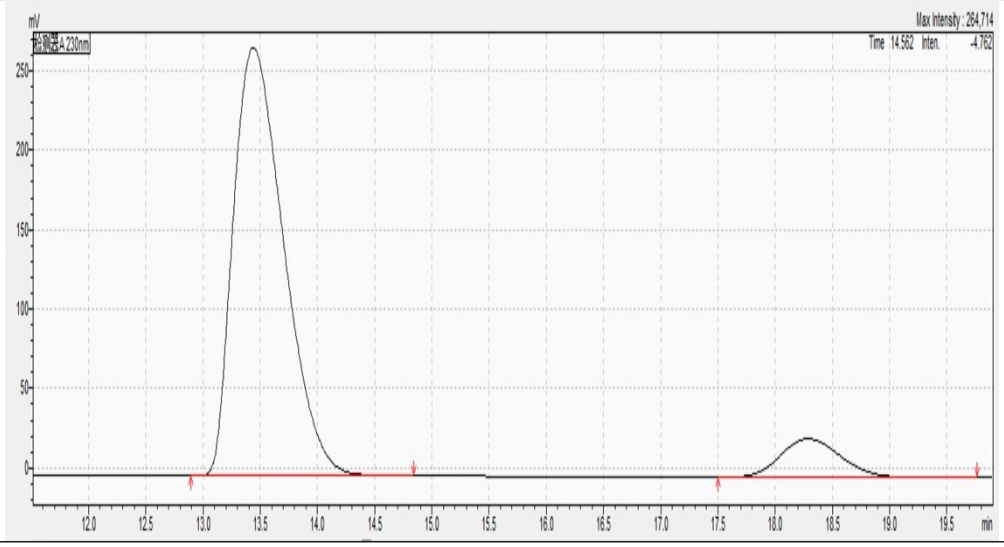
Peak	Rentention Time/min	Area %
1	16.818	10.438
2	17.606	89.562



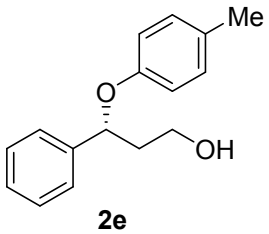
2d



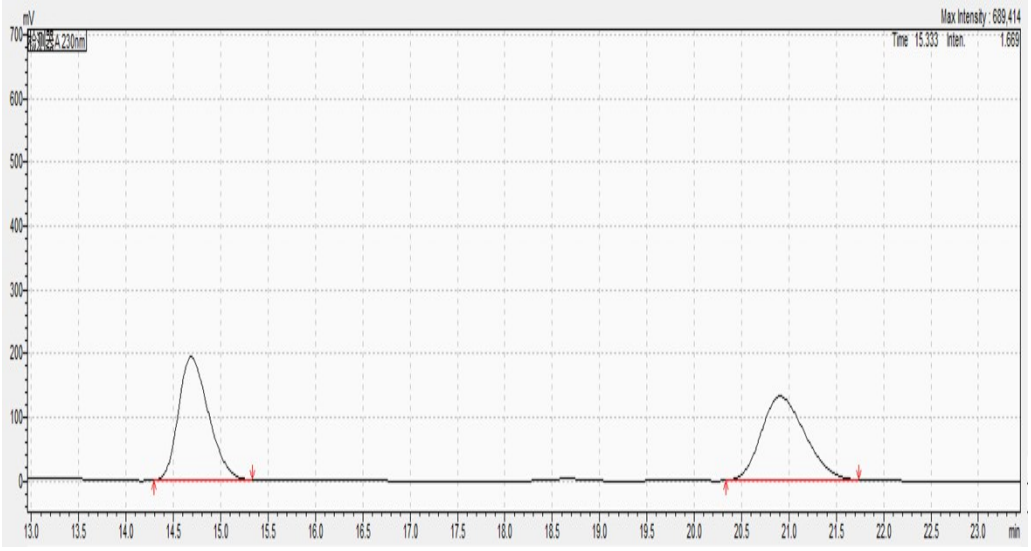
Peak	Rentention Time/min	Area %
1	12.029	50.498
2	16.198	49.502



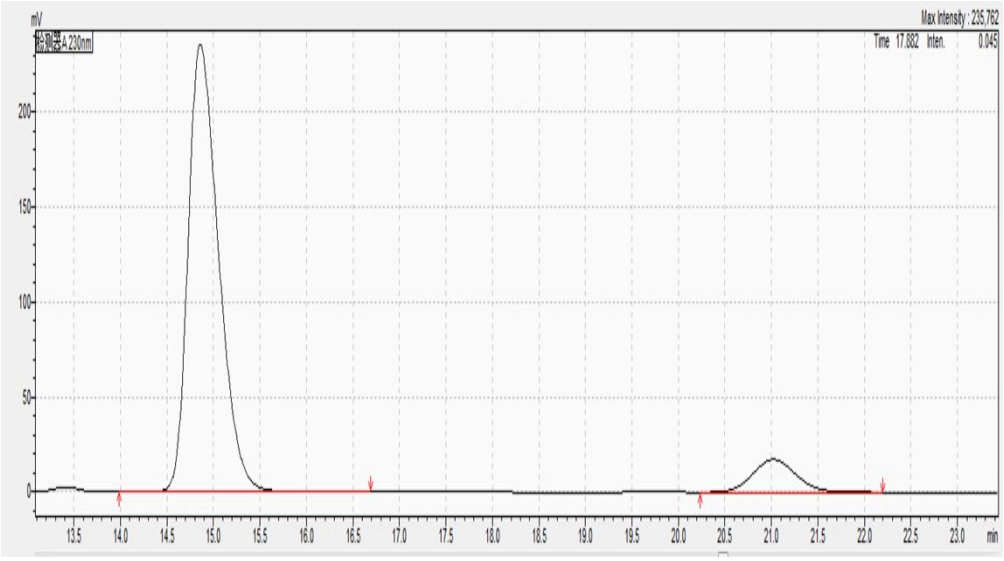
Peak	Rentention Time/min	Area %
1	13.440	90.540
2	18.288	9.460



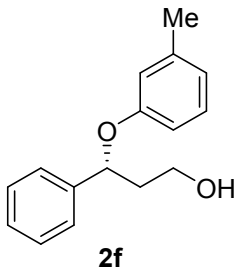
2e



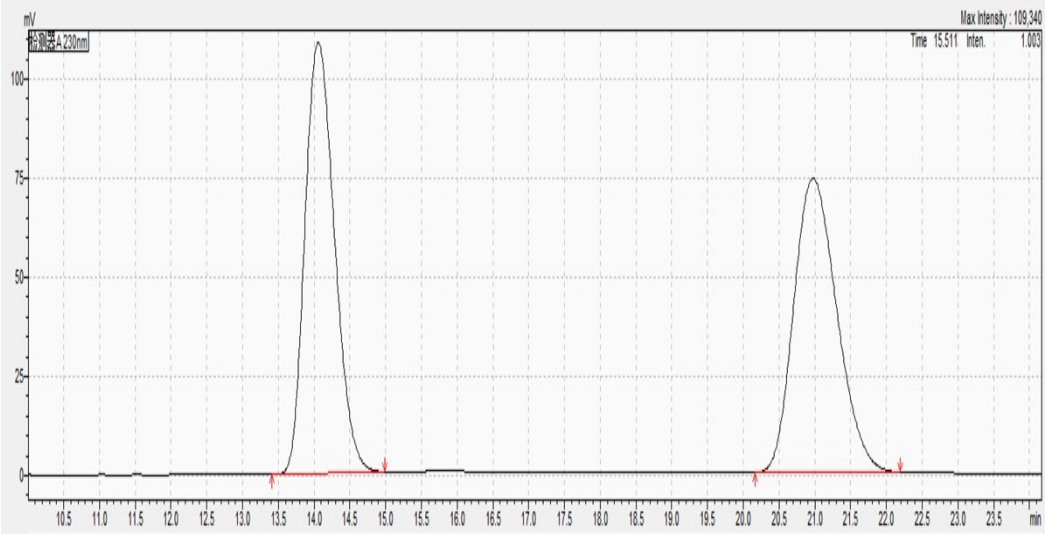
Peak	Rentention Time/min	Area %
1	14.687	49.736
2	20.908	50.264



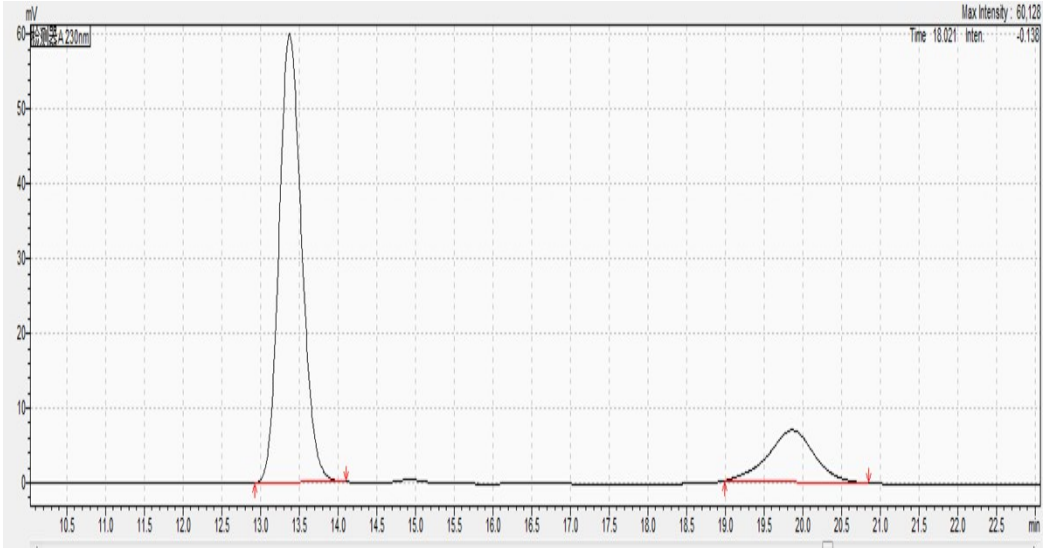
Peak	Rentention Time/min	Area %
1	14.859	90.582
2	21.021	9.418



