Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information for:

Rhodium-Catalyzed Asymmetric Hydroboration of γ,δ-Unsaturated Amide Derivatives: δ-Borylated Amides

Gia L. Hoang,[‡] Shuyang Zhang[‡] and James M. Takacs*[‡]

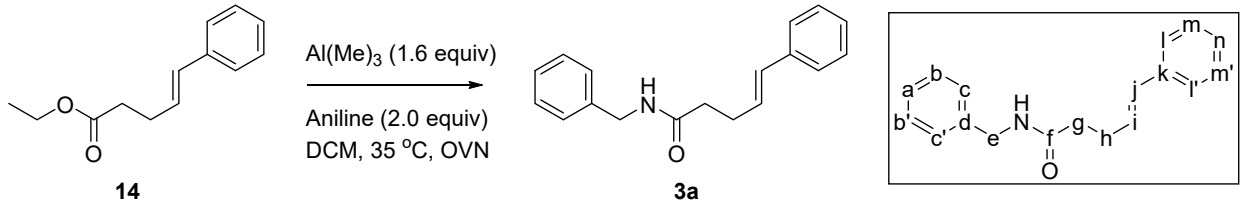
[‡] Department of Chemistry, University of Nebraska–Lincoln, Lincoln, NE 68588, USA

* jtakacs1@unl.edu

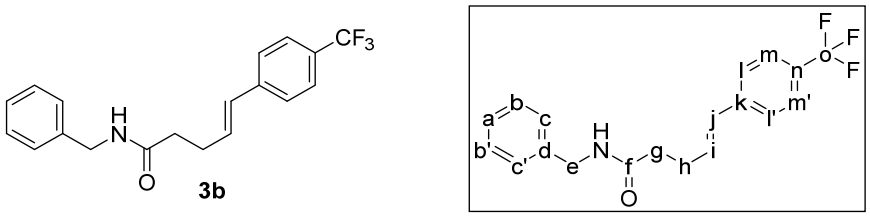
Table of Contents

General procedures. Reactions were carried out in a dry nitrogen atmosphere. Tetrahydrofuran (THF) was freshly distilled under sodium metal and benzophenone. HPLC solvents were filtered through Millipore filter paper. 4,4,5,5-tetramethyl-1,3,2-dioxaborolane (pinBH) was distilled immediately before use. All synthesized compounds were purified with flash chromatography using EMD Silica Gel 60 Geduran®, distilled via short path distillation, or triturated. Thin Layer Chromatography analyses were performed on Analtech Silica Gel HLF (0.25 mm) precoated analytical plates and visualized with use of handheld short wavelength UV light, Iodine stain (I_2 and EMD Silica Gel 60 Geduran®) and Vanillin stain (Ethanol, H_2SO_4, and vanillin). HPLC analyses were performed with use of an ISCO model 2360 HPLC and Chiral Technologies, Inc. chiral HPLC columns (Chiralcel-AD, column: 250 x 4.6 mm; Chiralcel-OD, column: 250 x 4.6 mm; Chiralpak-IC, column: 250 x 4.6 mm) and monitored with UV-VIS detector (Shimadzu SPD-10A$_{VP}$/10A$_{VP}$, λ = 210 nm). Data were recorded and analyzed with ChromPerfect chromatography software (version 5.1.0). NMR spectra were recorded on 400 MHz and 300 MHz Bruker Advance NMR spectrometers using residue $CHCl_3$ (δ 7.27 ppm) or $CDCl_3$ (δ 77.0 ppm) for reference unless otherwise specified. Peaks are expressed as m (unresolved multiplet), q (quartet), t (triplet), d (doublet) or s (singlet). IR spectra were recorded using an Avatar 360 FT-IR. Optical rotations were measured as solutions, 1.0 g/100 mL in chloroform or methanol unless indicated otherwise, and recorded using an Autopol III automatic polarimeter. HRMS analyses were performed by the Nebraska Center for Mass Spectrometry. Ligands **B1–5** are prepared as previously reported.[1]

General procedure for the preparation of (*E*)-γ,δ-unsaturated amides via trimethylaluminum mediated amide bond formation (GP1)

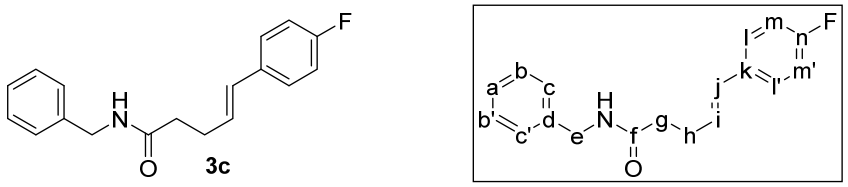


(*E*)-5-phenyl-4-pentenecarboxylic acid benzyl amide (3a). To a solution of benzyl amine (2 equiv, 10 mmol, 1.1 mL) in DCM (20 mL) was slowly added trimethylaluminum (1.62 equiv, 2.0 M in hexanes, 8.1 mmol, 4.05 mL) at room temperature. The resulting mixture was stirred at room temp for 30 mins, and the corresponding ethyl ester ((*E*)-5-phenyl-4-pentenecarboxylic acid ethyl ester, (*E*)-**14**, (5 mmol, 1.02 g)) was added dropwise. The resultant mixture was heated overnight at 35 °C. After cooling to room temperature, the reaction mixture was quenched with HCl (1M) and extracted with DCM (3 x 20 mL). The combined organic extracts were dried (anhyd. Na_2SO_4) and concentrated under reduced pressure. Flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (1.05 g, 79%) as a white solid: m.p. 85.0–86.5 °C; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (10H, m, a,b,b',c,c',l,l',m,m',n), 6.46 (1H, d, *J* = 15.8 Hz, j), 6.22 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 6.07 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 2.55–2.65 (2H, m, h), 2.39 (2H, t, *J* = 7.4 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.10 (f), 138.40 (d), 137.42 (k), 131.30 (j), 128.81 (b,b'), 128.75 (i), 128.64 (l,l'), 127.90 (m,m'), 127.59 (n), 127.30 (a), 126.20 (c,c'), 43.72 (e), 36.51 (g), 29.13 (h); IR (neat) 3292 (N-H stretch), 3062, 3029, 1635 (C=O stretch), 1545, 1492, 1445 (C-N stretch), 1223, 967, 732, 671 cm^{-1}; HRMS (EI) calcd. for $C_{18}H_{19}NO$ (M): 265.1467, found 265.1476 *m/z*.

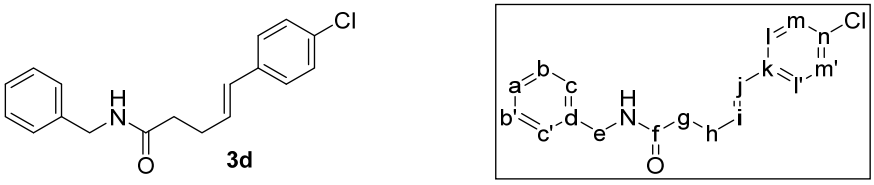


(*E*)-5-(4-trifluoromethyl)phenyl-4-pentenecarboxylic acid benzyl amide (3b). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(4-trifluoromethyl)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.36 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title

compound (1.18 g, 71%) as a white solid: m.p. 127.0–128.0 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{19}F NMR (376 MHz, $CDCl_3$) δ –62.44 (CF_3); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (2H, d, *J* = 8.2 Hz, l,l'), 7.41 (2H, d, *J* = 8.2 Hz, m,m'), 7.25–7.35 (5H, m, a,b,b',c,c'), 6.49 (1H, d, *J* = 15.8 Hz, j), 6.34 (1H, dt, *J* = 15.8 and 6.8 Hz, i), 5.81 (1H, br s, NH), 4.48 (2H, d, *J* = 5.7 Hz, e), 2.64 (2H, q, *J* = 7.1 Hz, h), 2.42 (2H, t, *J* = 7.3 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.75 (f), 140.90 (k), 138.32 (d), 131.62 (aryl), 130.08 (aryl), 128.83 (aryl), 127.92 (aryl), 127.68 (aryl), 126.31 (aryl), 125.57 (q, *J* = 4,1 Hz, aryl), 43.76 (e), 36.17 (g), 29.04 (h); IR (neat) 3292 (N-H stretch), 1634 (C=O stretch), 1603, 1542, 1327, 1165, 1109, 1068, 742, 691 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{18}F_3NO$ (M): 333.1340, found 333.1356 *m/z*.

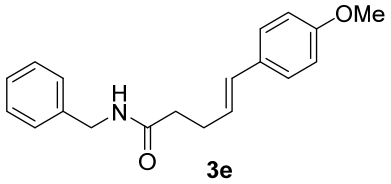


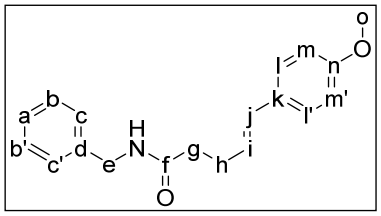
(*E*)-5-(4-fluoro)phenyl-4-pentenecarboxylic acid benzyl amide (3c). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(4-fluoro)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.11 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (1.06 g, 75%) as a white solid: m.p. 92.5–93.5 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{19}F NMR (376 MHz, $CDCl_3$) δ –115.10 to –115.25 (m, F); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.30 (7H, m, a,b,b',c,c',l,l'), 6.99 (2H, t, *J* = 8.7 Hz, m,m'), 6.41 (1H, d, *J* = 15.8 Hz, j), 6.13 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.99 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 2.58 (2H, q, *J* = 7.2 Hz, h), 2.39 (2H, t, *J* = 7.4 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.08 (f), 162.18 (d, *J* = 244 Hz, n), 138.40 (d), 133.61 and 133.58 (k), 130.10 (j), 128.80 (b,b'), 128.50 and 128.48 (i), 127.88 (l,l'), 127.68 (a), 127.60 (c,c'), 115.48 (d, *J* = 22 Hz, m,m'), 43.70 (e), 36.43 (g), 29.05 (h); IR (neat) 3286 (N-H stretch), 2917, 1632 (C=O stretch), 1544, 1507, 1224, 1157, 968, 849, 810, 738, 694 cm^{-1}; HRMS (EI) calcd. for $C_{18}H_{18}FNO$ (M): 283.1372, found 283.1383 *m/z*.



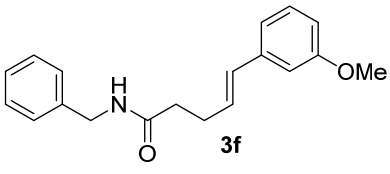
(*E*)-5-(4-chloro)phenyl-4-pentenecarboxylic acid benzyl amide (3d). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(4-chloro)phenyl-4-pentenecarboxylic acid ethyl ester (5

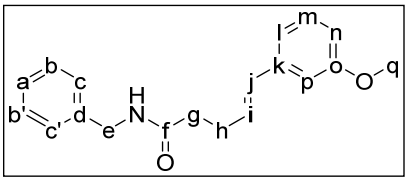
mmol, 1.19 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (1.17 g, 77%) as a white solid: m.p. 122.5–124.5 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.30 (9H, m, a,b,b',c,c',l,l',m,m'), 6.39 (1H, d, *J* = 15.8 Hz, j), 6.19 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 6.00 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 2.59 (2H, q, *J* = 7.0 Hz, h), 2.39 (2H, t, *J* = 7.4 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.00 (f), 138.39 (n), 135.93 (d), 132.83 (k), 130.07 (j), 129.52 (i), 128.81 (l,l'), 128.75 (b,b'), 127.89 (m,m'), 127.62 (a), 127.40 (c,c'), 43.71 (e), 36.30 (g), 29.06 (h); IR (neat) 3294 (N-H stretch), 1634 (C=O stretch), 1548, 1489, 1093, 1010, 970, 851, 806, 745, 696 cm^{-1}; HRMS (EI) calcd. for $C_{18}H_{18}ClNO$ (M): 299.1077, found 299.1082 *m/z*.



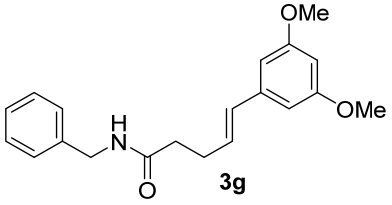


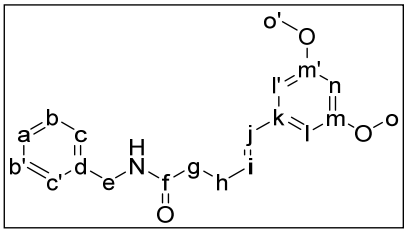
(*E*)-5-(4-methoxy)phenyl-4-pentenecarboxylic acid benzyl amide (3e). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(4-methoxy)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.17 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (1.11 g, 75%) as a white solid: m.p. 123.5–124.5 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.30 (7H, m, a,b,b',c,c',l,l'), 6.85 (2H, d, *J* = 8.7 Hz ,m,m'), 6.39 (1H, d, *J* = 15.8 Hz, j), 6.07 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 4.44 (2H, d, *J* = 5.8 Hz, e), 3.82 (3H, s, o), 2.57 (2H, q, *J* = 7.3 Hz, h), 2.38 (2H, t, *J* = 7.4 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.30 (f), 159.01 (n), 138.47 (d), 130.64 (j), 130.28 (k), 128.78 (l,l'), 127.87 (b,b'), 127.53 (a), 127.32 (c,c'), 126.57 (i), 114.06 (m,m'), 55.42 (o), 43.66 (e), 36.62 (g), 29.16 (h); IR (neat) 3292 (N-H stretch), 3065, 3032, 2918, 1636 (C=O stretch), 1605, 1545, 1509, 1454, 1439, 1248, 1221, 1174, 1029, 969, 845, 805, 693 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{21}NO_2$ (M): 295.1572, found 295.1567 *m/z*.



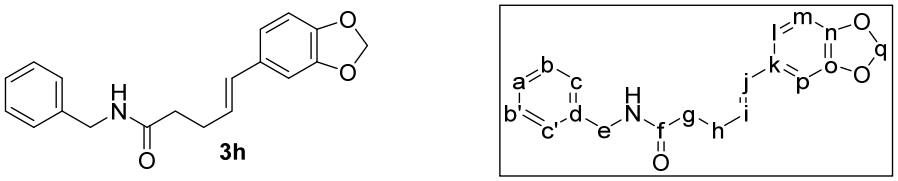


(*E*)-5-(3-methoxy)phenyl-4-pentenecarboxylic acid benzyl amide (3f). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(3-methoxy)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.17 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (1.07 g, 72%) as a white solid: m.p. 57.5–59.0 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.30 (6H, m, a,b,b',c,c',m), 6.93 (1H, d, *J* = 7.6 Hz ,l), 6.88 (1H, d, *J* = 2.1 Hz, p), 6.80 (1H, dd, *J* = 8.2 and 2.5 Hz, n), 6.43 (1H, d, *J* = 15.8 Hz, j), 6.22 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.95 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 3.82 (3H, s, q), 2.60 (2H, q, *J* = 7.5 Hz, h), 2.39 (2H, t, *J* = 7.5 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.12 (f), 159.92 (o), 138.90 (k), 138.38 (d), 131.19 (j), 129.61 (i), 129.12 (m), 128.81 (b,b'), 127.89 (c,c'), 127.59 (a), 118.87 (l), 112.94 (n), 111.59 (p), 55.33 (q), 43.72 (e), 36.43 (g), 29.08 (h); IR (neat) 3284 (N-H stretch), 3062, 3027, 2914, 2834, 1642 (C=O stretch), 1603, 1598, 1577, 1544, 1491, 1464, 1453, 1430, 1256, 1154, 1041, 965, 772, 733, 689 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{21}NO_2$ (M): 295.1572, found 295.1571 *m/z*.

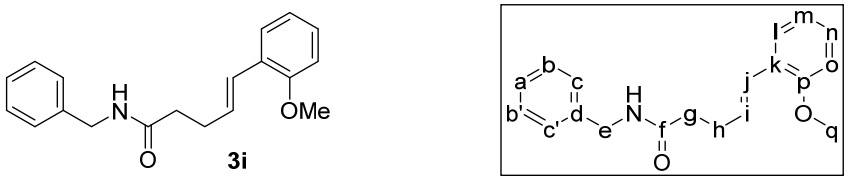




(*E*)-5-(3,5-dimethoxy)phenyl-4-pentenecarboxylic acid benzyl amide (3g). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(3,5-dimethoxy)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.32 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (1.16 g, 71%) as a white solid: m.p. 91.0–92.0 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.30 (5H, m, a,b,b',c,c'), 6.60 (2H, d, *J* = 2.2 Hz ,l,l'), 6.30–6.40 (2H, m, j,n), 6.20 (1H, dt, *J* = 15.8 and 6.8 Hz, i), 6.14 (1H, br s), 4.43 (2H, d, *J* = 5.8 Hz, e), 3.79 (6H, s, o,o'), 2.57 (2H, q, *J* = 6.9 Hz, h), 2.37 (2H, t, *J* = 7.5 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.18 (f), 161.01 (o,o'), 139.52 (k), 138.43 (d), 131.20 (j), 129.40 (i), 128.78 (b,b'), 127.83 (c,c'), 127.54 (a), 104.34 (l,l'), 99.60 (n), 55.43 (o,o'), 43.66 (e), 36.30 (g), 29.03 (h); IR (neat) 3287 (N-H stretch), 2936, 2359, 1629 (C=O stretch), 1587, 1536, 1454, 1422, 1203, 1149, 1058, 960, 819, 749, 697, 680 cm^{-1}; HRMS (EI) calcd. for $C_{20}H_{23}NO_3$ (M): 325.1678, found 325.1685 *m/z*.

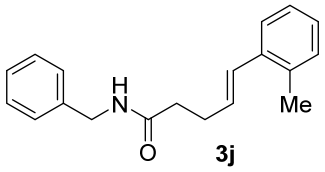


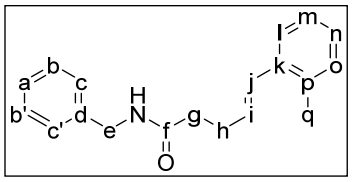
(*E*)-5-(benzo[*d*][1,3]dioxol-5-yl-4-pentenecarboxylic acid benzyl amide (3h). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(benzo[*d*][1,3]dioxol-5-yl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.24 g)) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (1.11 g, 72%) as a white solid: m.p. 100.5–102.0 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (5H, m, a,b,b',c,c'), 6.85–6.90 (1H, m, p), 6.70–6.75 (2H, m, l,m), 6.35 (1H, d, *J* = 15.8 Hz, j), 6.03 (1H, dt, *J* = 15.7 and 6.9 Hz, i), 6.03 (1H, br s, NH, *overlapped* with i), 5.95 (2H, s, q), 4.44 (2H, d, *J* = 5.7 Hz, e), 2.55 (2H, q, *J* = 7.0 Hz, h), 2.37 (2H, t, *J* = 7.5 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.20 (f), 148.08 (o), 146.97 (n), 138.43 (d), 131.97 (k), 130.82 (j), 128.80 (b,b'), 127.88 (c,c'), 127.58 (a), 126.99 (i), 120.65 (l), 108.34 (m), 105.59 (p), 101.10 (q), 43.69 (e), 36.55 (g), 29.04 (h); IR (neat) 3292 (N-H stretch), 2971, 2911, 2360, 1635 (C=O stretch), 1603, 1544, 1505, 1489, 1445, 1237, 1031, 967, 921, 801, 741, 694 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{19}NO_3$ (M): 309.1365, found 309.1365 *m/z*.



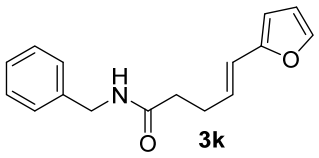
(*E*)-5-(2-methoxy)phenyl-4-pentenecarboxylic acid benzyl amide (3i). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(2-methoxy)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.17 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (1.05 g, 71%) as a white solid: m.p. 87.5–88.0 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (1H, dd, *J* = 7.8 and 1.6 Hz, n), 7.20–7.30 (6H, m, a,b,b',c,c',l), 6.93 (1H, t, *J* = 7.4 Hz , m), 6.88 (1H, d, *J* = 8.2 Hz, o), 6.80 (1H, d, *J* = 16.0 Hz, j), 6.23 (1H, dt, *J* = 16.0 and 6.9 Hz, i), 6.04 (1H, br s), 4.45 (2H, d, *J* = 5.8 Hz, e), 3.83 (3H, s, q), 2.61 (2H, q, *J* = 7.3 Hz, h), 2.40 (2H, t, *J* = 7.6 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.35 (f), 156.50 (p), 138.49 (d), 129.48 (j), 128.77 (b,b'), 128.33 (l), 127.88 (c,c'), 127.52 (a), 126.68 (n), 126.49 (k), 125.90 (i), 120.77 (m), 110.92 (o), 55.53 (q), 43.68 (e), 36.62 (g), 29.61 (h); IR (neat) 3307 (N-H stretch), 2923, 2359,

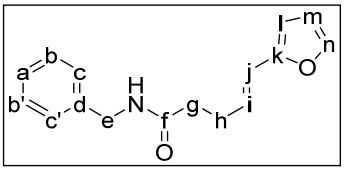
1636 (C=O stretch), 1544, 1488, 1242, 1027, 963, 752, 743, 729, 700 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{21}NO_2$ (M): 295.1572, found 295.1562 *m/z*.





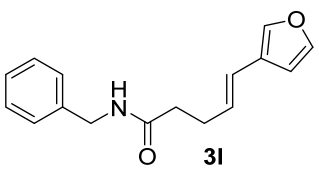
(*E*)-5-(2-methyl)phenyl-4-pentenecarboxylic acid benzyl amide (3j). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(2-methyl)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.09 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (992 mg, 71%) as a white solid: m.p. 67.5–68.5 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35–7.45 (1H, m, aryl), 7.25–7.35 (5H, m, aryl), 7.10–7.20 (3H, m, aryl), 6.67 (1H, d, *J* = 15.6 Hz, j), 6.10 (1H, dt, *J* = 15.6 and 6.9 Hz, i), 5.97 (1H, br s), 4.47 (2H, d, *J* = 5.7 Hz, e), 2.63 (2H, q, *J* = 7.3 Hz, h), 2.42 (2H, t, *J* = 7.5 Hz, g), 2.33 (3H, s, q); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.15 (f), 138.41 (aryl), 136.54 (aryl), 135.19 (aryl), 130.34 (aryl), 130.09 (i), 129.14 (j), 128.82 (aryl), 127.91 (aryl), 127.61 (aryl), 127.25 (aryl), 126.16 (aryl), 125.61 (aryl), 43.72 (e), 36.67 (g), 29.47 (h), 19.93 (q); IR (neat) 3297 (N-H stretch), 2914, 2359, 1633 (C=O stretch), 1533, 1242, 1173, 966, 747, 697 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{21}NO$ (M): 279.1623, found 279.1625 *m/z*.

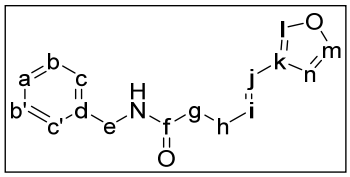




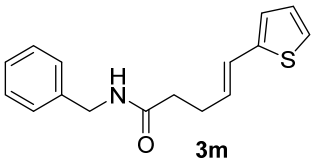
(*E*)-5-(2-furanyl)-4-pentenecarboxylic acid benzyl amide (3k). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(2-furanyl)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 970 mg) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (830 mg, 65%) as a light yellow solid: m.p. 93.5–94.5 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (6H, m, a,b,b',c,c',n), 6.37 (1H, dd, *J* = 3.2 and 1.9 Hz, m), 6.28 (1H, d, *J* = 15.9 Hz, j), 6.10–6.20 (2H, m, i and l, *overlapped*), 5.95 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 2.57 (2H, q, *J* = 7.2 Hz, h), 2.37 (2H, t, *J* = 7.5 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.03 (f), 152.89 (k), 141.63 (n), 138.36 (d), 128.78 (b,b'), 127.87 (c,c'), 127.64 (i), 127.57 (a), 119.92 (j), 111.28 (m), 106.90 (l), 43.71 (e), 36.37 (g), 28.90 (h); IR (neat) 3273 (N-H stretch), 2361, 1635

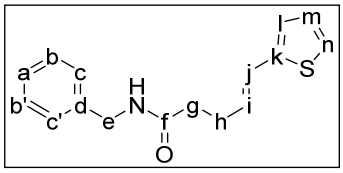
(C=O stretch), 1538, 1219, 1152, 1012, 957, 921, 726, 696 cm^{-1}; HRMS (EI) calcd. for $C_{16}H_{17}NO_2$ (M): 255.1259, found 255.1252 *m/z*.





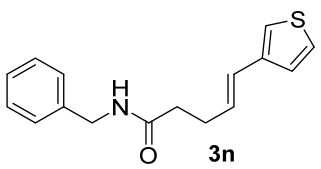
(*E*)-5-(3-furanyl) -4-pentenecarboxylic acid benzyl amide (3l). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(3-furanyl)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 970 mg) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (870 mg, 68%) as a light yellow solid: m.p. 91.0–92.5 °C; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (7H, m, a,b,b',c,c',l,m), 6.49 (1H, d, *J* = 0 Hz, n), 6.30 (1H, d, *J* = 15.8 Hz, j), 5.94 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.89 (1H, br s, NH), 4.46 (2H, d, *J* = 5.7 Hz, e), 2.55 (2H, q, *J* = 7.4 Hz, h), 2.37 (2H, t, *J* = 7.4 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.10 (f), 143.50 (m), 139.90 (l), 138.40 (d), 128.81 (b,b'), 128.33 (i), 127.89 (c,c'), 127.61 (a), 124.21 (k), 120.94 (j), 107.60 (n), 43.70 (e), 36.49 (g), 28.97 (h); IR (neat) 3294 (N-H stretch), 2360, 2342, 1635 (C=O stretch), 1548, 1225, 1158, 1072, 1021, 963, 873, 779, 746, 728, 694 cm^{-1}; HRMS (EI) calcd. for $C_{16}H_{17}NO_2$ (M): 255.1259, found 255.1259 *m/z*.

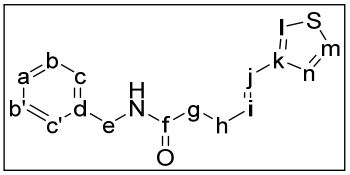




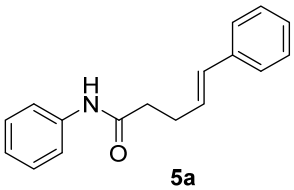
(*E*)-5-(2-thiophenyl)-4-pentenecarboxylic acid benzyl amide (3m). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(2-thiophenyl)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.05 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (936 mg, 69%) as a white solid: m.p. 88.5–90.0 °C; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.35 (5H, m, a,b,b',c,c'), 7.13 (1H, d, *J* = 5.0 Hz, n), 6.96 (1H, dd, *J* = 5.0 and 3.6 Hz, l), 6.89 (1H, d. *J* = 3.3 Hz, m), 6.58 (1H, d, *J* = 15.7 Hz, j), 6.06 (1H, dt, *J* = 15.7 and 7.0 Hz, i), 5.91 (1H, br s), 4.46 (2H, d, *J* = 5.7 Hz, e), 2.57 (2H, q, *J* = 7.2 Hz, h), 2.38 (2H, t, *J* = 7.3 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.98 (f), 142.57 (k), 138.33 (d), 128.81

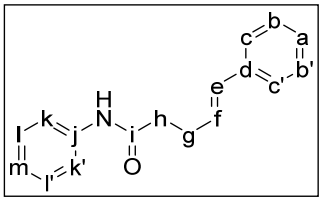
(b,b'), 128.56 (i), 127.87 (c,c'), 127.59 (a) , 127.39 (l), 124.98 (m), 124.60 (j). 123.67 (n), 43.73 (e), 36.36 (g), 28.92 (h); IR (neat) 3305 (N-H stretch), 2360, 1648 (C=O stretch), 1538, 1454, 953, 736, 694 cm^{-1}; HRMS (EI) calcd. for $C_{16}H_{17}NOS$ (M): 271.1031, found 271.1034 *m/z*.





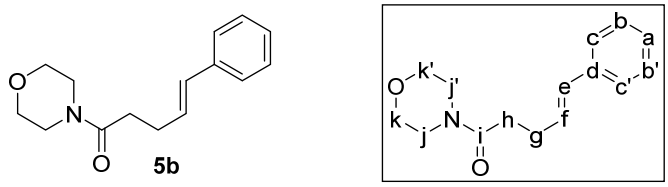
(*E*)-5-(3-thiophenyl)-4-pentenecarboxylic acid benzyl amide (3n). Following **GP1** with the corresponding ethyl ester ((*E*)-5-(3-thiophenyl)phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.05 g) and benzyl amine (2 equiv, 10 mmol, 1.1 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (963 mg, 79%) as a white solid: m.p. 111.5–112.5 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (6H, m, a,b,b',c,c',l,), 7.17 (1H, dd, *J* = 5.0 and 0 Hz, m), 7.07 (1H, dd, *J* = 2.2 and 0 Hz, n), 6.46 (1H, d, *J* = 15.8 Hz, j), 6.07 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.94 (1H, br s), 4.46 (2H, d, *J* = 5.7 Hz, e), 2.57 (2H, q, *J* = 7.3 Hz, h), 2.38 (2H, t, *J* = 7.4 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.11 (f), 140.03 (k), 138.40 (d), 128.81 (b,b'), 128.65 (i), 127.89 (c,c'), 127.60 (a), 126.01 (l), 125.60 (j), 125.03 (m), 121.23 (n), 43.70 (e), 36.49 (g), 29.01 (h); IR (neat) 3289 (N-H stretch), 2360, 1633 (C=O stretch), 1545, 1453, 1222, 964, 763, 746, 727, 693 cm^{-1}; HRMS (EI) calcd. for $C_{16}H_{17}NOS$ (M): 271.1031, found 271.1040 *m/z*.



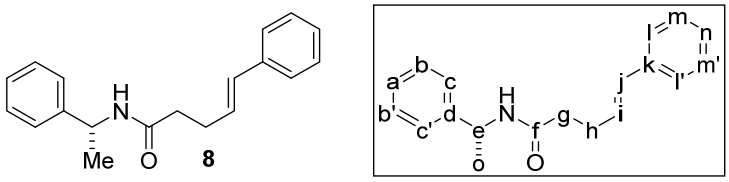


(*E*)-5-phenyl-4-pentenecarboxylic acid phenyl amide (5a). Following **GP1** with the corresponding ethyl ester ester ((*E*)-5-phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.02 g)) and aniline (2 equiv, 10 mmol, 0.91 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (892 mg, 71%) as a white solid: m.p. 112.5–114.0 ºC; TLC analysis R_f 0.65 (60:40 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (2H, d, *J* = 7.8 Hz, k,k'), 7.30–40 (7H, m, b,b',c,c',l,l',NH), 7.24 (1H, t, *J* = 7.0 Hz, a), 7.13 (1H, t, *J* = 7.3 Hz, m), 6.51 (1H, d, *J* = 15.8 Hz, e), 6.25 (1H, dt, *J* = 15.8 and 6.7 Hz, f), 2.67 (2H, q, *J* = 7.1 Hz, g), 2.55 (2H, t, *J* = 7.4 Hz, h); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.61 (i), 137.94 (j), 137.36 (d), 131.47 (e), 129.13 (l,l'), 128.67 (b,b'), 128.63 (m), 127.37 (f), 126.21 (c,c'), 124.44

(a), 120.04 (k,k'), 37.43 (h), 28.94 (g); IR (neat) 3279 (N-H stretch), 3026, 1650 (C=O stretch), 1598, 1526, 1441, 964, 738, 690 cm^{-1}; HRMS (EI) calcd. for $C_{17}H_7NO$ (M): 251.1310, found 251.1313 *m/z*.



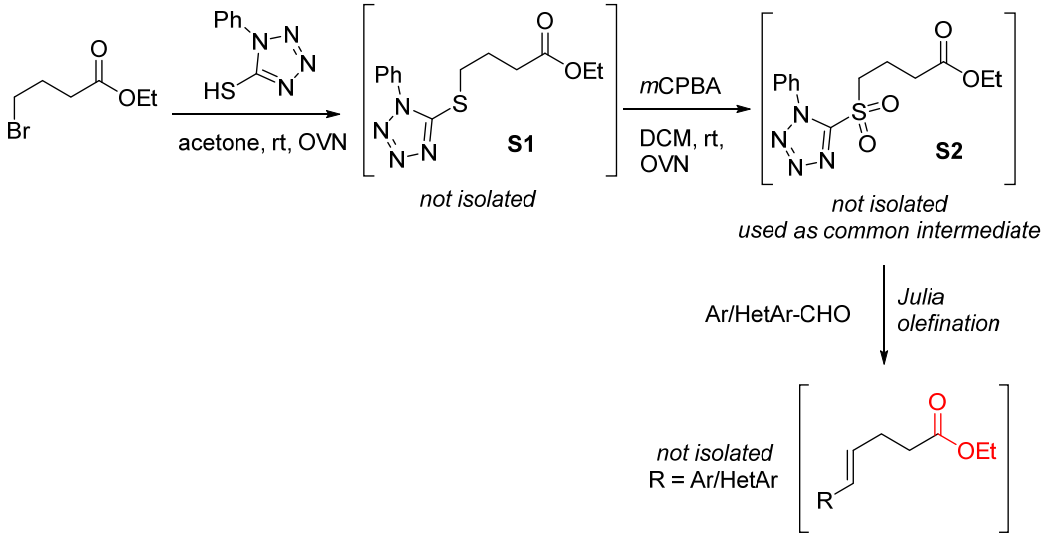
(*E*)-5-phenyl-4-pentenecarboxylic acid morpholine amide (5b). Following **GP1** the corresponding ethyl ester ester ((*E*)-5-phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.02 g)) and morpholine (2 equiv, 10 mmol, 0.87 mL) affords, after flash chromatography on silica gel (70:30–50:50 hexanes:ethyl acetate), the title compound (957 mg, 78%) as a yellow oil; TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (2H, d, *J* = 7..2 Hz, c,c'), 7.29 (2H, t, *J* = 7.3 Hz, b,b'), 7.20 (1H, t, *J* = 7.1 Hz, a), 6.44 (1H, d, *J* = 15.9 Hz, e), 6.26 (1H, dt, *J* = 15.9 and 6.8 Hz, f), 3.60–3.70 (6H, m, j,j',k,k'), 3.45–3.50 (2H, m, j,j'), 2.57 (2H, q, *J* = 6.8 Hz, h), 2.47 (2H, t, *J* = 7.5 Hz, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.91 (i), 137.50 (d), 130.94 (e), 129.24 (f), 128.64 (b,b'), 127.24 (a), 126.13 (c,c'), 67.02 and 66.72 (k,k'), 46.06 and 42.06 (j,j'), 32.86 (g), 28.67 (h); IR (neat) 2962, 2897, 2853, 1639 (C=O stretch), 1429, 1220, 1112, 1024, 964, 746, 693 cm^{-1}; HRMS (EI) calcd. for $C_{15}H_{19}NO_2$ (M): 245.1416, found 245.1413 *m/z*.



(*R,E*)-5-phenyl-4-pentenecarboxylic acid 1-phenylethyl amide (8). Following **GP1** with the corresponding ethyl ester ((*E*)-5-phenyl-4-pentenecarboxylic acid ethyl ester (5 mmol, 1.02 g) and (*R*)-(+)-α-methylbenzylamine (2 equiv, 10 mmol, 1.3 mL) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound (1.06 g, 76%) as a white solid: m.p. 77.5–79.0 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); $[\alpha]_D^{20}$ = +44.1 (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (10H, m, a,b,b',c,c',l,l',m,m',n), 6.45 (1H, d, *J* = 15.8 Hz, j), 6.21 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.94 (1H, d, *J* = 7.4 Hz, NH), 5.15–5.20 (1H, m, e), 2.58 (2H, q, *J* = 6.9 Hz, h), 2.30–2.40 (2H, m, g), 1.50 (3H, d, *J* = 6.9 Hz, o); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.33 (f), 143.32 (k), 137.44 (d), 131.25 (j), 128.82 (i), 128.77 (b,b'), 128. 128.64

(l,l'), 127.42 (n), 127.29 (a), 126.30 (m,m'), 126.19 (c,c'), 43.77 (e), 36.57 (g), 29.16 (h), 21.86 (o); IR (neat) 3304 (N-H stretch), 3059, 3025, 2981, 2359, 2341, 1638 (C=O stretch), 1534, 1493, 1444, 1374, 1217, 959, 743, 694 cm^{-1}; HRMS (ESI) calcd. for $C_{19}H_{21}NNaO$ (M+Na): 302.1521, found 302.1525 *m/z*.

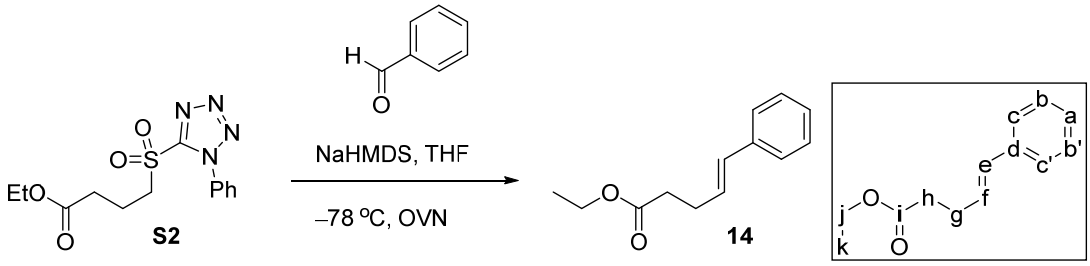
General procedure for the preparation of aryl/ heteroaryl substituted γ,δ-unsaturated esters via Julia-typed olefination (GP2)[2]



To a solution of 1-phenyl-1H-tetrazole-5-thiol (10.0 g, 56.1 mmol, 1 equiv) and K_2CO_3 (15.5 g, 112.3 mmol, 2.0 equiv) in acetone (300 mL) was added ethyl 4-bromobutyrate (8.8 mL, 61.7 mmol, 1.1 equiv) under nitrogen atmosphere. The resulting mixture was stirred at room temperature overnight then filtered through a pad of celite and concentrated under reduced pressure to afford light yellow oil crude **S1** (16.5 g, quantitative) used in the next step without further purification.

Inside a nitrogen glove bag, to a solution of **S1** (16.4 g, 56 mmol, 1 equiv) in DCM (200 mL) was added *m*-CPBA (48.4 g, 278 mmol, 5 equiv) and $NaHCO_3$ (11.7 g, 140 mmol, 2.5 equiv. After an overnight stir, the resulting mixture was filtered, washed with EtOAc, and concentrated under reduced pressure. The residue was then dissolved in the minimum amount of DCM and pushing through a plug of silica gel eluting with 1:1 EtOAc:hexanes to afford the Julia-typed intermediate **S2** as light yellow solid (17.3 g, 95%). Crude **S2** is used as a common intermediate for Julia olefination.

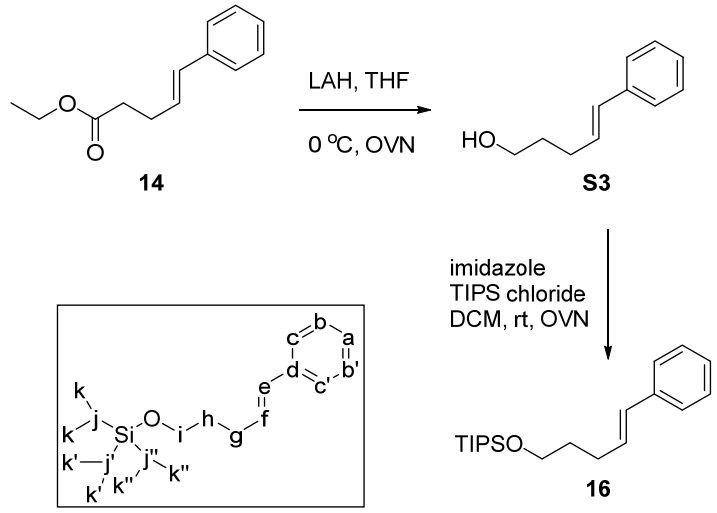
Representative Julia olefination example:



(*E*)- 5-phenyl-4-pentenecarboxylic acid ethyl ester (14). To a solution of Julia reagent **S2** (1.6 g, 4.93 mmol, 1 equiv) in THF (25 mL) was added NaHMDS (2M, 2.7 mL, 5.43 mmol, 1.1 equiv) at –78 ºC. The resulting mixture was stirred at the same temperature for 30 mins then benzaldehyde (0.75 mL, 7.4 mmol, 1.5 equiv) was added dropwise. The resultant mixture was slowly warmed to room temperature and stir overnight after quenching with water and EtOAc. The organic layer was separated and the aqueous layer was extract with EtOAc (10 mL x 2). The combined organic layers were dried (anhyr. Na_2SO_4) and concentrated under reduced pressure affords, after flash chromatography on silica gel (95:5 hexanes:ethyl acetate), the title compound (1.09 g, 72%) as a yellow liquid; TLC analysis R_f 0.7 (90:10 hexanes:ethyl acetate). Characterization data match with literature report:[3] ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.40 (4H, m, b,b',c,c'), 7.24 (1H, t, *J* = 7.0 Hz, a), 6.48 (1H, d, *J* = 15.8 Hz, e), 6.25 (1H, dt, *J* = 15.8 and 6.3 Hz, f), 4.19 (2H, q, *J* = 7.1 Hz, j), 2.50–2.60 (4H, m, g,h), 1.30 (3H, t, *J* = 7.1 Hz, k); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.08 (i), 137.54 (d), 131.09 (f), 128.63 (b,b',e), 127.26 (a), 126.19 (c,c'), 60.51 (j), 34.20 (h), 28.45 (g), 14.41 (k).

Ester **14** was isolated for screening purpose. All other esters prepared via this method were passed through a short pad of silica gel eluting with 10% EtOAc:hexanes, concentrated under reduced pressure, and used without further purification.

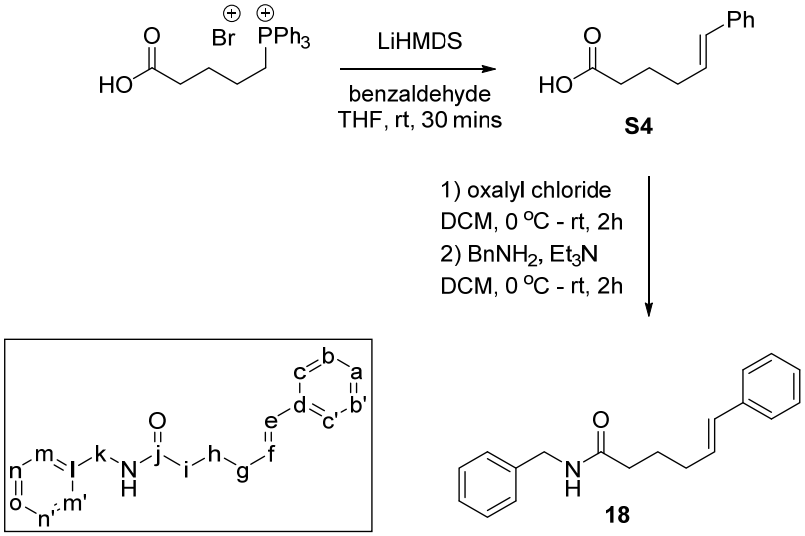
Preparation of OTIPS substrate 16



(*E*)-triisopropyl((5-phenylpent-4-en-1-yl)oxy)silane (16). To a cooled (0 °C) solution of (*E*)- 5-phenyl-4-pentenecarboxylic acid ethyl ester **14** (3.3 mmol, 679 mg, 1 equiv) in THF (15 mL) was added lithium aluminum hydride (LAH, 1.05 equiv, 3.5 mmol, 133 mg). The resulting mixture was warmed to room temperature and stirred overnight. The reaction mixture was quenched with 5N KOH (0.3 mL) and water (0.15 mL). Following by 30-min stir, the resultant mixture was filtered through a pad of celite and washed with ethyl acetate (2 x 5 mL). The filtrate was dried (anhyd. Na_2SO_4) and concentrated under reduced pressure to give the crude yellow oil **S3** in quantitative yield (535 mg) which was used in the next step without further purification.