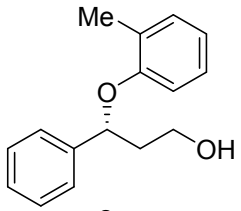
2f



Peak	Rentention Time/min	Area %
1	14.058	50.131
2	20.976	49.869



Peak	Rentention Time/min	Area %
1	13.375	80.498
2	19.864	19.502



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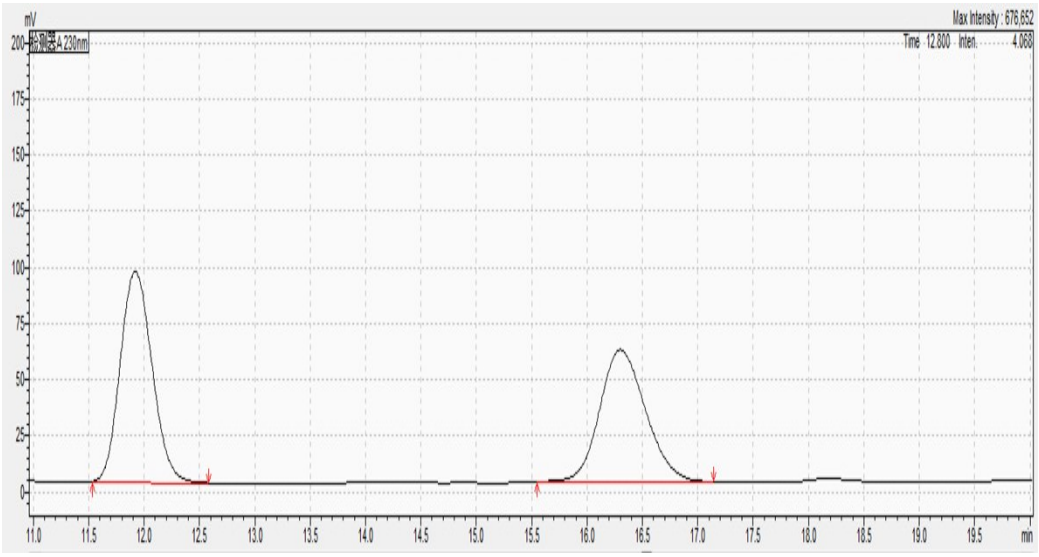
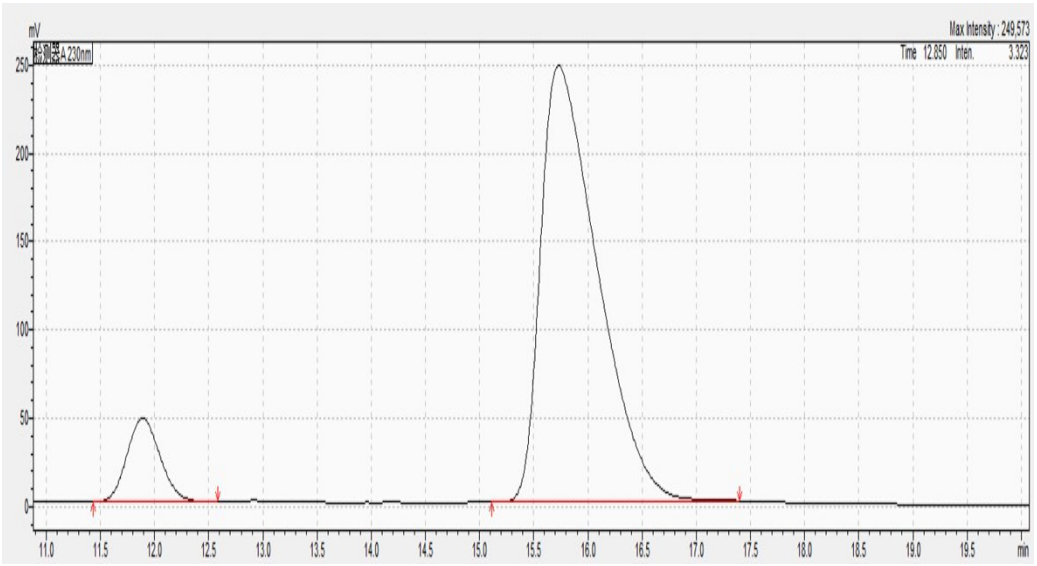
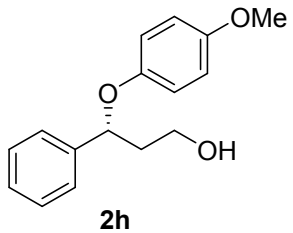


Peak	Rentention Time/min	Area %
1	11.920	50.842
2	16.301	49.158



Peak	Rentention Time/min	Area %
1	11.889	9.683
2	15.730	90.317



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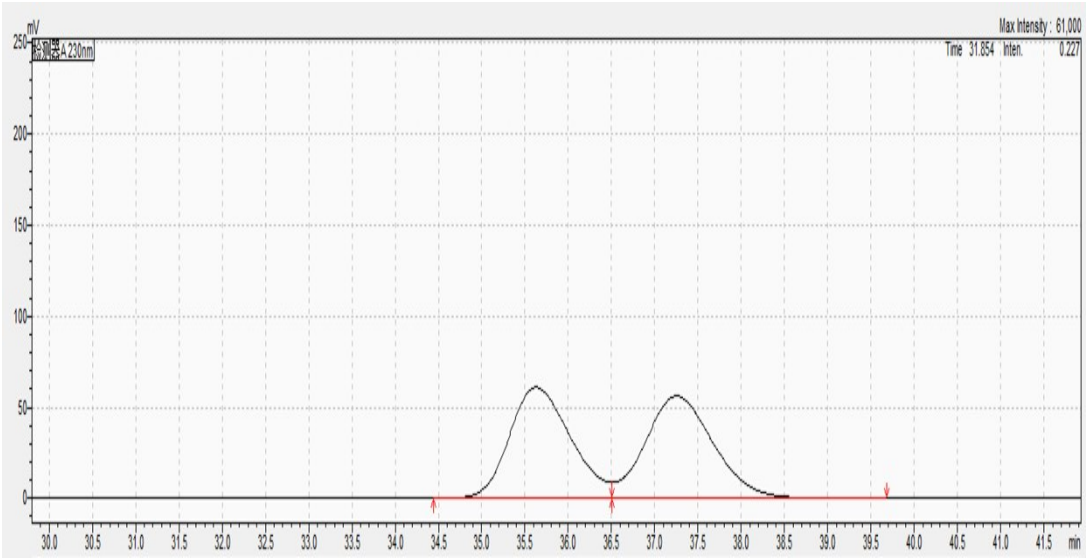
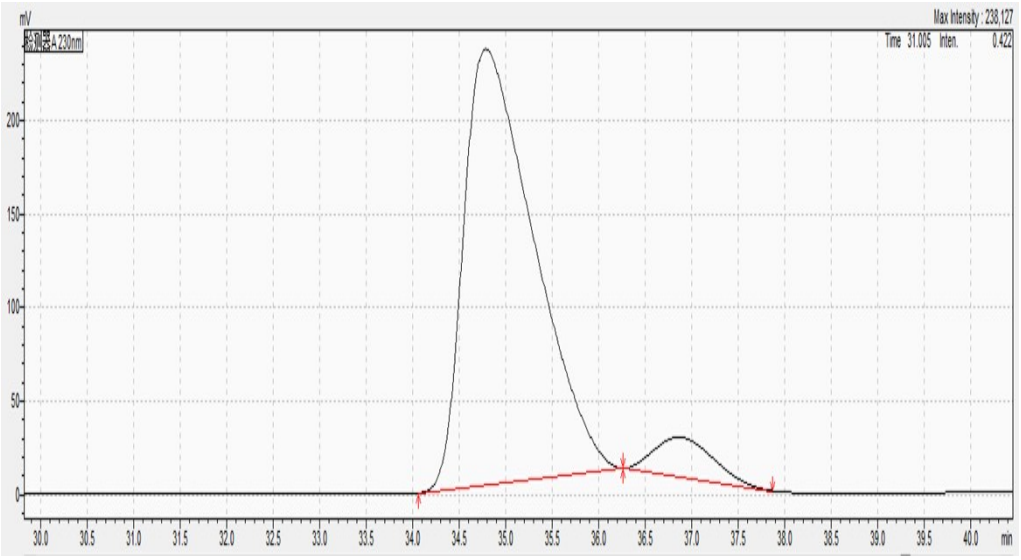
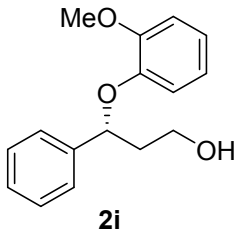