To the solution of alcohol **S3** (520 mg, 3.2 mmol, 1 equiv) and imidazole (435 mg, 6.4 mmol, 2 equiv) in DCM (10 mL) was added dropwise triisopropylsilyl chloride (645 mg, 3.36 mmol, 1.05 equiv) at room temperature. The resultant mixture was stirred overnight then concentrated under reduced pressure affords, after flash chromatography on silica gel (95:5 hexanes:ethyl acetate), the title compound (814 mg, 80%) as a colorless liquid; TLC analysis R_f 0.7 (90:10 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (2H, d, *J* = 7.2 Hz, c,c'), 7.36 (2H, t, *J* = 7.4 Hz, b,b'), 7.26 (1H, t, *J* = 7.0 Hz, a), 6.49 (1H, d, *J* = 15.8 Hz, e), 6.32 (1H, dt, *J* = 15.8 and 6.8 Hz, f), 3.83 (2H, d, *J* = 6.4 Hz, i), 2.35–2.45 (2H, m, g), 1.75–1.85 (2H, m, h), 1.15–1.20 (21H, m, j,j',j'',k,k',k''); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.06 (d), 130.78 (f), 130.21 (e), 128.61 (b,b'), 126.94 (a), 126.08 (c,c'), 62.88 (i), 32.81 (h), 29.54 (g), 18.22 (k,k',k''), 12.21 (j,j',j''); IR (neat) 2141, 2891, 2864, 1462, 1102, 962, 881, 679, 657 cm^{-1}; HRMS (EI) calcd. for $C_{17}H_{27}OSi$ (M–C_3H_7): 275.1831, found 275.1843 *m/z*.

Preparation of δ,ε-unsaturated benzyl amide substrate 18 involving general procedure for the preparation of unsaturated amides via acid chloride intermediates (GP3)

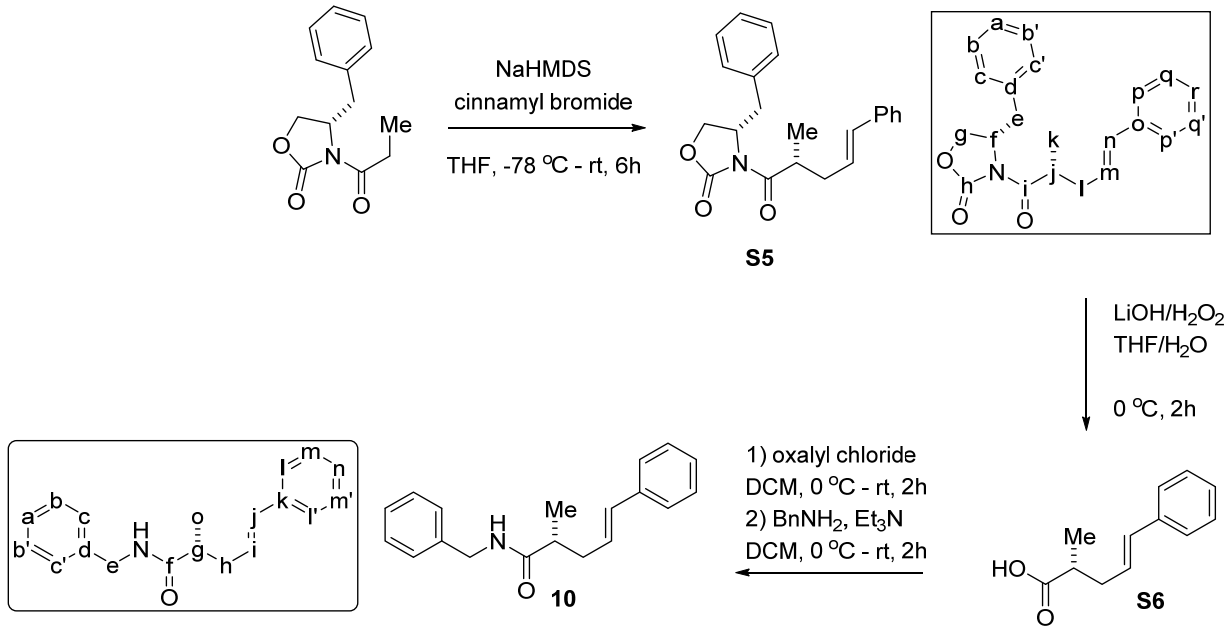


(*E*)-6-phenyl-5-hexanecarboxylic acid benzyl amide (18). To a solution of 4-(carboxybutyl)triphenylphosphonium bromide (2.0 g, 4.5 mmol, 1 equiv) in THF (10 mL) was added LiHMDS (1.8 mL, 9..5 mmol, 2.1 equiv) over 15 mins at room temperature. After 15 min, benzaldehyde (0.5 mL, 4.5 mmol) was added dropwise at room temperature. After 15 min, water (15 mL) and ether (15 mL) were added. The organic phase was rinsed with water (10 mL). The combined aqueous solution was washed with EtOAc (15 mL), acidified (10% HCl), and extracted with EtOAc (2 x 15 mL). The combined organic extracts were dried and concentrated under reduced pressure to afford crude **S4**[4] (771 mg, 90%).

(**GP3**) To a cooled solution (0 °C) of the crude carboxylic acid **S4** (750 mg, 3.94 mmol) in dichloromethane (20 mL) was added dropwise oxalyl chloride (1.35 mL, 4.0 equiv, 15.7 mmol) followed by 3 drops of DMF. The resultant mixture was slowly warmed to room temperature and stir for the total of 1.5 h. The reaction mixture was then concentrated. The resulting acid chloride was redissolved in dichloromethane (20 mL) and cooled to 0 °C. The resultant mixture was then added dropwise aniline (0.55 mL, 1.5 equiv, 5.85 mmol) followed by dropwise addition of triethylamine (1.1 mL, 2.0 equiv, 7.8 mmol). The reaction mixture was slowly warm to room temp and stir for the total of 1.5 h. Whereupon it was diluted, it was washed twice with HCl (3M), once with satd. $NaHCO_3$ solution, and once with brine. The organic extracts were dried (Na_2SO_4) and concentrated under reduced pressure affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the mixture of 7:1 *E/Z* **18** as a yellow solid; TLC analysis R_f 0.4

(60:40 hexanes:ethyl acetate). The mixture of 7:1 *E/Z* **18** was then diluted with a minimum amount of EtOAc following by addition of hexanes (9:1 hexanes:EtOAc). After 2 hours, the resultant mixture was filtered to afford the title compound (672 mg, 61%) as a white solid: m.p. 103.5–105.0 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.40 (9H, m, a,b,b',c,c',m,m',n,n'), 7.22 (1H, t, *J* = 6.7 Hz, o), 6.39 (1H, d, *J* = 15.8 Hz, e), 6.20 (1H, dt, *J* = 15.8 and 6.9 Hz, f), 5.74 (1H, br s, NH), 4.46 (2H, d, *J* = 5.7 Hz, k), 2.25–2.35 (4H, m, g, i), 1.85–1.95 (2H, m, h); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.65 (j), 138.45 (l), 137.62 (d), 130.93 (e), 129.80 (f), 128.86 (n,n'), 128.63 (b,b'), 128.00 (c,c'), 128.67 (o), 127.14 (a), 126.08 (m,m'), 43.77 (k), 36.01 (i), 32.52 (g), 25.24 (h); IR (neat) 3278 (N-H stretch), 1630 (C=O stretch), 1552, 1546, 1451, 965, 728, 689 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{21}NO$ (M): 279.1623, found 279.1632 *m/z*.

Preparation sequence of pre-installed α-chiral center aryl substituted γ.δ-unsaturated amide 10



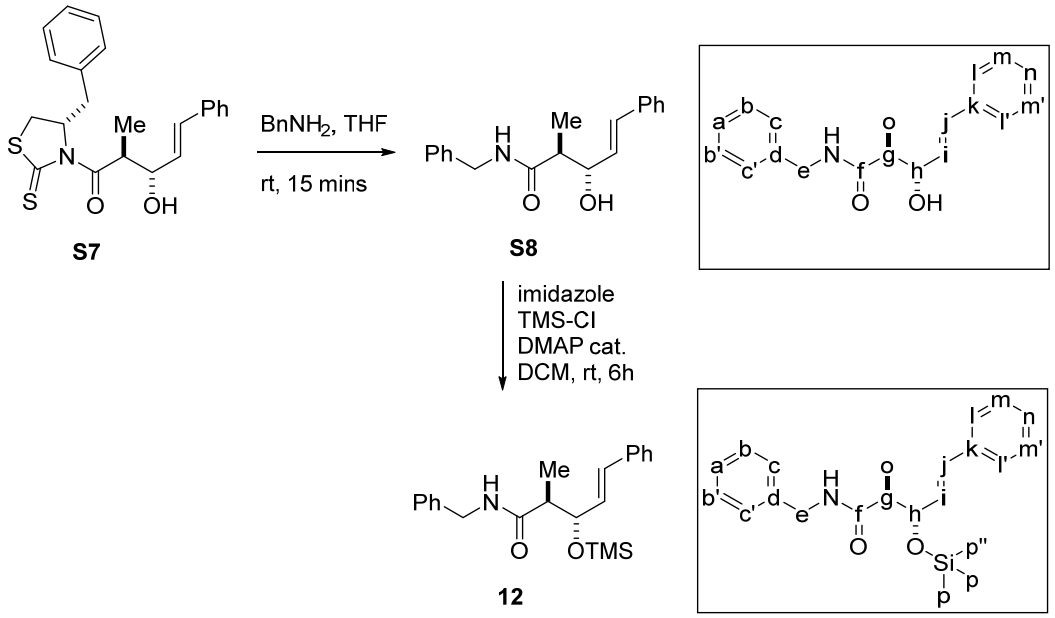
To a cooled (–78 °C) solution of (*S*)-4-benzyl-3-propionyloxazolidin-2-one[5] (4.4 mmol, 1.03 g) in THF (15 mL) was added NaHMDS (1.1 equiv, 2M, 4.84 mmol, 2.4 mL) dropwise. The resultant mixture was stirred at –78 °C for 1h, and cinnamyl bromide (2 equiv, 8.8 mmol, 1.73 g) was added dropwise. The resulting mixture was slowly warmed to room temp and stirred for another 5h before quenching with water. The crude reaction was extracted with EtOAc (3 x 30

mL). The organic extracts were combined, dried (anhyd. Na_2SO_4), and concentrated under reduced pressure. Flash chromatography on silica gel (75:25 hexanes:ethyl acetate) affords the title compound **S5** (1.18 g, 77%) as a white solid: 98.5–99.5 ºC; TLC analysis R_f 0.4 (70:30 hexanes:ethyl acetate); $[\alpha]_D^{20}$ = +14.6º (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10–7.50 (10H, m, aryl), 6.49 (1H, d, *J* = 15.8 Hz, n), 6.28 (1H, dd, *J* = 15.5 and 7.3 Hz, m), 4.65–4.75 (1H, m, f), 4.20 (1H, dd, *J* = 8.7 and 8.7 Hz, g), 4.14 (1H, dd, *J* = 9.1 and 3.0 Hz, g), 3.95–4.05 (1H, m, e), 3.29 (1H, dd, *J* = 13.4 and 3.3 Hz, e), 2.60–2.75 (2H, m, j,l), 2.35–2.50 (1H, m, l), 1.27 (3H, d, *J* = 6.8 Hz, k); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.71 (j), 153.30 (h), 137.41 (d), 135.45 (o), 132.60 (n), 129.48 (aryl), 129.03 (aryl), 128.66 (aryl), 127.41 (aryl), 127.33 (aryl), 127.15 (m), 126.24 (aryl), 66.13 (g), 55.45 (f), 38.12 (e), 37.79 (j), 37.63 (l), 16.77 (k); IR (neat) 3025, 2971, 1780 (C=O stretch), 1692 (C=O stretch), 1384, 1348, 1247, 1215, 1097, 967, 744, 697 cm^{-1}; HRMS (ESI) calcd. for $C_{22}H_{23}NNaO_3$ (M+Na): 372.1576, found 372.1582 *m/z*.

To cooled (0 ºC) a solution of **S5** (665 mg, 1.9 mmol) in THF (30 mL) and water (10 mL) was added LiOH (150 mg, 3.8 mmol) and hydrogen peroxide (30%, 1.4 mL). The resulting mixture was stirred at the same temperature for 2h before quenching with aqueous Na_2SO_3 (1.5M, 13 mL). The resultant mixture was then acidified with dilute HCl (1M) to pH=1. Following by extraction with DCM (20 mL x 3), the combined organic layers dried (anhyd. Na_2SO_4), and concentrated under reduced pressure to afford crude acid **S6** in quantitative yield used in the next step without further purification.

Following **GP3** for the preparation of unsaturated amides via acid chloride intermediates, crude acid **S6** (1.9 mmol) affords, after flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate), the title compound **10** (425 mg, 80%) as a white solid: m.p. 92.5–93.5 ºC; TLC analysis R_f 0.4 (60:40 hexanes:ethyl acetate); $[\alpha]_D^{20}$ = +4.7º (*c* 2.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20–7.35 (10H, m, a,b,b',c,c',l,l',m,m',n), 6.44 (1H, d, *J* = 15.8 Hz, j), 6.21 (1H, dt, *J* = 15.8 and 7.3 Hz, i), 5.91 (1H, br s, NH), 4.55 (1H, dd, *J* = 14.7 and 8.2 Hz, e), 4.37(1H, dd, *J* = 14.7 and 8.2 Hz, e) 2.50–2.70 (1H, m, h), 2.30–2.50 (2H, m, g,h), 1.25 (3H, d, *J* = 6.4 Hz, o); ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.49 (f), 138.41 (d), 137.33 (k), 132.17 (j), 128.68 (b,b'), 128.53 (l,l'), 127.75 (m,m'), 127.49 (i), 127.41 (a) 127.21 (n), 126.13 (c,c'), 43.44 (e), 41.92 (g), 37.74 (h), 17.68 (o); IR (neat) 3303 (N-H stretch), 2966, 2360, 1633 (C=O stretch), 1603, 1539, 1495, 1453, 962, 739, 724, 691 cm^{-1}; HRMS (ESI) calcd. for $C_{19}H_{21}NNaO$ (M+Na): 302.1521, found 302.1526 *m/z*.

Preparation sequence of pre-installed α- and β– chiral centers aryl substituted γ.δ-unsaturated amide 12[1]



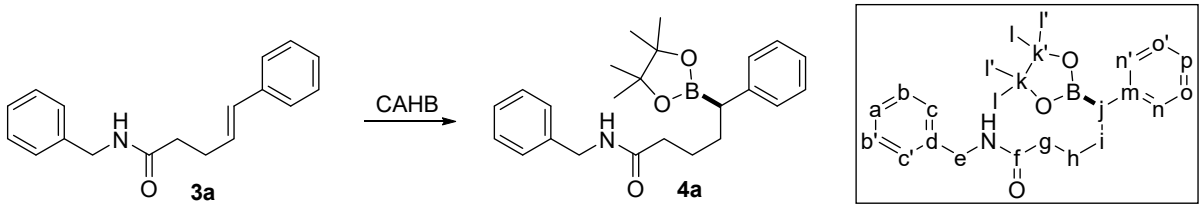
Chiral auxiliary **S7** was prepared as reported in literature.[6] Benzyl amine (2.5 mmol, 0.28 mL) was added to a solution of chiral auxiliary **S7** (2.5 mmol, 995 mg) in THF (5 mL). The resultant mixture was stirred at room temperature for 15 mins and poured into 1% aqueous $NaHCO_3$ (100 mL). After a 5 min-stir, dicholoromethane (DCM, 30 mL) was added and the two layers were separated. The aqueous layer was extracted with DCM (2 x 30 mL). The combined organic layers were dried (anhyd. Na_2SO_4) and concentrated under reduced pressures. Flash chromatography on silica gel (90:10–70:30 DCM:ethyl acetate) affords the title compound **S8** (673 mg, 91%) as a white solid: m.p. 137.5–139.0 ºC; TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); $[\alpha]_D^{20}$ = +10.5º (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (10H, m, aryl), 6.66 (1H, d, *J* = 15.8 Hz, j), 6.22 (1H, br s, NH, *overlapped with* i), 6.22 (1H, dd, *J* = 15.9 and 6.6 Hz, i), 4.54 (1H, dd, *J* = 14.8 and 6.1 Hz, e), 4.35–4.45 (2H, m, e,h), 3.61 (1H, d, *J* = 5.9 Hz, OH), 2.40–2.50 (1H, m, g), 1.32 (3H, d, *J* = 7.1 Hz, o); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.37 (f), 138.12 (d), 136.58 (k), 131.75 (j), 130.15 (i), 128.83 (aryl), 128.70 (aryl), 127.92 (aryl), 127.73 (aryl), 127.60 (aryl), 126.71 (aryl), 75.06 (h), 46.58 (g), 43.47 (e), 15.46 (o); IR (neat) 3336, 3281 (N-H

[1] The compound is unstable; it is quickly self-deprotected back to the corresponding alcohol **S8** at room temperature, and thus should be used immediately after being prepared.

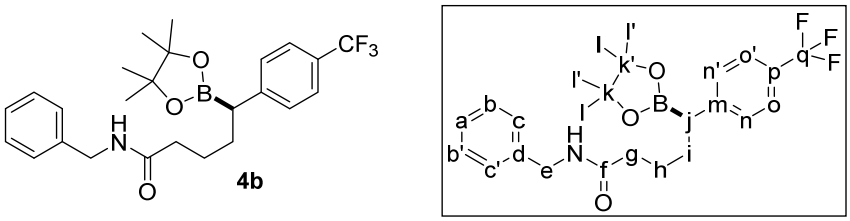
stretch), 3025, 2979, 2359, 2343, 1647 (C=O stretch), 1556, 1494, 1454, 1415, 1361, 1258, 1226, 1110, 978, 965, 736, 686 cm^{-1}; HRMS (ESI) calcd. for $C_{19}H_{21}NNaO_2$ (M+Na): 318.1470, found 318.1476 *m/z.*

To a solution of benzyl amide **S8** (2 mmol, 590 mg), 1 crystal of DMAP, imidazole (3 mmol (3 mmol, 1.5 equiv, 205 mg) in THF (10 mL) was added dropwise trimethylsilyl chloride (2.2 mmol, 1.1 equiv, 0.28 mL). The resultant mixture was stirred 6h at room temperature and quenched with water. The aqueous layer was extracted with DCM (2 x 30 mL). The combined organic layers were dried (anhyd. Na_2SO_4) and concentrated under reduced pressures. Flash chromatography on silica gel (80:20–60:40 hexanes:ethyl acetate) affords the title compound **12** (640 mg, 87%) as a white solid: m.p. 106.0–107.5 ºC; TLC analysis R_f 0.5 (60:40 hexanes:ethyl acetate); $[\alpha]_D^{20}$ = +77.7º (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (10H, m, aryl), 6.54 (1H, d, *J* = 4.9 Hz, NH, *overlapped with* j), 6.54 (1H, d, *J* = 15.7 Hz, j), 6.15 (1H, dd, *J* = 15.9 and 7.1 Hz, i), 4.54 (1H, dd, *J* = 14.8 and 5.9 Hz, e), 4.35–4.45 (2H, m, e,h), 2.40–2.50 (1H, m, g), 1.22 (3H, d, *J* = 7.1 Hz, o), 0.13 (9H, s, p,p',p''); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.49 (f), 138.68 (d), 136.64 (k), 131.58 (j), 130.42 (i), 128.75 (aryl), 128.72 (aryl), 127.87 (aryl), 127.43 (aryl), 126.67 (aryl), 76.08 (h), 48.42 (g), 43.57 (e), 15.19 (o), 0.39 (p,p',p''); IR (neat) 3260 (N-H stretch), 2958, 1642 (C=O stretch), 1563, 1247, 1051, 970, 883, 838, 736, 692 cm^{-1}.

General procedure for CAHB without subsequent oxidation. (GP4)

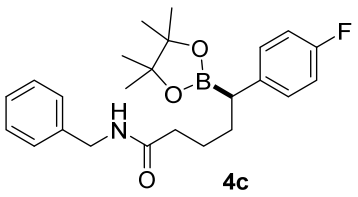


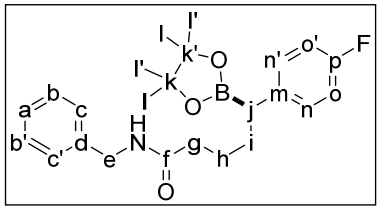
(*R*)-5-phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4a). To a yellow solution of 0.5 mol% [Rh(nbd)$_2$BF$_4$/ 2(*R*)-**B1**] (i.e., Rh(nbd)$_2$BF$_4$ (1.0 mg, 2.64 μmol) and (*R*)-(BINOL)PN(Me)Ph **B1** (2.23 mg, 5.28 μmol)) in THF (2.0 mL) was added γ,δ-unsaturated amide **3a** (140 mg, 0.528 mmol) as a solution in THF (2.0 mL). To the resulting solution was added dropwise a solution of 4,4,5,5-tetramethyl-1,3,2-dioxaborolane (pinBH, 101 mg, 0.8 mmol, 1.5 equiv) in THF (1.0 mL). The mixture was then stirred at 40 ºC for 12h. Afterwards, the reaction was concentrated under reduced pressure and purified via flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate) to afford the title compound (185 mg, 89%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10–7.40 (10H, m, a,b,b',c,c',n,n',o,o',p), 5.76 (1H, br s, NH), 4.44 (2H, d, J = 5.6 Hz, e), 2.32 (1H, t, J = 7.3 Hz, j), 2.23 (2H, t, J = 7.4 Hz, g), 1.80–1.95 (1H, m, h), 1.60–1.75 (3H, m, h,i), 1.21 and 1.19 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.76 (f), 142.94 (m), 138.55 (d), 128.79 (b,b'), 128.48 (n,n',o,o'), 127.93 (c,c'), 127.56 (a), 125.43 (p), 83.52 (k,k'), 43.64 (e), 36.81 (g), 32.25 (h), 25.44 (i), 24.73 and 24.70 (l,l').



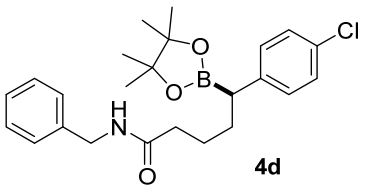
(*R*)-5-(4-trifluoromethyl)phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) pentanecarboxylic acid benzyl amide (4b). Following **GP4** with (*R*)-**B3** (2.55 mg, 5.28 μmol) and **3b** (176 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (190 mg, 78%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (2H, d, J = 8.1 Hz, o,o'), 7.25–7.35 (7H, m, a,b,b',c'c',n,n'), 5.87 (1H, br s, NH), 4.43 (2H, d, J = 5.5 Hz, e), 2.40 (1H, t, J = 7.2 Hz, j), 2.22 (2H, t, J = 7.4 Hz, g), 1.80–2.00 (1H, m, h), 1.60–1.80 (3H, m, h,i), 1.21 and 1.20 (12H,

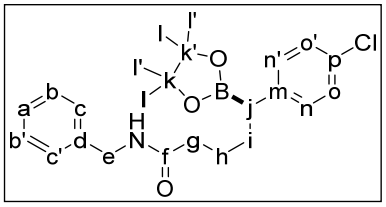
s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.62 (f), 147.35 (m), 138.49 (d), 128.80 (aryl), 128.67 (aryl), 127.92 (aryl), 127.60 (aryl), 125.37 (q, *J* = 3.7 Hz, aryl), 83.79 (k,k'), 43.66 (e), 36.63 (g), 31.93 (h), 25.33 (i), 24.71 and 24.69 (l,l').





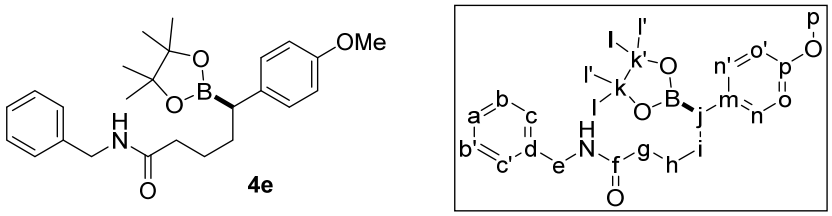
(*R*)-5-(4-fluoro)phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4c). Following **GP4** with **3c** (150 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (167 mg, 77%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.40 (5H, m, aryl), 7.14 (2H, dd, *J* = 8.6 and 5.5 Hz, aryl), 6.94 (2H, *J* = 8.8 Hz, aryl), 5.81 (1H, br s, NH), 4.44 (2H, d, *J* = 5.9 Hz, e), 2.30 (1H, t, *J* = 7.3 Hz, j), 2.22 (2H, t, *J* = 7.4 Hz, g), 1.80–1.90 (1H, m, h), 1.60–1.75 (3H, m, f,h), 1.21 and 1.19 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.69 (f), (d, *J* = 242 Hz, p), 138.54 (d), 129.74 (aryl), 129.66 (aryl), 128.79 (aryl), 127.83 (aryl), 127.59 (aryl), 115.19 (d, *J* = 21 Hz, o,o'), 83.59 (k,k'), 43.65 (e), 36.73 (g), 32.35 (h), 25.33 (i), 24.72 and 24.69 (l,l').



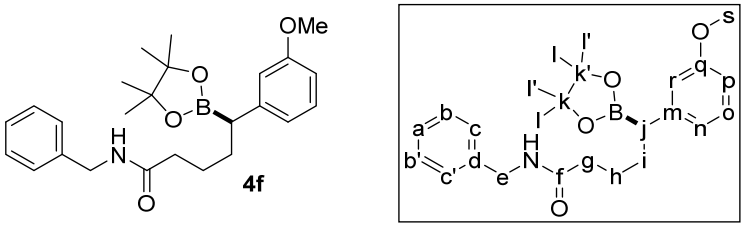


(*R*)-5-(4-chloro)phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4d). Following **GP4** with (*R*)-**B2** (2.63 mg, 5.28 μmol) and **3d** (158 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (169 mg, 75%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.35 (5H, m, a,b,b',o,o',p), 7.22 (2H, d, *J* = 8.4 Hz, c,c'), 7.12 (2H, d, *J* = 8.4 Hz, n,n'), 5.90 (1H, br s, NH), 4.42 (2H, d, *J* = 5.7 Hz, e), 2.29 (1H, t, *J* = 7.3 Hz, j), 2.20 (2H, t, *J* = 7.4 Hz, g), 1.80–1.90 (1H, m, h), 1.50–1.70 (3H, m, f,h), 1.21 and 1.19 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.68 (f), 141.50 (m), 138.55 (d), 131.09 (p). 129.78 (n,n'),

128.79 (b,b'), 128.56 (o,o'), 127.92 (c,c'), 127.57 (a), 83.66 (k,k'), 43.63 (e), 36.67 (g), 32.10 (h), 25.33 (i), 24.73 and 24.70 (l,l').

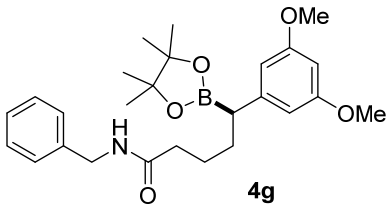


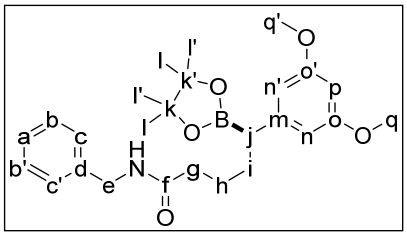
(*R*)-5-(4-methoxy)phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4e). Following **GP4** with (*R*)-**B2** (2.63 mg, 5.28 μmol) and **3e** (156 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (177 mg, 79%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (5H, m, a,b,b',c,c'), 7.10 (2H, d, J = 8.6 Hz, n,n'), 6.80 (2H, d, J = 8.6 Hz, o,o'), 5.94 (1H, br s, NH), 4.41 (2H, d, J = 5.7 Hz, e), 3.77 (3H, s, s), 2.15–2.30 (3H, m, g,j), 1.80–1.90 (1H, m, h), 1.55–1.70 (3H, m, h,i), 1.21 and 1.19 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.92 (f), 157.51 (q), 138.59 (d), 134.90 (m), 129.34 (n,n'), 128.75 (b,b'), 127.90 (c,c,'), 127.51 (a), 113.94 (o,o'), 83.46 (k,k'), 55.28 (s), 43.59 (e), 36.76 (g), 32.49 (h), 25.39 (i), 24.73 and 24.71 (l,l').



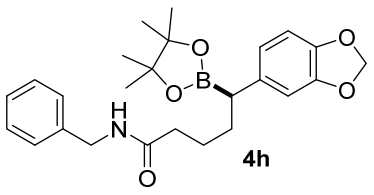
(*R*)-5-(3-methoxy)phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4f). Following **GP4** with **3f** (156 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (161 mg, 72%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (5H, m, aryl), 7.17 (1H, t, J = 7.7 Hz, aryl), 6.75–6.85 (2H, m, aryl), 6.71 (1H, d, J = 7.8 Hz, aryl), 5.87 (1H, br s, NH)), 4.43 (2H, d, J = 5.5 Hz, e), 3.79 (3H, s, s), 2.15–2.35 (3H, m, g,j), 1.80–1.95 (1H, m, h), 1.55–1.80 (3H, m, h,i), 1.22 and 1.20 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.81 (f), 159.72 (q), 144.61 (m), 138.85 (d), 129.35 (o), 128.77 (b,b'), 127.90 (c,c,'),

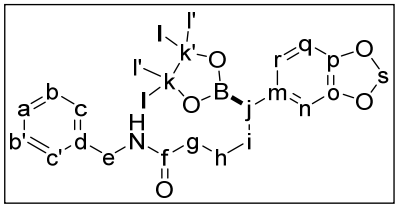
127.53 (a), 120.99 (n), 114.14 (p), 110.90 (r), 83.53 (k,k'), 55.18 (s), 43.62 (e), 36.79 (g), 32.26 (h), 25.46 (i), 24.73 (l,l').





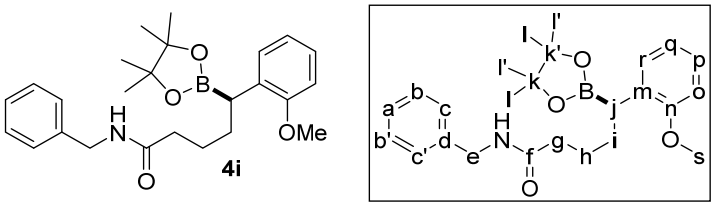
(*R*)-5-(3,5-dimethoxy)phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4g). Following **GP4** with (*R*)-**B2** (2.63 mg, 5.28 μmol) and **3g** (172 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (191 mg, 80%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.35 (5H, m, a,b,b',c,c'), 6.38 (2H, d, J = 2.2 Hz, n,n'), 6.37 (1H, t, J = 2.2 Hz, p), 5.80 (1H, br s, NH), 4.44 (2H, d, J = 5.7 Hz, e), 3.77 (6H, s, q,q'), 2.15–2.30 (3H, m, g,j), 1.80–1.90 (1H, m, h), 1.60–1.75 (3H, m, h,i), 1.22 and 1.21 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.79 (f), 160.78 (o,o'), 145.39 (m), 138.55 (d), 128.78 (b,b'), 127.90 (c,c,'), 127.55 (a), 106.54 (n,n'), 97.71 (p), 83.55 (k,k'), 55.30 (s), 43.64 (e), 36.82 (g), 32.25 (h), 25.45 (i), 24.77 and 24.71 (l,l').



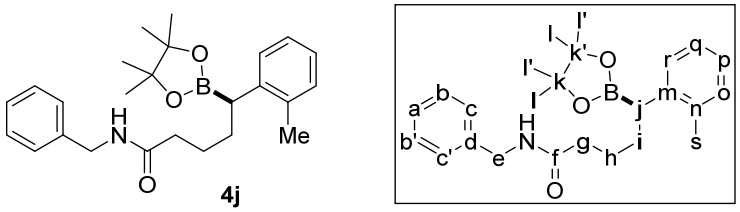


(*R*)-5-(benzo[*d*][1,3]dioxol-5-yl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4h). Following **GP4** with (*R*)-**B2** (2.63 mg, 5.28 μmol) and **3h** (163 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (164 mg, 71%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.35 (5H, m, a,b,b',c,c'), 6.70–6.75 (2H, m, aryl), 6.60–6.65 (1H, m, aryl), 5.90–5.95 (2H, m, s), 5.86 (1H, br s, NH), 4.43 (2H, d, J = 5.7 Hz, e), 2.15–2.25 (3H, m, g,j), 1.80–1.90 (1H, m, h), 1.60–1.70 (3H, m, h,i), 1.22 and 1.20 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.80 (f), 147.67 (o), 145.38 (p), 138.56 (d), 136.74

(m), 128.78 (b,b'), 127.91 (c,c,'), 127.56 (a), 121.28 (aryl), 108.91 (aryl), 108.31 (aryl), 100.77 (s), 83.53 (k,k'), 43.63 (e), 36.77 (g), 32.57 (h), 25.31 (i), 24.74 and 24.72 (l,l').

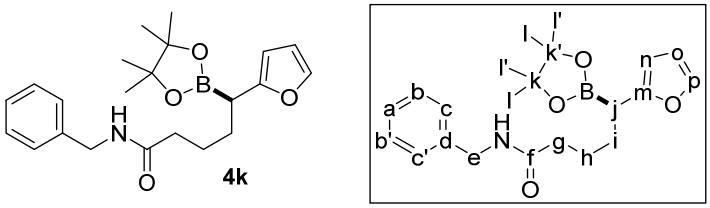


(*R*)-5-(2-methoxy)phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4i). Following **GP4** with **3i** (156 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (183 mg, 82%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.35 (2H, m, b,b'), 7.25–7.30 (3H, m, a,c,c'), 7.10–7.20 (2H, m, q,r), 6.88 (1H, t, J = 7.0 Hz, p), 6.82 (1H, d, J = 8.3 Hz, o), 5.95 (1H, br s, NH), 4.42 (2H, dd, J = 5.5 and 1.8 Hz, e), 3.77 (3H, s, s), 2.43 (1H, t, J = 6.9 Hz, j), 2.22 (2H, td, J = 8.1 and 2.8 Hz, g), 1.80–1.95 (1H, m, h), 1.55–1.75 (3H, m, h,i), 1.24 and 1.21 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.14 (f), 157.04 (n), 138.68 (d), 131.87 (m), 130.12 (r), 128.74 (b,b'), 127.87 (c,c,'), 127.47 (a), 126.64 (q), 120.74 (p), 110.20 (o), 83.23 (k,k'), 55.15 (s), 43.56 (e), 36.84 (g), 30.54 (h), 25.47 (i), 24.90 and 24.77 (l,l').

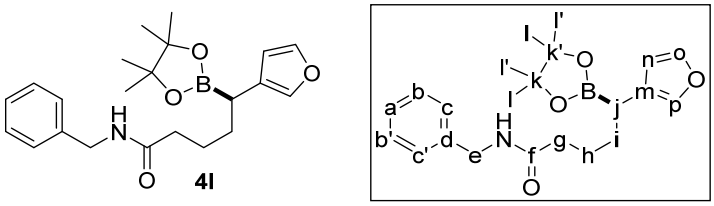


(*R*)-5-(2-methyl)phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4j). Following **GP4** with (*R*)-**B2** (2.63 mg, 5.28 μmol) and **3j** (148 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (181 mg, 84%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.35 (2H, m, b,b'), 7.25–7.30 (3H, m, c,c',o), 7.19 (1H, d, J = 7.5 Hz, a), 7.10–7.15 (2H, m, q,r), 7.00–7.10 (1H, m, p), 5.92 (1H, br s, NH), 4.43 (2H, d, J = 5.9 Hz, e), 2.54 (1H, t, J = 7.0 Hz, j), 2.34 (3H, s, s), 2.23 (2H, t, J = 6.8 Hz, g), 1.85–2.00 (1H, m, h), 1.60–1.80 (3H, m, h,i), 1.22 and 1.20 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.84 (f),

141.44 (m), 138.61 (d), 136.08 (n), 130.35 (o), 128.77 (b,b'), 127.91 (c,c,'), 127.86 (r), 127.53 (a), 126.07 (p), 125.25 (q), 83.44 (k,k'), 43.61 (e), 36.90 (g), 31.86 (h), 25.64 (i), 24.80 and 24.70 (l,l'), 20.30 (s).

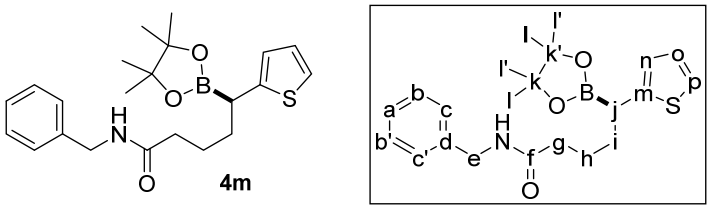


(*R*)-5-(2-furanyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4k). Following **GP4** with (*R*)-**B3** (2.55 mg, 5.28 μmol) and 3 equiv pinBH (203 mg, 0.16 mmol) and **3k** (135 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (140 mg, 69%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.40 (2H, m, b,b'), 7.25–7.30 (4H, m, a,c,c',p), 6.27 (1H, t, *J* = 2.5 Hz, o), 6.03 (1H, d, *J* = 2.9 Hz, n), 5.84 (1H, br s, NH), 4.45 (2H, d, *J* = 5.6 Hz, e), 2.49 (1H, t, *J* = 7.1 Hz, j), 2.23 (2H, t, *J* = 7.4 Hz, g), 1.75–1.85 (2H, m, h), 1.65–1.75 (2H, m, i), 1.26 and 1.25 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.74 (f), 156.37 (m), 140.86 (p), 138.54 (d), 128.78 (b,b'), 127.91 (c,c,'), 127.55 (a), 110.29 (o), 105.10 (n), 83.83 (k,k'), 43.85 (e), 36.65 (g), 29.81 (h), 25.20 (i), 24.80 and 24.73 (l,l').

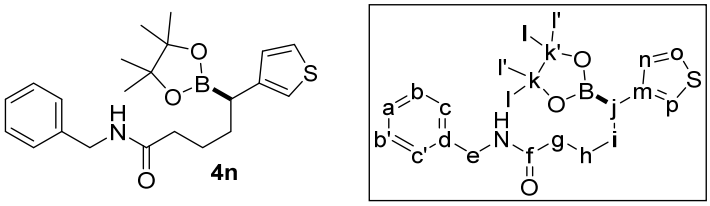


(*R*)-5-(3-furanyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4l). Following **GP4** with with (*R*)-**B2** (2.63 mg, 5.28 μmol) and 3 equiv pinBH (203 mg, 0.16 mmol) and **3l** (135 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (146 mg, 72%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (7H, m, a,b,b',c,c',o,p), 6.28 (1H, d, *J* = 0.0 Hz, n), 5.90 (1H, br s, NH), 4.43 (2H, d, *J* = 5.6 Hz, e), 2.15–2.30 (3H, m, g,j), 1.60–1.80 (2H, m, h,i), 1.23 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.90

(f), 142.62 (o), 138.89 (p), 138.55 (d), 128.77 (b,b'), 127.89 (c,c,'), 127.54 (a), 125.20 (m), 111.13 (n), 83.59 (k,k'), 43.62 (e), 36.69 (g), 31.32 (h), 25.26 (i), 24.79 and 24.74 (l,l').

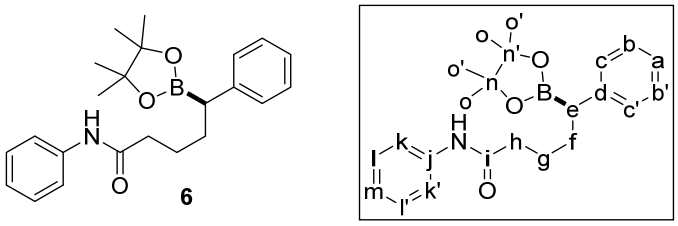


(*R*)-5-(2-thiophenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4m). Following **GP4** with with (*R*)-**B2** (2.63 mg, 5.28 μmol) and 3 equiv pinBH (203 mg, 0.16 mmol) and **3m** (143 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (152 mg, 72%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.40 (2H, m, b,b'), 7.25–7.30 (3H, m, a,c,c'), 7.09 (1H, dd, *J* = 5.1 and 0.9 Hz, p), 6.91 (1H, dd, *J* = 5.1 and 3.4 Hz, o), 6.81 (1H, d, *J* = 3.4 Hz, n), 5.82 (1H, br s, NH), 4.44 (2H, d, *J* = 5.7 Hz, e), 2.66 (1H, t, *J* = 7.1 Hz, j), 2.24 (2H, t, *J* = 7.3 Hz, g), 1.85–1.95 (1H, m, h), 1.65–1.80 (3H, m, h,i), 1.24 and 1.23 (12H, s, l,l'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.66 (f), 145.87 (m), 138.53 (d), 128.80 (b,b'), 127.92 (c,c,'), 127.57 (a), 126.88 (o), 124.09 (n), 122.78 (p), 83.84 (k,k'), 43.66 (e), 36.67 (g), 33.28 (h), 25.18 (i), 24.74 and 24.73 (l,l').

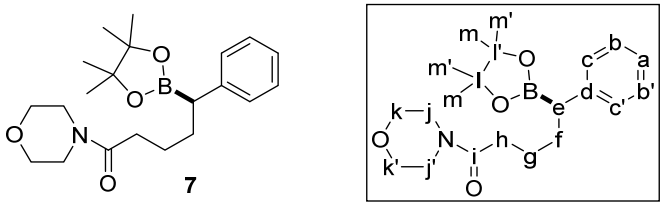


(*R*)-5-(2-thiophenyl)-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic acid benzyl amide (4n). Following **GP4** with with (*R*)-**B2** (2.63 mg, 5.28 μmol) and 3 equiv pinBH (203 mg, 0.16 mmol) and **3n** (143 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (154 mg, 73%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.35 (2H, m, b,b'), 7.25–7.30 (3H, m, a,c,c'), 7.22 (1H, dd, *J* = 4.8 and 3.0 Hz, o), 6.90–7.00 (2H, m, n,p), 5.91 (1H, br s, NH), 4.43 (2H, d, *J* = 5.7 Hz, e), 2.48 (1H, t, *J* = 7.1 Hz, j), 2.22 (2H, t, *J* = 7.4 Hz, g), 1.80–1.90 (1H, m, h), 1.60–1.80 (3H, m, h,i), 1.22 and 1.21 (12H, s, l,l'); ^{13}C NMR (100 MHz,

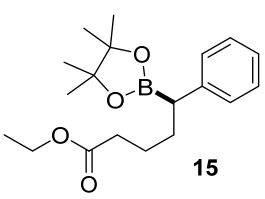
$CDCl_3$) δ 172.82 (f), 142.67 (m), 138.57 (d), 128.78 (b,b'), 128.30 (n), 127.92 (c,c,'), 127.55 (a), 125.07 (o), 119.70 (p), 83.58 (k,k'), 43.63 (e), 36.72 (g), 31.88 (h), 25.40 (i), 24.77 and 24.72 (l,l').

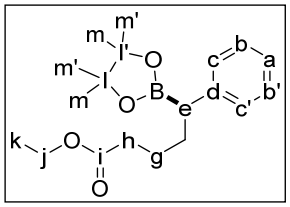


(*R*)-5-phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic phenyl amide (6). Following **GP4** with **5a** (133 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (152 mg, 76%) as a yellow oil: TLC analysis R_f 0.6 (50:50 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (2H, d, J = 7.8 Hz, k,k'), 7.20–7.35 (6H, m, b,b'c,c',l,l'), 7.16 (1H, t, J = 7.1 Hz, a), 7.11 (1H, t, J = 7.1 Hz, m), 2.30–2.40 (3H, e,h), 1.85–2.00 (1H, m, g), 1.65–1.80 (3H, m, f,g), 1.23 and 1.21 (12H, s, o,o'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.31 (i), 142.91 (d), 138.11 (j), 129.04 (l,l'), 128.52 (b,b',c,c'), 125.49 (a), 124.23 (m), 120.00 (k,k'), 83.58 (n,n'), 37.79 (g), 32.14 (h), 25.36 (f), 24.76 and 24.73 (o,o').

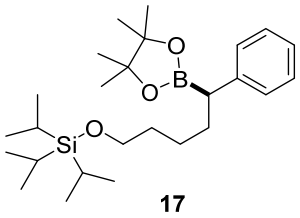


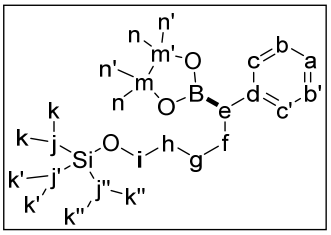
(*R*)-5-phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic phenyl amide (7). Following **GP4** with (*R*)-**B2** (2.63 mg, 5.28 μmol) and **5b** (130 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (154 mg, 78%) as a yellow oil: TLC analysis R_f 0.4 (30:70 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15–7.25 (4H, m, b,b',c,c'), 7.12 (1H, t, J = 7.0 Hz, a), 3.55–3.65 (6H, m, j,j',k,k'), 3.30–3.35 (2H, m, j,j'), 2.20–2.35 (3H, m, e,h), 1.85–1.95 (1H, m, g), 1.65–1.75 (1H, m, g), 1.55–1.65 (2H, m, f), 1.20 and 1.18 (12H, s, m,m'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.76 (i), 142.90 (d), 128.48 (b,b'), 128.43 (c,c'), 125.39 (a), 83.46 (l,l'), 67.00 and 67.71 (k,k'), 46.10 and 41.91 (j, j'), 33.32 (g), 32.22 (h), 24.86 (f), 24.74 and 24.71 (m,m').



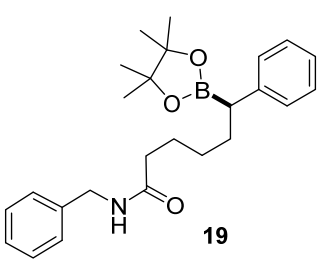


(*R*)-5-phenyl-5-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)pentanecarboxylic ethyl ester (15). Following **GP4** with **14** (108 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5 hexanes:ethyl acetate), the title compound (135 mg, 77%) as a yellow oil: TLC analysis R_f 0.7 (80:20 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.30 (2H, m, b,b'), 7.15–7.25 (2H, m, c,c'), 7.10–7.15 (1H, m, a), 4.12 (2H, q, J = 7.1 Hz, j), 2.20–2.40 (3H, m, e,h), 1.85–1.95 (1H, m, g), 1.65–1.75 (1H, m, g), 1.55–1.65 (2H, m, f), 1.25 (3H, t, J = 7.2 Hz, k), 1.23 and 1.21 (12H, s, m,m'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.74 (i), 142.94 (d), 128.46 (b,b'), 128.43 (c,c'), 125.37 (a), 83.45 (l,l'), 60.23 (j), 34.48 (g), 32.10 (h), 24.74 and 24.69 (f,m,m'), 14.34 (k).

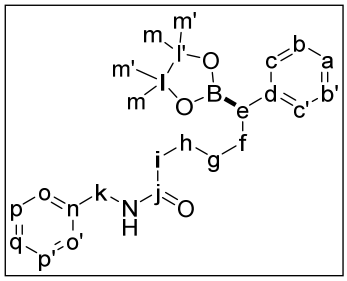




(*R*)-2-(5-((3-isopropyl-2,4-dimethylpentan-3-yl)oxy)-1-phenylpentyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (17). Following **GP4** with **16** (168 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5 hexanes:ethyl acetate), the title compound (170 mg, 75%) as a colorless oil: TLC analysis R_f 0.7 (80:20 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.30 (4H, m, b,b',c,c'), 7.10–7.20 (1H, m, a), 3.67 (2H, t, J = 6.6 Hz, l), 2.34 (1H, t, J = 7.9 Hz, e), 1.85–2.00 (1H, m, h), 1.65–1.75 (1H, m, h), 1.50–1.65 (2H, m, g), 1.30–1.45 (2H, m, f), 1.24 and 1.22 (12H, s, n,n'), 1.05–1.10 (21H, m, j,j',j'',k,k',k''); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.51 (d), 128.49 (b,b'), 128.34 (c,c'), 125.20 (a), 83.33 (m,m'), 63.57 (l), 33.24 (g), 32.70 (h), 25.77 (f), 24.74 and 24.71 (n,n'), 18.17 (k,k',k''), 12.14 (j,j',j'').



19



(*R*)-6-phenyl-6-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)hexanecarboxylic benzyl amide (19). Following **GP4** with **18** (148 mg, 0.528 mmol) affords, after flash chromatography on silica gel (95:5–90:10 DCM:ethyl acetate), the title compound (168 mg, 78%) as a yellow oil: TLC analysis R_f 0.4 (40:60 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15–7.40 (9H, m, b,b',c,c',o,o',p,p',q), 7.14 (1H, t, *J* = 7.1 Hz, a), 5.72 (1H, br s, NH), 4.42 (2H, d, *J* = 5.7 Hz, k), 2.30 (1H, t J = 7.8 Hz, e), 2.18 (2H, t, *J* = 7.2 Hz, i), 1.80–1.90 (1H, m, h), 1.60–1.80 (3H, m, f,h), 1.25–1.40 (2H, m, g), 1.22 and 1.20 (12H, s, m,m'); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.97 (j), 143.25 (n), 138.54 (d), 128.81 (b,b'), 128.48 (p,p'), 128.44 (c,c'), 127.92 (q), 125.30 (a), 83.41 (l,l'), 43.66 (k), 36.79 (i), 32.24 (h), 28.97 (g), 25.87 (f), 24.75 and 24.69 (m,m').

General procedure for oxidation of secondary benzylic boronic esters for further characterizations (GP5)

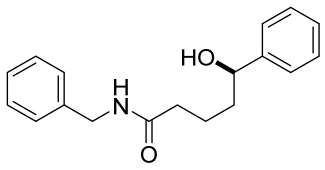
GP5A (mild conditions): for boronic esters containing versatile directing groups (i.e., ethylesters and morpholino amides). To a solution of boronic ester (0.1056 mmol) in THF (1.0 mL) was added $NaBO_3$ (100 mg) and water (1.0 mL) at room temperature. The resultant mixture was stirred for 5 hours then extracted with ethyl acetate (3 x 2 mL), dried (anhyd. Na_2SO_4), and concentrated under reduced pressure. Flash chromatography on silica gel affords the title compound.

GP5B (harsh conditions): To a solution of boronic ester (0.1056 mmol) in THF (1.0 mL) was added MeOH (1.0 mL), NaOH (3M, 1.5 mL), and H_2O_2 (30%, 0.2 mL) at room temperature. The resultant mixture was shaken (with glass ball) or stirred for 1 hour then extracted with DCM (3 x 2 mL), dried (anhyd. Na_2SO_4), and concentrated under reduced pressure. Flash chromatography on silica gel affords the title compound.

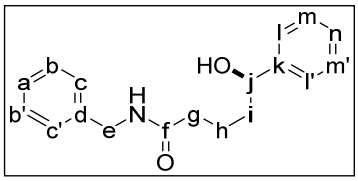
Isolated yields range from 92–99%.

Column chromatography conditions:

1. **For ethylester-, OTIPS-, and phenyl amide-containing boronic esters:** 90:10–60:40 hexanes:ethyl acetate.
2. **For benzyl amide-containing boronic esters:** 60:40 hexanes:ethyl acetate until all pinacol diol (visible with Vanillin stain) side-product came out, then switch to 100% ethyl acetate.
3. **For morpholino-containing boronic esters:** 60:40 hexanes:ethyl acetate until all pinacol diol (visible with Vanillin stain) side-product came out, then switch to 100% acetone.

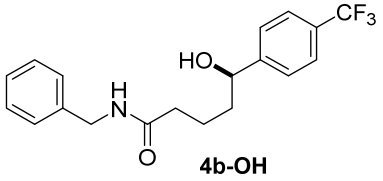


4a-OH

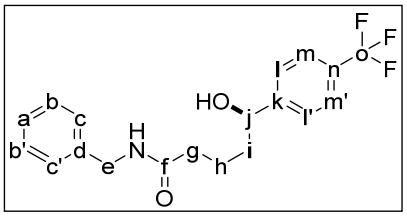


(*R*)-5-hydroxy-5-phenylpentanecarboxylic acid benzyl amide (4a-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a semi-solid; $[\alpha]_D^{20}$ = –16.3° (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (10H, m, a,b,b',c,c',l,l',m,m',n),

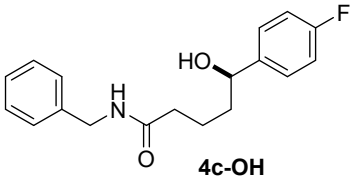
6.24 (1H, br s, NH), 4.60–4.70 (1H, m, j), 4.37 (2H, d, J = 5.7 Hz, e), 3.17 (1H, br s, OH), 2.22 (2H, t, J = 6.8 Hz, g), 1.65–1.85 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.19 (f), 144.92 (k), 138.48 (d), 128.78 (m,m'), 128.52 (b,b'), 127.90 (l,l'), 127.56 (n), 127.51 (a), 125.93 (c,c'), 73.97 (j), 43.63 (e), 38.56 (i), 36.20 (g), 22.04 (h); Enantiomeric ratio was checked by converting to the corresponding Mosher's ester **4a-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.36 (major 95%) and –71.61 (minor 5%); IR (neat) 3285 (O–H stretch), 3029 (N–H stretch), 2925, 1642 (C=O stretch), 1544, 1494, 1453, 1247, 1027, 749, 696 cm^{-1}; HRMS (EI) calcd. for $C_{18}H_{21}NO_2$ (M): 283.1572, found 283.1578 *m/z*.



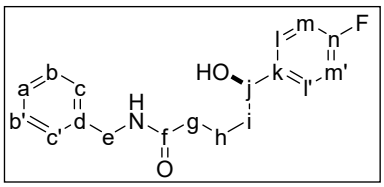
4b-OH



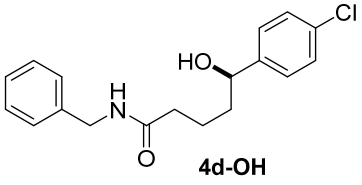
(*R*)-5-hydroxy-5-(4-(trifluoromethyl)phenyl)pentanecarboxylic acid benzyl amide (4b-OH). Following **GPxB** affords, after flash chromatography on silica gel the title compound as a white solid: 71.5–72.5 ºC; $[\alpha]_D^{20}$ = –22.5º (*c* 1.0, $CHCl_3$); ^{19}F NMR (376 MHz, $CDCl_3$) δ –62.39 (CF_3); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (2H, d, J = 8.1 Hz, m,m'), 7.44 (2H, d, J = 8.1 Hz, c,c'), 7.20–7.35 (5H, m, a,b,b',l,l'), 6.00 (1H, br s, NH), 4.70–4.80 (1H, m, j), 4.41 (2H, d, J = 5.7 Hz, e), 3.34 (1H, br s, OH), 2.28 (2H, t, J = 6.7 Hz, g), 1.70–1.85 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.00 (f), 148.91 (k), 138.25 (d), 129.86 (n), 129.60 (q, J = 32.1 Hz, o), 128.85 (b,b'), 127.91 (l,l'), 127.70 (a), 126.15 (c,c'), 125.43 (q, J = 3.8 Hz, m,m'), 73.23 (j), 43.76 (e), 38.63 (i), 35.97 (g), 21.59 (h); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4b-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.28 (major 95%) and –71.47 (minor 5%); IR (neat) 3296 (O–H stretch), 2924, 1644 (C=O stretch), 1545, 1323, 1161, 1110, 1066, 1016, 840, 730, 697, 603 cm^{-1}; HRMS (ESI) calcd. for $C_{19}H_{20}F_3NNaO_2$ (M+Na): 374.1344, found 374.1351 *m/z*.



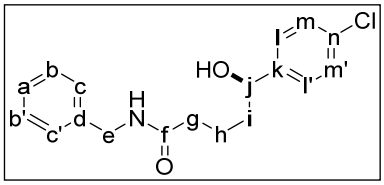
4c-OH



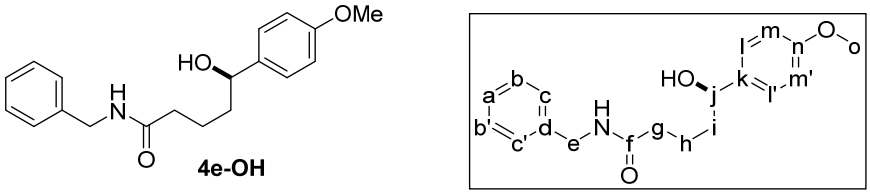
(*R*)-5-(4-fluorophenyl)-5-hydroxypentanecarboxylic acid benzyl amide (4c-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a white solid: 64.5–65.5 °C; $[\alpha]_D^{20}$ = –20.1° (*c* 2.0, $CHCl_3$); ^{19}F NMR (376 MHz, $CDCl_3$) δ –115.30 to –115.40 ($C(sp^2)$–F); ^1H NMR (400 MHz, $CDCl_3$) δ 7.25–7.35 (7H, m, a,b,b',c,c',l,l'), 7.00 (2H, t, *J* = 8.7 Hz, m,m'), 6.11 (1H, br s, NH), 4.60–4.70 (1H, m, j), 4.39 (2H, d, *J* = 5.7 Hz, e), 3.09 (1H, br s, OH), 2.24 (2H, t, *J* = 6.9 Hz, g), 1.65–1.80 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.07 (f), 162.17 (d, *J* = 243 Hz, n), 140.65 and 140.62 (k), 138.36 (d), 128.82 (b,b'), 127.90 (l,l'), 127.63 and 127.56 and 127.48 (a,c,c'), 115.28 (d, *J* = 21 Hz, m,m'), 73.30 (j), 43.69 (e), 38.64 (i), 36.11 (g), 21.87 (h); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4c-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.25 (major 95%) and –71.51 (minor 5%); IR (neat) 3283 (O–H stretch), 2921, 1643 (C=O stretch), 1548, 1508, 1219, 1155, 1076, 1014, 835, 730, 697 cm^{-1}; HRMS (ESI) calcd. for $C_{18}H_{20}FNNaO_2$ (M+Na): 324.1376, found 324.1385 *m/z*.



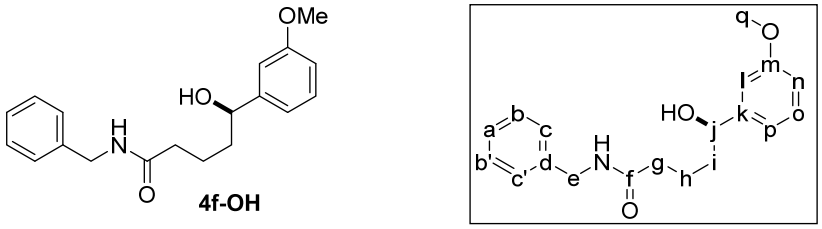
4d-OH



(*R*)-5-(4-chlorophenyl)-5-hydroxypentanecarboxylic acid benzyl amide (4d-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a white solid: 108.5–110.0 °C; $[\alpha]_D^{20}$ = –21.1° (*c* 2.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (9H, m, a,b,b',c,c',l,l',m,m'), 6.02 (1H, br s, NH), 4.60–4.80 (1H, m, j), 4.40 (2H, d, *J* = 5.7 Hz, e), 3.10 (1H, br s, OH), 2.25 (2H, t, *J* = 6.4 Hz, g), 1.65–1.85 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.89 (f), 143.31 (k), 138.32 (d), 133.14 (n), 128.86 (b,b'), 128.66 (m,m'), 127.96 (l,l'), 127.70 (a), 127.29 (c,c'), 73.32 (j), 43.77 (e), 38.58 (i), 36.10 (g), 21.71 (h). Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4d-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.35 (major 95%) and –71.57 (minor 5%); IR (neat) 3281 (O–H stretch), 2915, 2360, 1629 (C=O stretch), 1531, 1486, 1454, 1077, 1009, 936, 831, 799, 749, 698 cm^{-1}; HRMS (EI) calcd. for $C_{18}H_{20}ClNO_2$ (M): 317.1183, found 317.1189 *m/z*.

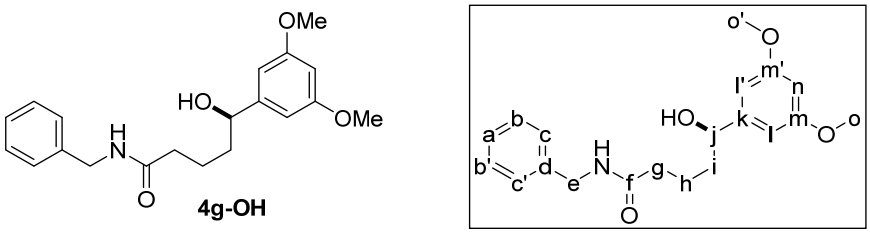


(*R*)-5-hydroxy-5-(4-methoxyphenyl)pentanecarboxylic acid benzyl amide (4e-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a colorless oil; $[\alpha]_D^{20}$ = –21.0° (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (7H, m, a,b,b',c,c',l,l'), 6.86 (2H, d, *J* = 8.6 Hz, m,m'), 6.11 (1H, br s, NH), 4.61 (1H, dd, *J* = 6.8 and 4.7 Hz, j), 4.39 (2H, d, *J* = 5.7 Hz, e), 3.80 (3H, s, o), 2.78 (1H, br s, OH), 2.23 (2H, t, *J* = 6.8 Hz, g), 1.65–1.85 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.94 (f), 159.11 (n), 138.44 (d), 136.94 (k), 128.82 (b,b'), 127.95 (l,l'), 127.61 (a), 127.16 (c,c'), 113.95 (m,m'), 73.76 (j), 55.40 (o), 43.71 (e), 38.44 (i), 36.31 (g), 22.06 (h); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4e-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.40 (major 96%) and –71.68 (minor 4%); IR (neat) 3287 (O–H stretch), 2924, 1643 (C=O stretch), 1611, 1543, 1510, 1454, 1242, 1174, 1029, 831, 732, 697 cm^{-1}; HRMS (ESI) calcd. for $C_{19}H_{23}NNaO_3$ (M+Na): 336.1576, found 336.1588 *m/z*.

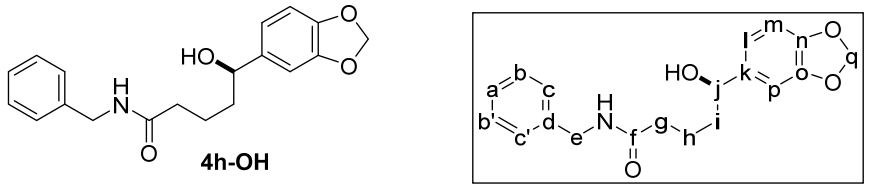


(*R*)-5-hydroxy-5-(3-methoxyphenyl)pentanecarboxylic acid benzyl amide (4f-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a colorless oil; $[\alpha]_D^{20}$ = –15.9° (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (6H, m, a,b,b',c,c',o), 6.85–6.90 (2H, m, l,p), 6.79 (1H, dd, *J* = 8.0 and 2.0 Hz, n), 6.32 (1H, dd, *J* = 5.3 and 0.0 Hz, NH), 4.61 (1H, dd, *J* = 7.4 and 4.3 Hz, j), 4.35 (2H, d, *J* = 5.7 Hz, e), 3.78 (3H, s, q), 3.36 (1H, br s, OH), 2.21 (2H, t, *J* = 6.8 Hz, g), 1.60–1.85 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.30 (f), 159.78 (m), 146.71 (k), 138.46 (d), 129.52 (o), 128.76 (b,b'), 127.86 (c,c'), 127.53 (a), 118.29 (p), 112.85 (n), 111.51 (l), 73.81 (j), 55.31 (q), 43.61 (e), 38.54 (i), 36.18 (g), 22.05 (h); Chiral HPLC analysis (Chiralcel-AD, 60:40 hexanes:isopropanol, flowrate = 1.0 mL/min) showed peaks at 15 minutes

(97.0% (*R*)) and 19 minutes (3.0% (*S*)); IR (neat) 3299 (O–H stretch), 2917, 1643 (C=O stretch), 1602, 1585, 1540, 1487, 1454, 1433, 1317, 1253, 1153, 1040, 783, 731, 696 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{23}NO_3$ (M): 313.1678, found 313.1680 *m/z*.

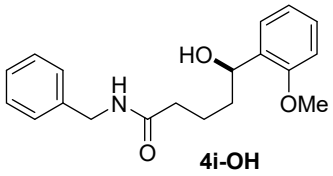


(*R*)-5-(3,5-dimethoxyphenyl)-5-hydroxypentanecarboxylic acid benzyl amide (4g-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a white solid: 110.0–110.5 ºC; $[\alpha]_D^{20}$ = –14.1º (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (5H, m, a,b,b',c,c',o), 6.50 (2H, d, *J* = 2.2 Hz, l,l'), 6.36 (1H, t, *J* = 2.2 Hz, n), 6.04 (1H, br s, NH), 4.55–4.65 (1H, m, j), 4.40 (2H, d, *J* = 5.7 Hz, e), 3.78 (6H, s, o,o'), 2.86 (1H, br s, OH), 2.25 (2H, t, *J* = 6.8 Hz, g), 1.65–1.85 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.02 (f), 160.95 (m,m'), 147.52 (k), 138.42 (d), 128.80 (b,b'), 127.91 (c,c'), 127.59 (a), 103.84 (l,l'), 99.41 (n), 74.08 (j), 55.44 (o,o'), 43.69 (e), 38.48 (i), 36.26 (g), 21.98 (h); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4g-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.34 (major 96.5%) and –71.50 (minor 3.5%); IR (neat) 3305, 3276 (O–H stretch), 2943, 2833, 2359, 2342, 1633 (C=O stretch), 1598, 1537, 1464, 1452, 1204, 1192, 1148, 1083, 1051, 859, 822, 747, 693 cm^{-1}; HRMS (EI) calcd. for $C_{20}H_{25}NO_4$ (M): 343.1784, found 343.1799 *m/z*.

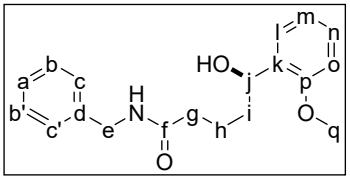


(*R*)-5-(benzo[d][1,3]dioxol-5-yl)-5-hydroxypentanecarboxylic acid benzyl amide (4h-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a colorless oil; $[\alpha]_D^{20}$ = –12.9º (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.40 (5H, m, a,b,b',c,c'), 6.80–6.90 (1H, m, p), 6.75–6.80 (2H, m, l,m), 5.99 (1H, br s, NH), 5.94 (2H, s, q), 4.59 (1H, dd, *J* = 7.4 and 4.7 Hz, j), 4.42 (2H, d, *J* = 5.7 Hz, e), 2.66 (1H, br s, OH), 2.25 (2H, t, *J* = 6.8 Hz, g),

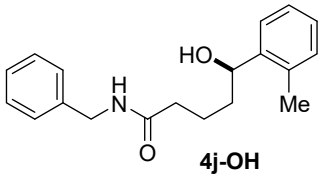
1.65–1.85 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.95 (f), 147.87 (o), 146.97 (n), 138.90 (k), 138.41 (d), 128.83 (b,b'), 127.95 (c,c'), 127.64 (a), 119.31 (l), 108.17 (m), 106.44 (p), 101.07 (q), 73.97 (j), 43.72 (e), 38.52 (i), 36.26 (g), 21.99 (h); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4h-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.33 (major 92%) and –71.64 (minor 8%); IR (neat) 3295 (O–H stretch), 2923, 1644 (C=O stretch), 1545, 1501, 1486, 1440, 1238, 1036, 933, 809, 729, 697 cm^{-1}; HRMS (ESI) calcd. for $C_{19}H_{21}NNaO_4$ (M+Na): 350.1368, found 350.1375 *m/z*.



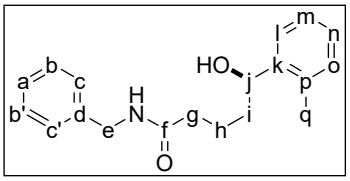
4i-OH



(*R*)-5-hydroxy-5-(2-methoxyphenyl)pentanecarboxylic acid benzyl amide (4i-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a white solid: 79.5–81.0 ºC; $[\alpha]_D^{20}$ = –10.8º (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (7H, m, a,b,b',c,c',l,m), 6.96 (1H, t, *J* = 7.1 Hz, n), 6.87 (1H, d, *J* = 8.2 Hz, o), 6.13 (1H, br s, NH), 4.85–5.00 (1H, m, j), 4.41 (2H, d, *J* = 5.7 Hz, e), 3.82 (3H, s, q), 3.02 (1H, br s, OH), 2.20–2.30 (2H, m, g), 1.70–1.90 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.16 (f), 156.44 (p), 138.57 (d), 132.61 (k), 128.77 (b,b'), 128.37 (l), 127.91 (c,c'), 127.52 (a), 126.85 (n), 120.90 (m), 110.58 (o), 70.03 (j), 55.38 (q), 43.63 (e), 36.74 (i), 36.43 (g), 22.41 (h); Chiral HPLC analysis (Chiralcel-AD, 60:40 hexanes:isopropanol, flowrate = 1.0 mL/min) showed peaks at 17 minutes (85.5% (*R*)) and 23 minutes (14.5% (*S*)); IR (neat) 3294 (O–H stretch), 2923, 1643 (C=O stretch), 1601, 1586, 1544, 1490, 1454, 1438, 1285, 1237, 1049, 1027, 752, 697 cm^{-1}; HRMS (ESI) calcd. for $C_{19}H_{23}NNaO_3$ (M+Na): 336.1576, found 336.1583 *m/z*.

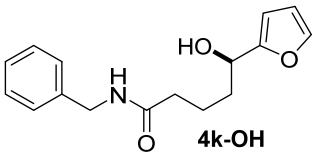


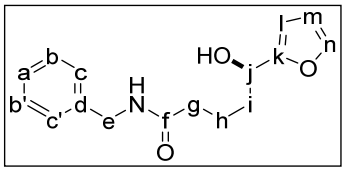
4j-OH



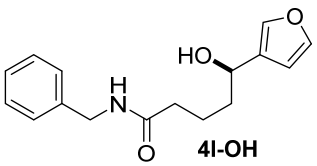
(*R*)-5-hydroxy-5-(2-methylphenyl)pentanecarboxylic acid benzyl amide (4j-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a semi solid; $[\alpha]_D^{20}$ = –24.6º (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (1H, d, *J* = 7.3 Hz, o), 7.10–7.35 (8H,

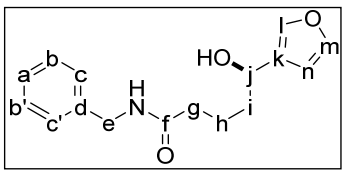
m, a,b,b',c,c',l,m,n), 6.20 (1H, br s, NH), 4.85–4.95 (1H, m, j), 4.39 (2H, d, *J* = 5.7 Hz, e), 2.91 (1H, br s, OH), 2.31 (3H, s, q), 2.20–2.30 (2H, m, g), 1.70–1.90 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.15 (f), 143.03 (k), 138.47 (d), 134.32 (p), 130.45 (n), 128.79 (b,b'), 127.91 (c,c'), 127.57 (a), 127.19 (l), 126.36 (m), 125.26 (o), 70.28 (j), 43.65 (e), 37.44 (i), 36.24 (g), 22.29 (h), 19.16 (q); Chiral HPLC analysis (Chiralpak-IC, 60:40 hexanes:isopropanol, flowrate = 1.4 mL/min) showed peaks at 14.5 minutes (93.0% (*R*)) and 16.5 minutes (7.0% (*S*)); IR (neat) 3292 (O–H stretch), 2920, 2360, 2342, 1635 (C=O stretch), 1538, 1495, 1453, 1059, 748, 724, 696 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1725 *m/z*.





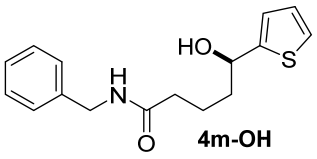
(*R*)-5-(2-furanyl)-5-hydroxypentanecarboxylic acid benzyl amide (4k-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a colorless oil; $[\alpha]_D^{20}$ = –14.2° (*c* 1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.40 (6H, m, a,b,b',c,c',n), 6.33 (1H, dd, *J* = 3.2 and 1.8 Hz, m), 6.24 (1H, d, *J* = 3.2 Hz, l), 6.03 (1H, br s), 4.69 (1H, dd, *J* = 11.1 and 6.2, j), 4.43 (2H, d, *J* = 5.7 Hz, e), 2.82 (1H, d, *J* = 4.7 Hz, OH), 2.28 (2H, t, *J* = 7.2 Hz, g), 1.75–1.95 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.93 (f), 156.77 (k), 141.95 (n), 138.41 (d), 128.82 (b,b'), 127.94 (c,c'), 127.62 (a), 110.28 (m), 106.94 (l), 67.36 (j), 43.72 (e), 36.08 (g), 34.96 (i), 21.60 (h); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4k-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.59 (major 97%) and –72.00 (minor 3%); IR (neat) 3283 (O–H stretch), 2926, 2360, 1640 (C=O stretch), 1544, 1454, 1250, 1070, 1008, 733, 697 cm^{-1}; HRMS (ESI) calcd. for $C_{16}H_{19}NNaO_3$ (M+Na): 296.1263, found 296.1265 *m/z*.

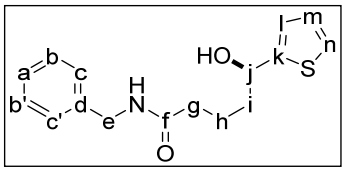




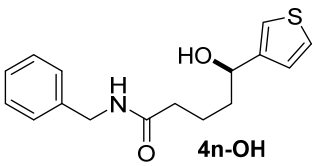
(*R*)-5-(3-furanyl)-5-hydroxypentanecarboxylic acid benzyl amide (4l-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a colorless oil; $[\alpha]_D^{20}$ = –

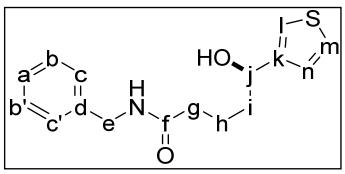
19.6° (*c* 1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.25–7.40 (7H, m, a,b,b',c,c',l,m), 6.39 (1H, m, n), 5.97 (1H, br s), 4.60–4.70 (1H, m, j), 4.44 (2H, d, *J* = 5.7 Hz, e), 2.57 (1H, br s, OH), 2.29 (2H, t, *J* = 6.4 Hz, g), 1.75–1.85 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.95 (f), 143.42 (m), 139.09 (l), 138.38 (d), 129.16 (k), 128.84 (b,b'), 127.95 (c,c'), 127.66 (a), 108.59 (n), 66.54 (j), 43.75 (e), 37.26 (i), 36.13 (g), 21.63 (h); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4l-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.49 (major 92%) and –71.72 (minor 8%); IR (neat) 3281 (O–H stretch), 2928, 1632 (C=O stretch), 1547, 1453, 1007, 735, 696 cm^{-1}; HRMS (ESI) calcd. for $C_{16}H_{19}NNaO_3$ (M+Na): 296.1263, found 296.1263 *m/z*.





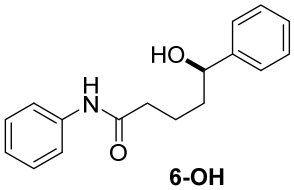
(*R*)-5-hydroxy-5-(2-thiophenyl)pentanecarboxylic acid benzyl amide (4m-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a colorless oil; $[\alpha]_D^{20}$ = –9.3° (*c* 1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (6H, m, a,b,b',c,c',n), 6.90–7.00 (2H, m, l,m), 6.23 (1H, br s), 4.85–4.95 (1H, m, j), 4.38 (2H, d, *J* = 5.7 Hz, e), 3.43 (1H, d, *J* = 4.3 Hz, OH), 2.24 (2H, t, *J* = 7.2 Hz, g), 1.70–1.90 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.16 (f), 148.99 (k), 138.42 (d), 128.80 (b,b'), 127.90 (c,c'), 127.58 (a), 126.74 (m), 124.42 (n), 123.61 (l), 69.74 (j), 43.67 (e), 38.73 (i), 36.03 (g), 21.88 (h); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4m-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.32 (major 92.5%) and –71.74 (minor 7.5%); IR (neat) 3292 (O–H stretch), 2916, 1640 (C=O stretch), 1539, 1495, 1454, 1357, 1250, 1077, 1028, 694 cm^{-1}; HRMS (ESI) calcd. for $C_{16}H_{19}NNaO_2S$ (M+Na): 312.1034, found 312.1039 *m/z*.



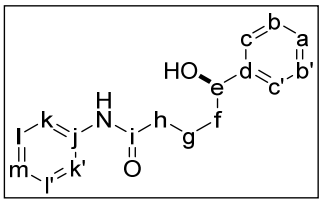


(*R*)-5-hydroxy-5-(3-thiophenyl)pentanecarboxylic acid benzyl amide (4n-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a white solid: 81.0–

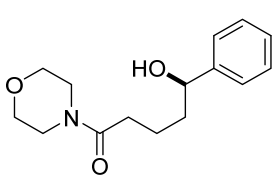
82.5 ºC; $[\alpha]_D^{20}$ = –18.2º (*c* 1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.25–7.35 (7H, m, a,b,b',c,c',m), 7.16 (1H, d, *J* = 2.6 Hz, n), 7.05 (1H, d, *J* = 5.0 Hz, l), 6.07 (1H, br s), 4.70–4.80 (1H, m, j), 4.41 (2H, d, *J* = 5.7 Hz, e), 2.90 (1H, d, *J* = 3.6 Hz, OH), 2.26 (2H, t, *J* = 6.7 Hz, g), 1.70–1.90 (4H, m, h,i); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.04 (f), 146.30 (k), 138.43 (d), 128.82 (b,b'), 127.93 (c,c'), 127.62 (a), 126.15 (m), 125.79 (n), 120.73 (l), 70.13 (j), 43.70 (e), 37.80 (i), 36.16 (g), 21.78 (h); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4n-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.34 (major 94%) and –71.60 (minor 6%); IR (neat) 3284 (O–H stretch), 2360, 2341, 1630 (C=O stretch), 1537, 1454, 1068, 844, 789, 747, 696 cm^{-1}; HRMS (ESI) calcd. for $C_{16}H_{19}NNaO_2S$ (M+Na): 312.1034, found 312.1036 *m/z*.



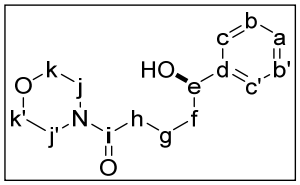
6-OH



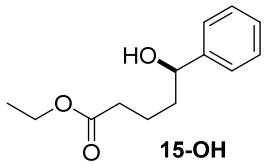
(*R*)-5-hydroxy-5-phenylpentanecarboxylic acid phenyl amide (6-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a white solid: 84.5–86.0 ºC; $[\alpha]_D^{20}$ = –21.6º (*c* 1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (1H, br s, NH), 7.51 (2H, d, *J* = 7.9 Hz, k,k'), 7.25–7.40 (7H, m, a,b,b',c,c',l,l'), 7.11 (1H, t, *J* = 7.4 Hz, m), 4.65–4.80 (1H, m, e), 2.79 (1H, br s, OH), 2.40 (2H, t, *J* = 6.4 Hz, h), 1.75–1.95 (4H, m, f,g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.64 (i), 144.68 (d), 138.06 (j), 129.07 (l,l'), 128.61 (b,b'), 127.68 (m), 125.91 (c,c'), 124.34 (a), 120.03 (k,k'), 74.32 (e), 38.28 (g), 37.28 (h), 22.04 (f); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **6-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.41 (major 94%) and –71.61 (minor 6%); IR (neat) 3294 (O–H stretch), 2360, 2342, 1668 (C=O stretch), 1655, 1600, 1557, 1499, 1489, 1444, 1088, 951, 699, 691 cm^{-1}; HRMS (EI) calcd. for $C_{17}H_{19}NO_2$ (M): 269.1416, found 269.1422 *m/z*.



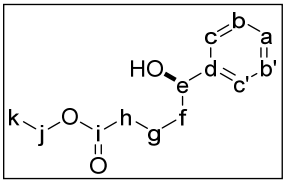
7-OH



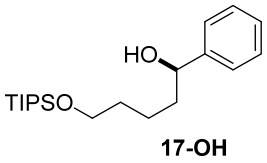
(*R*)-5-hydroxy-5-phenylpentanecarboxylic acid morpholino amide (7-OH). Following **GP5A** affords, after flash chromatography on silica gel the title compound as a light yellow oil; $[\alpha]_D^{20}$ = –22.7° (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (5H, m, aryl), 4.60–4.70 (1H, m, e), 3.55–3.65 (6H, m, j,j',k'k'), 3.35–3.45 (2H, m, k,k'), 3.25 (1H, br s, OH), 2.25–2.35 (2H, m, h), 1.60–1.85 (4H, m, f,g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.81 (i), 144.99 (d), 128.47 (b,b'), 127.45 (a), 125.96 (c,c'), 73.90 (e), 66.97 and 66.68 (k,k'), 46.03 and 42.01 (j,j'), 38.80 (g), 32.80 (h), 21.32 (f); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **7-Mosher** (general procedure **GP6** *vide infra*); ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.22 (major 95.5%) and –71.61 (minor 4.5%); IR (neat) 3410 (O–H stretch), 2915, 2856, 1621 (C=O stretch), 1433, 1271, 1237, 1113, 1066, 1026, 701 cm^{-1}; HRMS (EI) calcd. for $C_{15}H_{21}NO_3$ (M): 263.1521, found 263.1523 *m/z*.



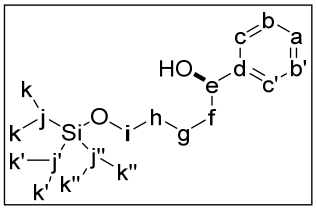
15-OH



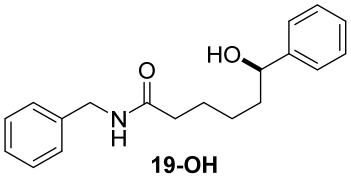
(*R*)-5-hydroxy-5-phenylpentanecarboxylic acid ethyl ester (15-OH). Following **GP5A** affords, after flash chromatography on silica gel the title compound as a colorless oil; $[\alpha]_D^{20}$ = –19.5° (*c* 2.0, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.25–7.45 (5H, m, aryl), 4.65–4.75 (1H, m, e), 4.13 (2H, q, *J* = 9.5 Hz, j), 2.34 (2H, t, *J* = 8.7 Hz, h), 2.24 (1H, br s, OH), 1.60–1.85 (4H, m, f,g), 1.26 (3H, t, *J* = 9.5 Hz, k); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.67 (i), 144.54 (d), 128.49 (b,b'), 127.59 (a), 125.85 (c,c'), 74.12 (e), 60.34 (j), 38.37 (f), 34.03 (h), 21.21 (g), 14.23 (k); Enantiomeric excess was checked by converting to the corresponding Mosher's ester **15-Mosher** (general procedure **GP6** *vide infra*); crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.44 (major 85%) and –71.63 (minor 15%); IR (neat) 3342 (O–H stretch), 2979, 2936, 1731 (C=O stretch), 1453, 1373, 1238, 1185, 1146, 1065, 1026, 736, 700 cm^{-1}; HRMS (EI) calcd. for $C_{13}H_{18}O_3$ (M): 222.1256, found 222.1262 *m/z*.



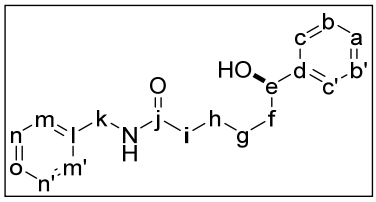
17-OH



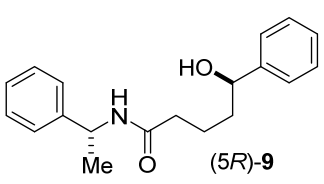
(*R*)-1-phenyl-5-((triisopropylsilyl)oxy)pentan-1-ol (17-OH). Following **GP5B** affords, after flash chromatography on silica gel the title compound as a colorless oil; $[\alpha]_D^{20}$ = –11.0º (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35–7.40 (4H, m, b,b',c,c'), 7.25–7.35 (1H, m, a), 4.70 (1H, dd, *J* = 7.2 and 6.0 Hz, e), 3.69 (2H, d, *J* = 6.4 Hz, i), 1.97 (1H, br s, OH), 1.80–1.90 (1H, m, f), 1.70–1.80 (1H, m, f), 1.55–1.65 (2H, m, h), 1.45–1.55 (1H, m, g), 1.35–1.45 (1H, m, g), 1.05–1.10 (21H, m, j,j',j'',k,k',k''); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.96 (d), 128.53 (b,b'), 127.59 (a), 126.01 (c,c'), 74.76 (e), 63.36 (i), 38.99 (f), 32.89 (h), 22.26 (g), 18.15 (k,k',k''), 12.12 (j,j',j''); Chiral HPLC analysis (Chiralcel-OD, 99:1 hexanes:isopropanol, flowrate = 1.0 mL/min) showed peaks at 35 minutes (10.0% (*S*)) and 42 minutes (90.0% (*R*)); IR (neat) 3369 (O–H stretch), 2940, 2891, 2864, 1462, 1101, 1069, 881, 759, 698, 679, 657 cm^{-1}; HRMS (ESI) calcd. for $C_{20}H_{36}NaO_2Si$ (M+Na): 359.2382, found 359.2385 *m/z*.

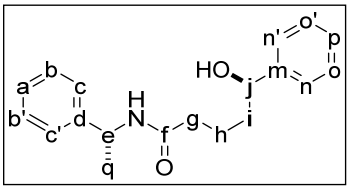


19-OH

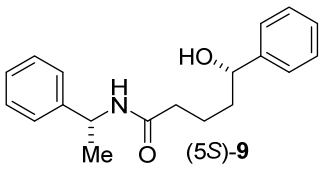


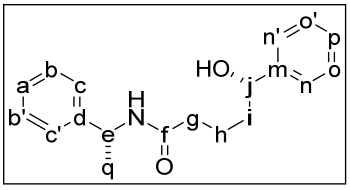
(*R*)-6-hydroxy-6-phenylhexanecarboxylic acid benzyl amide (19-OH). Following **GPxB** affords, after flash chromatography on silica gel the title compound as a semi-solid; $[\alpha]_D^{20}$ = –16.8º (*c* 2.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (10H, m, a,b,b',c,c',l,m,m',n,n',o), 5.91 (1H, br s, NH), 4.67 (1H, dd, *J* = 7.6 and 5.6 Hz, e), 4.40 (2H, d, *J* = 5.7 Hz, k), 2.40 (1H, br s, OH), 2.19 (2H, t, *J* = 7.5 Hz, i), 1.75–1.85 (1H, m, f), 1.65–1.75 (3H, m, f,h), 140–1.55 (1H, m, g), 1.30–1.40 (1H, m, g); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.98 (j), 144.96 (d), 138.49 (l), 128.81 (n,n'), 128.55 (b,b'), 1)27.93 (c,c'), 127.59 (a and o), 125.97 (m,m'), 74.32 (e), 43.67 (k), 38.79 (f), 36.60 (i), 25.54 (g and h); Chiral HPLC analysis (Chiralpak-IC, 60:40 hexanes:isopropanol, flowrate = 1.0 mL/min) showed peaks at 47 minutes (90.5% (*R*)) and 56 minutes (9.5% (*S*)); IR (neat) 3281 (O–H stretch), 2929, 2858, 1642 (C=O stretch), 1544, 1494, 1453, 1028, 747, 696 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1742 *m/z*.





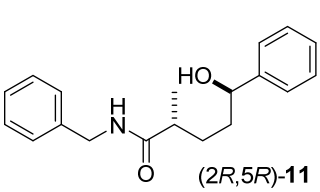
(*R*)-5-hydroxy-5-phenyl-*N*-((*R*)-1-phenylethyl)pentanamide ((5*R*)-9). Following **GP4** with **8** (74 mg, 0.264 mmol) without purification then **GP5B** affords, after flash chromatography on silica gel (80:20-0:100 hexanes:ethyl acetate), the title compound (64 mg, 82%) as a white solid: 65.5–67.0 ºC; $[\alpha]_D^{20}$ = +43.6º (*c* 2.0, $CHCl_3$); TLC analysis R_f 0.4 (0:100 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (10H, m, aryl), 6.05 (1H, d, *J* = 7.6 Hz, NH), 5.05–5.15 (1H, m, e), 4.60–4.70 (1H, m, j), 2.91 (1H, br s, OH), 2.10–2.30 (2H, m, g), 1.60–1.80 (4H, m, h,i), 1.47 (3H, d, *J* = 6.9 Hz, q); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.24 (f), 144.92 (m), 143.40 (d), 128.77, 128.53, 127.53, 127.43, 126.31, 125.92, 74. 02 (j), 48.78 (e), 38.52 (i), 36. 29 (g), 22.02 (minor q, 5.5%), 21.97 (major q, 94.5%), 21.86 (h); IR (neat) 3338 (O–H stretch), 2360, 2340, 1639 (C=O stretch), 1620, 1529, 1493, 1448, 1066, 1029, 758, 698 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1737 *m/z*.