Peak	Rentention Time/min	Area %
1	35.628	49.477
2	37.252	50.523



Peak	Rentention Time/min	Area %
1	34.783	88.710
2	36.857	11.290



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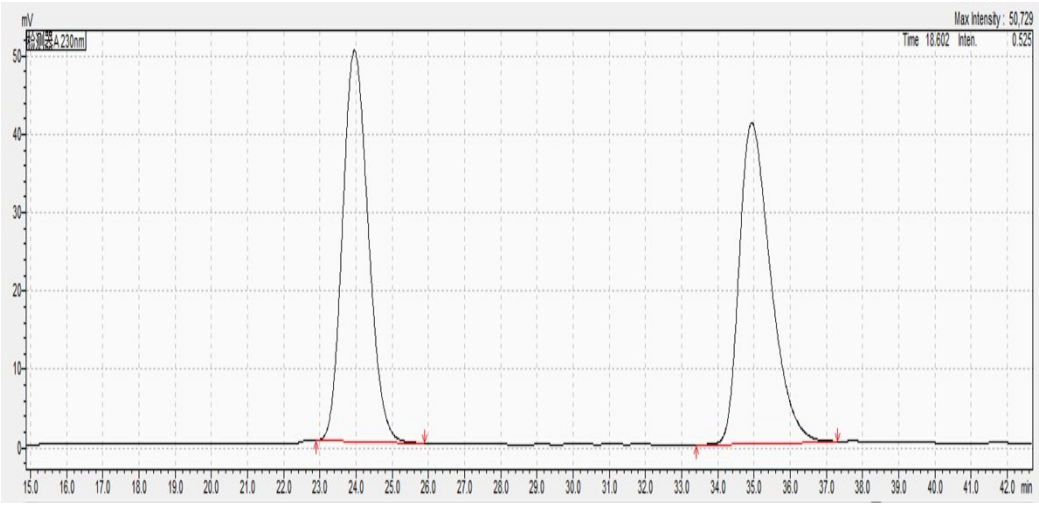
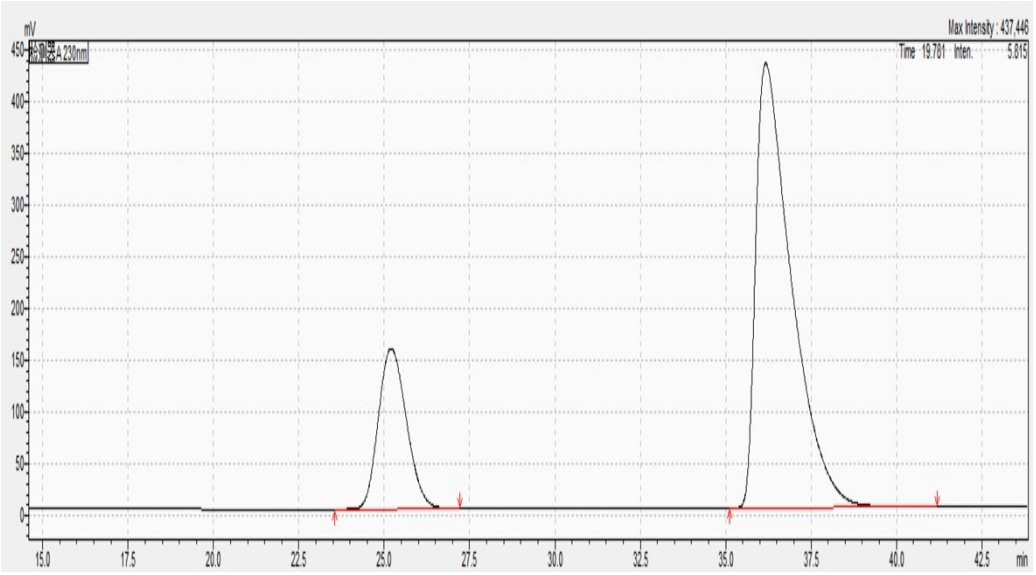
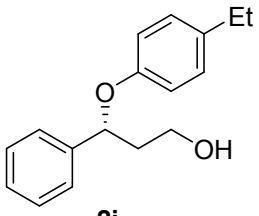


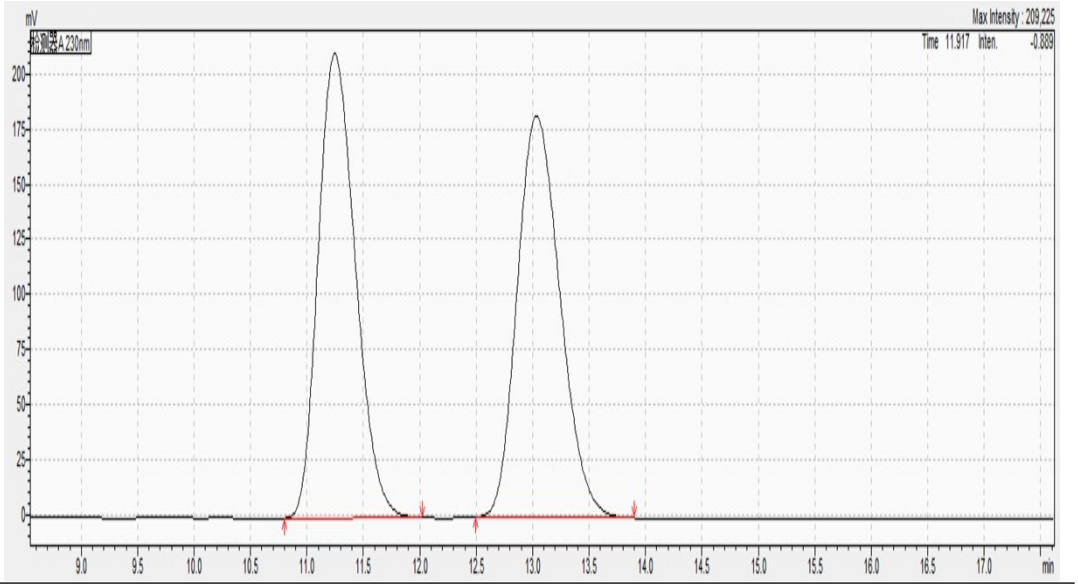
Peak	Rentention Time/min	Area %
1	23.953	50.020
2	34.936	49.980



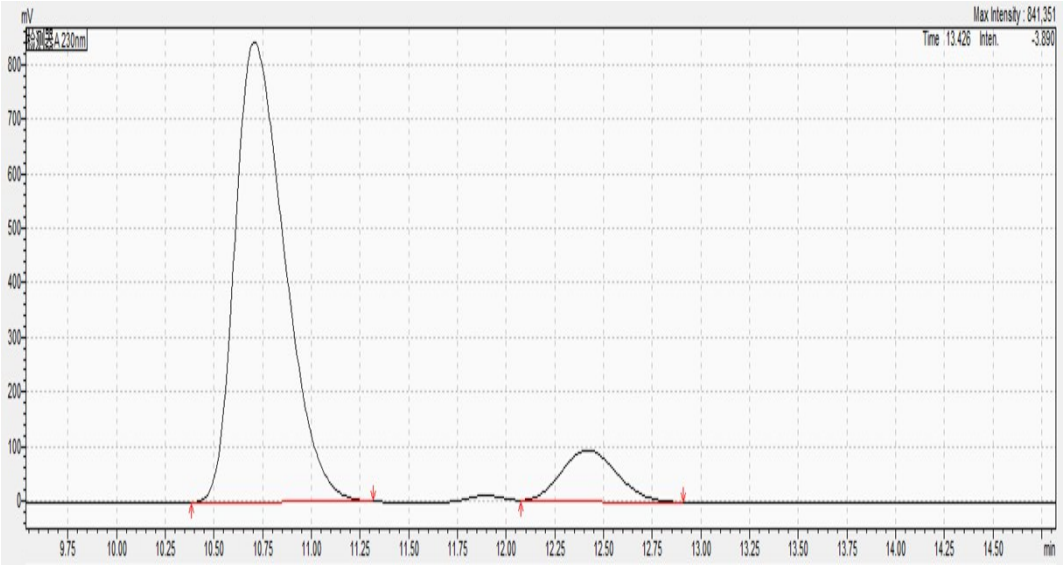
Peak	Rentention Time/min	Area %
1	25.203	22.097
2	36.174	77.903