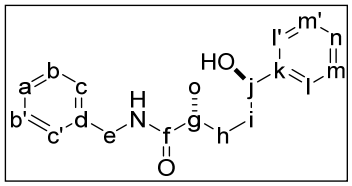




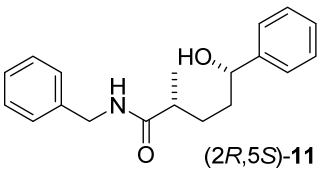
(*S*)-5-hydroxy-5-phenyl-*N*-((*R*)-1-phenylethyl)pentanamide ((5*S*)-9). Following **GP4** with (*S*)-**B1** and **8** (74 mg, 0.264 mmol) without purification then **GP5B** affords, after flash chromatography on silica gel (80:20-0:100 hexanes:ethyl acetate), the title compound (63 mg, 80%) as a white solid: 96.5–97.5 ºC; $[\alpha]_D^{20}$ = +91.3º (*c* 2.0, $CHCl_3$); TLC analysis R_f 0.4 (0:100 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (10H, m, aryl), 6.04 (1H, d, *J* = 7.6 Hz, NH), 5.05–5.15 (1H, m, e), 4.60–4.70 (1H, m, j), 2.88 (1H, br s, OH), 2.21 (2H, t, *J* = 6.6 Hz, g), 1.60–1.85 (4H, m, h,i), 1.47 (3H, d, *J* = 6.9 Hz, q); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.23 (f), 144.90 (m), 143.39 (d), 128.76, 128.53, 127.53, 127.43, 126.32, 125.91, 74. 07 (j), 48.78 (e), 38.50 (i), 36. 33 (g), 22.02 (major q, 9.0%), 21.96 (major q, 91%), 21.86 (h); IR (neat) 3318 (O–H stretch),

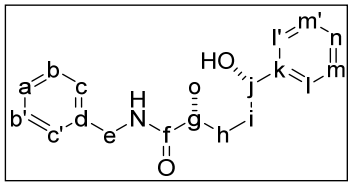
2360, 2341, 1640 (C=O stretch), 1532, 1493, 1449, 1074, 1028, 753, 697 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1717 *m/z*.





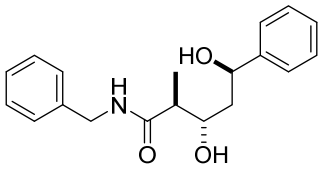
(2*R*,5*R*)-5-hydroxy-2-methyl-5-phenylpentanecarboxylic acid benzyl amide ((2*R*,5*R*)-11). Following **GP4** with **10** (74 mg, 0.264 mmol) without purification then **GP5B** affords, after flash chromatography on silica gel (80:20-0:100 hexanes:ethyl acetate), the title compound (62 mg, 79%) as a white solid: 63.0–64.5 °C; $[\alpha]_D^{20}$ = –35.6° (*c* 2.0, $CHCl_3$); TLC analysis R_f 0.4 (0:100 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.35 (10H, m, aryl), 6.21 (br s, minor 8.0%, NH), 6.13 (br s, major 92.0%, NH), 4.63 (1H, dd, *J* = 6.2 and 6.2 Hz, j), 4.38 (2H, d, *J* = 5.7 Hz, e), 2.90 (1H, br s, OH), 2.20–2.30 (1H, m, g), 1.70–1.90 (3H, m, h,i), 1.55–1.65 (0.08H, m, minor i), 1.35–1.50 (0.92H, m, major i), 1.13 (3H, d, *J* = 6.9 Hz, o); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.46 (f), 144.88 (k), 138.61 (d), 128.78 (aryl), 128.52 (aryl), 127.87 (aryl), 127.54 (aryl), 127.53 (aryl), 125.97 (aryl, 92.0%, major), 125.88 (aryl, 8.0%, minor), 74.31 (j), 43.49 (e), 41.27 (g), 36.95 (i), 30.42 (h), 18.16 (o); IR (neat) 3571, 3542, 3446, 3307 (O–H stretch), 2916, 1640 (C=O stretch), 1545, 1494, 1243, 1056, 761, 728, 695 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1724 *m/z*.



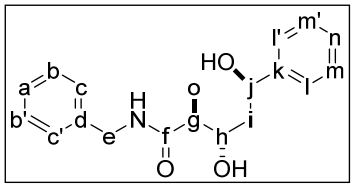


(2*R*,5*S*)-5-hydroxy-2-methyl-5-phenylpentanecarboxylic acid benzyl amide ((2*R*,5*S*)-11). Following **GP4** with (*S*)-**B1** and **10** (74 mg, 0.264 mmol) without purification then **GP5B** affords, after flash chromatography on silica gel (80:20-0:100 hexanes:ethyl acetate), the title compound (62 mg, 79%) as a white solid: 93.0–95.0 °C; $[\alpha]_D^{20}$ = +22.9° (*c* 2.0, $CHCl_3$); TLC analysis R_f 0.4 (0:100 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20–7.40 (10H, m, aryl), 6.17 (br s, major 91.0%, NH), 6.08 (br s, minor 9.0%, NH), 4.60–4.65 (1H, m, j), 4.39 (2H, d, *J* = 5.6 Hz, e), 2.82 (1H, br s, OH), 2.20–2.35 (1H, m, g), 1.65–1.80 (3H, m, h,i), 1.50–1.65 (0.91H, m, major

i), 1.35–1.45 (0.09H, m, minor i), 1.15 (3H, d, J = 6.8 Hz, o); ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.54 (f), 144.97 (k), 138.61 (d), 128.79 (aryl), 128.53 (aryl), 127.90 (aryl), 127.53 (aryl), 127.53 (aryl), 12.96 (aryl, 8.0%, minor), 125.87 (aryl, 92.0%, major), 74.35 (j), 43.51 (e), 41.16 (g), 36.91 (i), 30.82 (h), 18.24 (o); IR (neat) 3285 (O–H stretch), 2919, 1636 (C=O stretch), 1548, 1495, 1451, 1350, 1225, 1089, 1056, 1027, 753, 735, 695 cm^{-1}; HRMS (EI) calcd. for $C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1719 *m/z*.

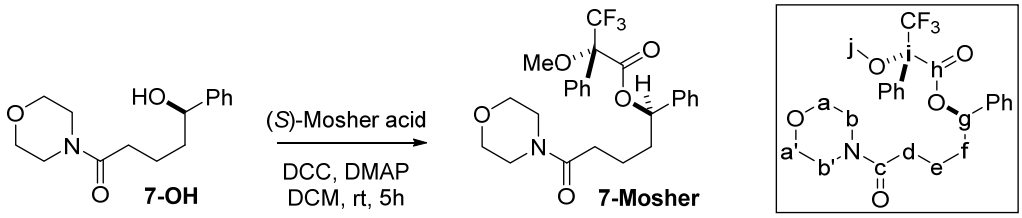


(2*S*,3*S*,5*R*)-**13**



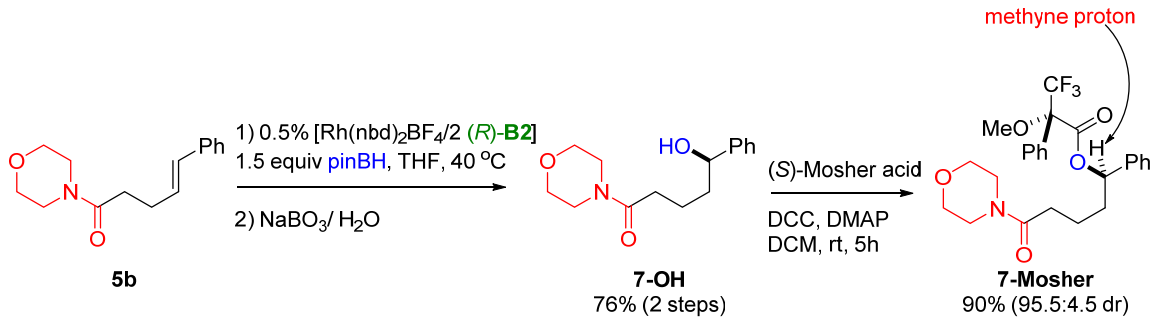
(2*S*,3*S*,5*R*)-3,5-dihydroxy-2-methyl-5-phenylpentanecarboxylic acid benzyl amide ((2*S*,3*S*,5*R*)-13). Following **GP4** with 3.0 mol% [$Rh(nbd)_2BF_4$/ 2(*R*)-**B1**] and **12** (97 mg, 0.264 mmol) affords, after flash chromatography on silica gel (80:20-0:100 hexanes:ethyl acetate), the title compound (62 mg, 75%) as a white solid: 109.0–110.0 ºC; $[\alpha]_D^{20}$ = –14.5º (*c* 0.775, $CHCl_3$); TLC analysis R_f 0.3 (0:100 hexanes:ethyl acetate); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.40 (10H, m, aryl), 6.39 (br s, minor 13.0%, NH), 6.31 (br s, major 87.0%, NH), 5.07 (0.87H, dd, J = 7.3 and 4.1 Hz, j), 4.97 (0.13H, dd, J = 9.6 and 3.3 Hz, j), 4.44 (2H, d, J = 5.7 Hz, e), 3.80–4.20 (2H, m, h,OH), 3.36 (1H, br s, OH), 2.30–2.40 (1H, m, g), 1.85–1.95 (2H, m, i), 1.26 (3H, d, J = 7.2 Hz, o); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.99 (f), 144.65 (k), 138.09 (d), 128.88 (aryl), 128.57 (aryl), 127.80 (aryl), 127.69 (aryl), 127.41 (aryl), 125.60 (aryl), 74.44 (j), 71.23 (h), 46.09 (g), 43.52 (i), 43.48 (e), 15.56 (o); IR (neat) 3295 (O–H stretch), 2925, 2359, 2341, 1636 (C=O stretch), 1537, 1495, 1453, 1369, 1028, 751, 696 cm^{-1}; HRMS (ESI) calcd. for $C_{19}H_{23}NNaO_3$ (M+Na): 336.1576, found 336.1583 *m/z*.

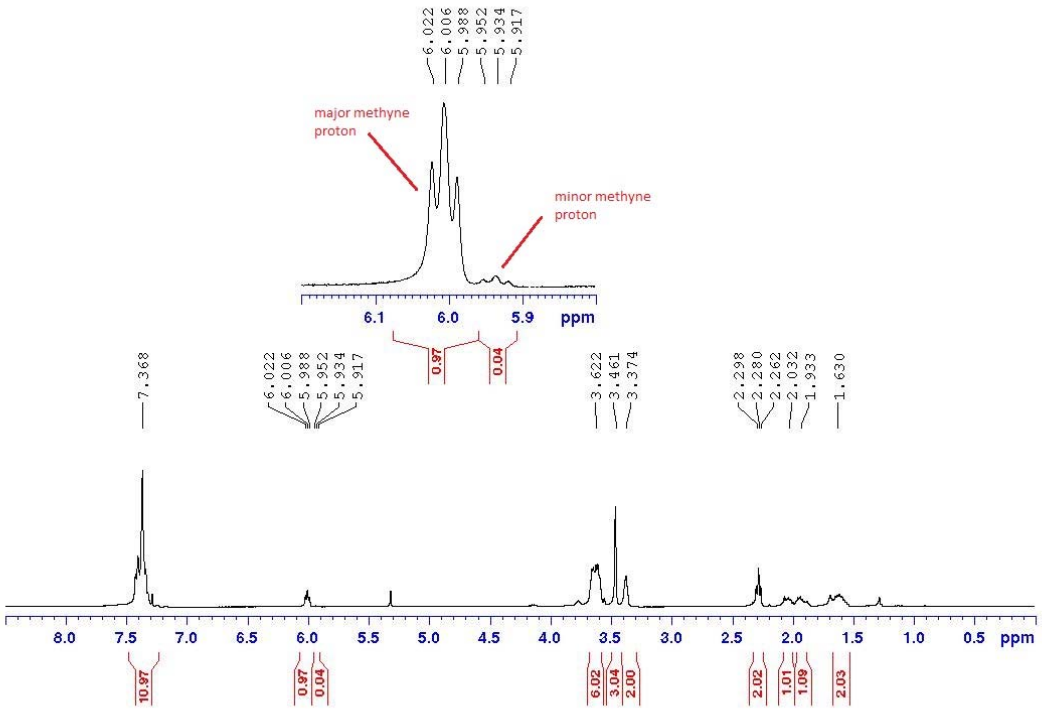
General procedure for preparation of Mosher's ester via DCC condensation with benzylic alcohol and (*S*)-Mosher acid (GP6): checking ee and determining absolute configuration purposes.



(*R*)-5-morpholino-5-oxo-1-phenylpentyl (*S*)-3,3,3-trifluoro-2-methoxy-2-phenylpropanoate (7-Mosher). To a solution of **7-OH** (0.06 mmol, 15.8 mg) and (*S*)-Mosher acid (3.1 equiv, 0.186 mmol, 44 mg) in DCM (1 mL) was added DCC (3.1 equiv, 0.186 mmol, 38.6 mg) and DMAP (3.1 equiv, 0.186 mmol, 22.8 mg). The resultant mixture was stirred at room temp for 5h then concentrated under vacuum. Flash column chromatography (80:20-50:50 hexanes:ethyl acetate) the title compound (26 mg, 90%) as a white semi-solid; $[\alpha]_D^{20}$ = –48.0° (*c* 1.0, $CHCl_3$); TLC analysis R_f 0.5 (50:50 hexanes:ethyl acetate); ^{19}F NMR (375 MHz, $CDCl_3$) δ –71.22 (s, CF_3, major 95.5%) and –71.61 (s, CF_3, minor 4.5%); ^1H NMR (400 MHz, $CDCl_3$) δ 7.20–7.45 (10H, m, aryl), 6.01 (0.955H, t, *J* = 6.4 Hz, g major), 5.93 (0.045H, t, *J* = 7.0 Hz, g minor), 3.50–3.70 (6H, m, a,a',b,a'), 3.46 (3H, s, j), 3.35–3.40 (2H, m, b,b'), 2.28 (2H, t, *J* =7.3 Hz, d), 2.00–2.10 (1H, m, f), 1.85–2.00 (1H, m, f), 1.55–1.75 (2H, m, e); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.91, 166.11, 138.88, 132.38, 129.66, 128.73, 128.70, 128.45, 127.55, 127.08, 78.55, 67.01, 66.68, 55.49, 45.98, 41.98, 35.41, 32.45, 20.93; IR (neat) 2953, 2925, 2851, 1743 (C=O stretch), 1644 (C=O stretch), 1452, 1433, 1269, 1237, 1165, 1113, 1014, 989, 763, 717, 698 cm^{-1}; HRMS (ESI) calcd. for $C_{25}H_{28}F_3NNaO_5$ (M+Na): 502.1817, found 502.1825 *m/z*.

The absolute configuration of δ-borylation of γ,δ-unsaturated amides was obtained via ^1H NMR analysis of Mosher's ester **7-Mosher**; the latter is synthesized from DCC condensation of the δ-hydroxy morpholine amide **7-OH** (obtained via CAHB/ oxidation of **5b** using 0.5% $[Rh(nbd)_2BF_4$/ 2(*R*)-**B2**]) with (*S*)-Mosher acid as shown in the above **GP6** scheme. The secondary benzylic Mosher's ester (e.g., **7-Mosher**) is deemed a trustworthy source for determining absolute configurations. According to Feng Shao's[7] and co-workers, the major benzylic methyne proton is more downfield, it is on the same side of the methoxy group and on the opposite side of the phenyl group resulting in *R,S*-configuration of **7-Mosher** and thus *R*-configuration of **7-OH**.





The above Mosher's ester (i.e., **7-Mosher**) was isolated for proof of CAHB absolute configuration purpose; other benzylic alcohols applied this method to determine the ee were run at 0.02 mmol scale and the Mosher's esters were not isolated; the crude ^{19}F NMR were essentially clean to obtain the ee.

References

[1] (a) Smith, S. M.; Thacker, N. C.; Takacs, J. M. *J. Am. Chem. Soc.* **2008**, *130,* 3734–3735; (b) Hoang, G. L.; Yang, Z.-D.; Smith, S. M.; Pal, R.; Miska, J. L.; Perez, D. E.; Pelter, L. S.; Zeng, X. C.; Takacs, J. M. *Org. Lett.* **2015**, *17,* 940–943; (c) Bernsmann, H.; Berg, M. V. D.; Hoen, R.; Minnaard, A. J.; Mehler, G.; Reetz, M. T.; Vries, J. G. D.; Feringa, B. L. *J. Org. Chem.* **2005**, *70,* 943–951; (d) Liu, W.-B.; Zheng, C.; Zhuo, C.-X.; Dai, L.-X.; You, S.-L. *J. Am. Chem. Soc.* **2012**, *134,* 4812–4821; (e) Zhang, F.; Li, Y.; Li, Z.-W.; He, Y.-M.; Zhu, S.-F.; Fan, Q.-H.; Zhou, Q.-L. *Chem. Commun.* **2008**, 6048–6050.

[2] Musacchio, A. J.; Nguyen, L. Q.; Beard, G. H.; Knowles, R. R. *J. Am. Chem. Soc.,* **2014**, *136*, 12217–12220.

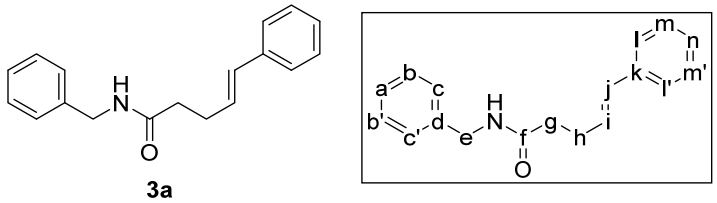
[3] Li, G.; Han, A.; Pulling, M. E.; Estes, D. P.; Norton, J. R. *J. Am. Chem. Soc.* **2012**, *134*, 14662–14665.

[4] Allen, A. D.; Fenwick, M. F.; Henry-Riyad, H.; Tidwell, T. T. J. Org. Chem. **2001**, *66*, 5759–5765.

[5] Speltz, T. E.; Fanning, S. W.; Mayne, C. G.; Fowler, C.; Tajkhorshid, E.; Greene, G. L.; Moore, T. W. *Angew. Chem., Int. Ed.* **2016**, *55*, 4252–4255.

[6] Evans, D. A.; Tedrow, J. S.; Shaw, J. T.; Downey, C. W. *J. Am. Chem. Soc.* **2002**, *124,* 392–393.

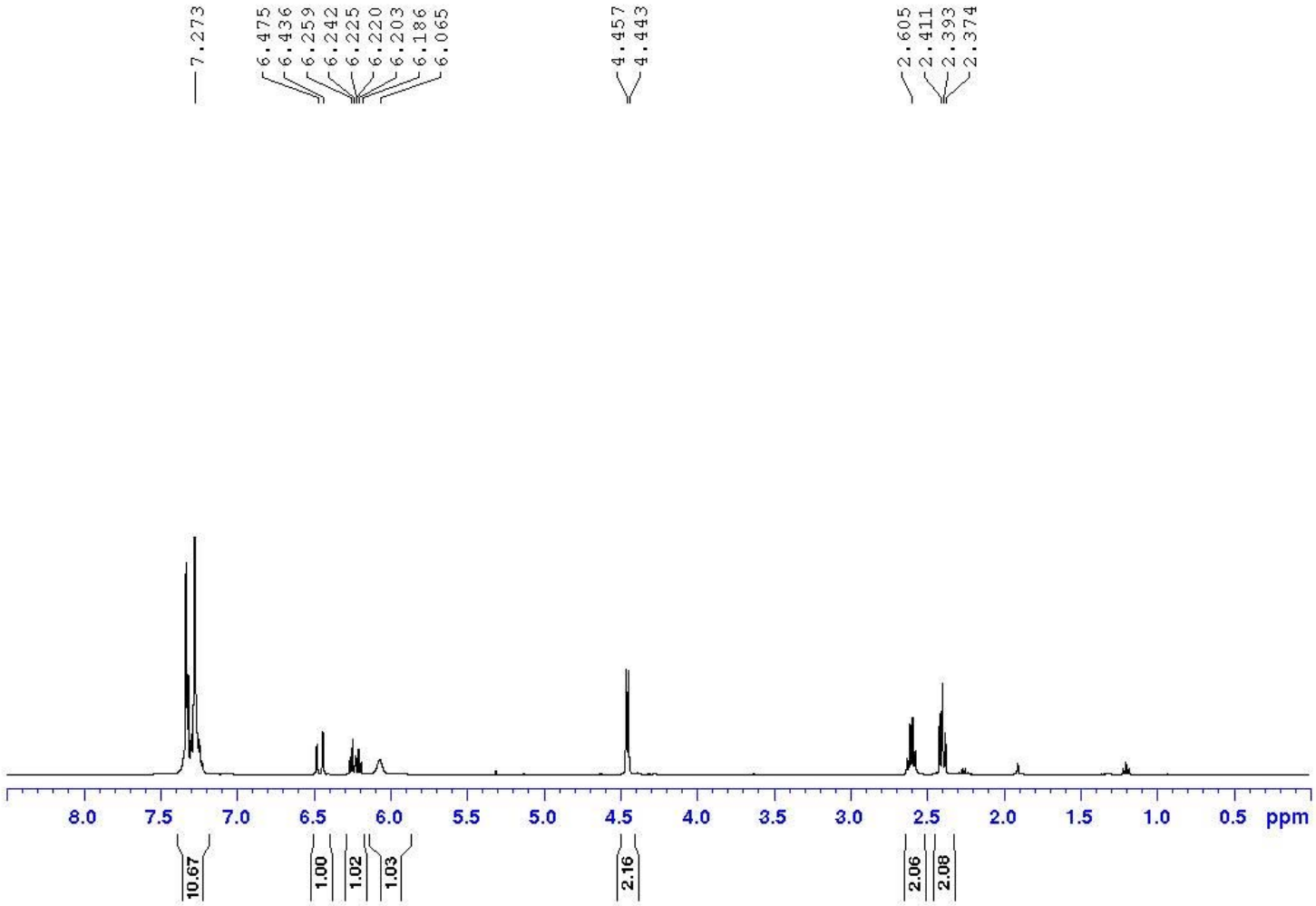
[7] Hoye, T. R.; Jeffrey, C. S.; Shao, F. *Nat. Prot.* **2007**, *2,* 2451–2458.



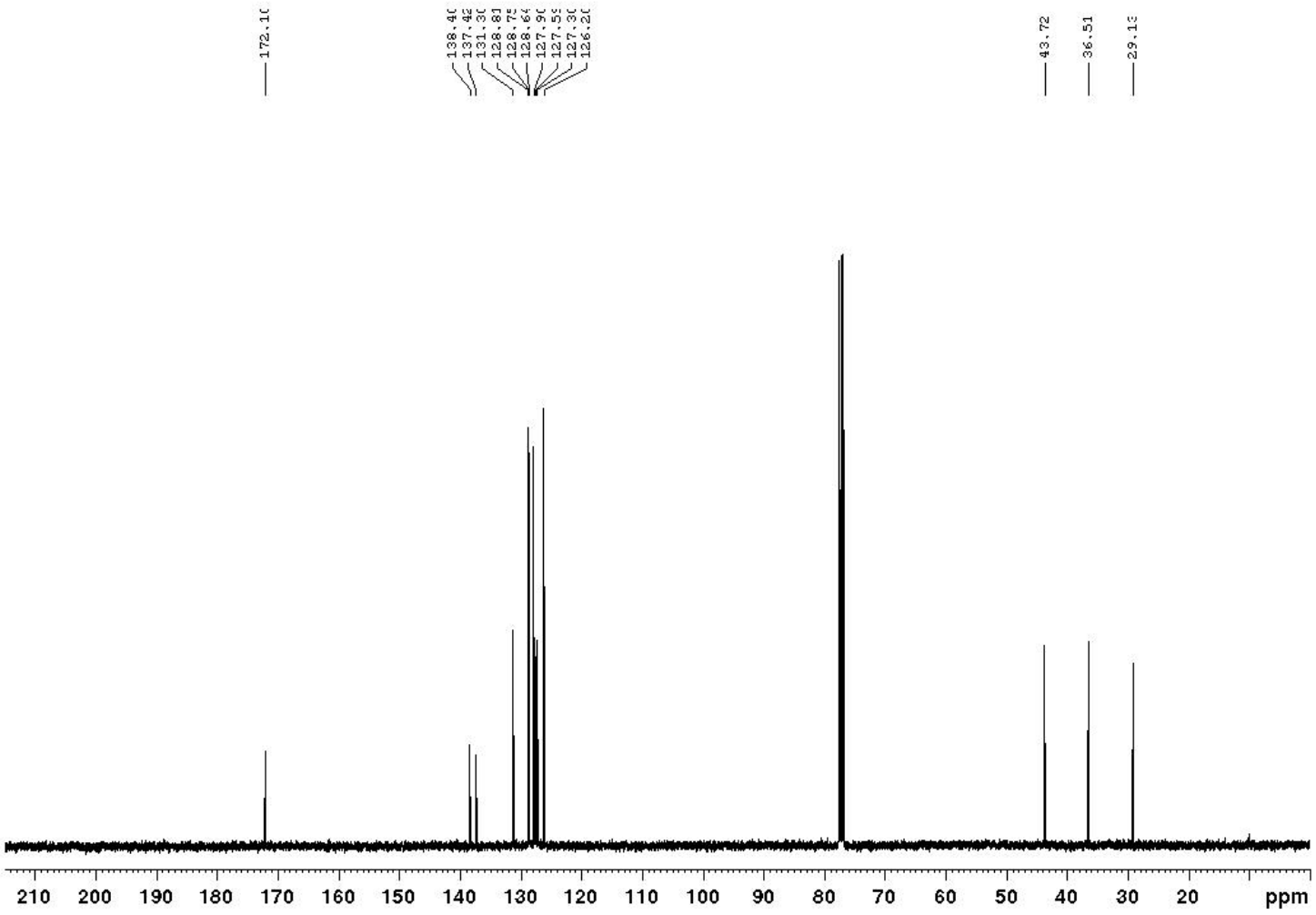
3a

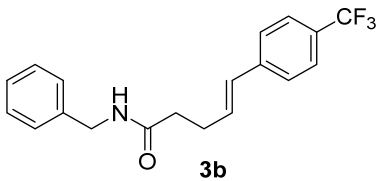
m.p.	85.0–86.5 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (10H, m, a,b,b',c,c',l,l',m,m',n), 6.46 (1H, d, *J* = 15.8 Hz, j), 6.22 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 6.07 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 2.55–2.65 (2H, m, h), 2.39 (2H, t, *J* = 7.4 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.10 (f), 138.40 (d), 137.42 (k), 131.30 (j), 128.81 (b,b'), 128.75 (i), 128.64 (l,l'), 127.90 (m,m'), 127.59 (n), 127.30 (a), 126.20 (c,c'), 43.72 (e), 36.51 (g), 29.13 (h)
IR (neat)	3292 (N-H stretch), 3062, 3029, 1635 (C=O stretch), 1545, 1492, 1445 (C-N stretch), 1223, 967, 732, 671 cm^{-1}
HRMS (EI)	$C_{18}H_{19}NO$ (M): 265.1467, found 265.1476 *m/z*

^{1}H NMR

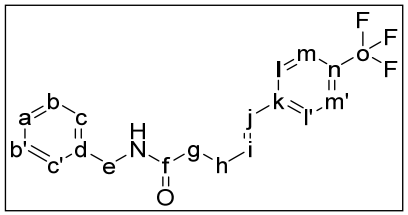

7.273
6.475
6.436
6.259
6.242
6.225
6.220
6.203
6.186
6.065
4.457
4.443
2.605
2.411
2.393
2.374
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.67
1.00
1.02
1.03
2.16
2.06
2.08

^{13}C NMR


172.10
138.40
137.42
131.30
128.81
128.75
128.64
127.90
127.53
127.30
126.20
43.72
36.51
29.13
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

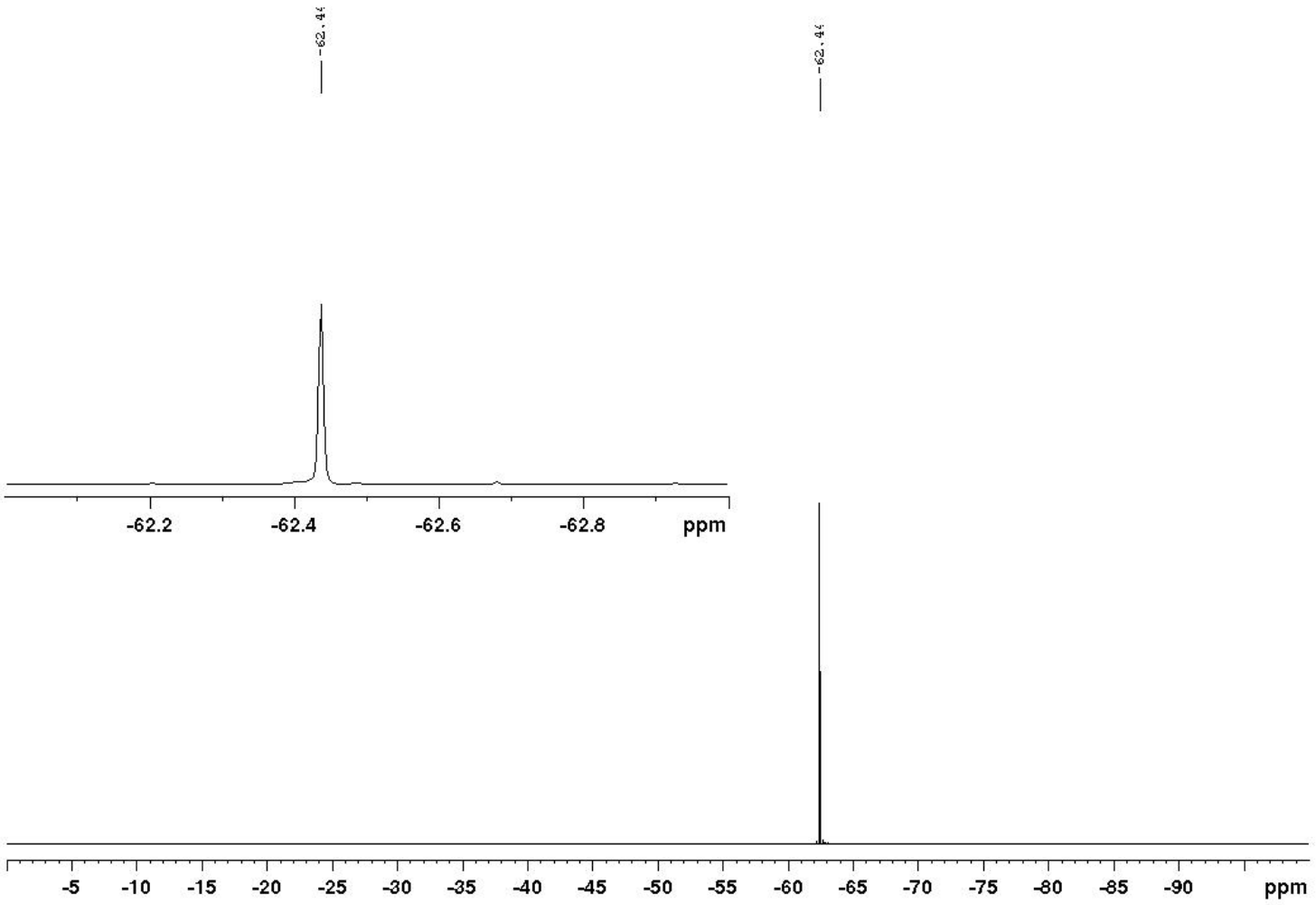


3b

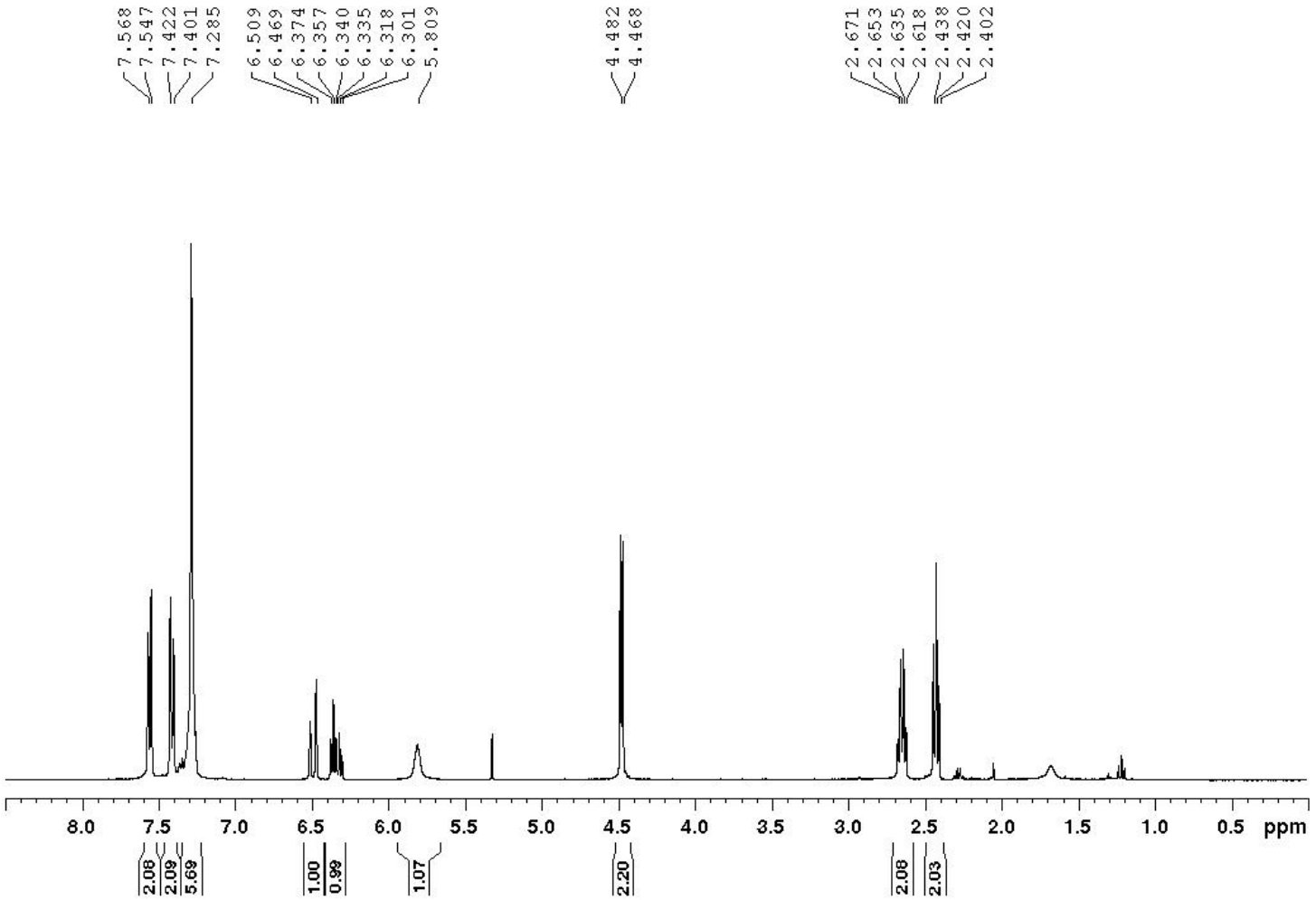


m.p.	127.0–128.0 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{19}F NMR (376 MHz, $CDCl_3$)	δ –62.44 (CF_3)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.56 (2H, d, *J* = 8.2 Hz, l,l'), 7.41 (2H, d, *J* = 8.2 Hz, m,m'), 7.25–7.35 (5H, m, a,b,b',c,c'), 6.49 (1H, d, *J* = 15.8 Hz, j), 6.34 (1H, dt, *J* = 15.8 and 6.8 Hz, i), 5.81 (1H, br s, NH), 4.48 (2H, d, *J* = 5.7 Hz, e), 2.64 (2H, q, *J* = 7.1 Hz, h), 2.42 (2H, t, *J* = 7.3 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.75 (f), 140.90 (k), 138.32 (d), 131.62 (aryl), 130.08 (aryl), 128.83 (aryl), 127.92 (aryl), 127.68 (aryl), 126.31 (aryl), 125.57 (q, *J* = 4,1 Hz, aryl), 43.76 (e), 36.17 (g), 29.04 (h)
IR (neat)	3292 (N-H stretch), 1634 (C=O stretch), 1603, 1542, 1327, 1165, 1109, 1068, 742, 691 cm^{-1}
HRMS (EI)	$C_{19}H_{18}F_3NO$ (M): 333.1340, found 333.1356 *m/z*

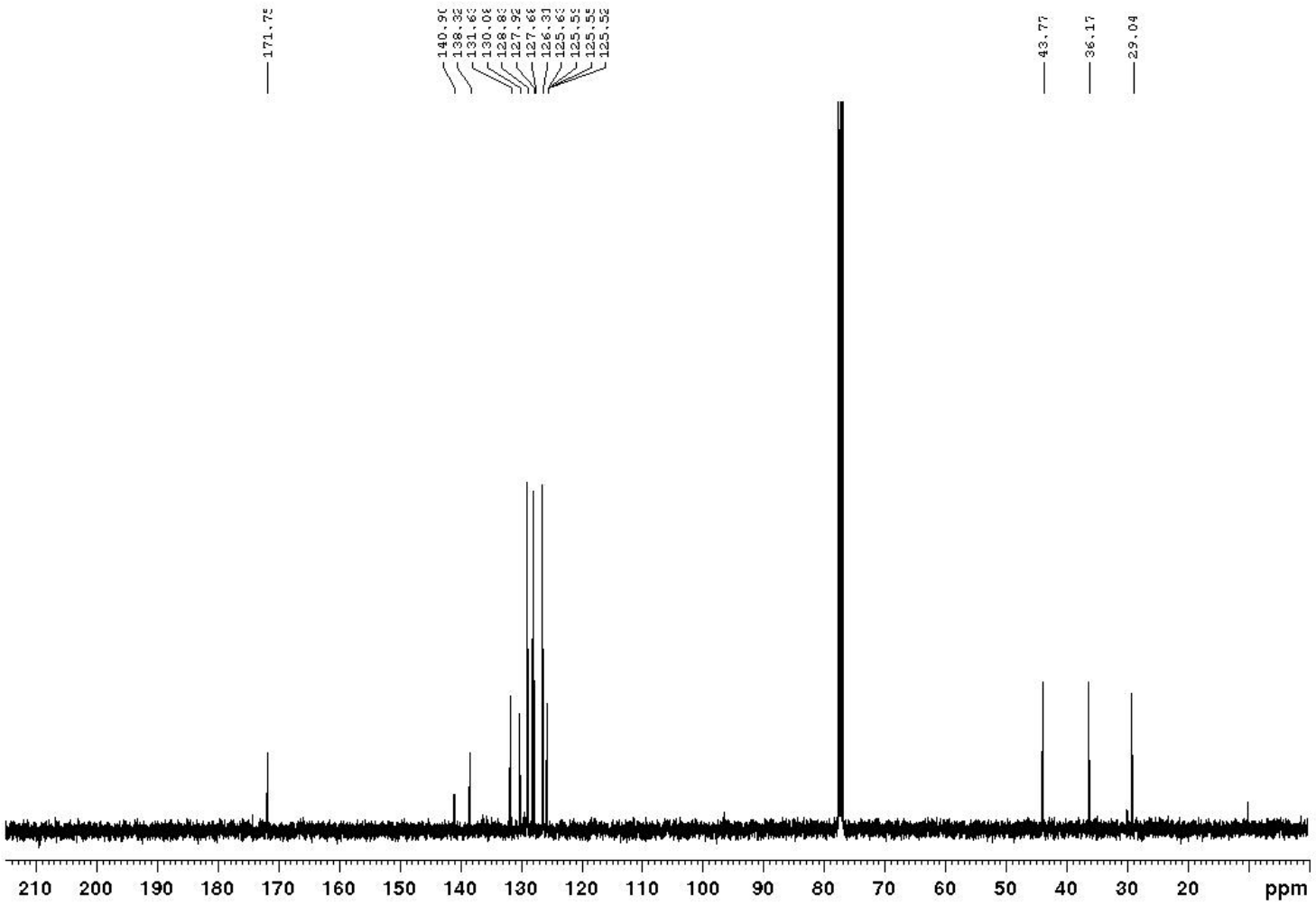
^{19}F NMR

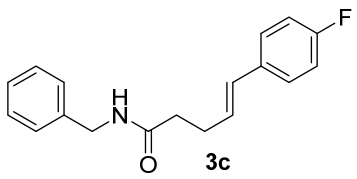

-62.44
-62.44
-62.2
-62.4
-62.6
-62.8
ppm
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
-55
-60
-65
-70
-75
-80
-85
-90
ppm

^{1}H NMR

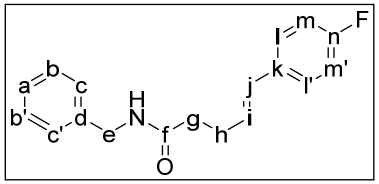

7.568
7.547
7.422
7.401
7.285
6.509
6.469
6.374
6.357
6.340
6.335
6.318
6.301
5.809
4.482
4.468
2.671
2.653
2.635
2.618
2.438
2.420
2.402
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.08
2.09
5.69
1.00
0.99
1.07
2.20
2.06
2.03

^{13}C NMR


171.75
140.90
138.32
131.63
130.08
128.80
127.92
127.68
126.31
125.63
125.55
125.55
125.52
43.77
36.17
29.04
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