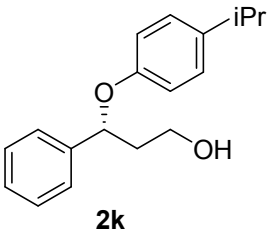
2j



Peak	Rentention Time/min	Area %
1	11.246	49.872
2	13.035	50.128



Peak	Rentention Time/min	Area %
1	10.708	89.254
2	12.417	10.746



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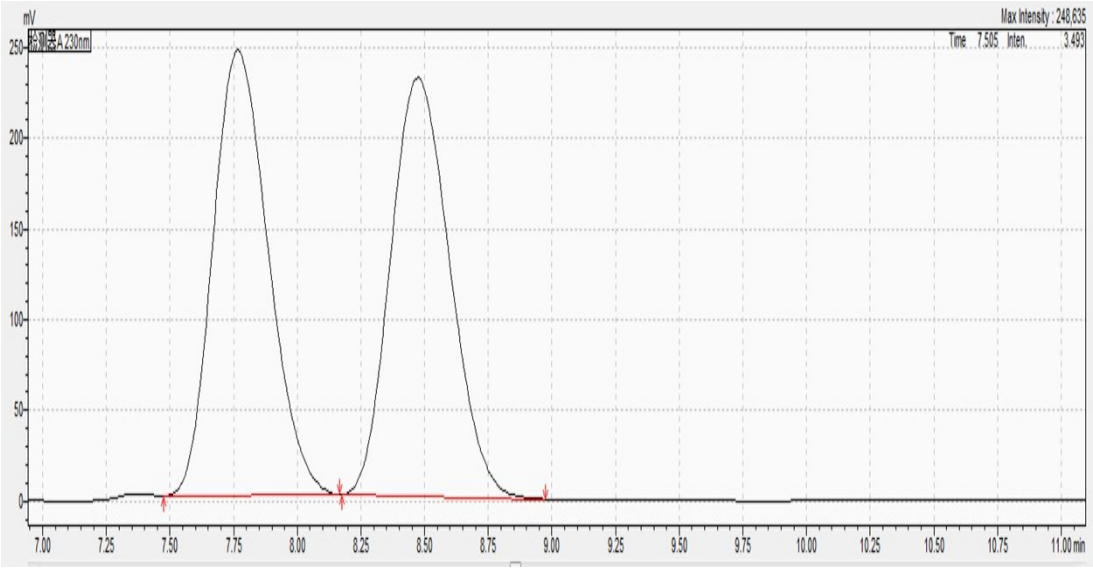
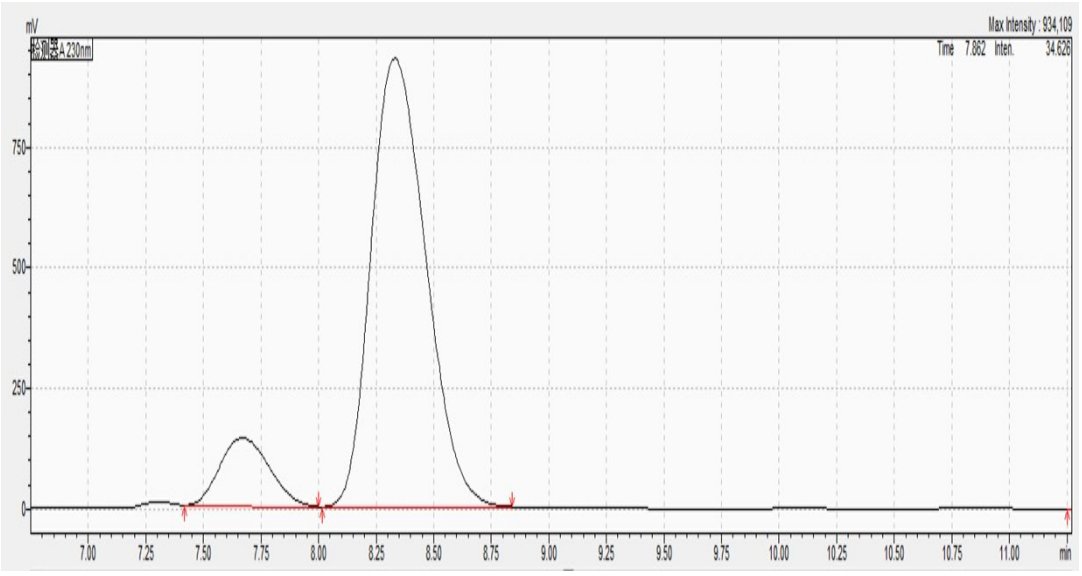
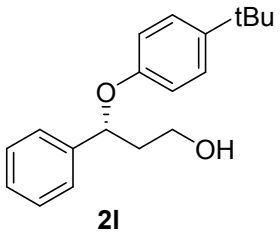


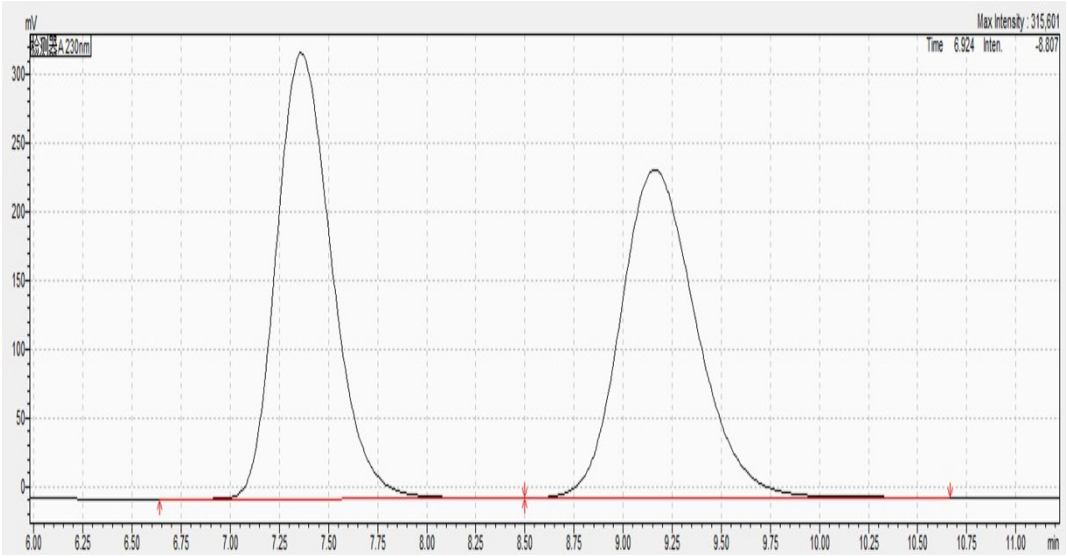
Peak	Rentention Time/min	Area %
1	7.766	49.688
2	8.473	50.312



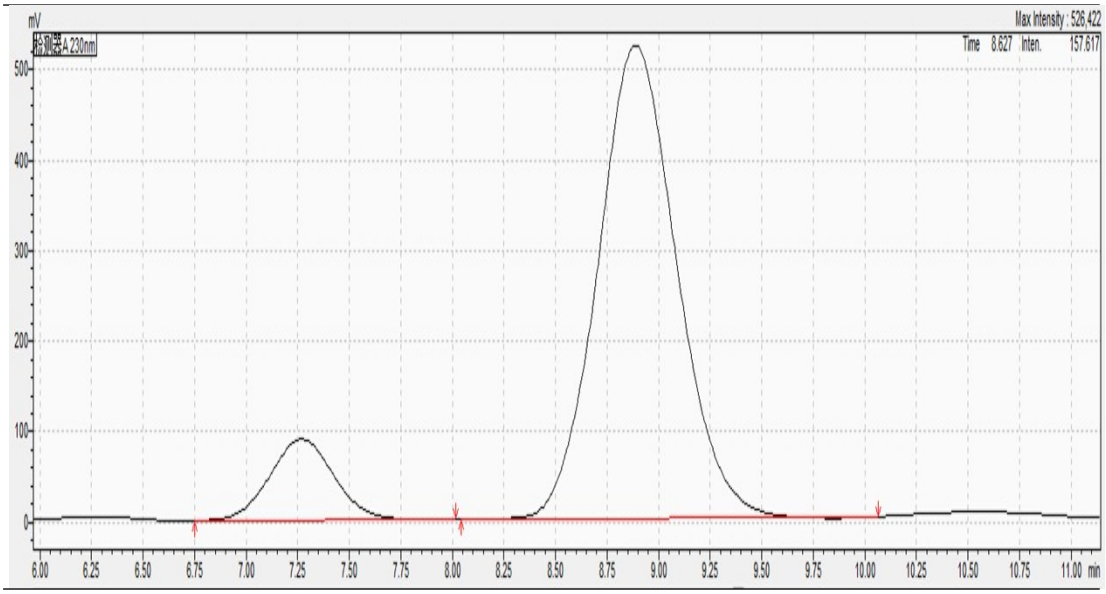
Peak	Rentention Time/min	Area %
1	7.670	11.867
2	8.331	88.133



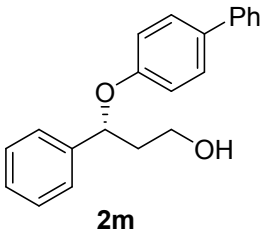
2l



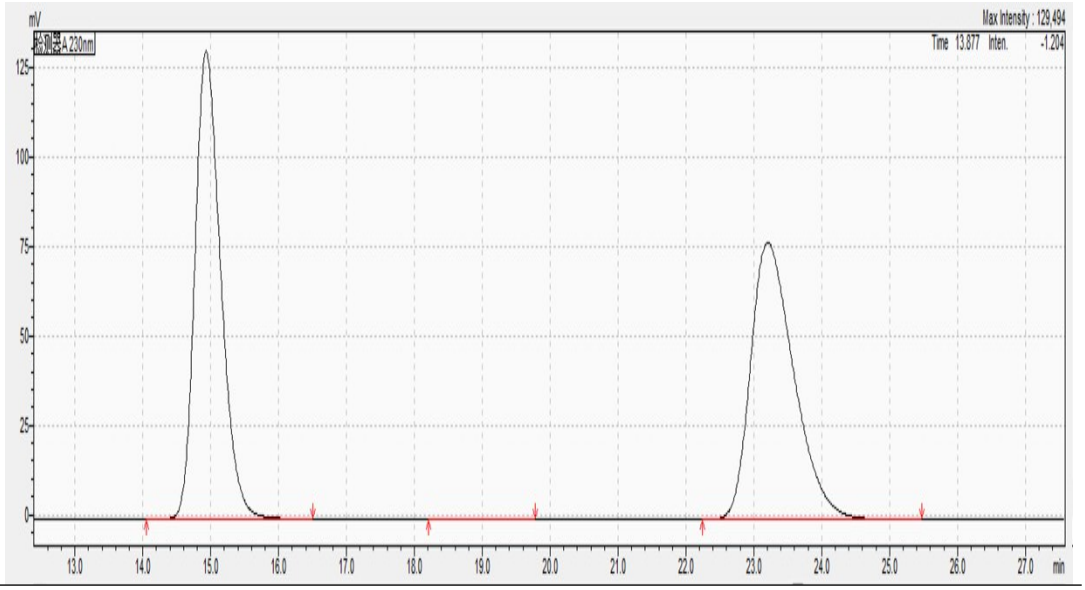
Peak	Rentention Time/min	Area %
1	7.361	49.930
2	9.162	50.070



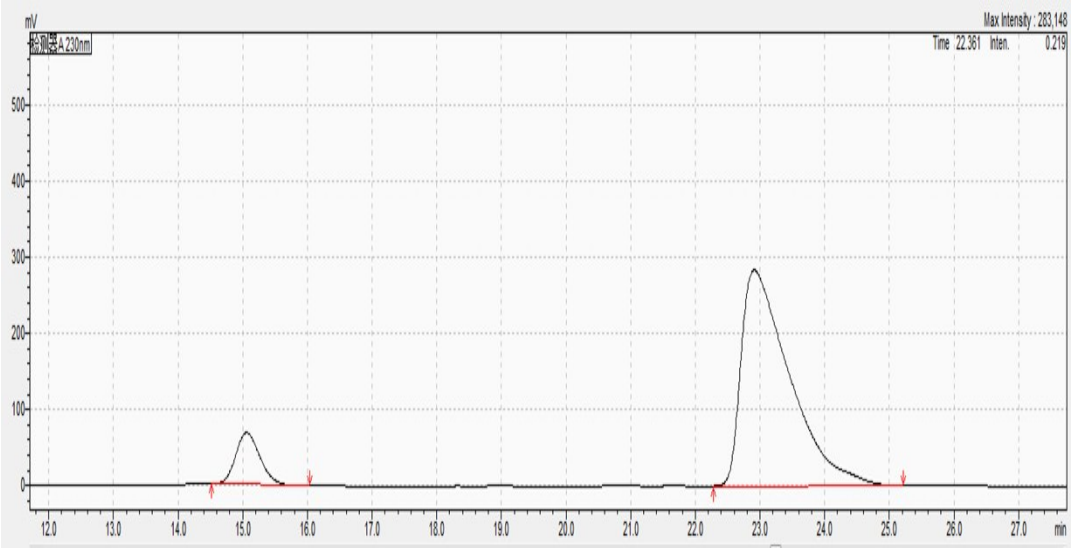
Peak	Rentention Time/min	Area %
1	7.270	11.945
2	8.887	88.055



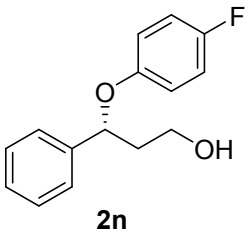
2m



Peak	Rentention Time/min	Area %
1	14.932	49.997
2	23.206	50.003



Peak	Rentention Time/min	Area %
1	15.058	10.600
2	22.911	89.400



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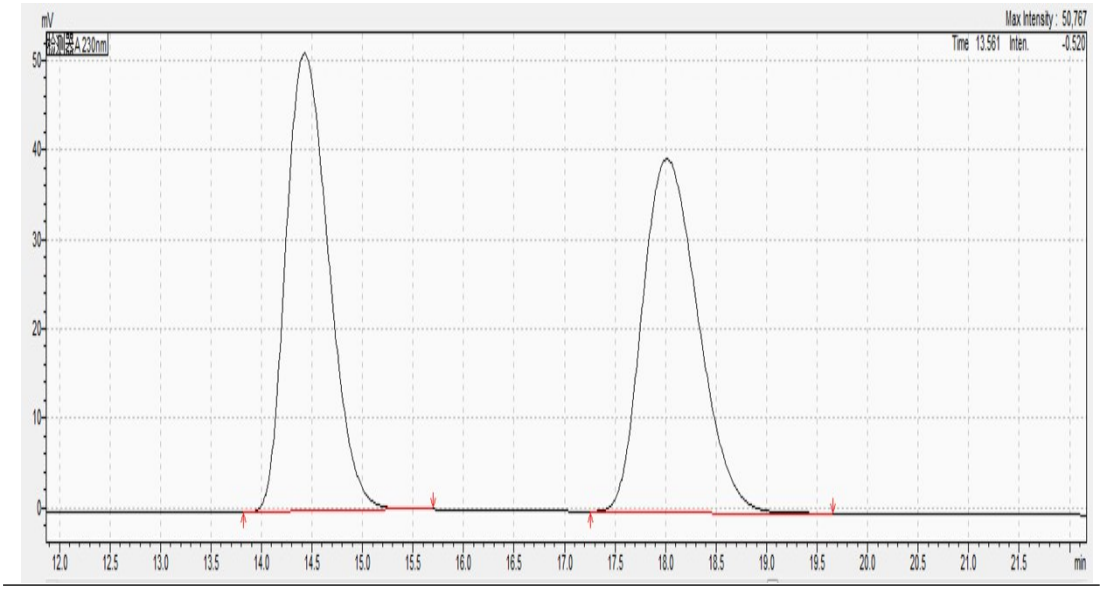
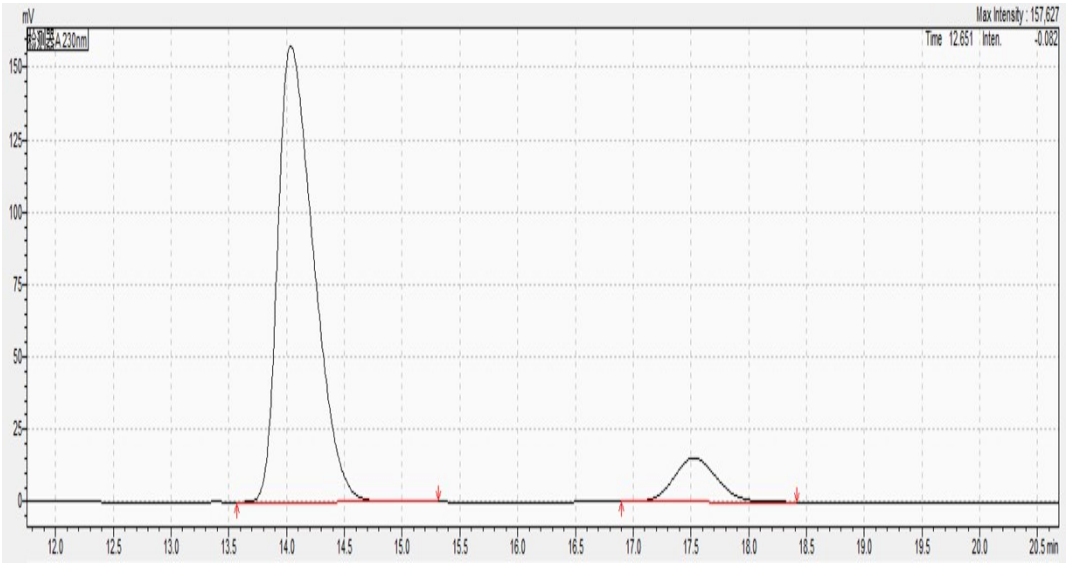
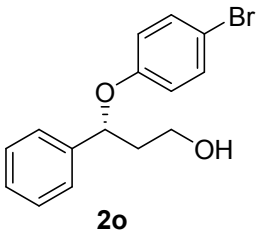


Peak	Rentention Time/min	Area %
1	14.428	49.971
2	18.015	50.029



Peak	Rentention Time/min	Area %
1	14.037	90.026
2	17.528	9.974



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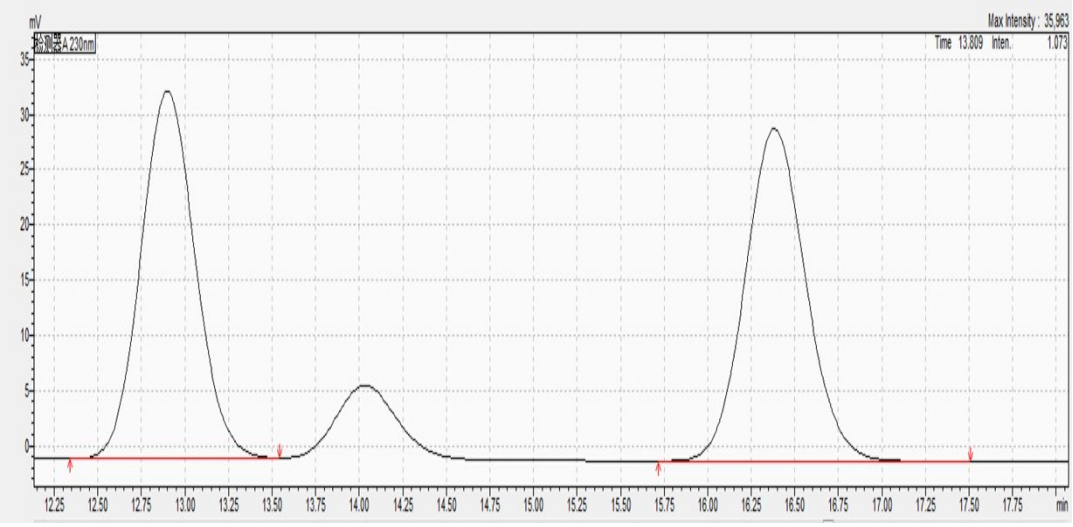
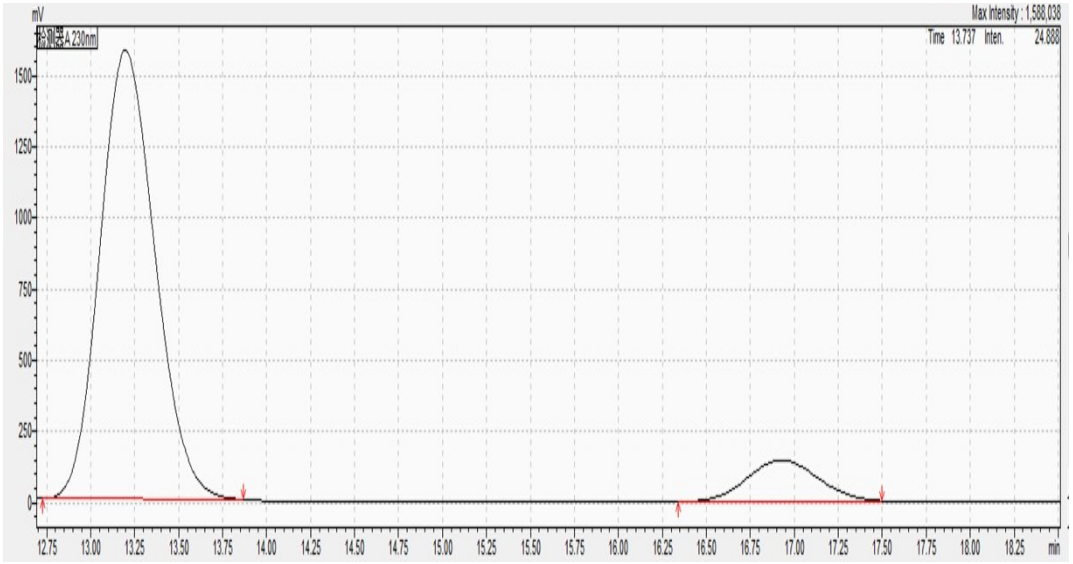
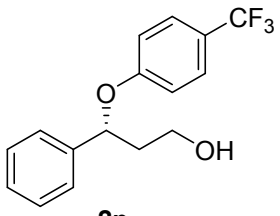