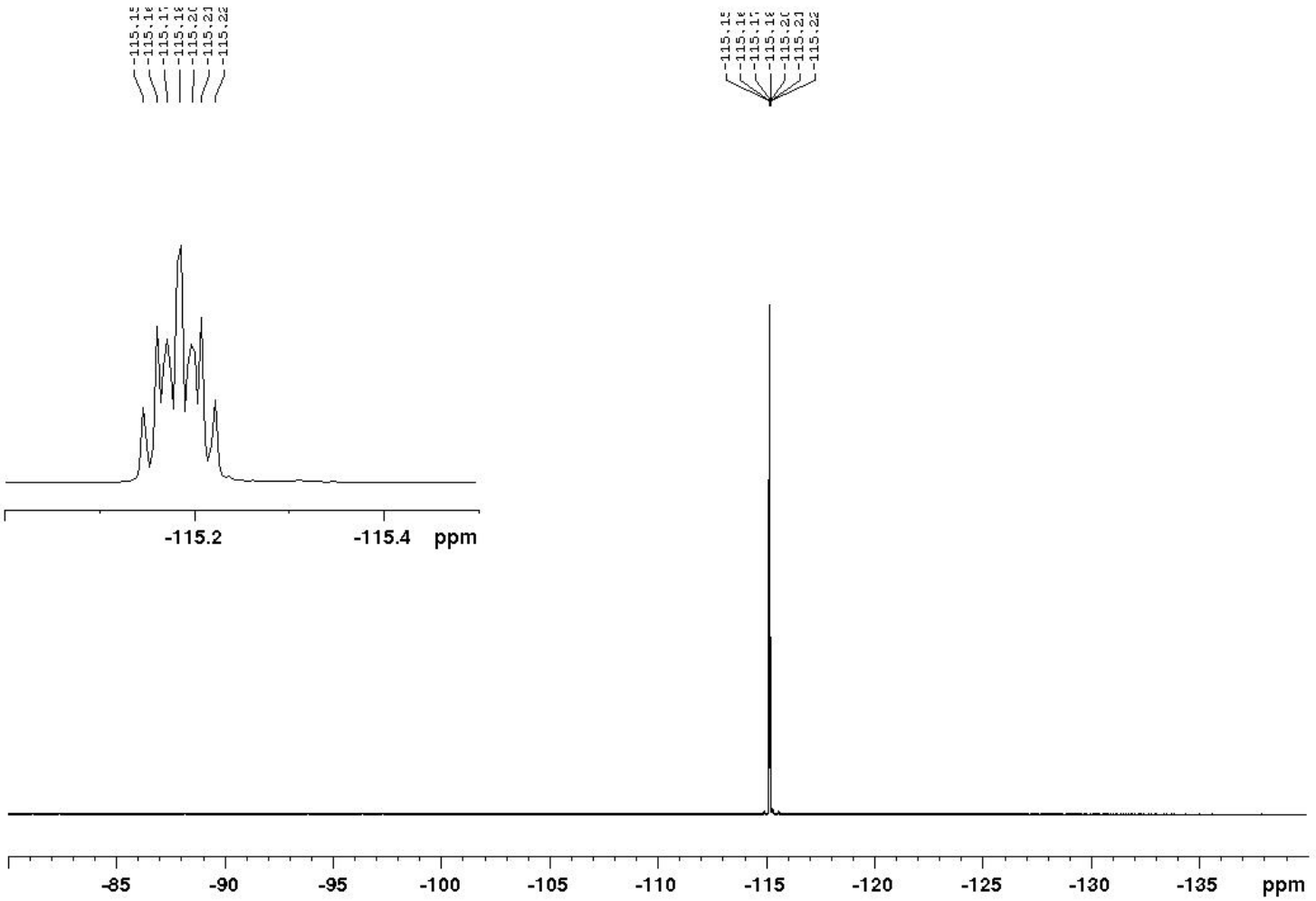


3c

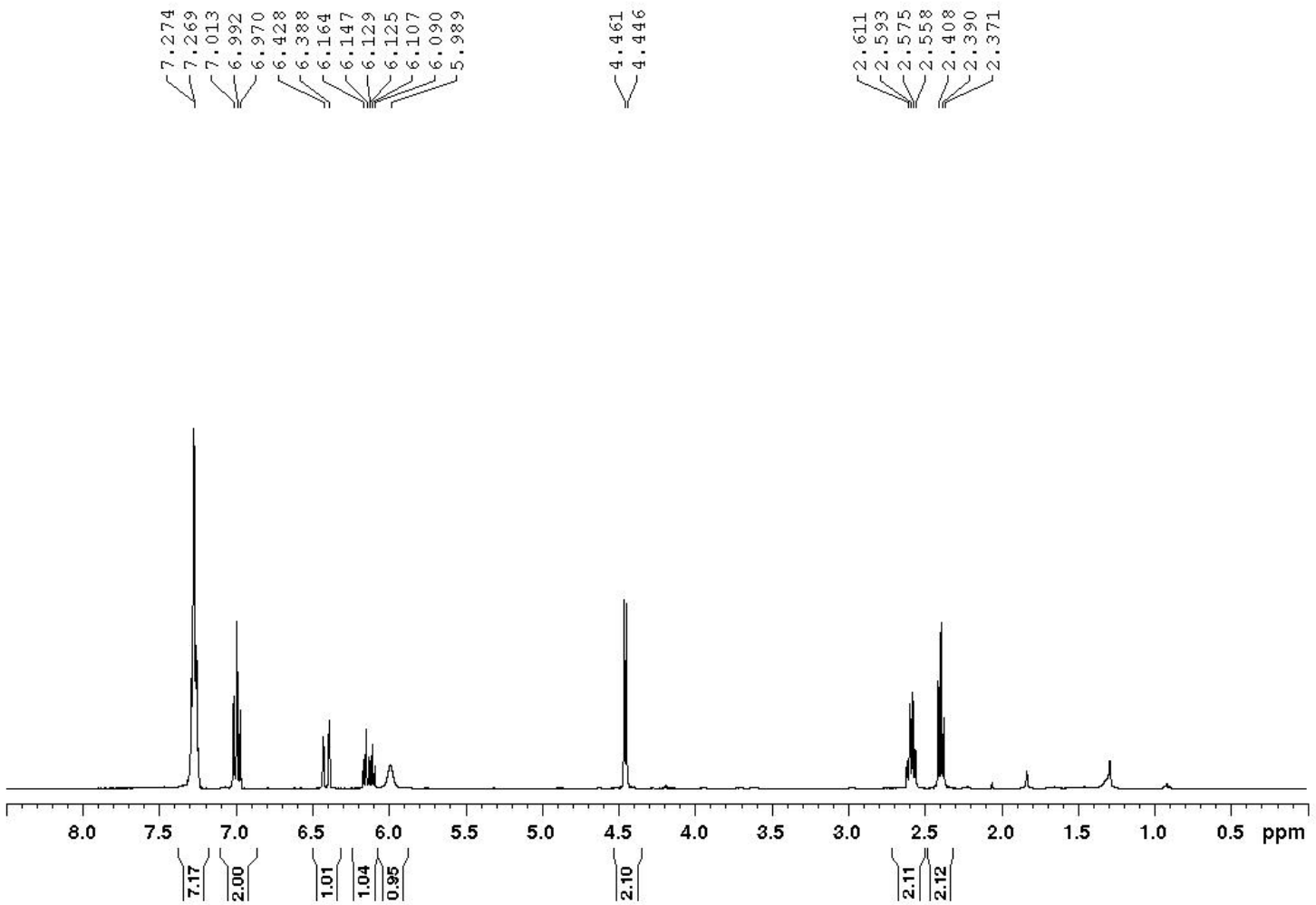


m.p.	92.5–93.5 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{19}F NMR (376 MHz, $CDCl_3$)	δ –115.10 to –115.25 (m, F)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.30 (7H, m, a,b,b',c,c',l,l'), 6.99 (2H, t, *J* = 8.7 Hz, m,m'), 6.41 (1H, d, *J* = 15.8 Hz, j), 6.13 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.99 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 2.58 (2H, q, *J* = 7.2 Hz, h), 2.39 (2H, t, *J* = 7.4 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.08 (f), 162.18 (d, *J* = 244 Hz, n), 138.40 (d), 133.61 and 133.58 (k), 130.10 (j), 128.80 (b,b'), 128.50 and 128.48 (i), 127.88 (l,l'), 127.68 (a), 127.60 (c,c'), 115.48 (d, *J* = 22 Hz, m,m'), 43.70 (e), 36.43 (g), 29.05 (h)
IR (neat)	3286 (N-H stretch), 2917, 1632 (C=O stretch), 1544, 1507, 1224, 1157, 968, 849, 810, 738, 694 cm^{-1}
HRMS (EI)	$C_{18}H_{18}FNO$ (M): 283.1372, found 283.1383 *m/z*

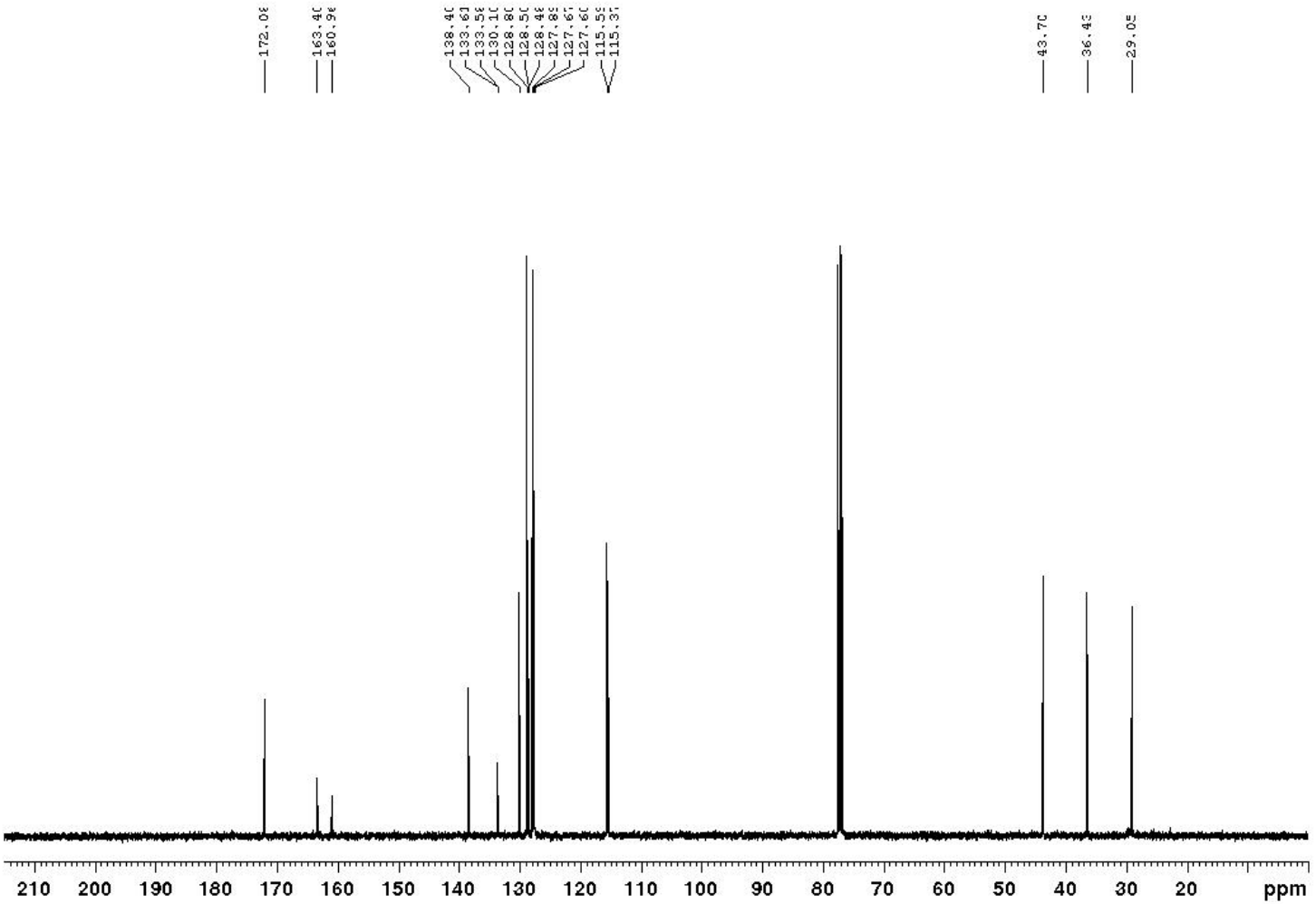
^{19}F NMR

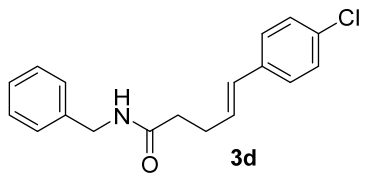


^{1}H NMR

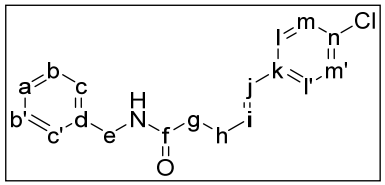

7.274
7.269
7.013
6.992
6.970
6.428
6.388
6.164
6.147
6.129
6.125
6.107
6.090
5.989
4.461
4.446
2.611
2.593
2.575
2.558
2.408
2.390
2.371
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
7.17
2.00
1.01
1.04
0.95
2.10
2.11
2.12

^{13}C NMR


172.08
163.40
160.94
138.40
133.61
133.58
130.10
128.80
128.50
128.48
127.85
127.67
127.60
115.55
115.31
43.70
36.43
29.05
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

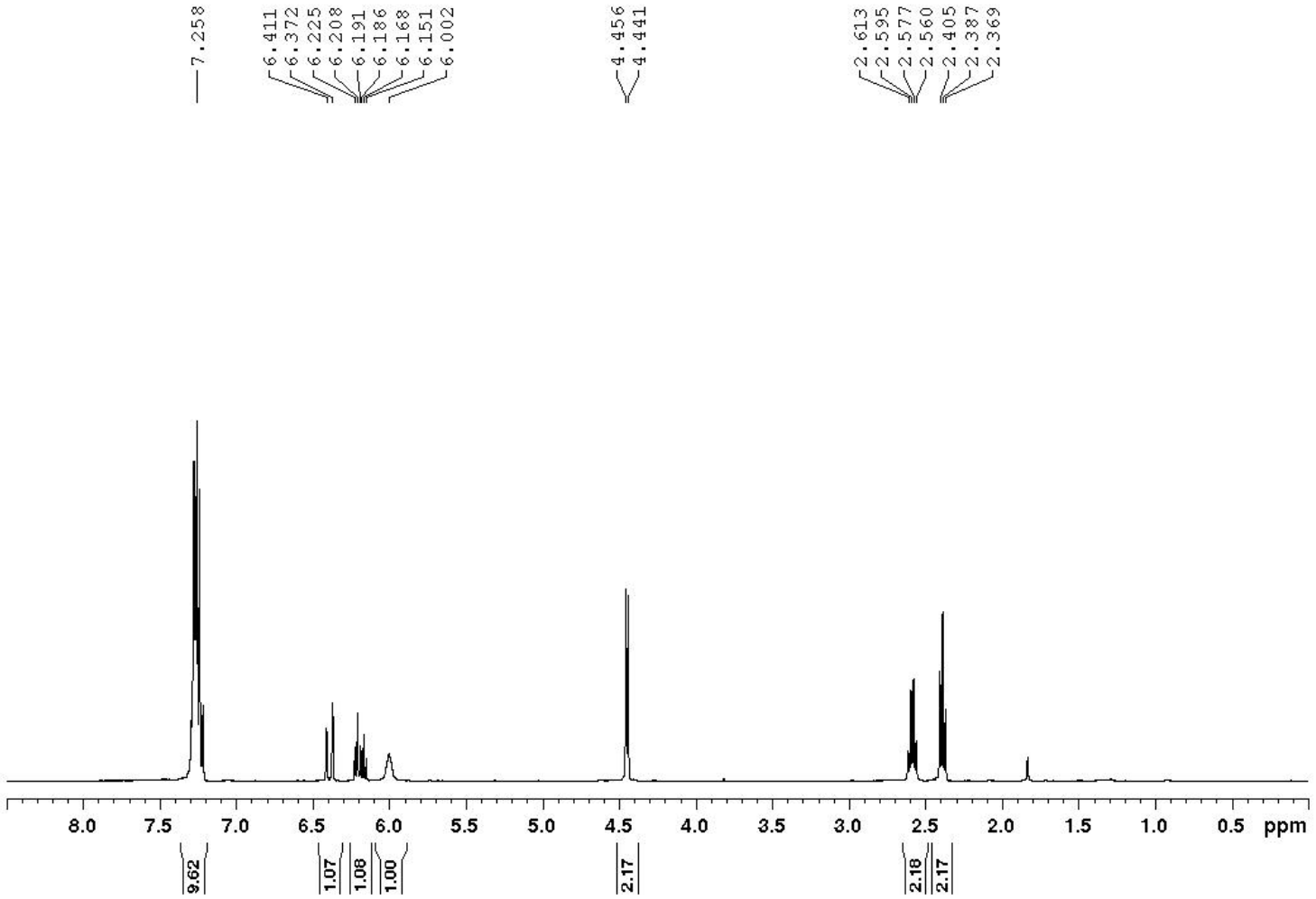


3d

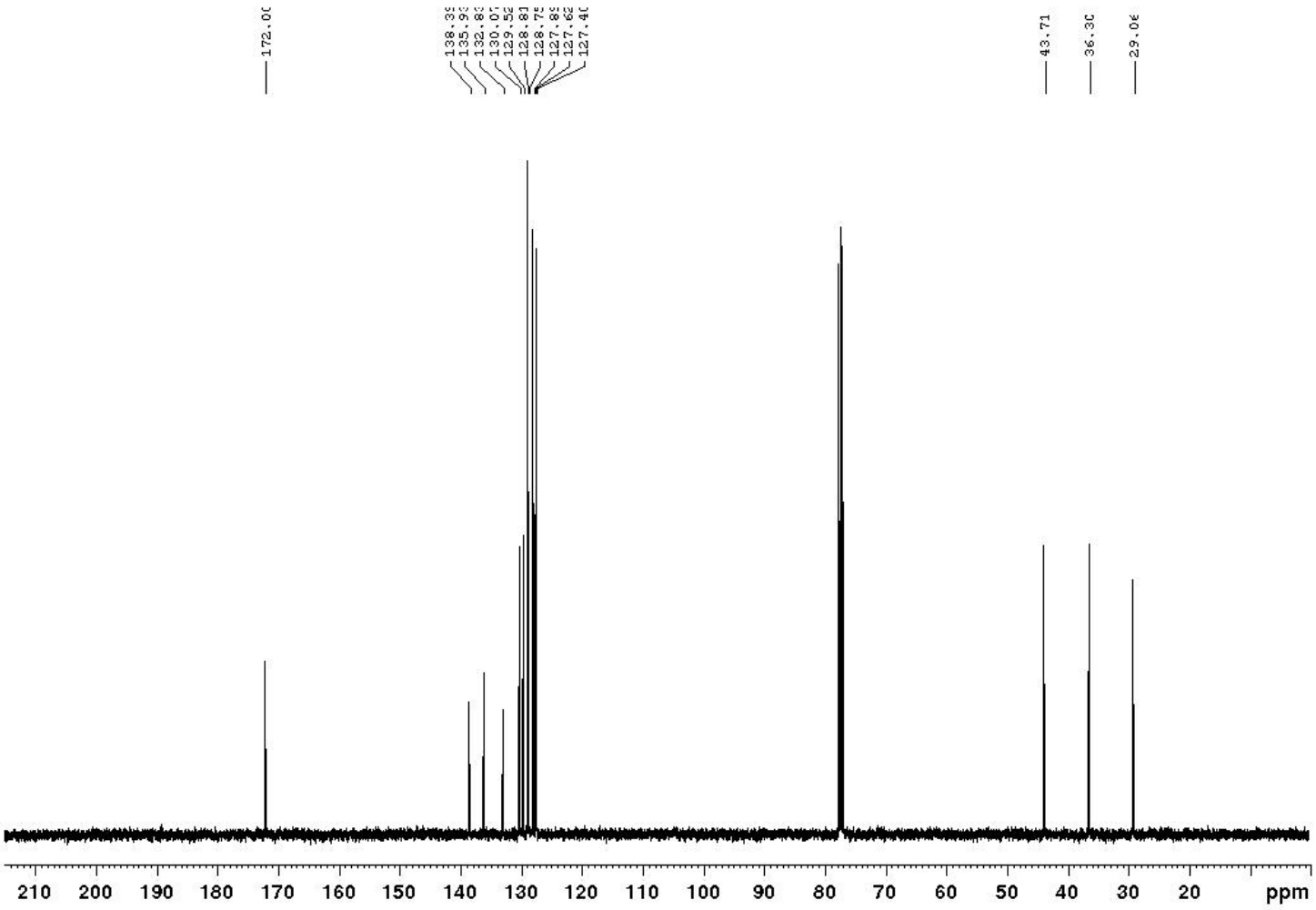


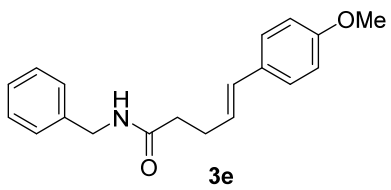
m.p.	122.5–124.5 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.30 (9H, m, a,b,b',c,c',l,l',m,m'), 6.39 (1H, d, *J* = 15.8 Hz, j), 6.19 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 6.00 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 2.59 (2H, q, *J* = 7.0 Hz, h), 2.39 (2H, t, *J* = 7.4 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.00 (f), 138.39 (n), 135.93 (d), 132.83 (k), 130.07 (j), 129.52 (i), 128.81 (l,l'), 128.75 (b,b'), 127.89 (m,m'), 127.62 (a), 127.40 (c,c'), 43.71 (e), 36.30 (g), 29.06 (h)
IR (neat)	3294 (N-H stretch), 1634 (C=O stretch), 1548, 1489, 1093, 1010, 970, 851, 806, 745, 696 cm^{-1}
HRMS (EI)	$C_{18}H_{18}ClNO$ (M): 299.1077, found 299.1082 *m/z*

^{1}H NMR

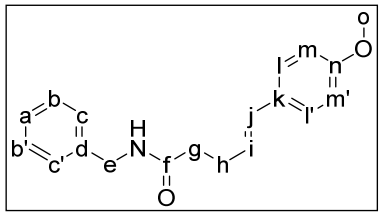

7.258
6.411
6.372
6.225
6.208
6.191
6.186
6.168
6.151
6.002
4.456
4.441
2.613
2.595
2.577
2.560
2.405
2.387
2.369
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
9.62
1.07
1.08
1.00
2.17
2.18
2.17

^{13}C NMR


172.0
138.3
135.9
132.8
130.0
129.5
128.8
128.7
127.8
127.6
127.4
43.71
36.30
29.04
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

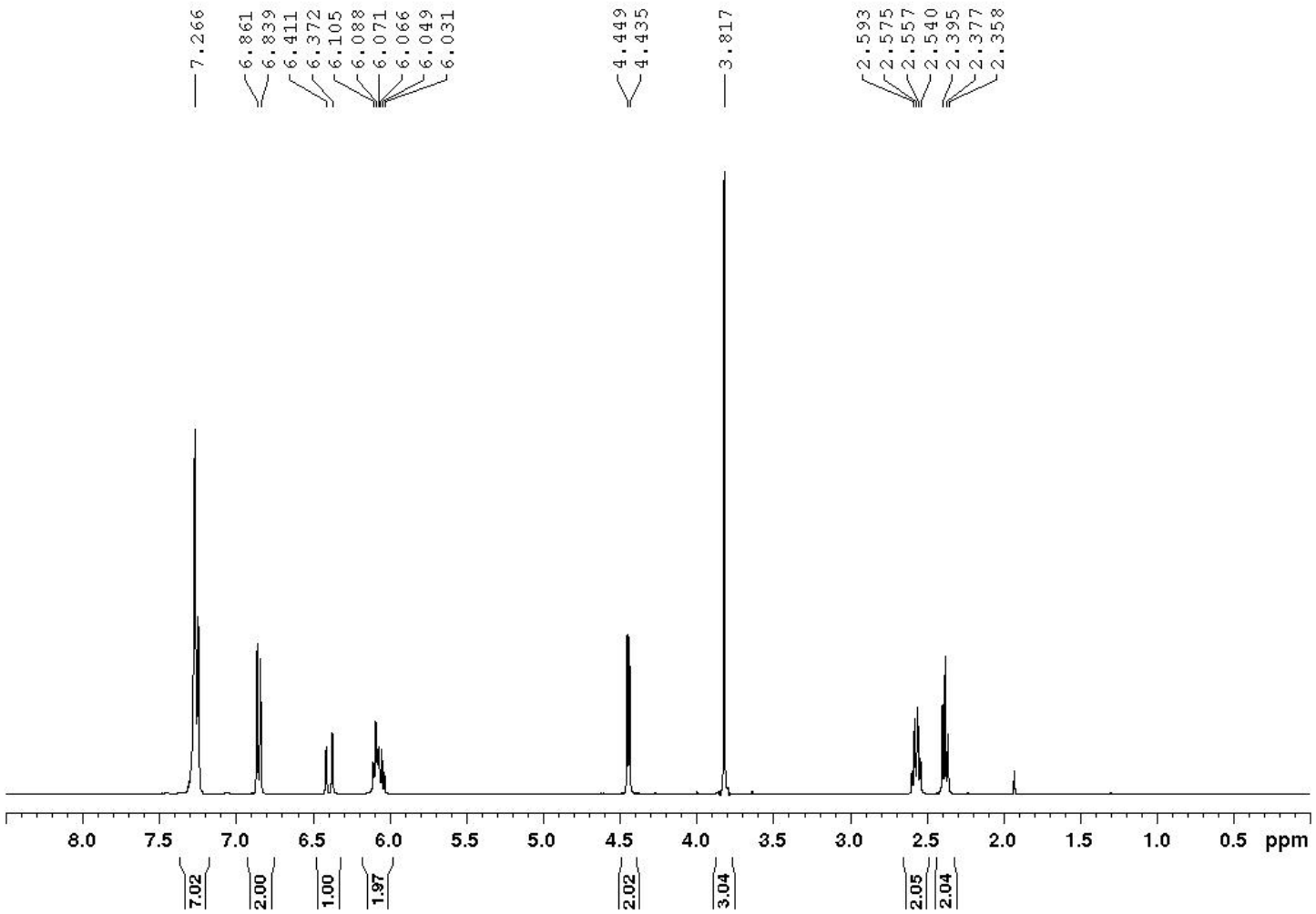


3e

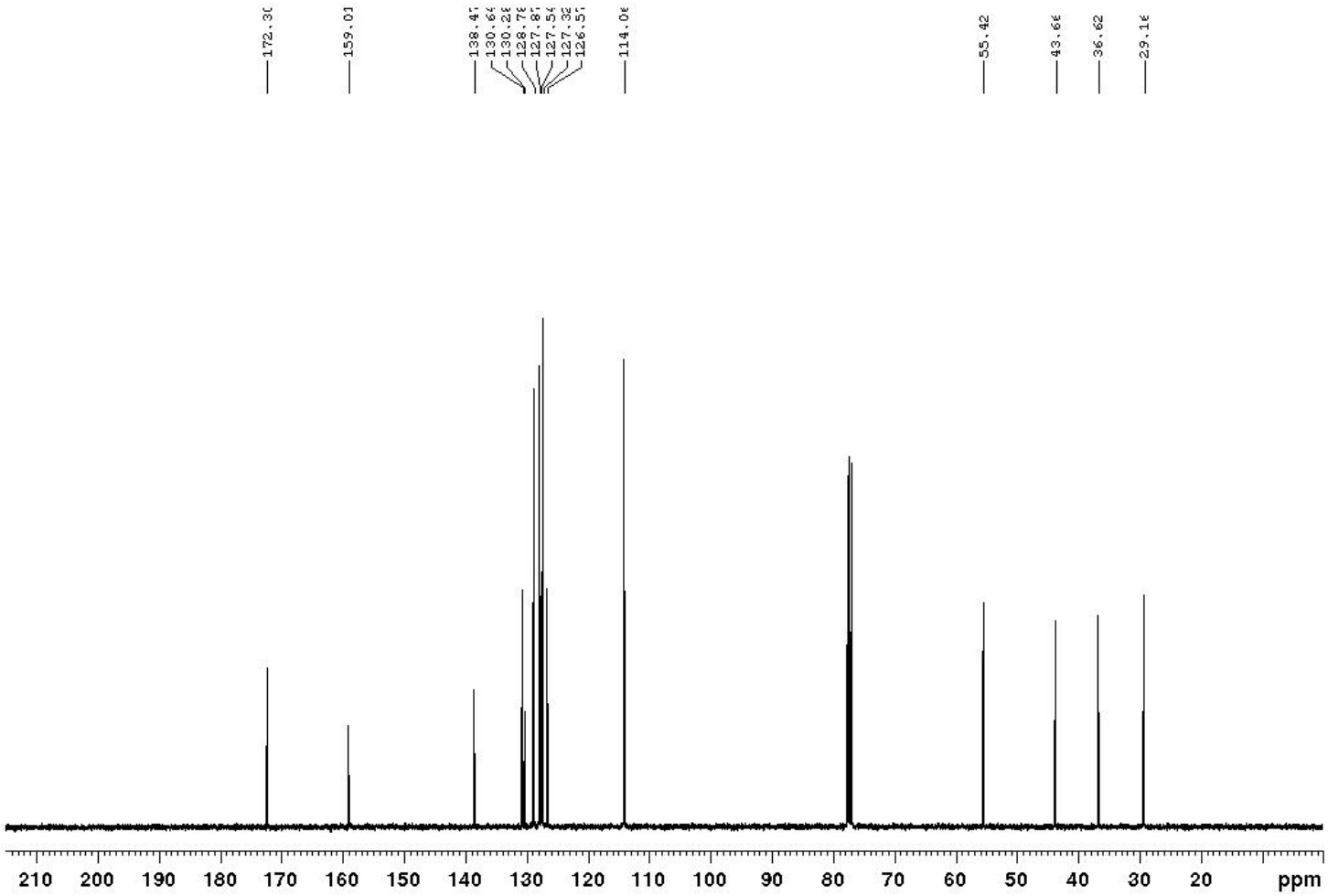


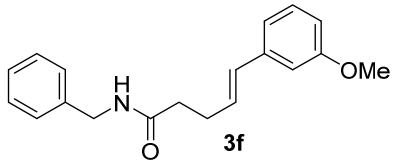
m.p.	123.5–124.5 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.30 (7H, m, a,b,b',c,c',l,l'), 6.85 (2H, d, *J* = 8.7 Hz ,m,m'), 6.39 (1H, d, *J* = 15.8 Hz, j), 6.07 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 4.44 (2H, d, *J* = 5.8 Hz, e), 3.82 (3H, s, o), 2.57 (2H, q, *J* = 7.3 Hz, h), 2.38 (2H, t, *J* = 7.4 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.30 (f), 159.01 (n), 138.47 (d), 130.64 (j), 130.28 (k), 128.78 (l,l'), 127.87 (b,b'), 127.53 (a), 127.32 (c,c'), 126.57 (i), 114.06 (m,m'), 55.42 (o), 43.66 (e), 36.62 (g), 29.16 (h)
IR (neat)	3292 (N-H stretch), 3065, 3032, 2918, 1636 (C=O stretch), 1605, 1545, 1509, 1454, 1439, 1248, 1221, 1174, 1029, 969, 845, 805, 693 cm^{-1}
HRMS (EI)	$C_{19}H_{21}NO_2$ (M): 295.1572, found 295.1567 *m/z*

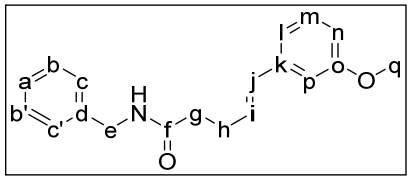
^{1}H NMR


7.266
6.861
6.839
6.411
6.372
6.105
6.088
6.071
6.066
6.049
6.031
4.449
4.435
3.817
2.593
2.575
2.557
2.540
2.395
2.377
2.358
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
7.02
2.00
1.00
1.97
2.02
3.04
2.05
2.04

^{13}C NMR

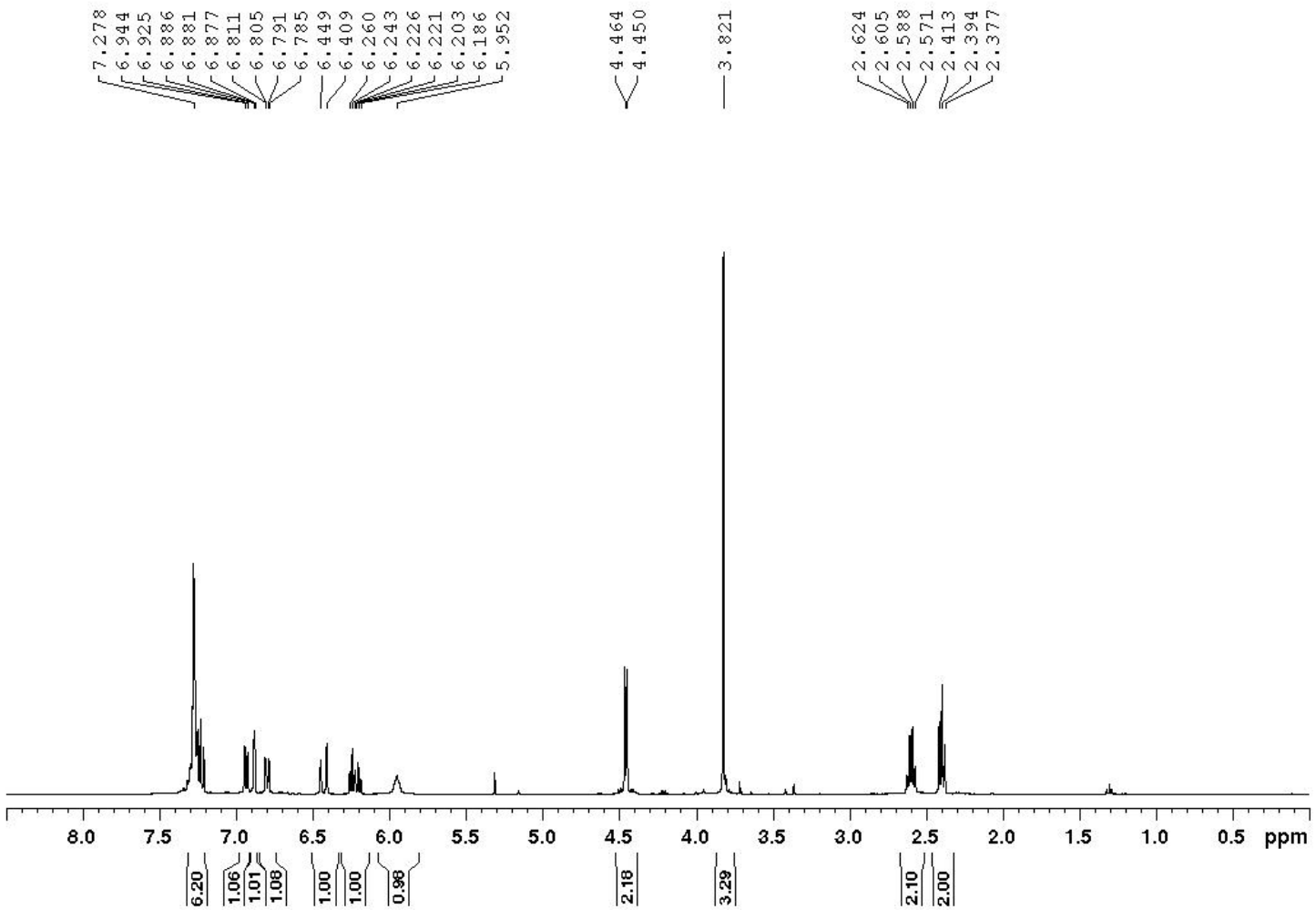

172.3
159.0
138.4
130.6
130.2
128.7
127.8
127.5
127.3
126.5
114.0
55.42
43.6
36.62
29.1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



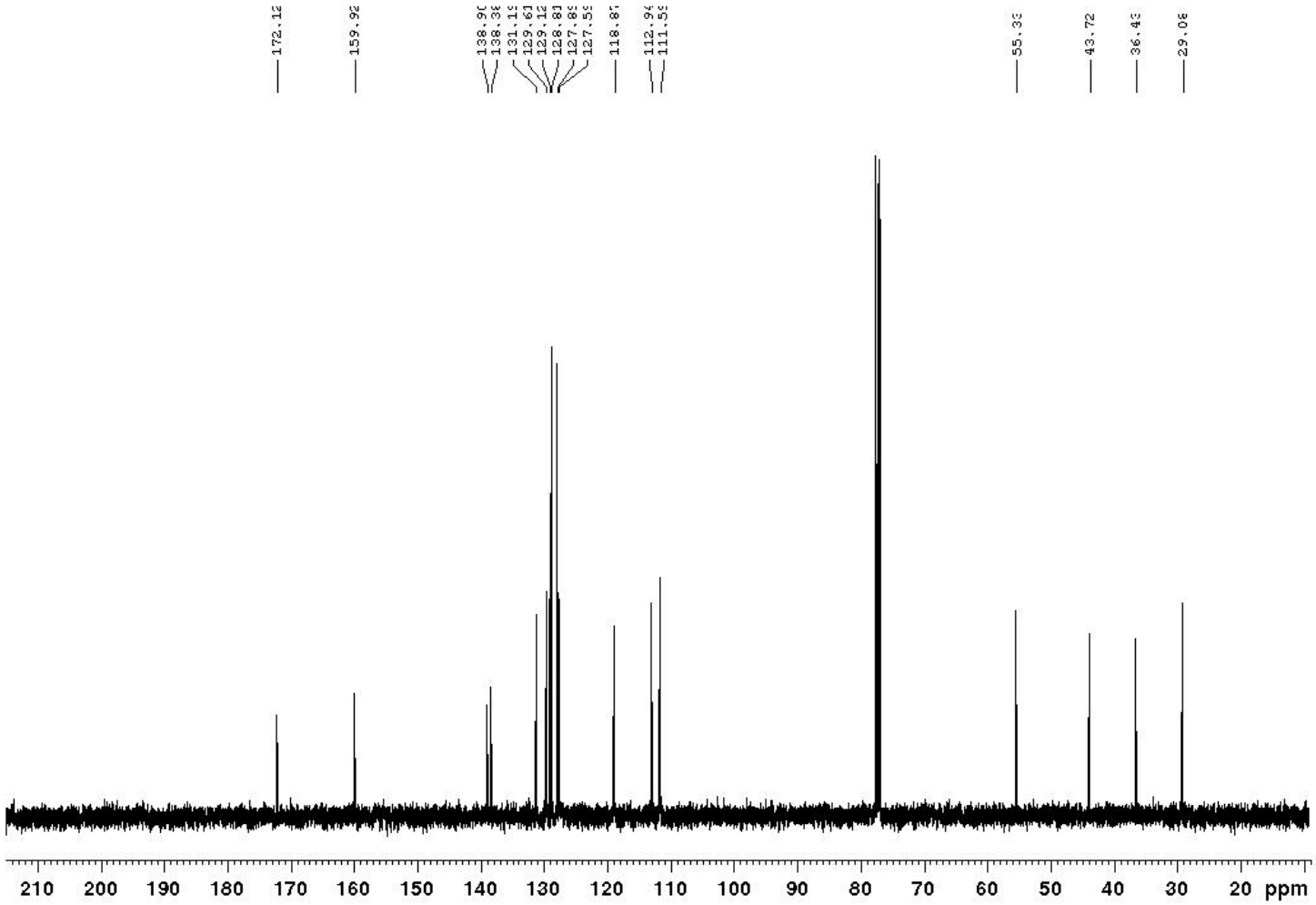


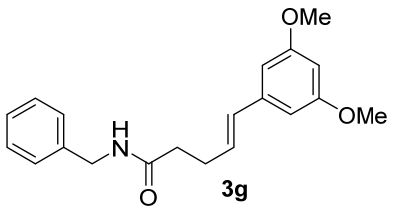
m.p.	57.5–59.0 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.30 (6H, m, a,b,b',c,c',m), 6.93 (1H, d, *J* = 7.6 Hz ,l), 6.88 (1H, d, *J* = 2.1 Hz, p), 6.80 (1H, dd, *J* = 8.2 and 2.5 Hz, n), 6.43 (1H, d, *J* = 15.8 Hz, j), 6.22 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.95 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 3.82 (3H, s, q), 2.60 (2H, q, *J* = 7.5 Hz, h), 2.39 (2H, t, *J* = 7.5 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.12 (f), 159.92 (o), 138.90 (k), 138.38 (d), 131.19 (j), 129.61 (i), 129.12 (m), 128.81 (b,b'), 127.89 (c,c'), 127.59 (a), 118.87 (l), 112.94 (n), 111.59 (p), 55.33 (q), 43.72 (e), 36.43 (g), 29.08 (h)
IR (neat)	3284 (N-H stretch), 3062, 3027, 2914, 2834, 1642 (C=O stretch), 1603, 1598, 1577, 1544, 1491, 1464, 1453, 1430, 1256, 1154, 1041, 965, 772, 733, 689 cm^{-1}
HRMS (EI)	$C_{19}H_{21}NO_2$ (M): 295.1572, found 295.1571 *m/z*

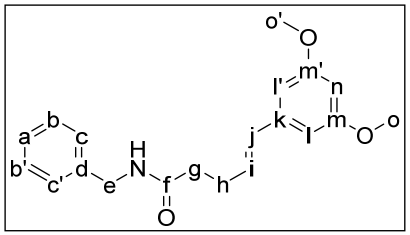
^{1}H NMR


7.278
6.944
6.925
6.886
6.881
6.877
6.811
6.805
6.791
6.785
6.449
6.409
6.260
6.243
6.226
6.221
6.203
6.186
5.952
4.464
4.450
3.821
2.624
2.605
2.588
2.571
2.413
2.394
2.377
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
6.20
1.06
1.01
1.08
1.00
1.00
0.98
2.18
3.29
2.10
2.00

^{13}C NMR

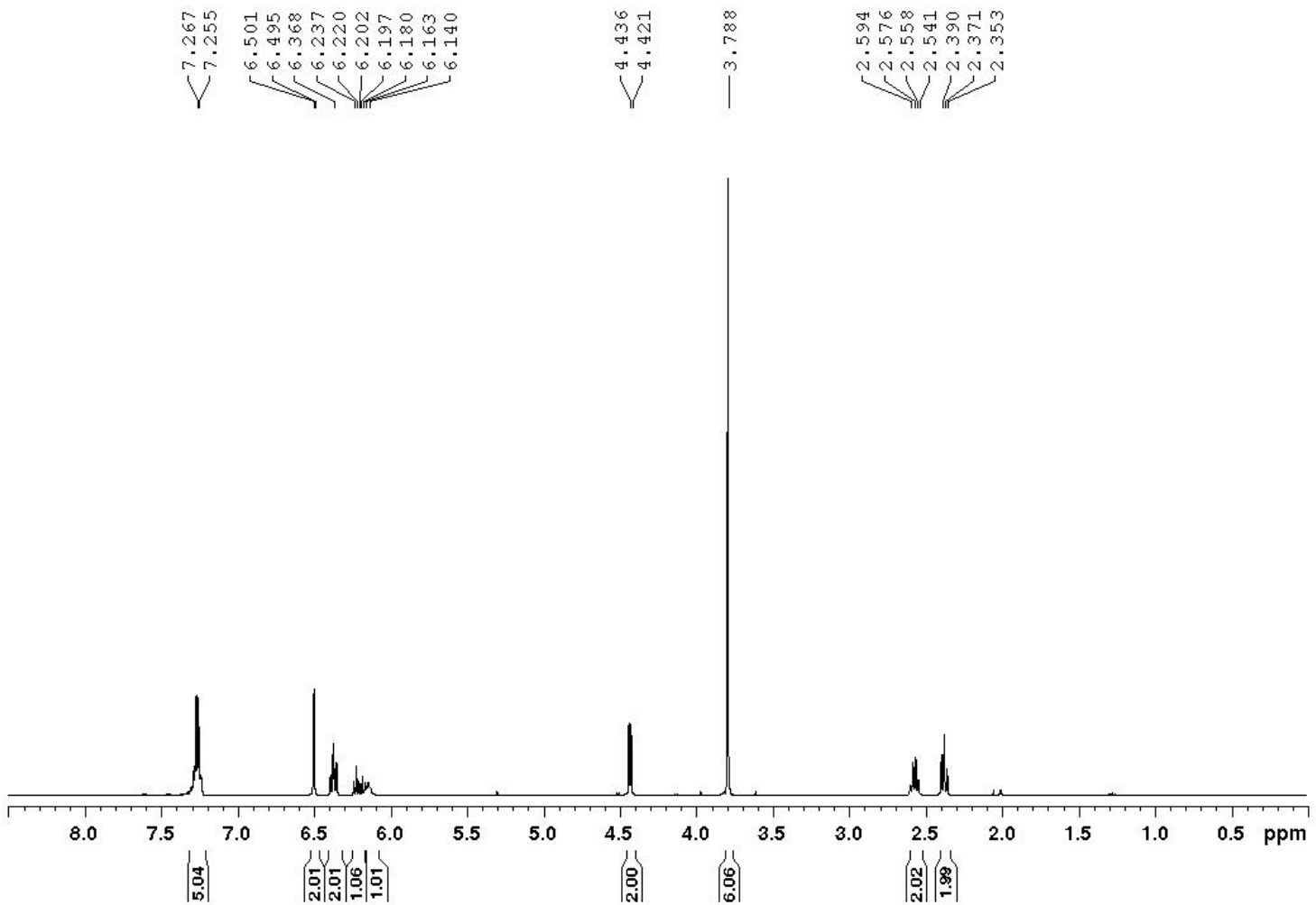

172.12
159.92
138.90
138.38
131.15
129.61
129.12
128.81
127.85
127.55
118.87
112.94
111.55
55.38
43.72
36.43
29.08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



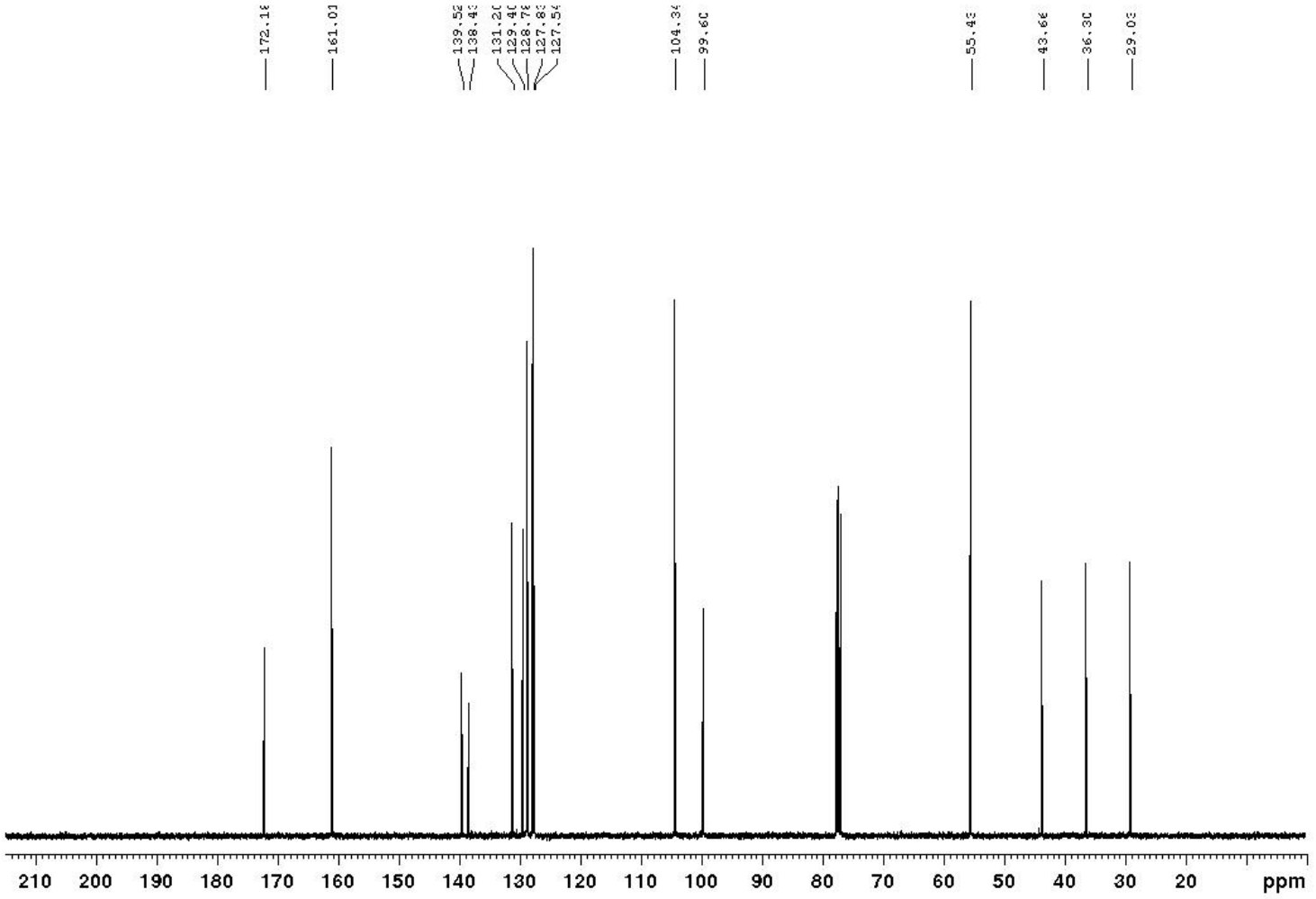


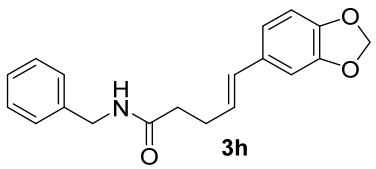
m.p.	91.0–92.0 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.30 (5H, m, a,b,b',c,c'), 6.60 (2H, d, *J* = 2.2 Hz ,l,l'), 6.30–6.40 (2H, m, j,n), 6.20 (1H, dt, *J* = 15.8 and 6.8 Hz, i), 6.14 (1H, br s), 4.43 (2H, d, *J* = 5.8 Hz, e), 3.79 (6H, s, o,o'), 2.57 (2H, q, *J* = 6.9 Hz, h), 2.37 (2H, t, *J* = 7.5 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.18 (f), 161.01 (o,o'), 139.52 (k), 138.43 (d), 131.20 (j), 129.40 (i), 128.78 (b,b'), 127.83 (c,c'), 127.54 (a), 104.34 (l,l'), 99.60 (n), 55.43 (o,o'), 43.66 (e), 36.30 (g), 29.03 (h)
IR (neat)	3287 (N-H stretch), 2936, 2359, 1629 (C=O stretch), 1587, 1536, 1454, 1422, 1203, 1149, 1058, 960, 819, 749, 697, 680 cm^{-1}
HRMS (EI)	$C_{20}H_{23}NO_3$ (M): 325.1678, found 325.1685 *m/z*

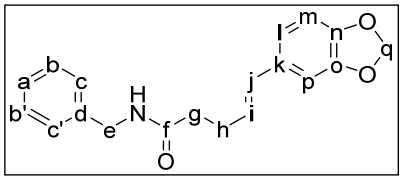
^{1}H NMR


7.267
7.255
6.501
6.495
6.368
6.237
6.220
6.202
6.197
6.180
6.163
6.140
4.436
4.421
3.788
2.594
2.576
2.558
2.541
2.390
2.371
2.353
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.04
2.01
2.01
1.06
1.01
2.00
6.06
2.02
1.99

^{13}C NMR

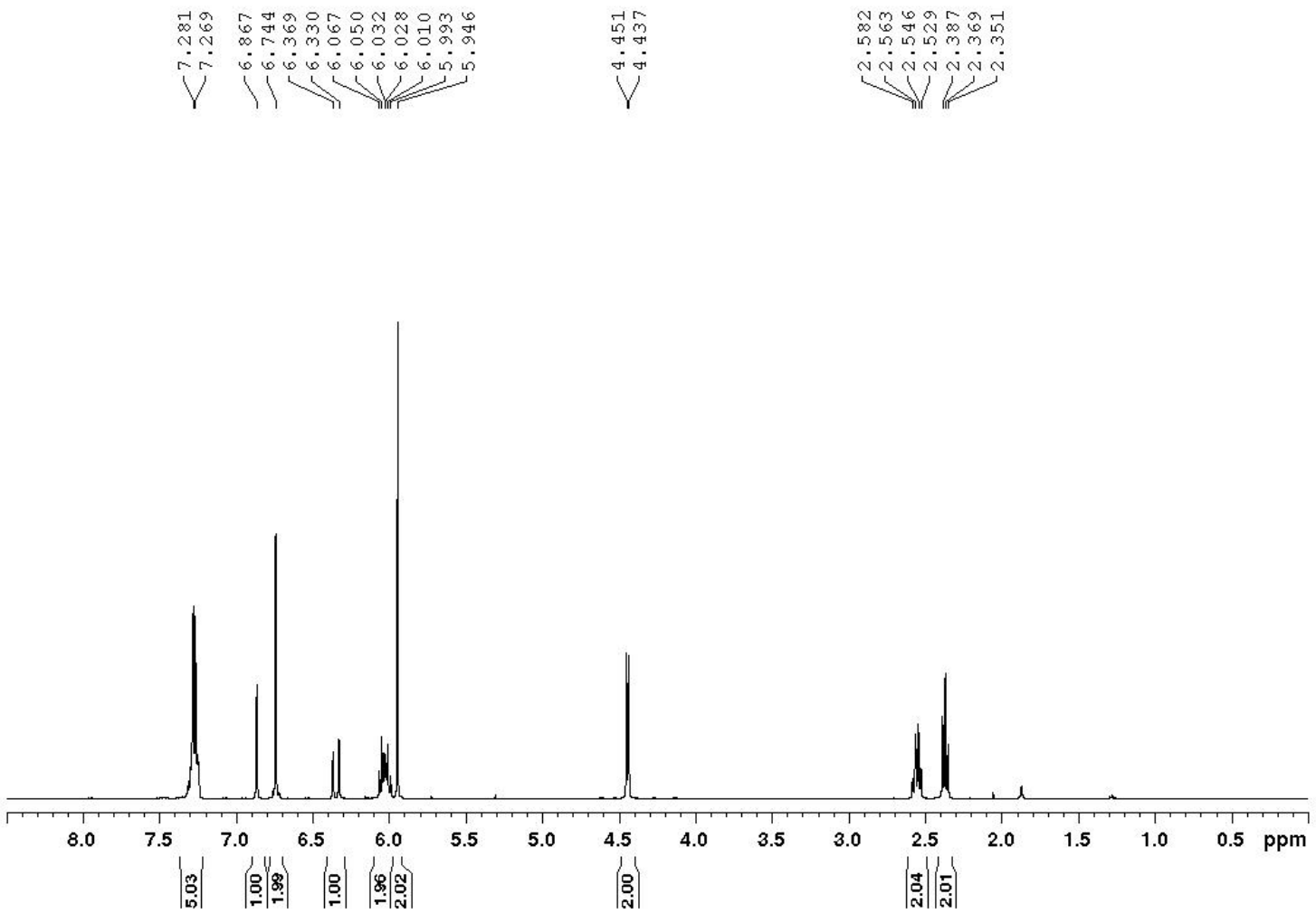

172.18
161.01
139.52
138.43
131.20
129.40
128.78
127.83
127.54
104.34
99.60
55.43
43.66
36.30
29.03
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



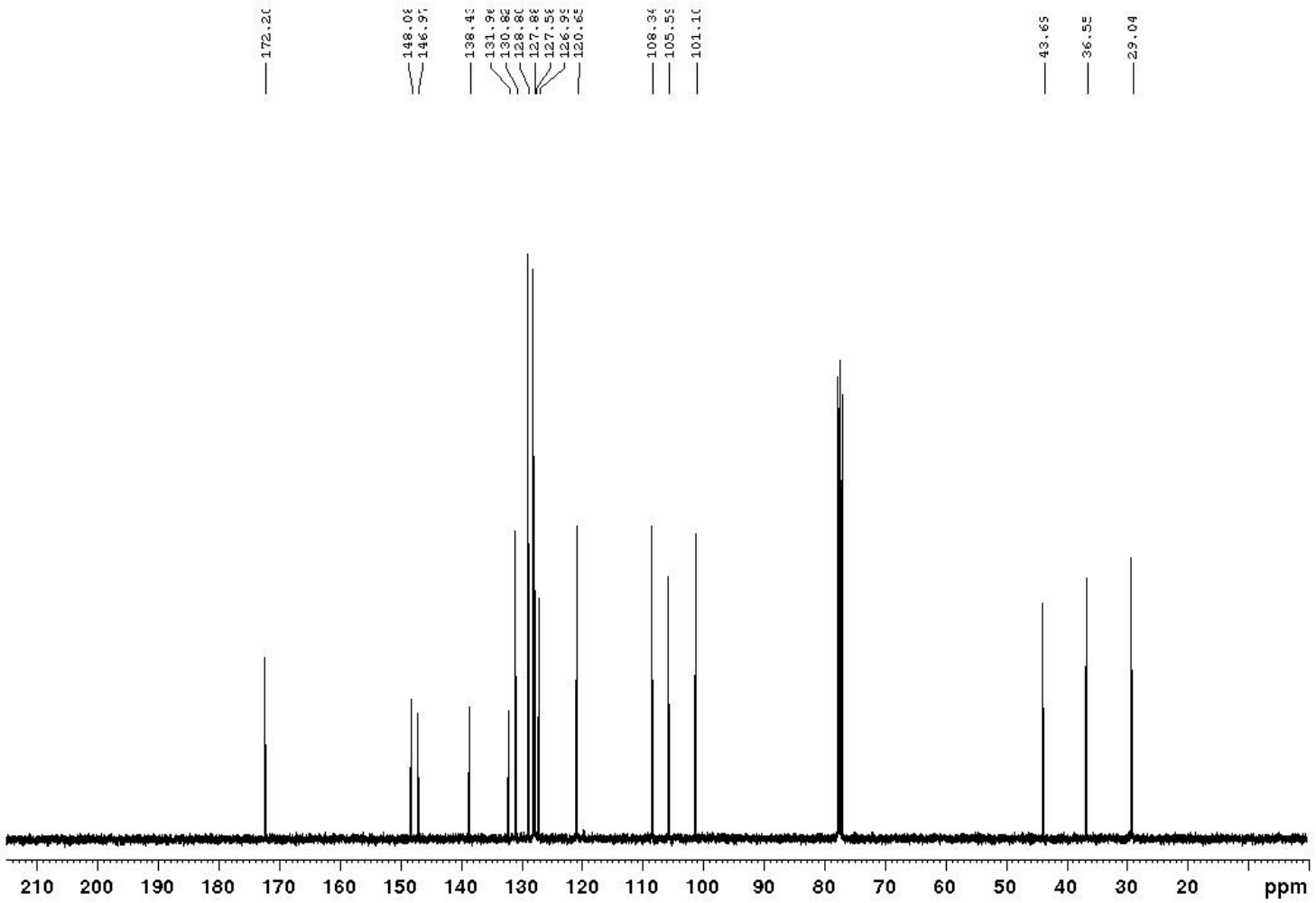


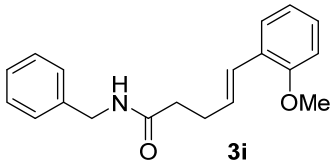
m.p.	100.5–102.0 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (5H, m, a,b,b',c,c'), 6.85–6.90 (1H, m, p), 6.70–6.75 (2H, m, l,m), 6.35 (1H, d, *J* = 15.8 Hz, j), 6.03 (1H, dt, *J* = 15.7 and 6.9 Hz, i), 6.03 (1H, br s, NH, *overlapped* with i), 5.95 (2H, s, q), 4.44 (2H, d, *J* = 5.7 Hz, e), 2.55 (2H, q, *J* = 7.0 Hz, h), 2.37 (2H, t, *J* = 7.5 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.20 (f), 148.08 (o), 146.97 (n), 138.43 (d), 131.97 (k), 130.82 (j), 128.80 (b,b'), 127.88 (c,c'), 127.58 (a), 126.99 (i), 120.65 (l), 108.34 (m), 105.59 (p), 101.10 (q), 43.69 (e), 36.55 (g), 29.04 (h)
IR (neat)	3292 (N-H stretch), 2971, 2911, 2360, 1635 (C=O stretch), 1603, 1544, 1505, 1489, 1445, 1237, 1031, 967, 921, 801, 741, 694 cm^{-1}
HRMS (EI)	$C_{19}H_{19}NO_3$ (M): 309.1365, found 309.1365 *m/z*

^{1}H NMR

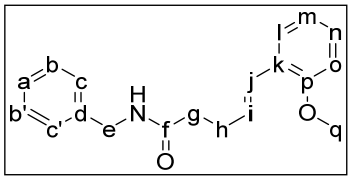

7.281
7.269
6.867
6.744
6.369
6.330
6.067
6.050
6.032
6.028
6.010
5.993
5.946
4.451
4.437
2.582
2.563
2.546
2.529
2.387
2.369
2.351
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.03
1.00
1.99
1.00
1.96
2.02
2.00
2.04
2.01

^{13}C NMR


172.20
148.08
146.97
138.43
131.98
130.82
128.80
127.88
127.58
126.95
120.65
108.34
105.59
101.10
43.65
36.55
29.04
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

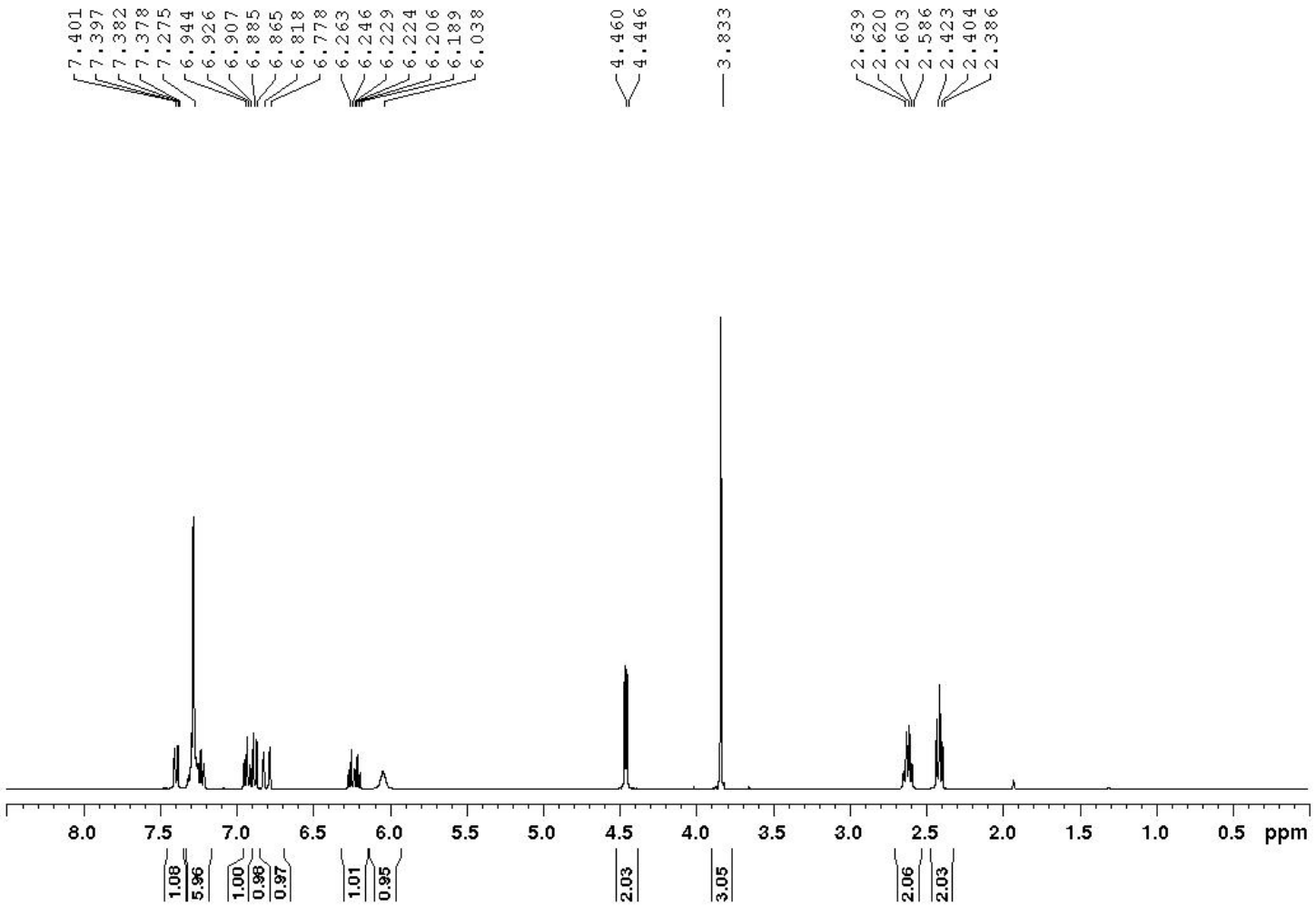


3i

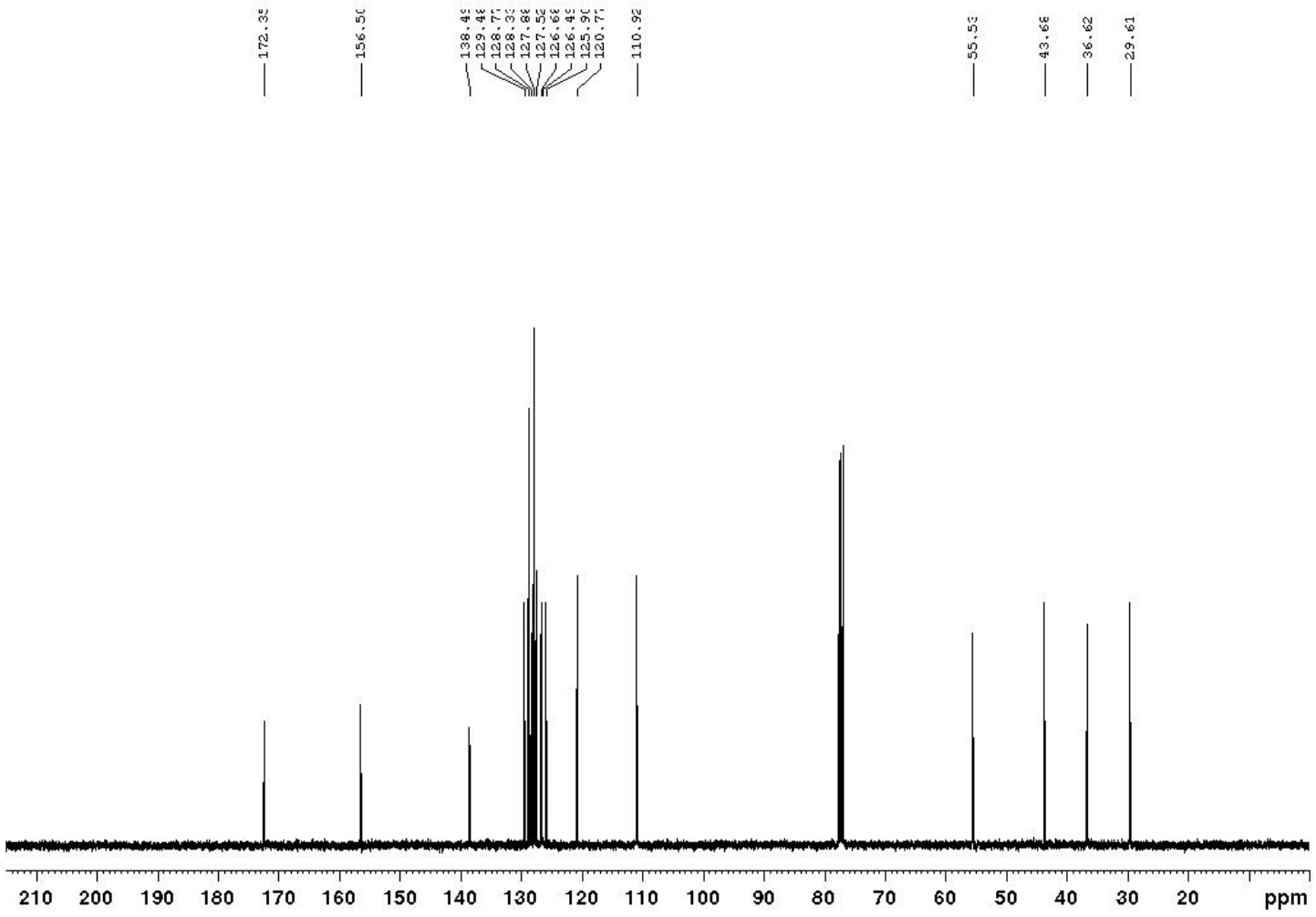


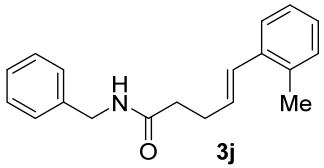
m.p.	87.5–88.0 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.39 (1H, dd, *J* = 7.8 and 1.6 Hz, n), 7.20–7.30 (6H, m, a,b,b',c,c',l), 6.93 (1H, t, *J* = 7.4 Hz , m), 6.88 (1H, d, *J* = 8.2 Hz, o), 6.80 (1H, d, *J* = 16.0 Hz, j), 6.23 (1H, dt, *J* = 16.0 and 6.9 Hz, i), 6.04 (1H, br s), 4.45 (2H, d, *J* = 5.8 Hz, e), 3.83 (3H, s, q), 2.61 (2H, q, *J* = 7.3 Hz, h), 2.40 (2H, t, *J* = 7.6 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.35 (f), 156.50 (p), 138.49 (d), 129.48 (j), 128.77 (b,b'), 128.33 (l), 127.88 (c,c'), 127.52 (a), 126.68 (n), 126.49 (k), 125.90 (i), 120.77 (m), 110.92 (o), 55.53 (q), 43.68 (e), 36.62 (g), 29.61 (h)
IR (neat)	3307 (N-H stretch), 2923, 2359, 1636 (C=O stretch), 1544, 1488, 1242, 1027, 963, 752, 743, 729, 700 cm^{-1}
HRMS (EI)	$C_{19}H_{21}NO_2$ (M): 295.1572, found 295.1562 *m/z*

^{1}H NMR

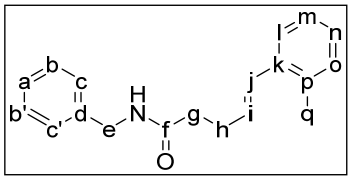

7.401
7.397
7.382
7.378
7.275
6.944
6.926
6.907
6.885
6.865
6.818
6.778
6.263
6.246
6.229
6.224
6.206
6.189
6.038
4.460
4.446
3.833
2.639
2.620
2.603
2.586
2.423
2.404
2.386
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.08
5.96
1.00
0.98
0.97
1.01
0.95
2.03
3.05
2.06
2.03

^{13}C NMR


172.35
156.50
138.45
129.48
128.77
128.33
127.88
127.52
126.68
126.45
125.90
120.77
110.92
55.58
43.68
36.62
29.61
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

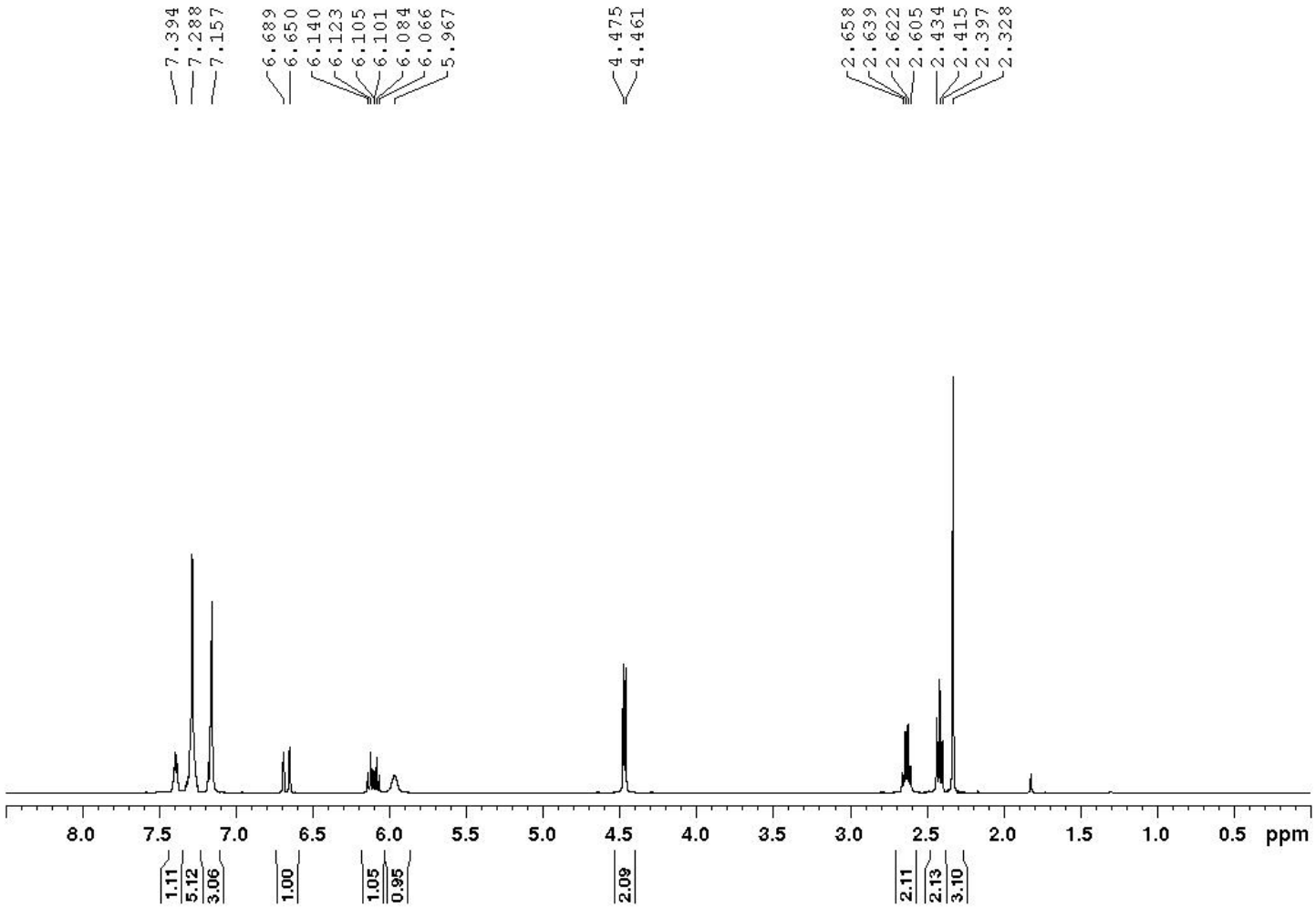


3j

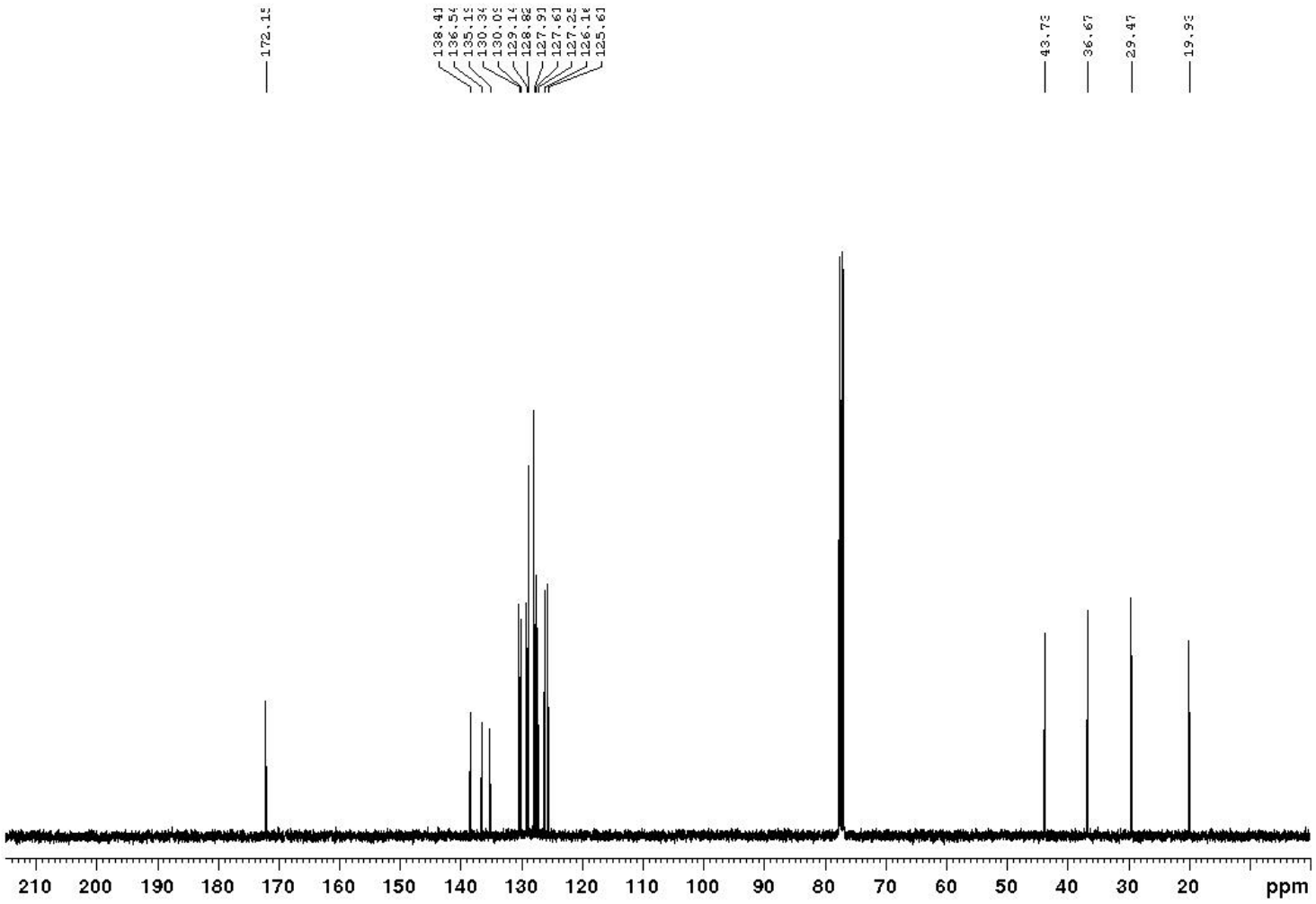


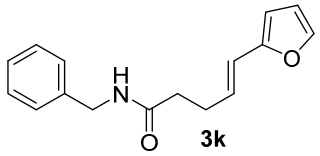
m.p.	67.5–68.5 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.35–7.45 (1H, m, aryl), 7.25–7.35 (5H, m, aryl), 7.10–7.20 (3H, m, aryl), 6.67 (1H, d, *J* = 15.6 Hz, j), 6.10 (1H, dt, *J* = 15.6 and 6.9 Hz, i), 5.97 (1H, br s), 4.47 (2H, d, *J* = 5.7 Hz, e), 2.63 (2H, q, *J* = 7.3 Hz, h), 2.42 (2H, t, *J* = 7.5 Hz, g), 2.33 (3H, s, q)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.15 (f), 138.41 (aryl), 136.54 (aryl), 135.19 (aryl), 130.34 (aryl), 130.09 (i), 129.14 (j), 128.82 (aryl), 127.91 (aryl), 127.61 (aryl), 127.25 (aryl), 126.16 (aryl), 125.61 (aryl), 43.72 (e), 36.67 (g), 29.47 (h), 19.93 (q)
IR (neat)	3297 (N-H stretch), 2914, 2359, 1633 (C=O stretch), 1533, 1242, 1173, 966, 747, 697 cm^{-1}
HRMS (EI)	$C_{19}H_{21}NO$ (M): 279.1623, found 279.1625 *m/z*

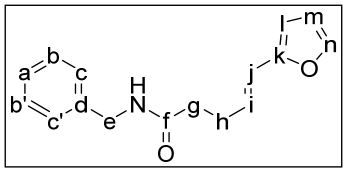
^{1}H NMR


7.394
7.288
7.157
6.689
6.650
6.140
6.123
6.105
6.101
6.084
6.066
5.967
4.475
4.461
2.658
2.639
2.622
2.605
2.434
2.415
2.397
2.328
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.11
5.12
3.06
1.00
1.05
0.95
2.09
2.11
2.13
3.10

^{13}C NMR

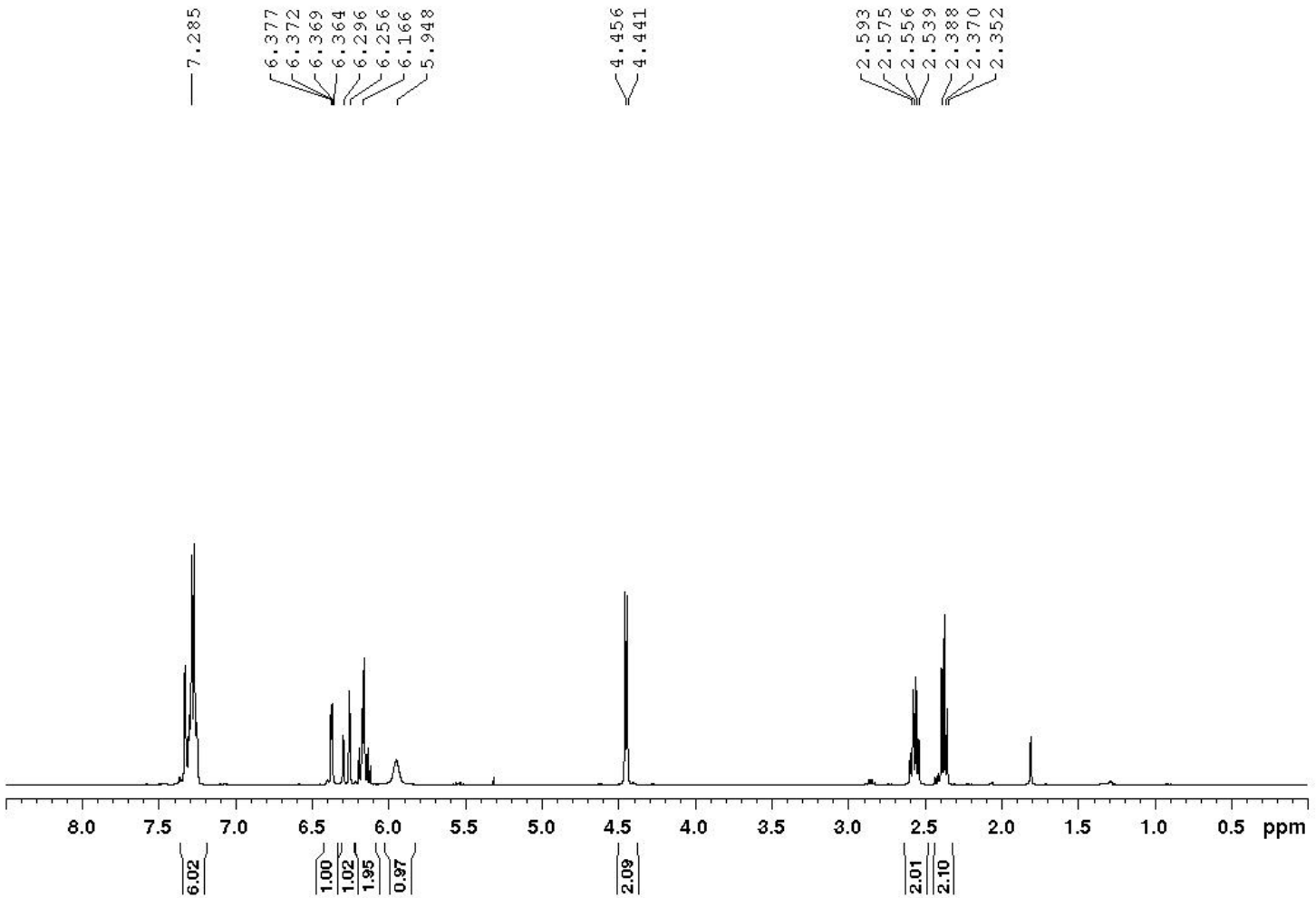

172.15
138.41
136.54
135.19
130.34
130.05
129.14
128.82
127.91
127.61
127.25
126.18
125.61
43.73
36.67
29.47
19.93
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



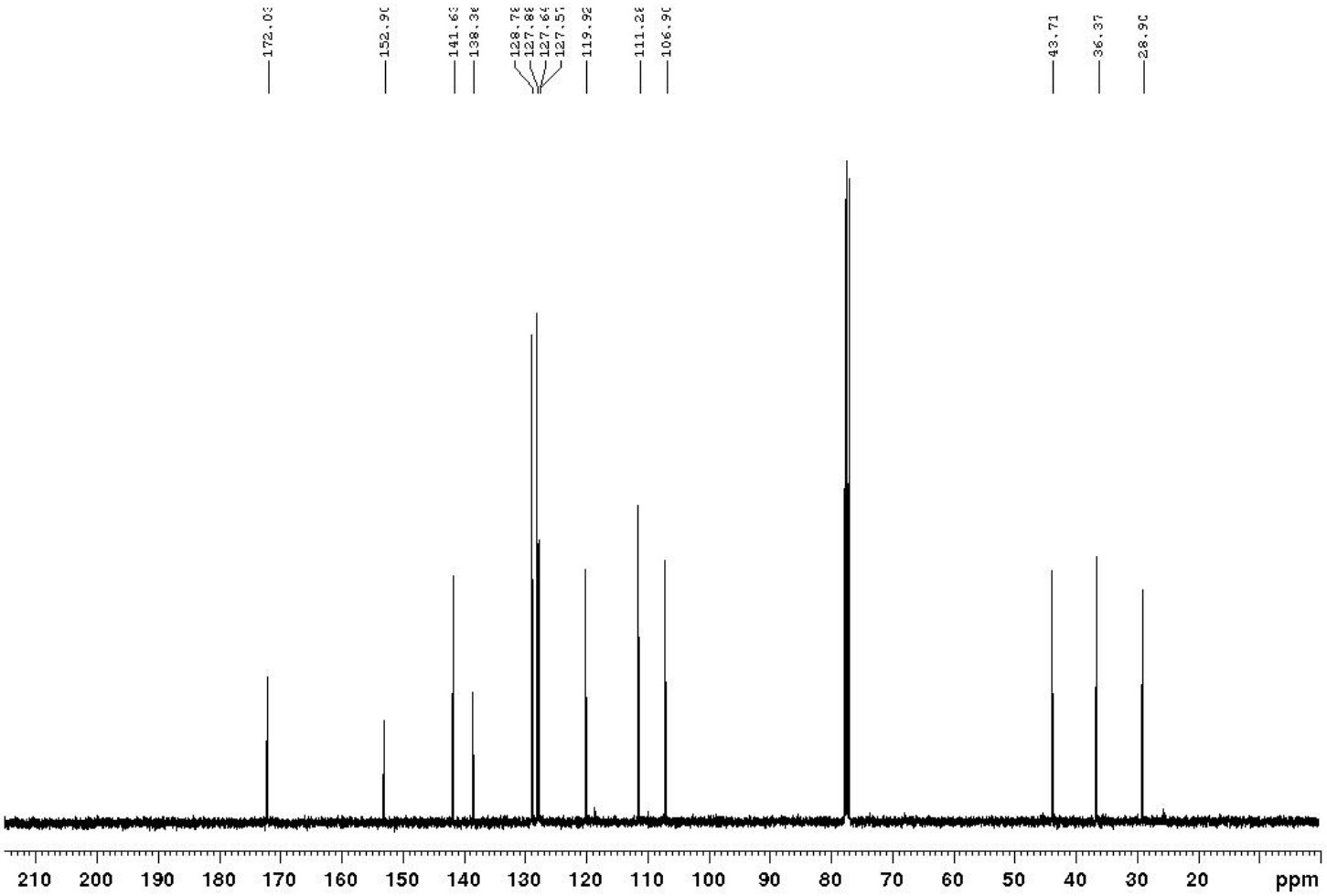


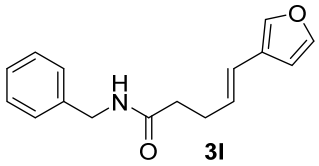
m.p.	93.5–94.5 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (6H, m, a,b,b',c,c',n), 6.37 (1H, dd, *J* = 3.2 and 1.9 Hz, m), 6.28 (1H, d, *J* = 15.9 Hz, j), 6.10–6.20 (2H, m, i and l, *overlapped*), 5.95 (1H, br s, NH), 4.45 (2H, d, *J* = 5.7 Hz, e), 2.57 (2H, q, *J* = 7.2 Hz, h), 2.37 (2H, t, *J* = 7.5 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.03 (f), 152.89 (k), 141.63 (n), 138.36 (d), 128.78 (b,b'), 127.87 (c,c'), 127.64 (i), 127.57 (a), 119.92 (j), 111.28 (m), 106.90 (l), 43.71 (e), 36.37 (g), 28.90 (h)
IR (neat)	3273 (N-H stretch), 2361, 1635 (C=O stretch), 1538, 1219, 1152, 1012, 957, 921, 726, 696 cm^{-1}
HRMS (EI)	$C_{16}H_{17}NO_2$ (M): 255.1259, found 255.1252 *m/z*

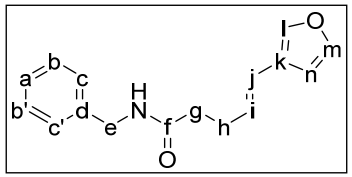
^{1}H NMR


7.285
6.377
6.372
6.369
6.364
6.296
6.256
6.166
5.948
4.456
4.441
2.593
2.575
2.556
2.539
2.388
2.370
2.352
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
6.02
1.00
1.02
1.95
0.97
2.09
2.01
2.10