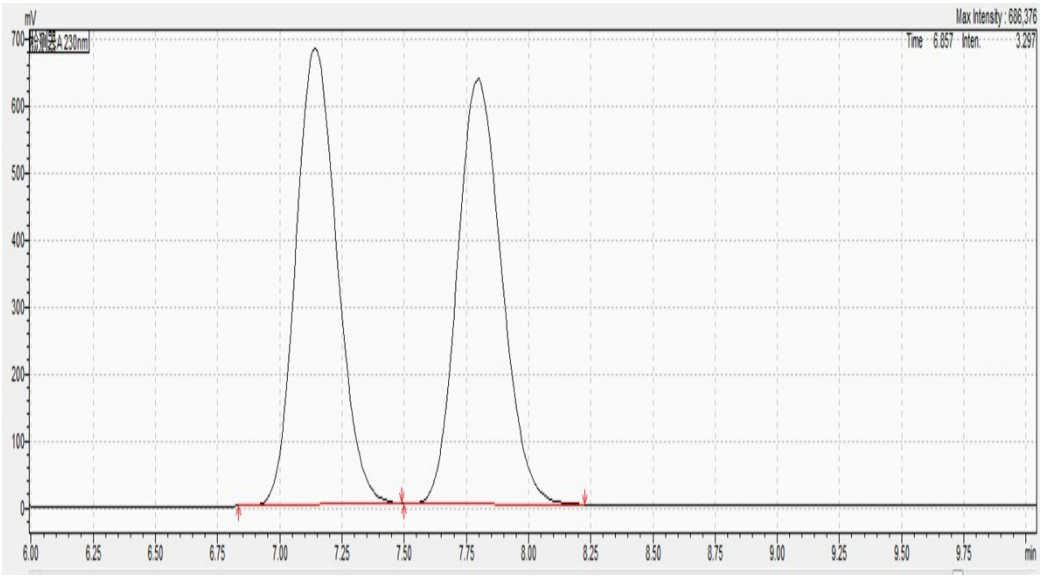
Peak	Rentention Time/min	Area %
1	12.898	50.216
2	16.383	49.784



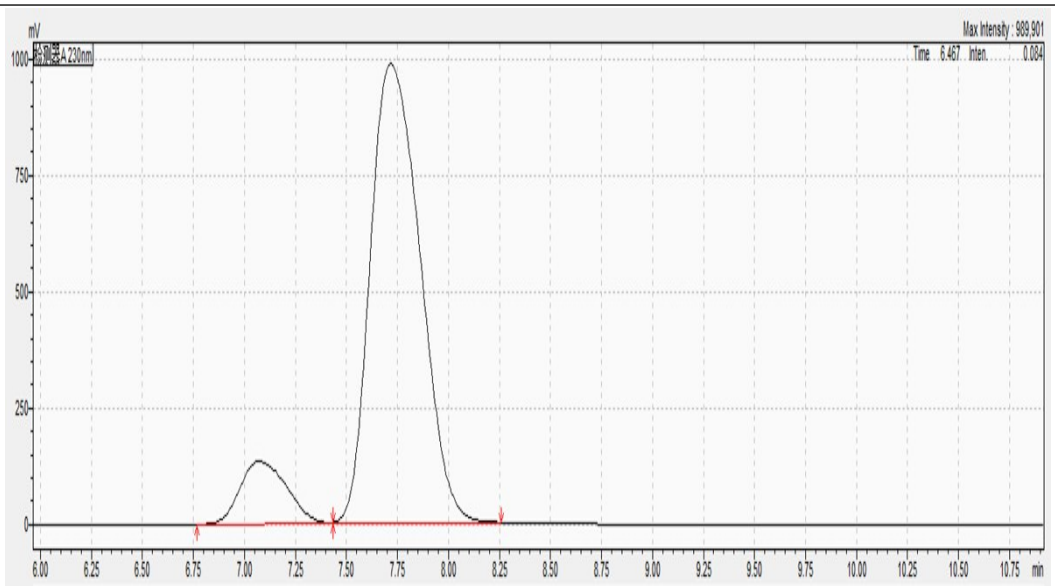
Peak	Rentention Time/min	Area %
1	13.197	89.737
2	16.928	10.263



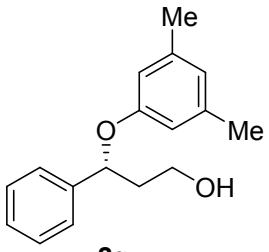
2p



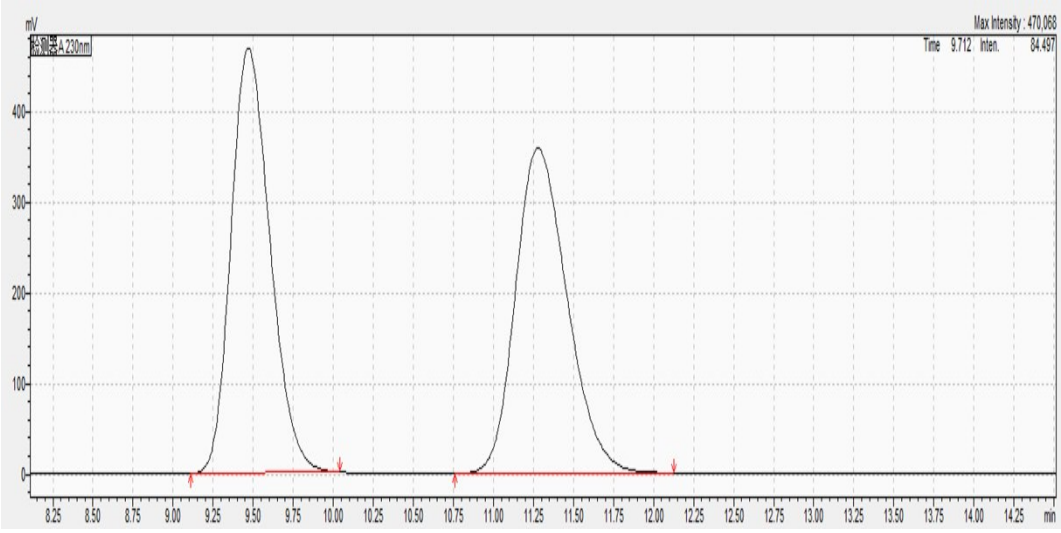
Peak	Rentention Time/min	Area %
1	7.141	49.818
2	7.796	50.182



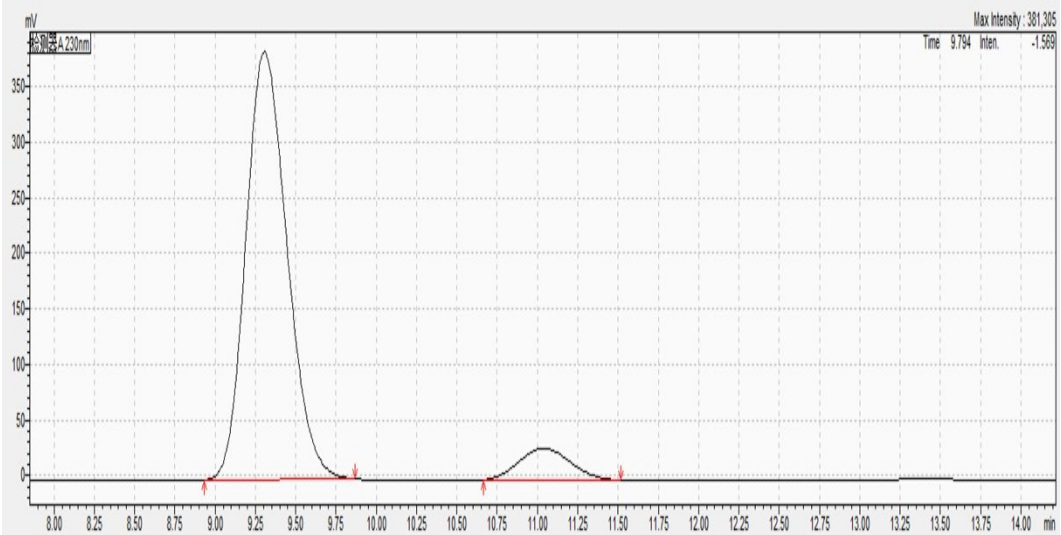
Peak	Rentention Time/min	Area %
1	7.073	10.865
2	7.718	89.135



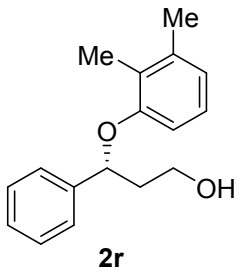
2q



Peak	Rentention Time/min	Area %
1	9.470	49.977
2	11.279	50.023



Peak	Rentention Time/min	Area %
1	9.306	91.484
2	11.039	8.516



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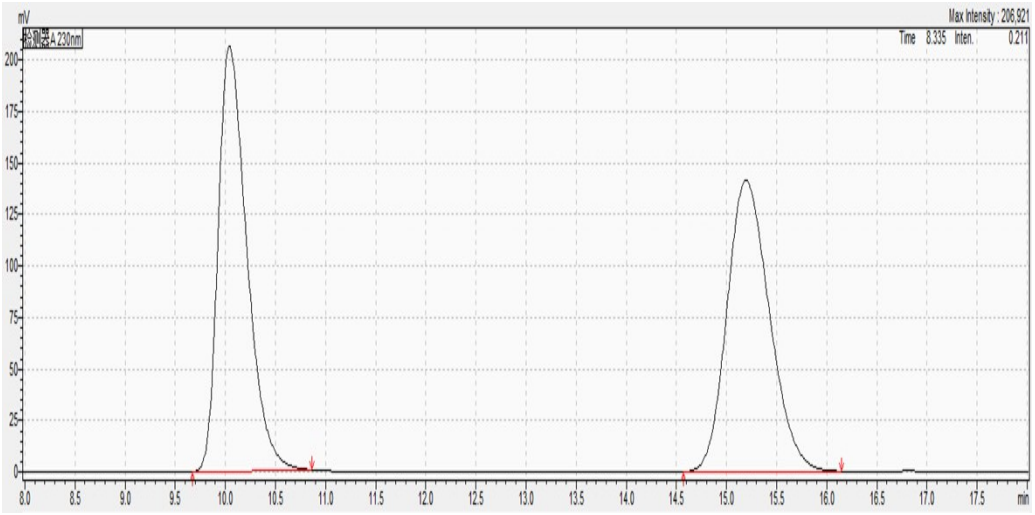
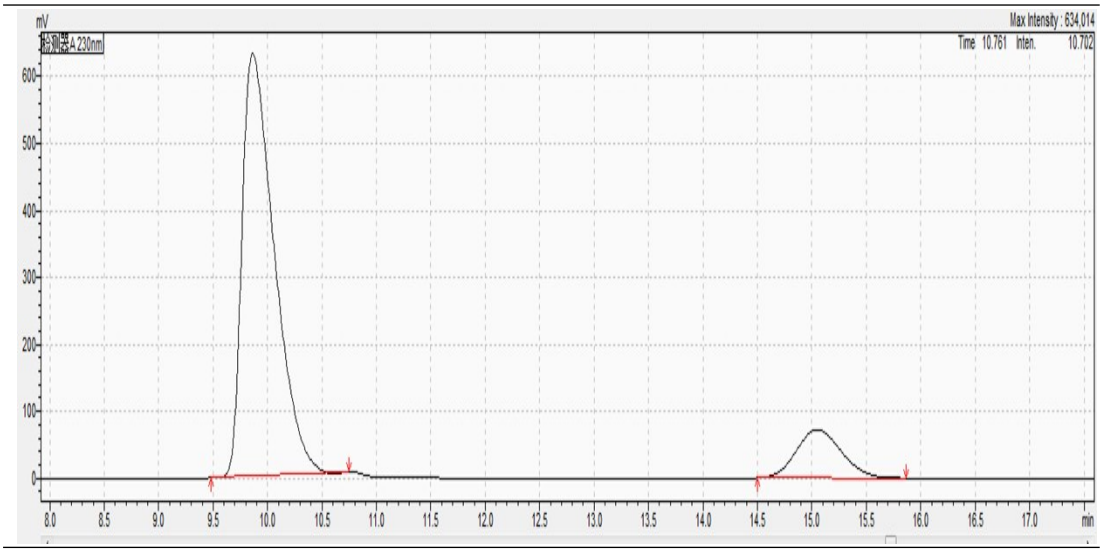
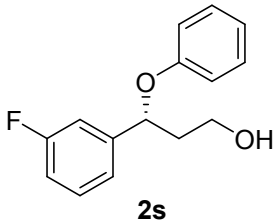


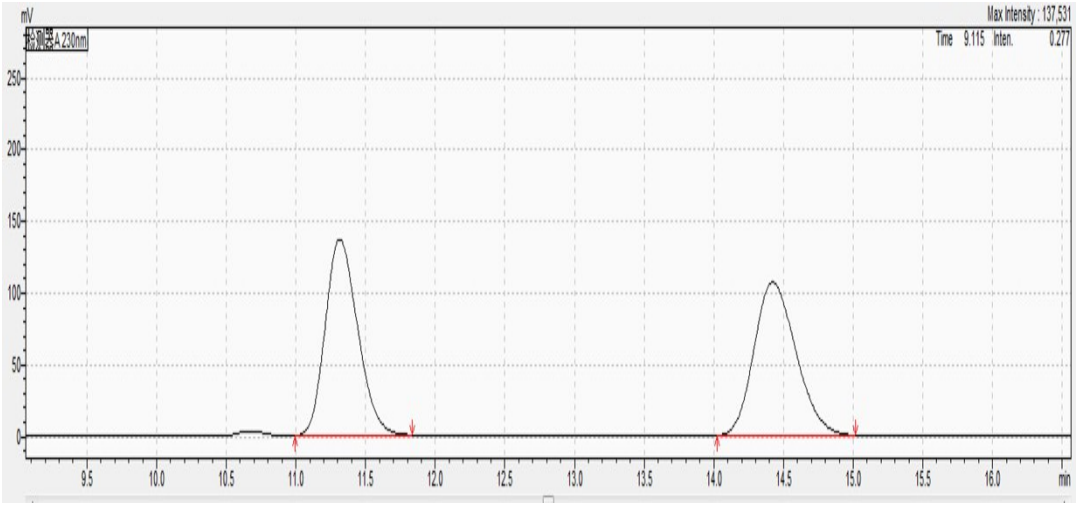
Peak	Rentention Time/min	Area %
1	10.042	49.784
2	15.194	50.216



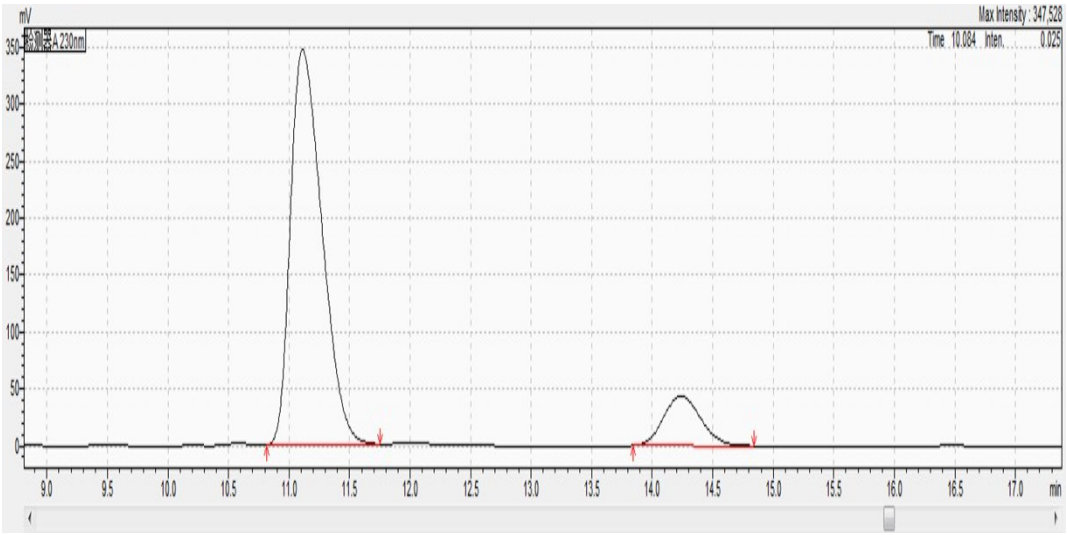
Peak	Rentention Time/min	Area %
1	9.864	86.749
2	15.050	13.251



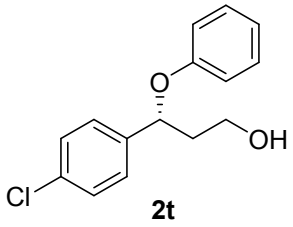
2s



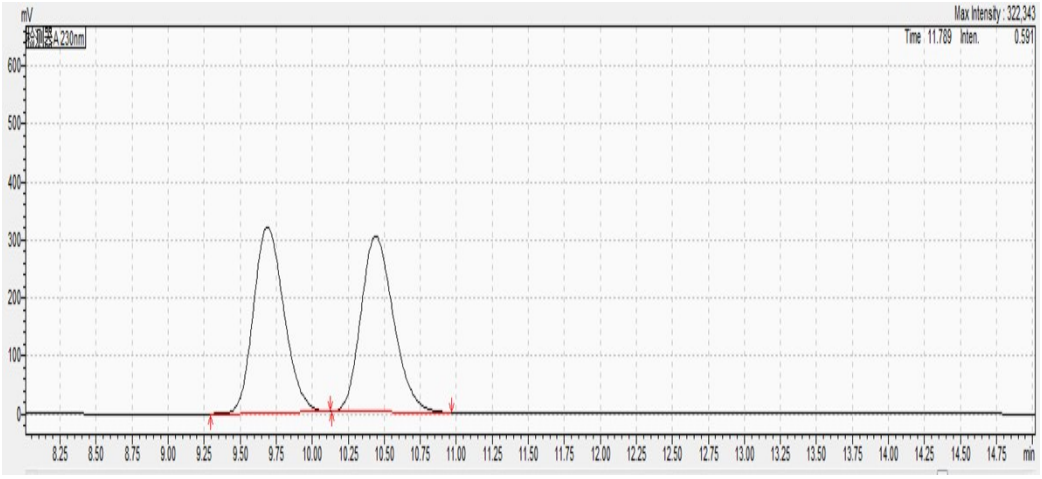
Peak	Rentention Time/min	Area %
1	11.314	49.671
2	14.422	50.329