^{13}C NMR

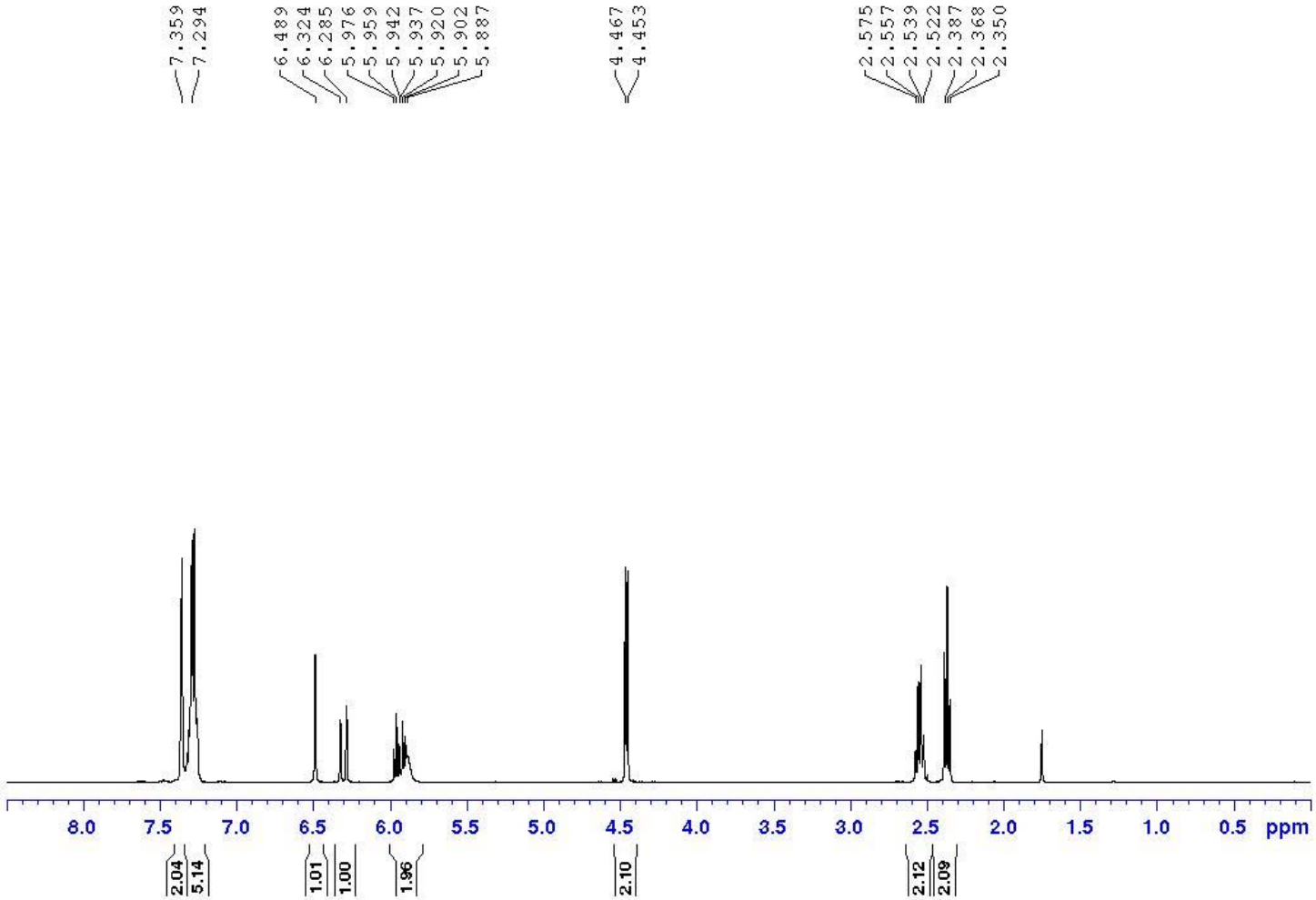

172.03
152.90
141.63
138.34
128.78
127.88
127.64
127.51
119.92
111.28
106.90
43.71
36.37
28.90
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



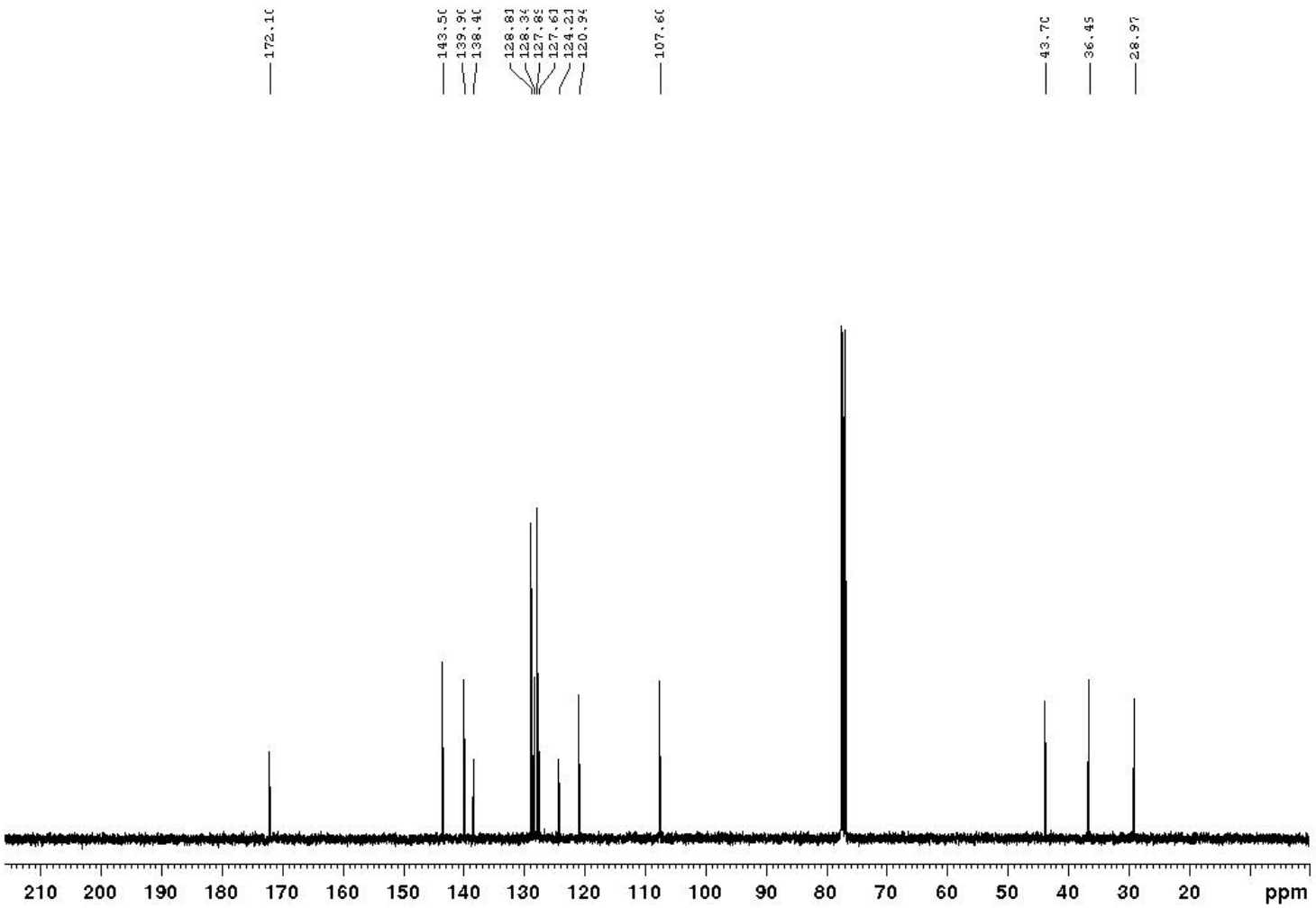


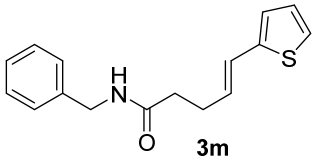
m.p.	91.0–92.5 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (7H, m, a,b,b',c,c',l,m), 6.49 (1H, d, *J* = 0 Hz, n), 6.30 (1H, d, *J* = 15.8 Hz, j), 5.94 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.89 (1H, br s, NH), 4.46 (2H, d, *J* = 5.7 Hz, e), 2.55 (2H, q, *J* = 7.4 Hz, h), 2.37 (2H, t, *J* = 7.4 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.10 (f), 143.50 (m), 139.90 (l), 138.40 (d), 128.81 (b,b'), 128.33 (i), 127.89 (c,c'), 127.61 (a), 124.21 (k), 120.94 (j), 107.60 (n), 43.70 (e), 36.49 (g), 28.97 (h)
IR (neat)	3294 (N-H stretch), 2360, 2342, 1635 (C=O stretch), 1548, 1225, 1158, 1072, 1021, 963, 873, 779, 746, 728, 694 cm^{-1}
HRMS (EI)	$C_{16}H_{17}NO_2$ (M): 255.1259, found 255.1259 *m/z*

^{1}H NMR

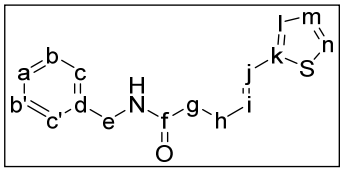

7.359
7.294
6.489
6.324
6.285
5.976
5.959
5.942
5.937
5.920
5.902
5.887
4.467
4.453
2.575
2.557
2.539
2.522
2.387
2.368
2.350
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.04
5.14
1.01
1.00
1.96
2.10
2.12
2.09

^{13}C NMR


172.1
143.5
139.9
138.4
128.8
128.3
127.8
127.6
124.2
120.9
107.6
43.7
36.4
28.9
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

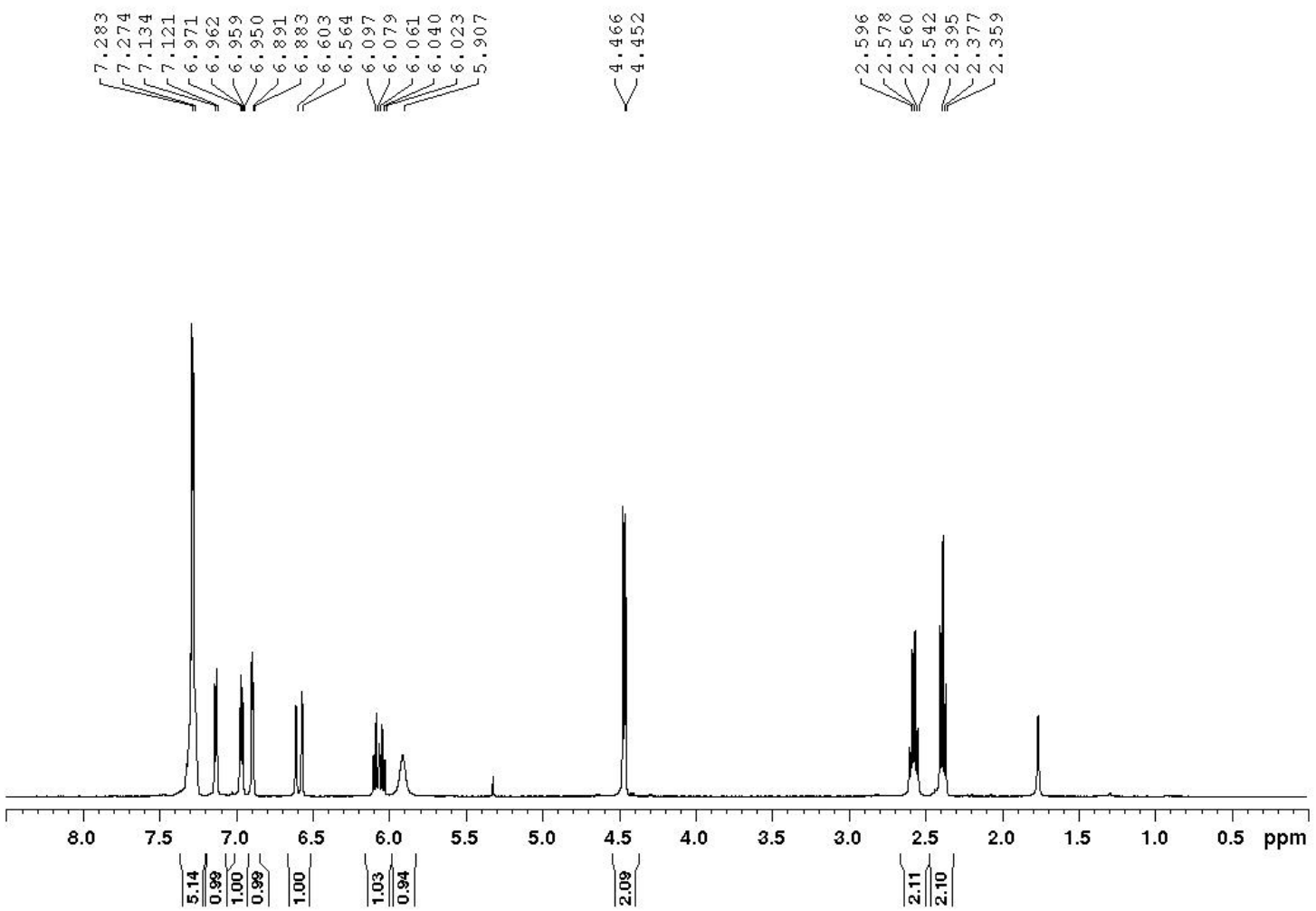


3m

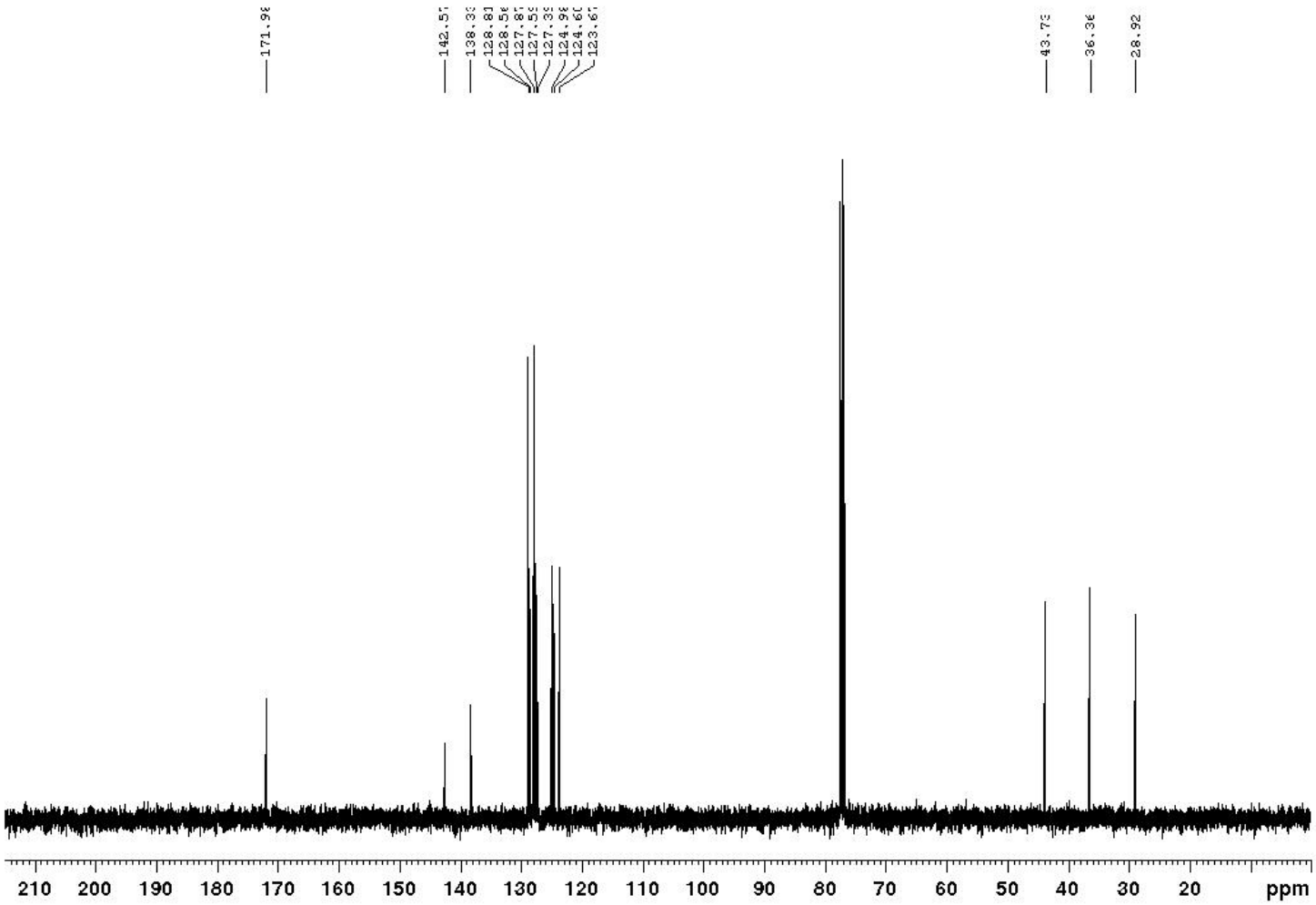


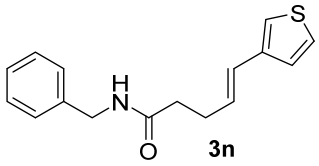
m.p.	88.5–90.0 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.35 (5H, m, a,b,b',c,c'), 7.13 (1H, d, *J* = 5.0 Hz, n), 6.96 (1H, dd, *J* = 5.0 and 3.6 Hz, l), 6.89 (1H, d. *J* = 3.3 Hz, m), 6.58 (1H, d, *J* = 15.7 Hz, j), 6.06 (1H, dt, *J* = 15.7 and 7.0 Hz, i), 5.91 (1H, br s), 4.46 (2H, d, *J* = 5.7 Hz, e), 2.57 (2H, q, *J* = 7.2 Hz, h), 2.38 (2H, t, *J* = 7.3 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.98 (f), 142.57 (k), 138.33 (d), 128.81 (b,b'), 128.56 (i), 127.87 (c,c'), 127.59 (a) , 127.39 (l), 124.98 (m), 124.60 (j). 123.67 (n), 43.73 (e), 36.36 (g), 28.92 (h)
IR (neat)	3305 (N-H stretch), 2360, 1648 (C=O stretch), 1538, 1454, 953, 736, 694 cm^{-1}
HRMS (EI)	$C_{16}H_{17}NOS$ (M): 271.1031, found 271.1034 *m/z*

^{1}H NMR

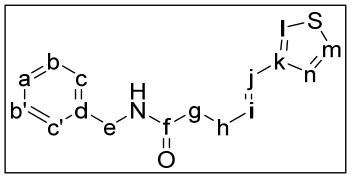

7.283
7.274
7.134
7.121
6.971
6.962
6.959
6.950
6.891
6.883
6.603
6.564
6.097
6.079
6.061
6.040
6.023
5.907
4.466
4.452
2.596
2.578
2.560
2.542
2.395
2.377
2.359
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.14
0.99
1.00
0.99
1.00
1.03
0.94
2.09
2.11
2.10

^{13}C NMR


171.98
142.57
138.33
128.83
128.58
127.81
127.55
127.33
124.98
124.66
123.67
43.73
36.36
28.92
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

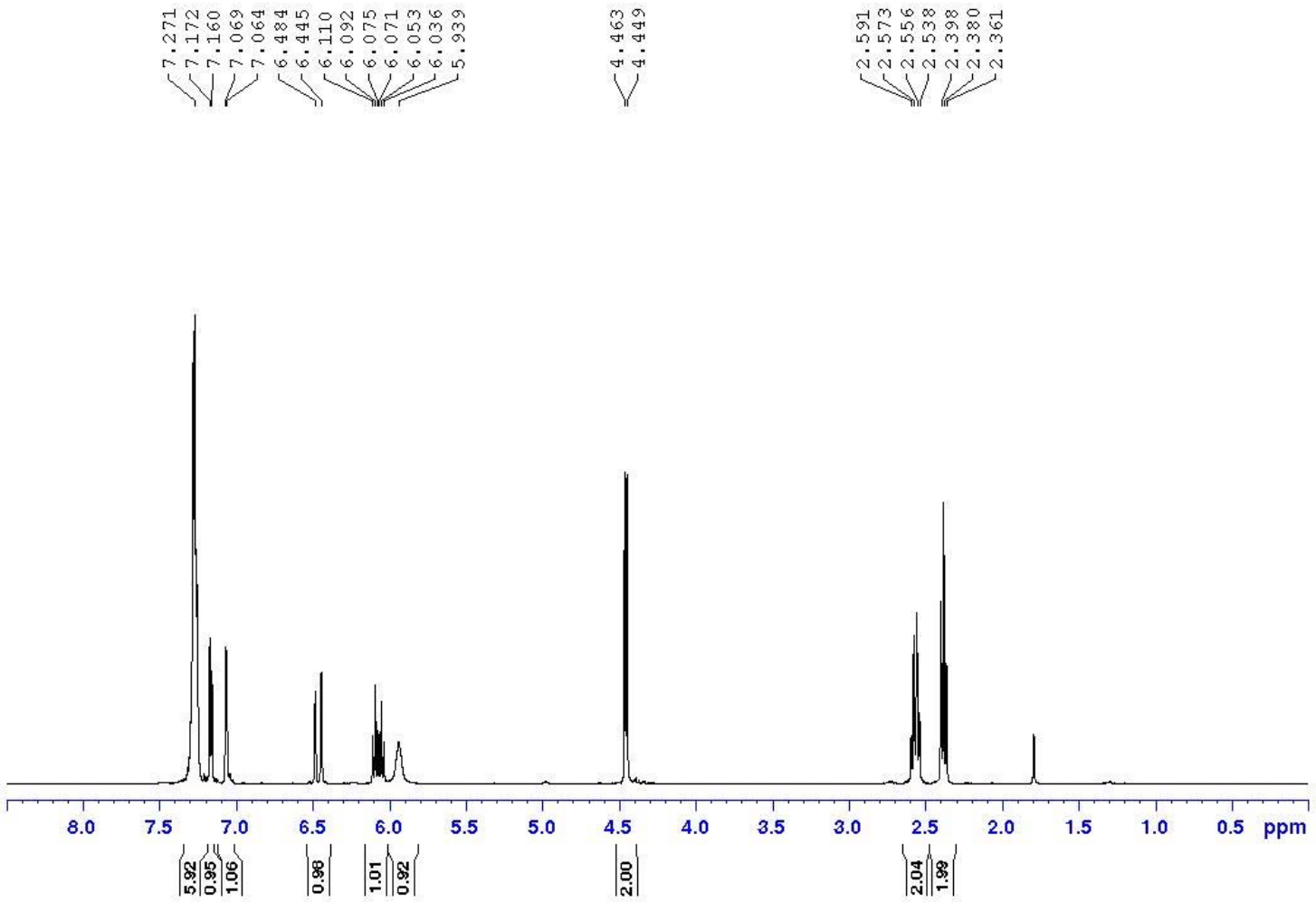


3n

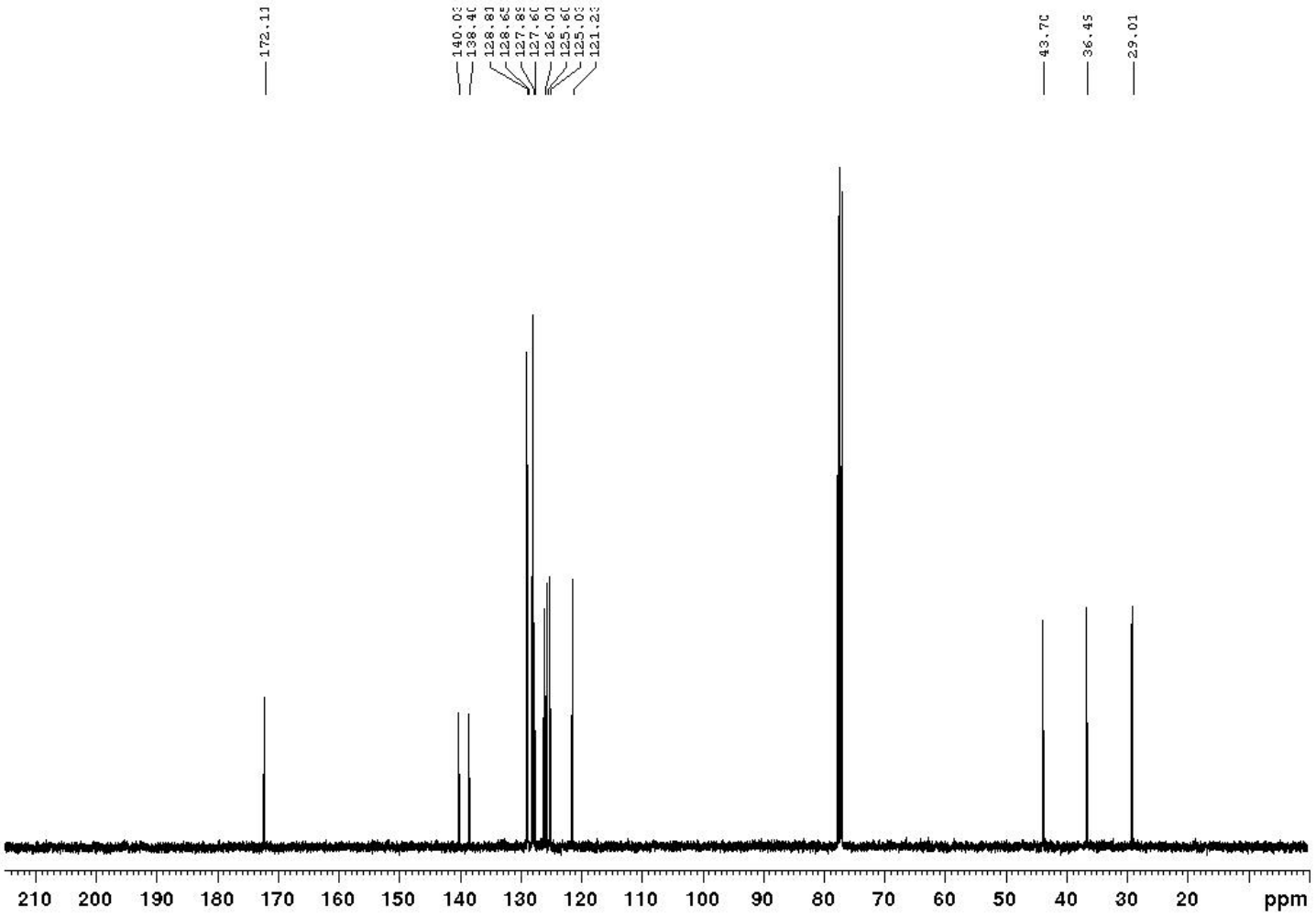


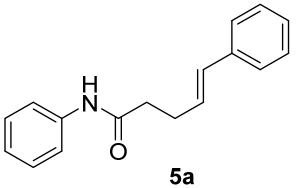
m.p.	111.5–112.5 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (6H, m, a,b,b',c,c',l,), 7.17 (1H, dd, *J* = 5.0 and 0 Hz, m), 7.07 (1H, dd, *J* = 2.2 and 0 Hz, n), 6.46 (1H, d, *J* = 15.8 Hz, j), 6.07 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.94 (1H, br s), 4.46 (2H, d, *J* = 5.7 Hz, e), 2.57 (2H, q, *J* = 7.3 Hz, h), 2.38 (2H, t, *J* = 7.4 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.11 (f), 140.03 (k), 138.40 (d), 128.81 (b,b'), 128.65 (i), 127.89 (c,c'), 127.60 (a), 126.01 (l), 125.60 (j), 125.03 (m), 121.23 (n), 43.70 (e), 36.49 (g), 29.01 (h)
IR (neat)	3289 (N-H stretch), 2360, 1633 (C=O stretch), 1545, 1453, 1222, 964, 763, 746, 727, 693 cm^{-1}
HRMS (EI)	$C_{16}H_{17}NOS$ (M): 271.1031, found 271.1040 *m/z*

^{1}H NMR

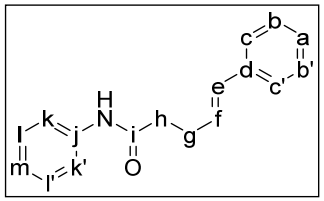

7.271
7.172
7.160
7.069
7.064
6.484
6.445
6.110
6.092
6.075
6.071
6.053
6.036
5.939
4.463
4.449
2.591
2.573
2.556
2.538
2.398
2.380
2.361
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.92
0.95
1.06
0.96
1.01
0.92
2.00
2.04
1.98

^{13}C NMR


172.11
140.03
138.40
128.81
128.65
127.85
127.60
126.01
125.60
125.03
121.23
43.70
36.45
29.01
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

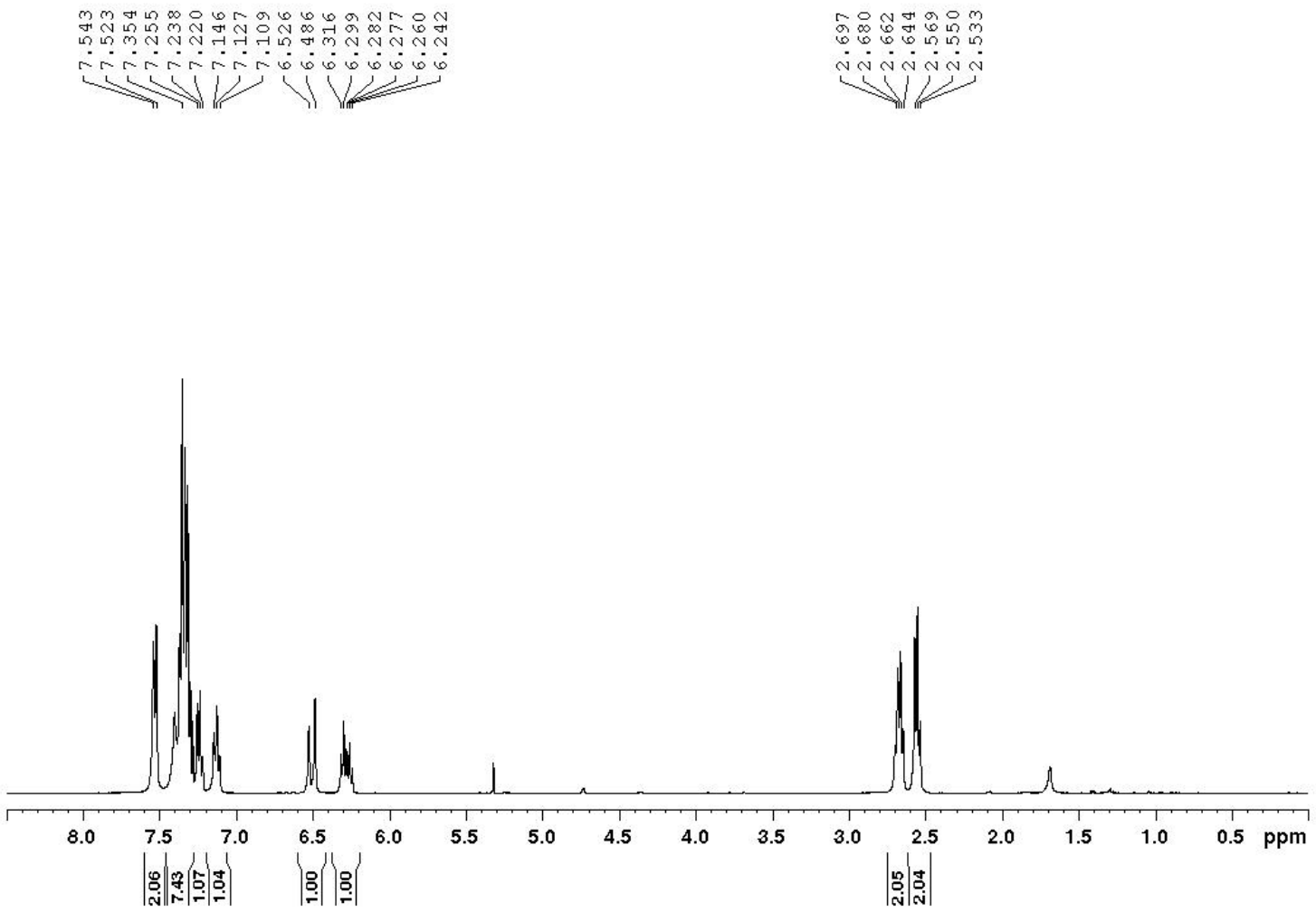


5a

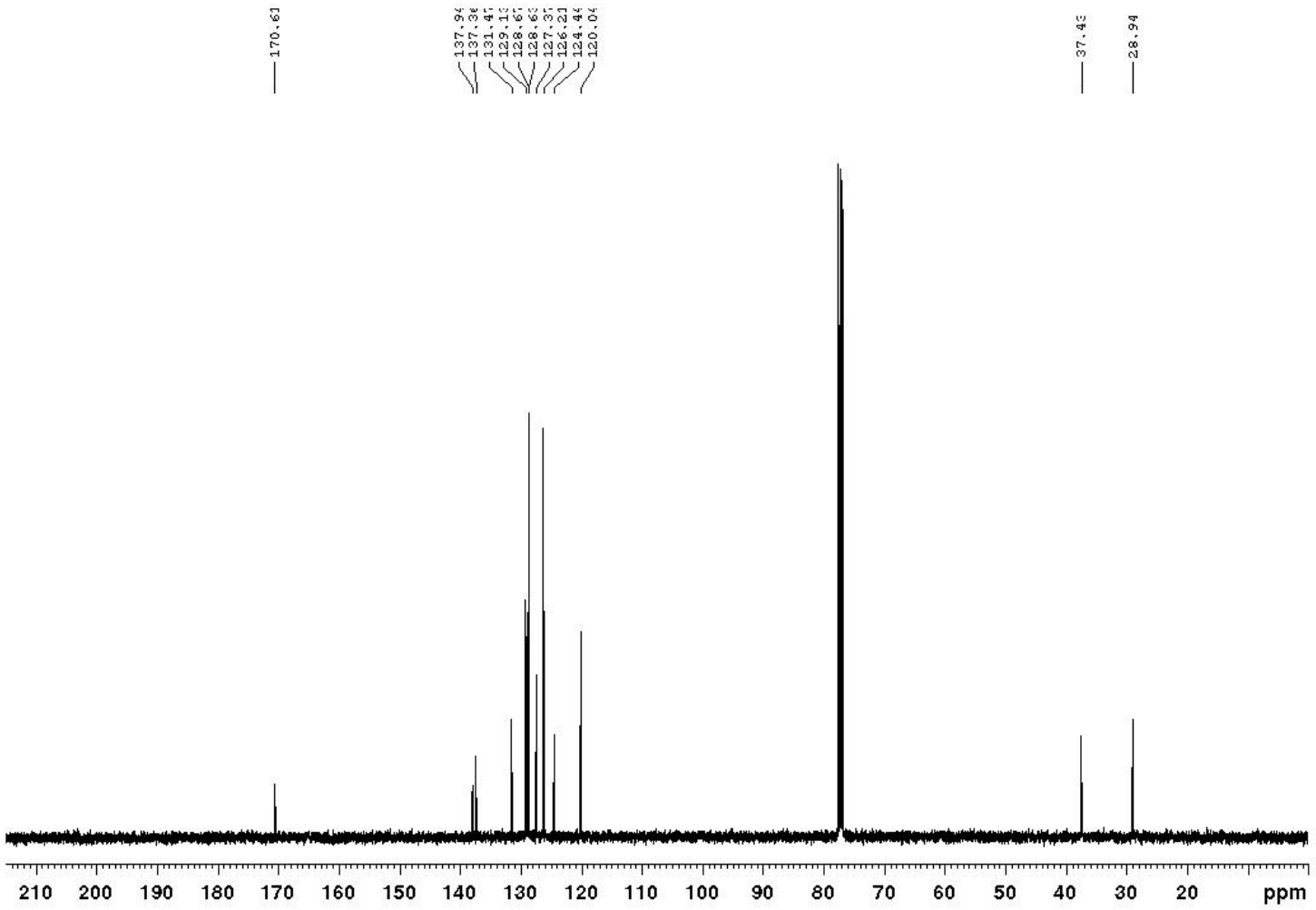


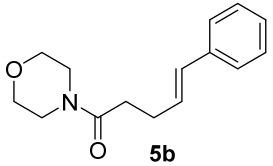
m.p.	112.5–114.0 ºC
TLC analysis	R_f 0.65 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.53 (2H, d, *J* = 7.8 Hz, k,k'), 7.30–40 (7H, m, b,b',c,c',l,l',NH), 7.24 (1H, t, *J* = 7.0 Hz, a), 7.13 (1H, t, *J* = 7.3 Hz, m), 6.51 (1H, d, *J* = 15.8 Hz, e), 6.25 (1H, dt, *J* = 15.8 and 6.7 Hz, f), 2.67 (2H, q, *J* = 7.1 Hz, g), 2.55 (2H, t, *J* = 7.4 Hz, h)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 170.61 (i), 137.94 (j), 137.36 (d), 131.47 (e), 129.13 (l,l'), 128.67 (b,b'), 128.63 (m), 127.37 (f), 126.21 (c,c'), 124.44 (a), 120.04 (k,k'), 37.43 (h), 28.94 (g)
IR (neat)	3279 (N-H stretch), 3026, 1650 (C=O stretch), 1598, 1526, 1441, 964, 738, 690 cm^{-1}
HRMS (EI)	$C_{17}H_7NO$ (M): 251.1310, found 251.1313 *m/z*

^{1}H NMR

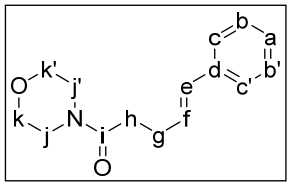

7.543
7.523
7.354
7.255
7.238
7.220
7.146
7.127
7.109
6.526
6.486
6.316
6.299
6.282
6.277
6.260
6.242
2.697
2.680
2.662
2.644
2.569
2.550
2.533
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.06
7.43
1.07
1.04
1.00
1.00
2.05
2.04

^{13}C NMR


170.61
137.94
137.38
131.45
129.13
128.67
128.63
127.35
126.21
124.44
120.04
37.45
28.94
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

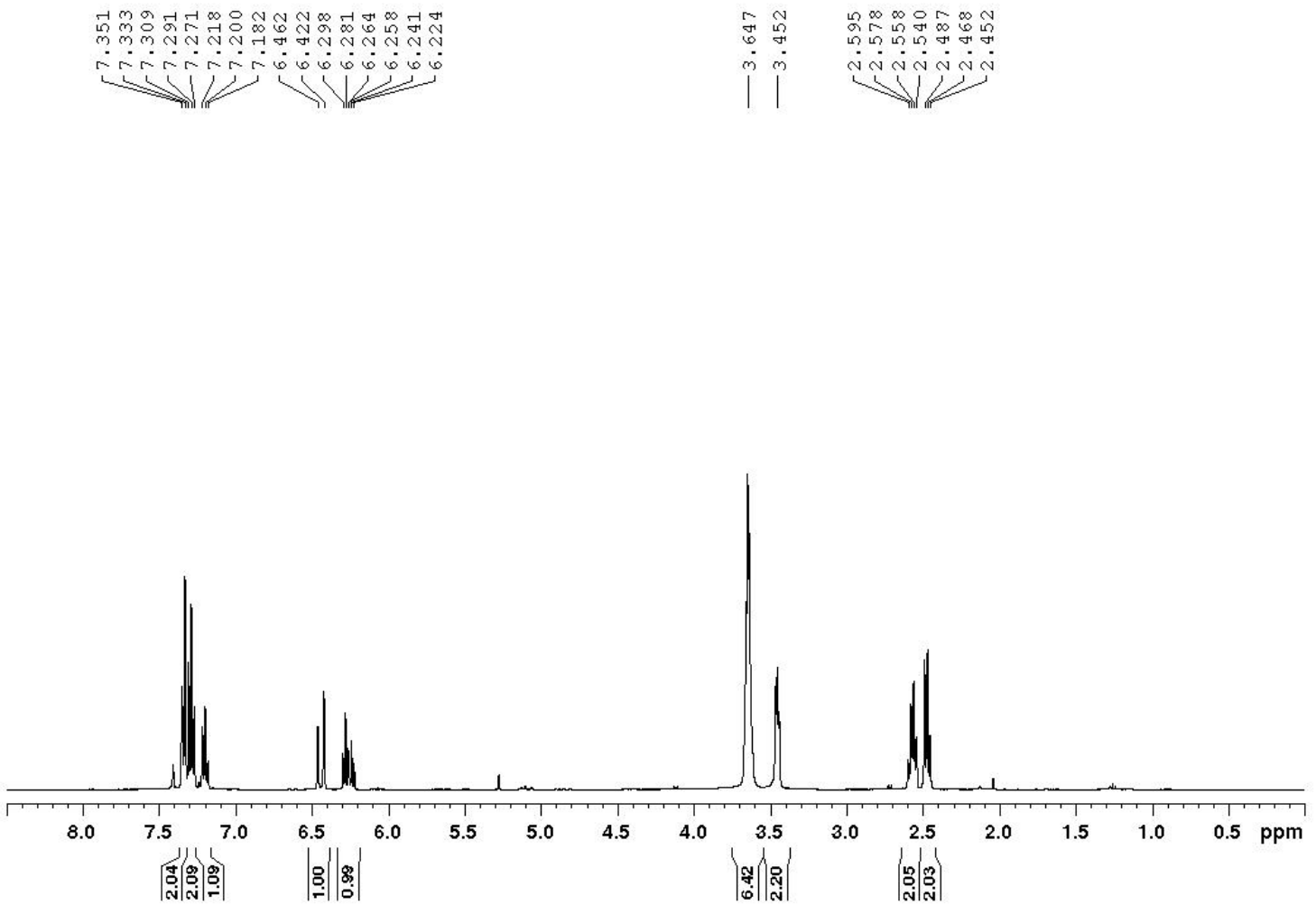


5b

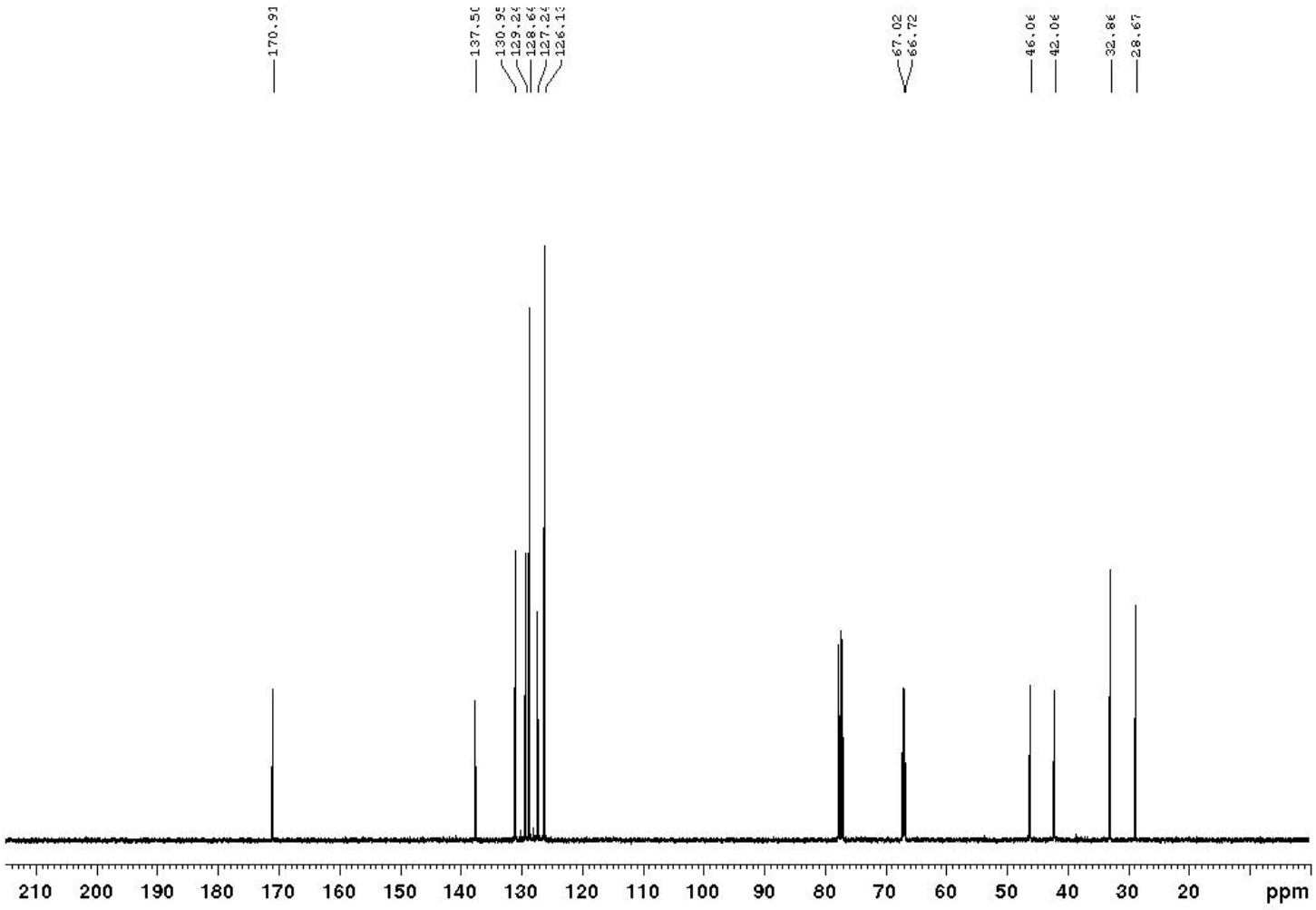


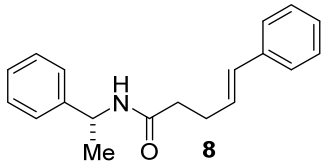
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.34 (2H, d, *J* = 7..2 Hz, c,c'), 7.29 (2H, t, *J* = 7.3 Hz, b,b'), 7.20 (1H, t, *J* = 7.1 Hz, a), 6.44 (1H, d, *J* = 15.9 Hz, e), 6.26 (1H, dt, *J* = 15.9 and 6.8 Hz, f), 3.60–3.70 (6H, m, j,j',k,k'), 3.45–3.50 (2H, m, j,j'), 2.57 (2H, q, *J* = 6.8 Hz, h), 2.47 (2H, t, *J* = 7.5 Hz, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 170.91 (i), 137.50 (d), 130.94 (e), 129.24 (f), 128.64 (b,b'), 127.24 (a), 126.13 (c,c'), 67.02 and 66.72 (k,k'), 46.06 and 42.06 (j,j'), 32.86 (g), 28.67 (h)
IR (neat)	2962, 2897, 2853, 1639 (C=O stretch), 1429, 1220, 1112, 1024, 964, 746, 693 cm^{-1}
HRMS (ESI)	$C_{15}H_{19}NO_2$ (M): 245.1416, found 245.1413 *m/z*