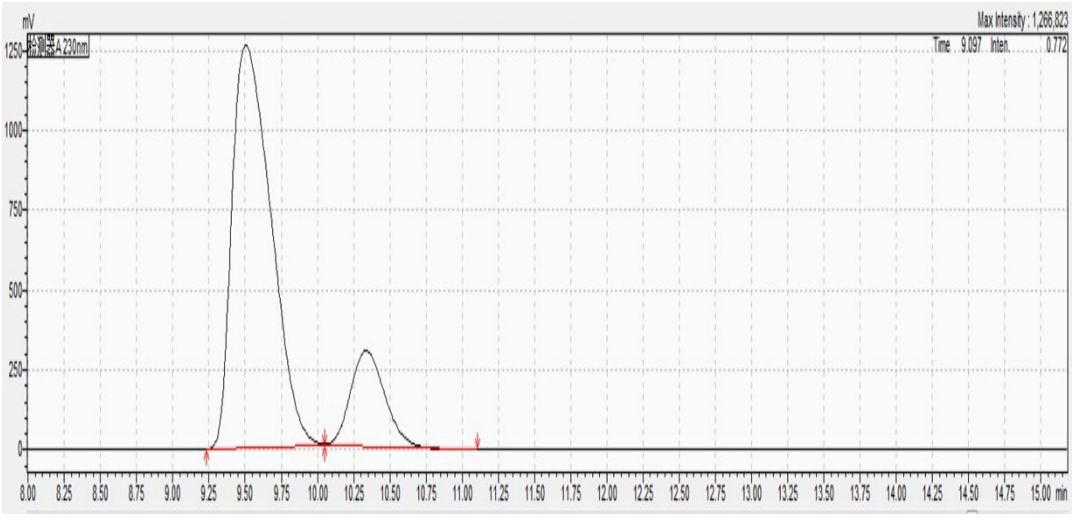
Peak	Rentention Time/min	Area %
1	11.112	87.077
2	14.237	12.923



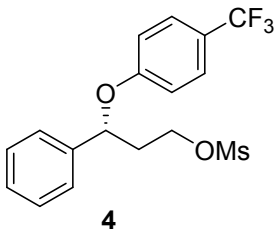
2t



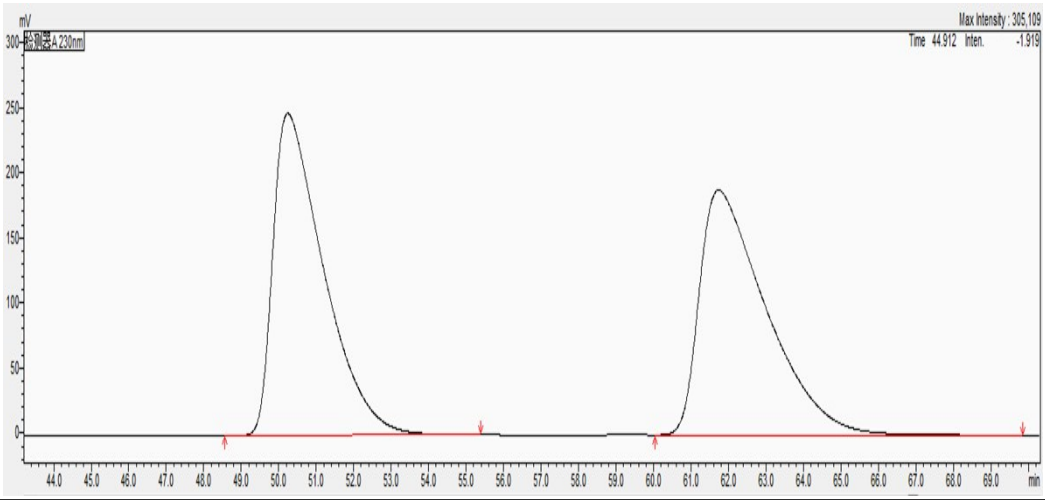
Peak	Rentention Time/min	Area %
1	9.689	49.628
2	10.440	50.372



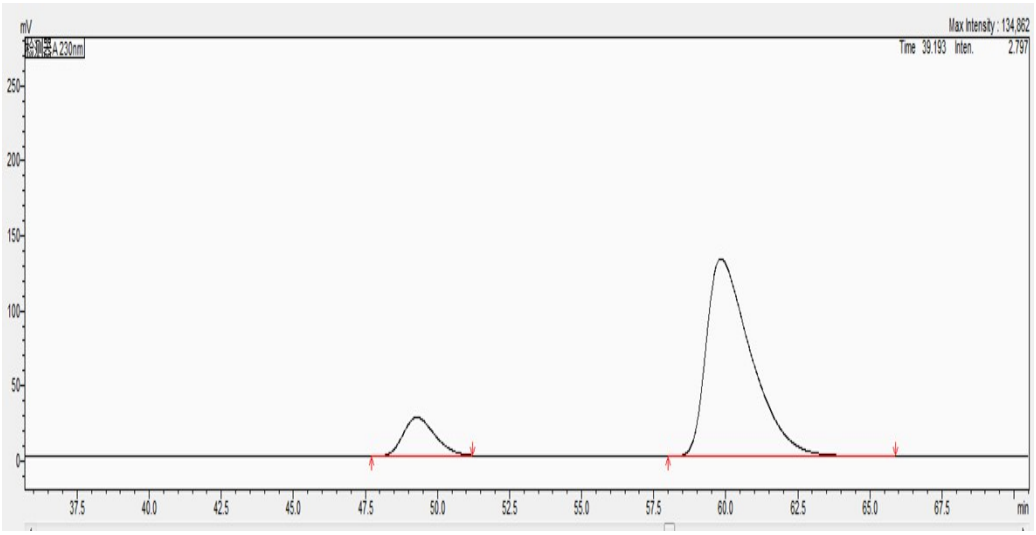
Peak	Rentention Time/min	Area %
1	9.504	82.845
2	10.335	17.155



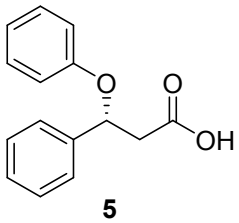
4



Peak	Rentention Time/min	Area %
1	50.243	49.896
2	61.737	50.104



Peak	Rentention Time/min	Area %
1	49.287	12.222
2	59.837	87.778



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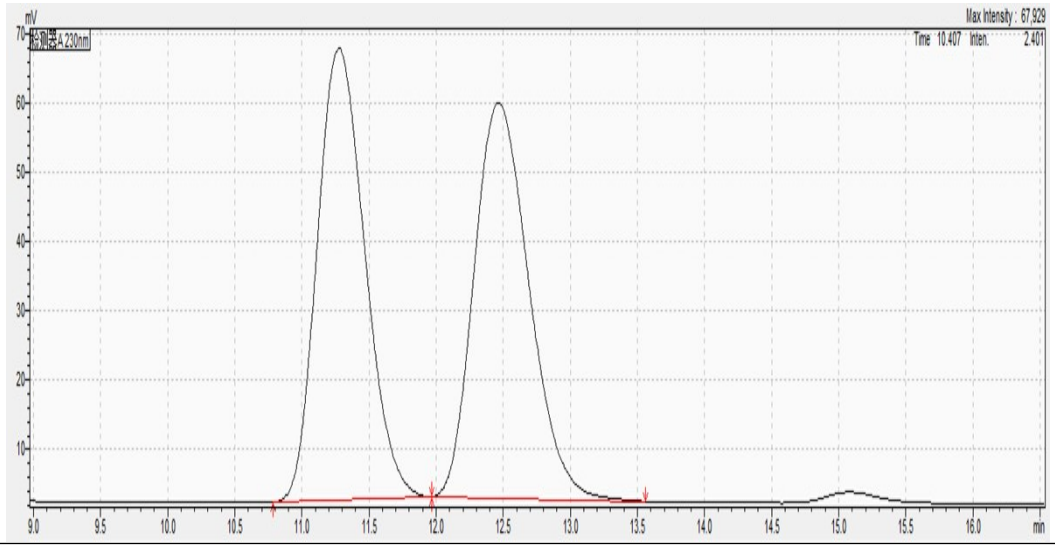
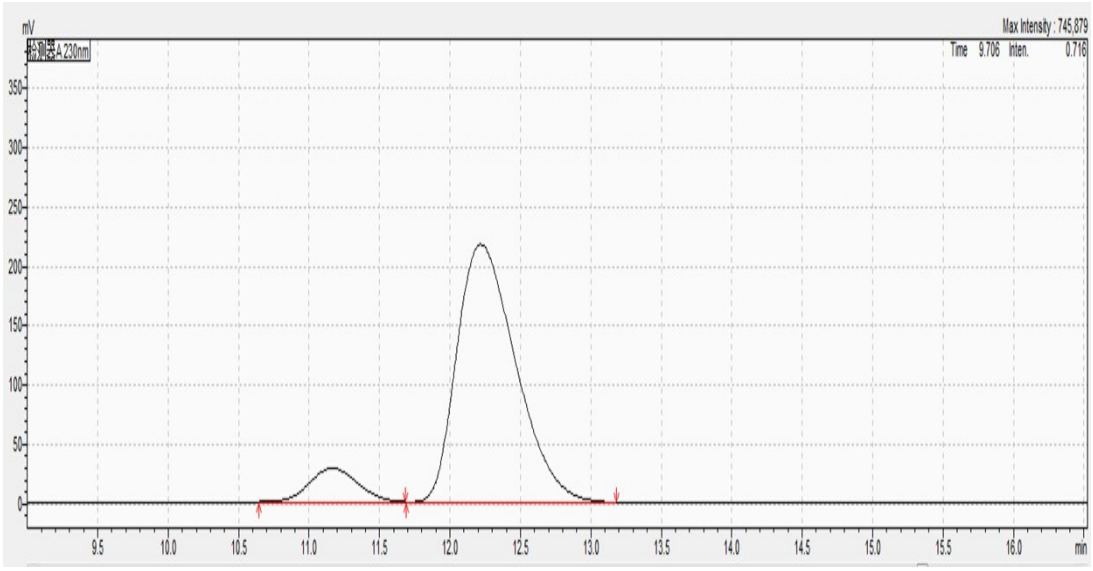


Peak	Rentention Time/min	Area %
1	11.276	49.843
2	12.465	50.157



Peak	Rentention Time/min	Area %
1	11.164	9.484
2	12.218	90.516