^{1}H NMR

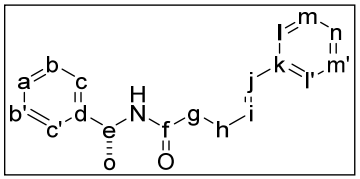

7.351
7.333
7.309
7.291
7.271
7.218
7.200
7.182
6.462
6.422
6.298
6.281
6.264
6.258
6.241
6.224
3.647
3.452
2.595
2.578
2.558
2.540
2.487
2.468
2.452
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.04
2.09
1.09
1.00
0.99
6.42
2.20
2.05
2.03

^{13}C NMR


170.93
137.50
130.95
129.24
128.66
127.24
126.13
67.02
66.72
46.06
42.06
32.86
28.67
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

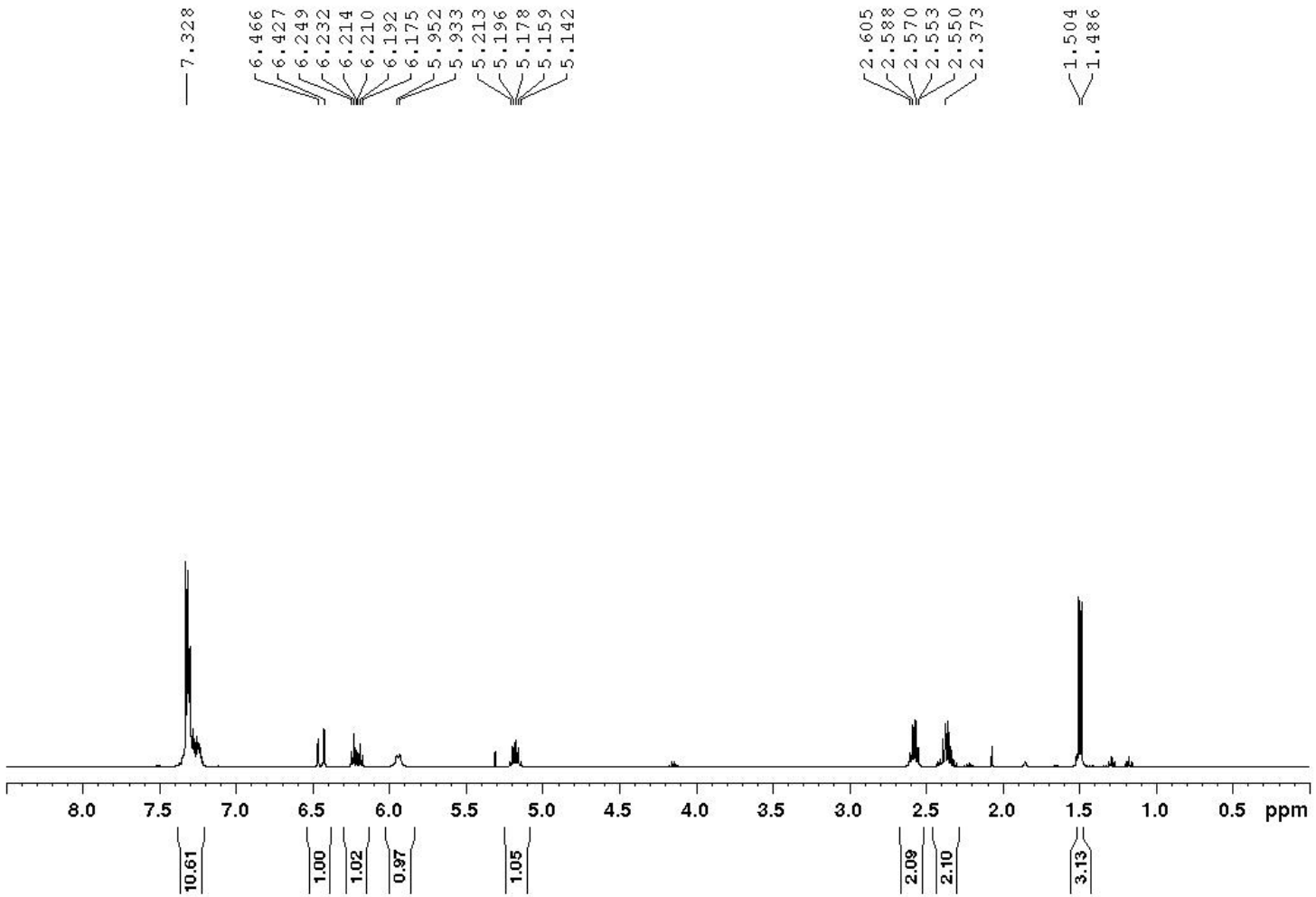


8

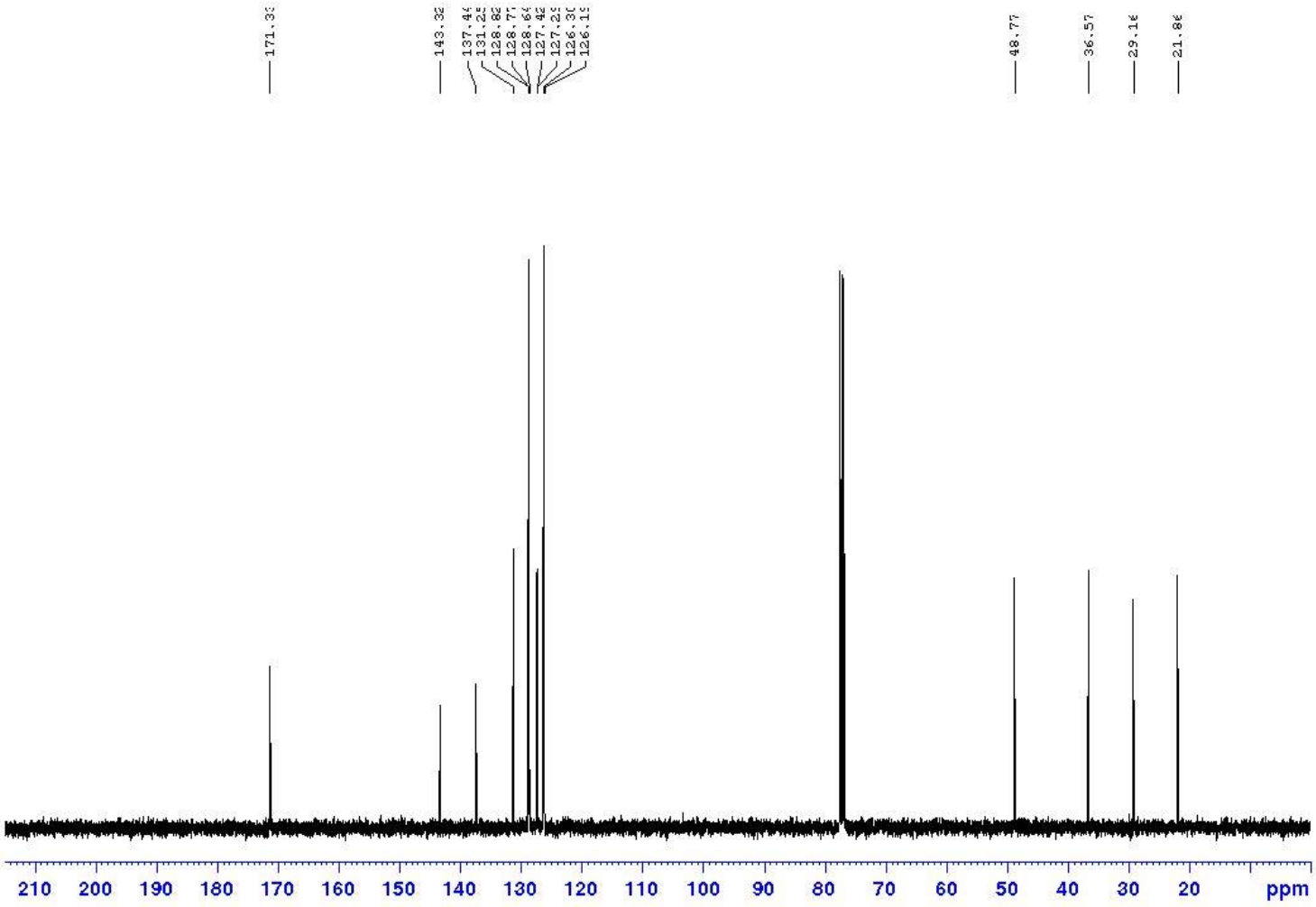


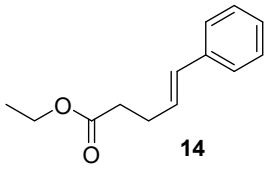
$[\alpha]_D^{20}$	+44.1 (*c* 2.0, $CHCl_3$)
m.p.	77.5–79.0 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (10H, m, a,b,b',c,c',l,l',m,m',n), 6.45 (1H, d, *J* = 15.8 Hz, j), 6.21 (1H, dt, *J* = 15.8 and 6.9 Hz, i), 5.94 (1H, d, *J* = 7.4 Hz, NH), 5.15–5.20 (1H, m, e), 2.58 (2H, q, *J* = 6.9 Hz, h), 2.30–2.40 (2H, m, g), 1.50 (3H, d, *J* = 6.9 Hz, o)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.33 (f), 143.32 (k), 137.44 (d), 131.25 (j), 128.82 (i), 128.77 (b,b'), 128. 128.64 (l,l'), 127.42 (n), 127.29 (a), 126.30 (m,m'), 126.19 (c,c'), 43.77 (e), 36.57 (g), 29.16 (h), 21.86 (o)
IR (neat)	3304 (N-H stretch), 3059, 3025, 2981, 2359, 2341, 1638 (C=O stretch), 1534, 1493, 1444, 1374, 1217, 959, 743, 694 cm^{-1}
HRMS (ESI)	$C_{19}H_{21}NNaO$ (M+Na): 302.1521, found 302.1525 *m/z*

^{1}H NMR

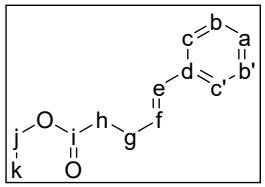

7.328
6.466
6.427
6.249
6.232
6.214
6.210
6.192
6.175
5.952
5.933
5.213
5.196
5.178
5.159
5.142
2.605
2.588
2.570
2.553
2.550
2.373
1.504
1.486
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.61
1.00
1.02
0.97
1.05
2.09
2.10
3.13

^{13}C NMR


171.3
143.3
137.4
131.2
128.8
128.7
128.6
127.4
127.2
126.3
126.1
48.7
36.5
29.1
21.8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

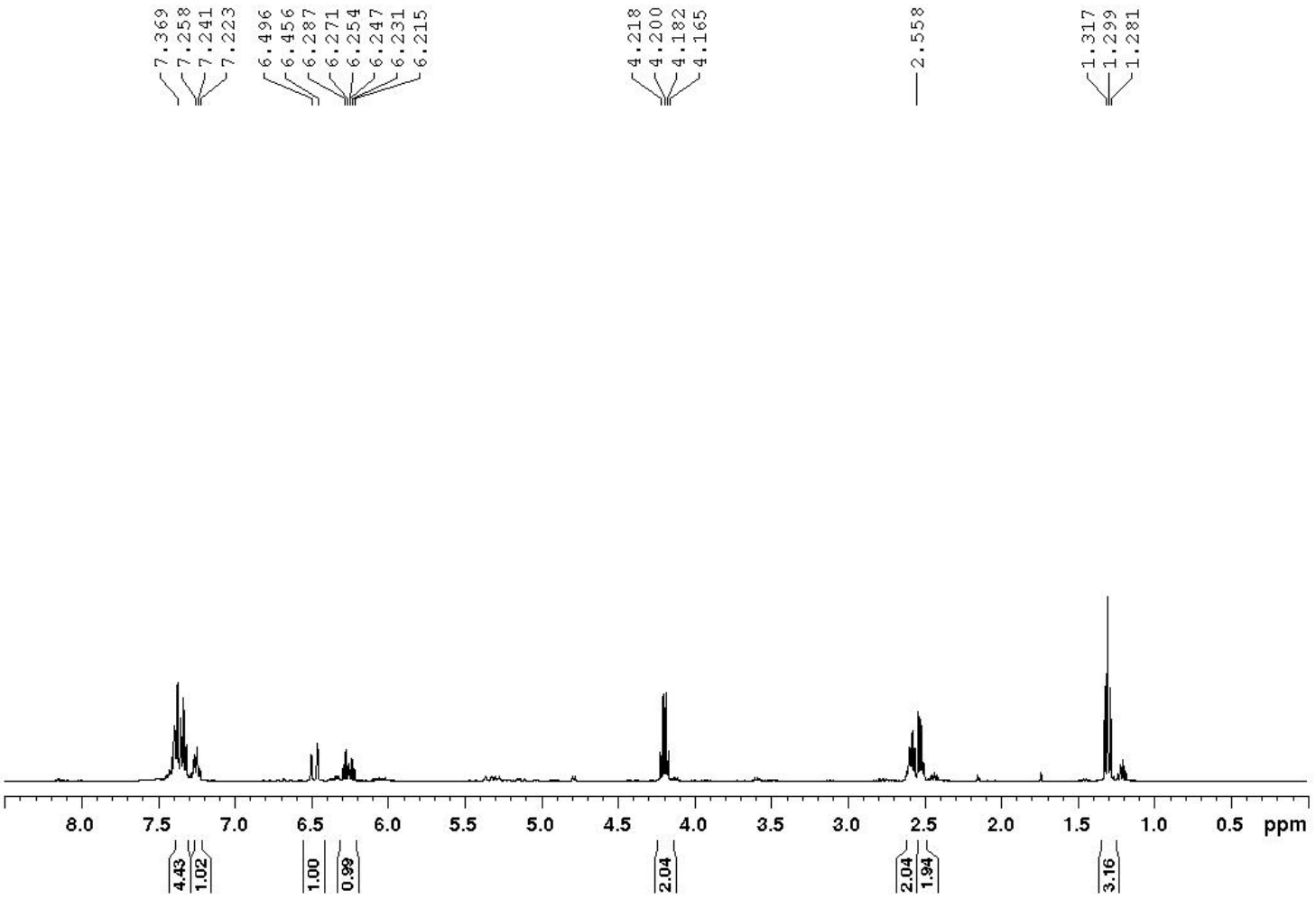


14

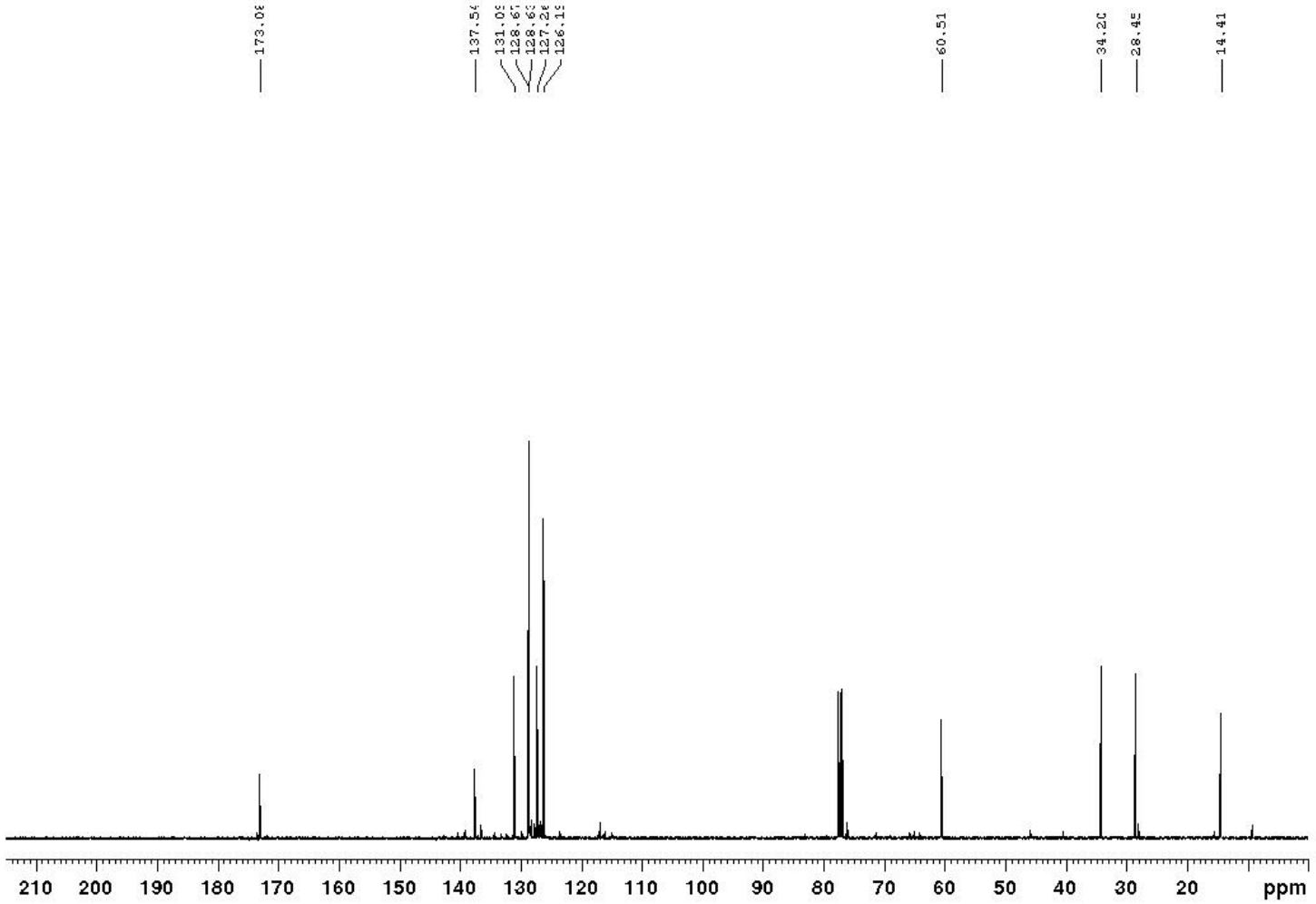


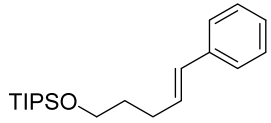
TLC analysis	R_f 0.7 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.30–7.40 (4H, m, b,b',c,c'), 7.24 (1H, t, *J* = 7.0 Hz, a), 6.48 (1H, d, *J* = 15.8 Hz, e), 6.25 (1H, dt, *J* = 15.8 and 6.3 Hz, f), 4.19 (2H, q, *J* = 7.1 Hz, j), 2.50–2.60 (4H, m, g,h), 1.30 (3H, t, *J* = 7.1 Hz, k)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.08 (i), 137.54 (d), 131.09 (f), 128.63 (b,b',e), 127.26 (a), 126.19 (c,c'), 60.51 (j), 34.20 (h), 28.45 (g), 14.41 (k)

^{1}H NMR

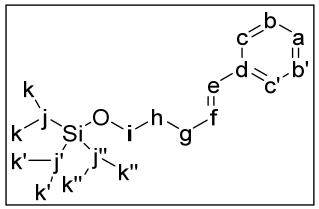

7.369
7.258
7.241
7.223
6.496
6.456
6.287
6.271
6.254
6.247
6.231
6.215
4.218
4.200
4.182
4.165
2.558
1.317
1.299
1.281
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.43
1.02
1.00
0.99
2.04
2.04
1.94
3.16

^{13}C NMR


173.0
137.5
131.0
128.6
128.6
127.2
126.1
60.51
34.2
28.4
14.41
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

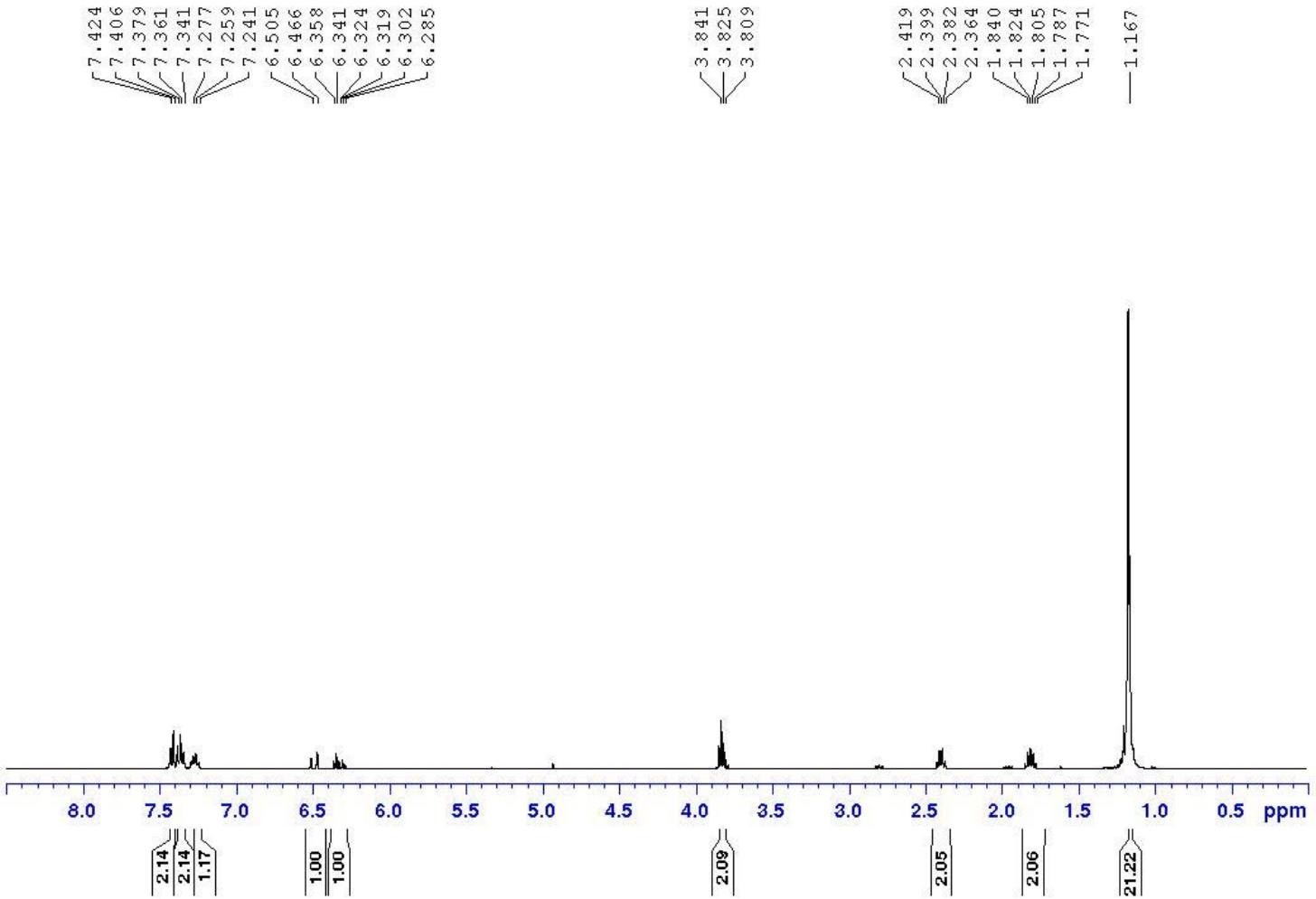


16

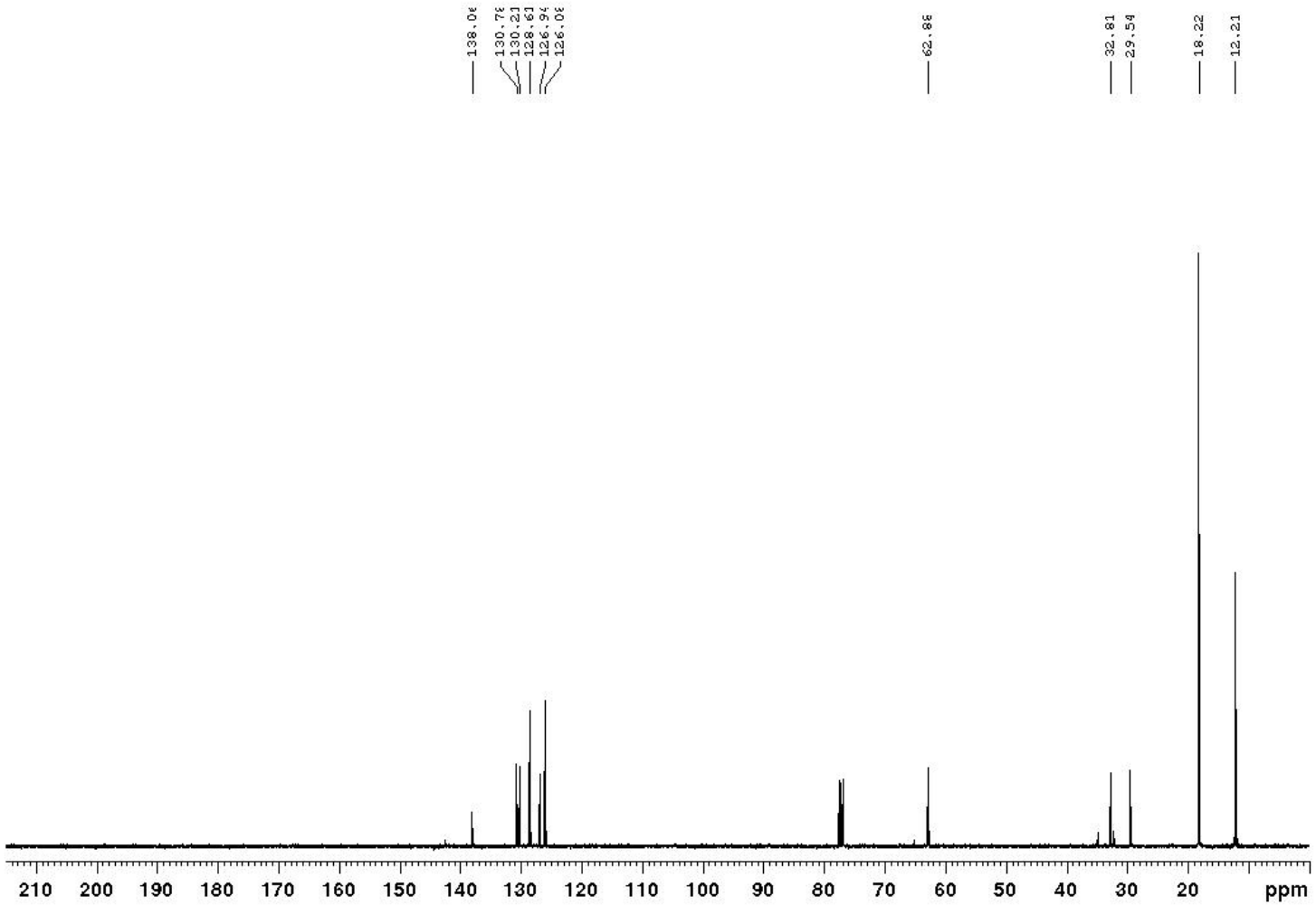


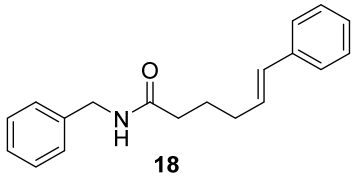
TLC analysis	R_f 0.7 (90:10 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.41 (2H, d, *J* = 7.2 Hz, c,c'), 7.36 (2H, t, *J* = 7.4 Hz, b,b'), 7.26 (1H, t, *J* = 7.0 Hz, a), 6.49 (1H, d, *J* = 15.8 Hz, e), 6.32 (1H, dt, *J* = 15.8 and 6.8 Hz, f), 3.83 (2H, d, *J* = 6.4 Hz, i), 2.35–2.45 (2H, m, g), 1.75–1.85 (2H, m, h), 1.15–1.20 (21H, m, j,j',j'',k,k',k'')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 138.06 (d), 130.78 (f), 130.21 (e), 128.61 (b,b'), 126.94 (a), 126.08 (c,c'), 62.88 (i), 32.81 (h), 29.54 (g), 18.22 (k,k',k''), 12.21 (j,j',j'')
IR (neat)	2141, 2891, 2864, 1462, 1102, 962, 881, 679, 657 cm^{-1}
HRMS (EI)	$C_{17}H_{27}OSi$ (M–C_3H_7): 275.1831, found 275.1843 *m/z*

^{1}H NMR

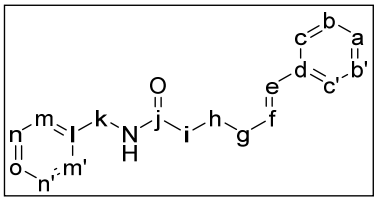

7.424
7.406
7.379
7.361
7.341
7.277
7.259
7.241
6.505
6.466
6.358
6.341
6.324
6.319
6.302
6.285
3.841
3.825
3.809
2.419
2.399
2.382
2.364
1.840
1.824
1.805
1.787
1.771
1.167
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.14
2.14
1.17
1.00
1.00
2.09
2.05
2.06
21.22

^{13}C NMR


138.0
130.7
130.2
128.6
126.9
126.0
62.8
32.8
29.5
18.2
12.2
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

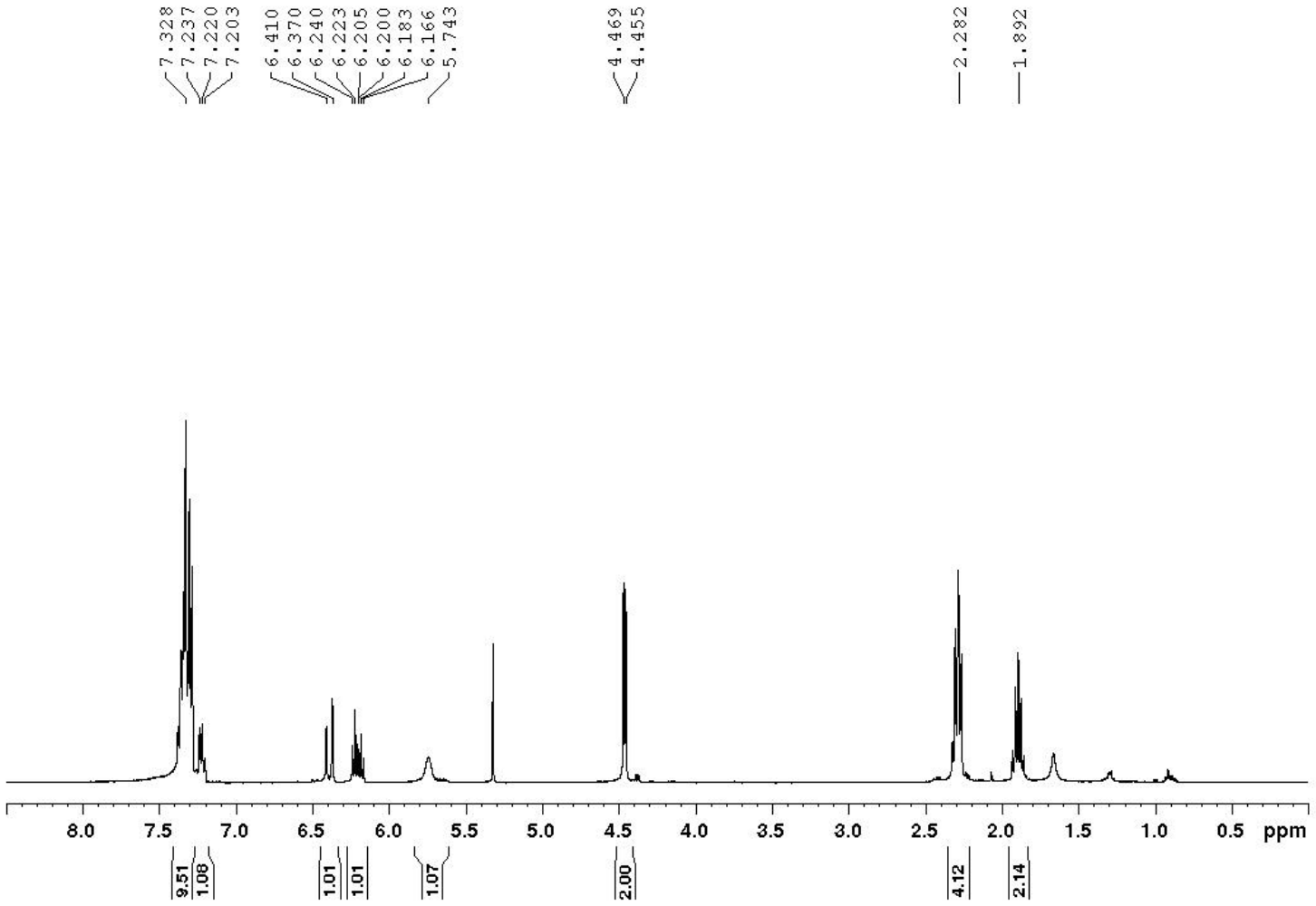


18

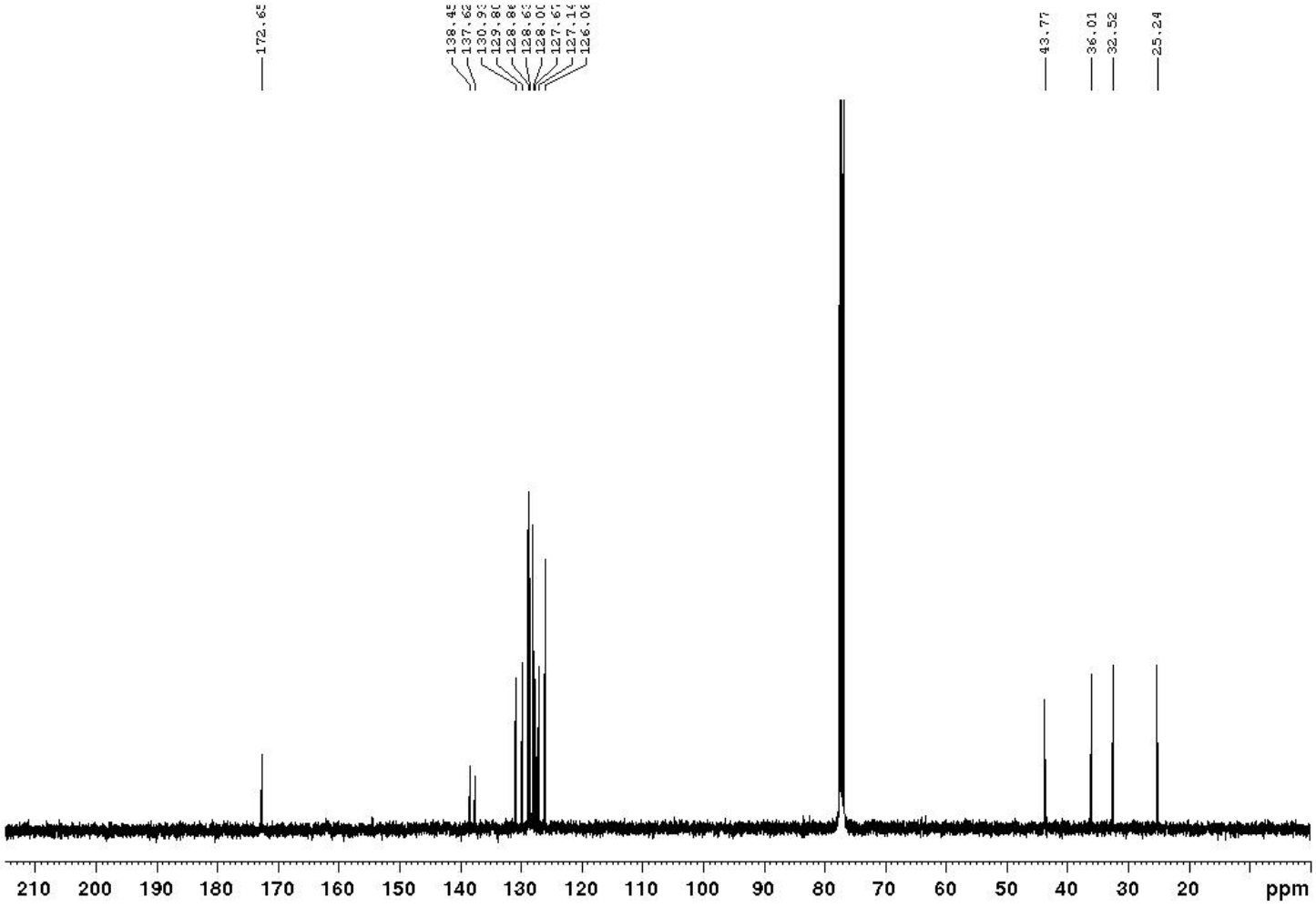


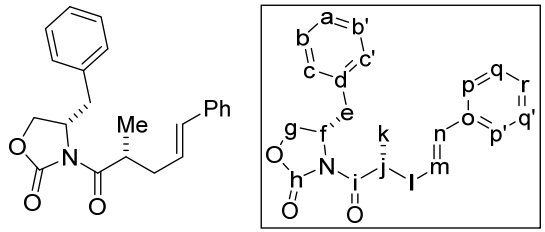
m.p.	103.5–105.0 ºC
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate).
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.40 (9H, m, a,b,b',c,c',m,m',n,n'), 7.22 (1H, t, *J* = 6.7 Hz, o), 6.39 (1H, d, *J* = 15.8 Hz, e), 6.20 (1H, dt, *J* = 15.8 and 6.9 Hz, f), 5.74 (1H, br s, NH), 4.46 (2H, d, *J* = 5.7 Hz, k), 2.25–2.35 (4H, m, g, i), 1.85–1.95 (2H, m, h)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.65 (j), 138.45 (l), 137.62 (d), 130.93 (e), 129.80 (f), 128.86 (n,n'), 128.63 (b,b'), 128.00 (c,c'), 128.67 (o), 127.14 (a), 126.08 (m,m'), 43.77 (k), 36.01 (i), 32.52 (g), 25.24 (h)
IR (neat)	3278 (N-H stretch), 1630 (C=O stretch), 1552, 1546, 1451, 965, 728, 689 cm^{-1}
HRMS (EI)	$C_{19}H_{21}NO$ (M): 279.1623, found 279.1632 *m/z*

^{1}H NMR


7.328
7.237
7.220
7.203
6.410
6.370
6.240
6.223
6.205
6.200
6.183
6.166
5.743
4.469
4.455
2.282
1.892
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
9.51
1.08
1.01
1.01
1.07
2.00
4.12
2.14

^{13}C NMR

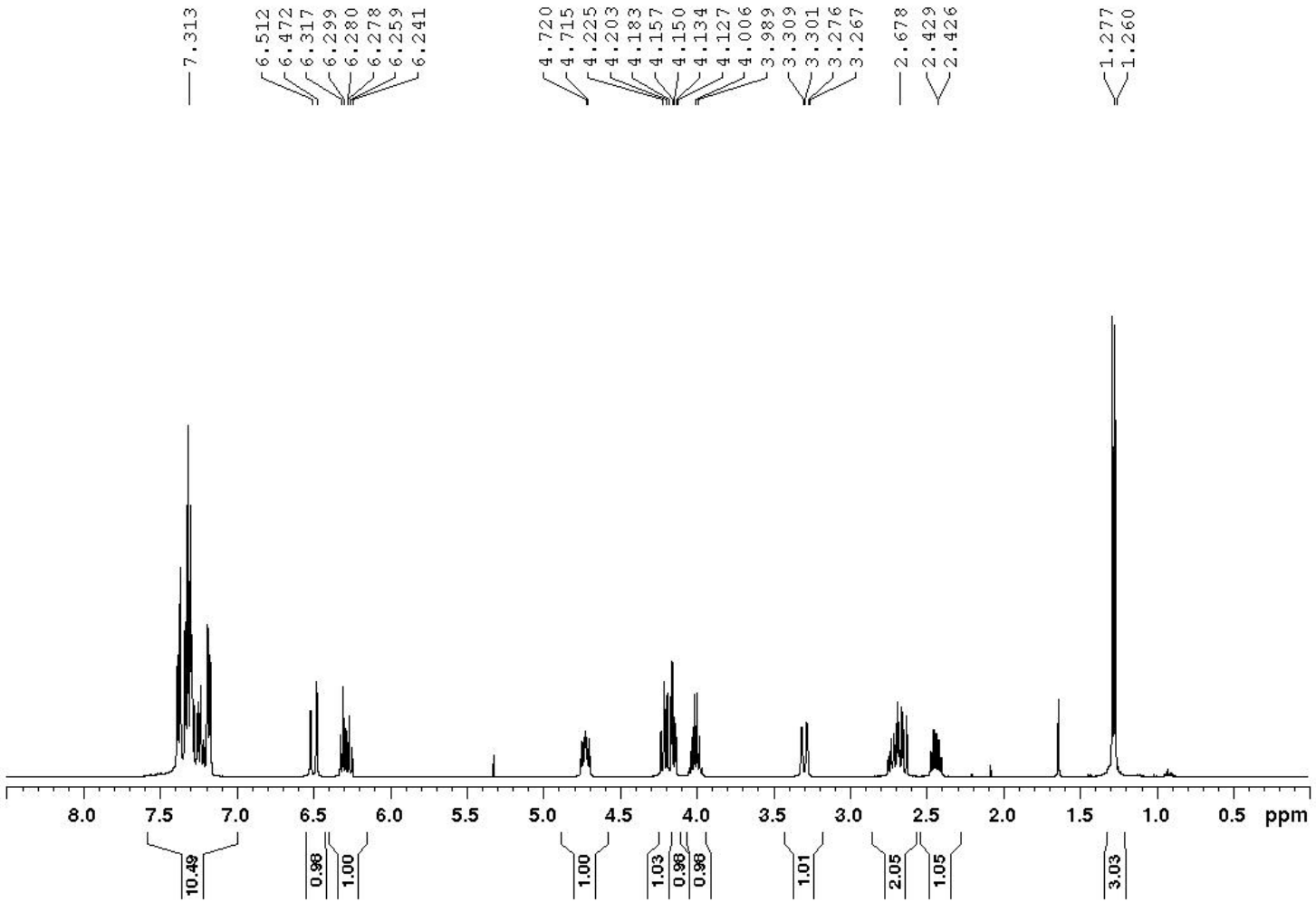

172.65
138.45
137.62
130.93
129.80
128.86
128.65
128.01
127.61
127.14
126.06
43.77
36.01
32.52
25.24
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



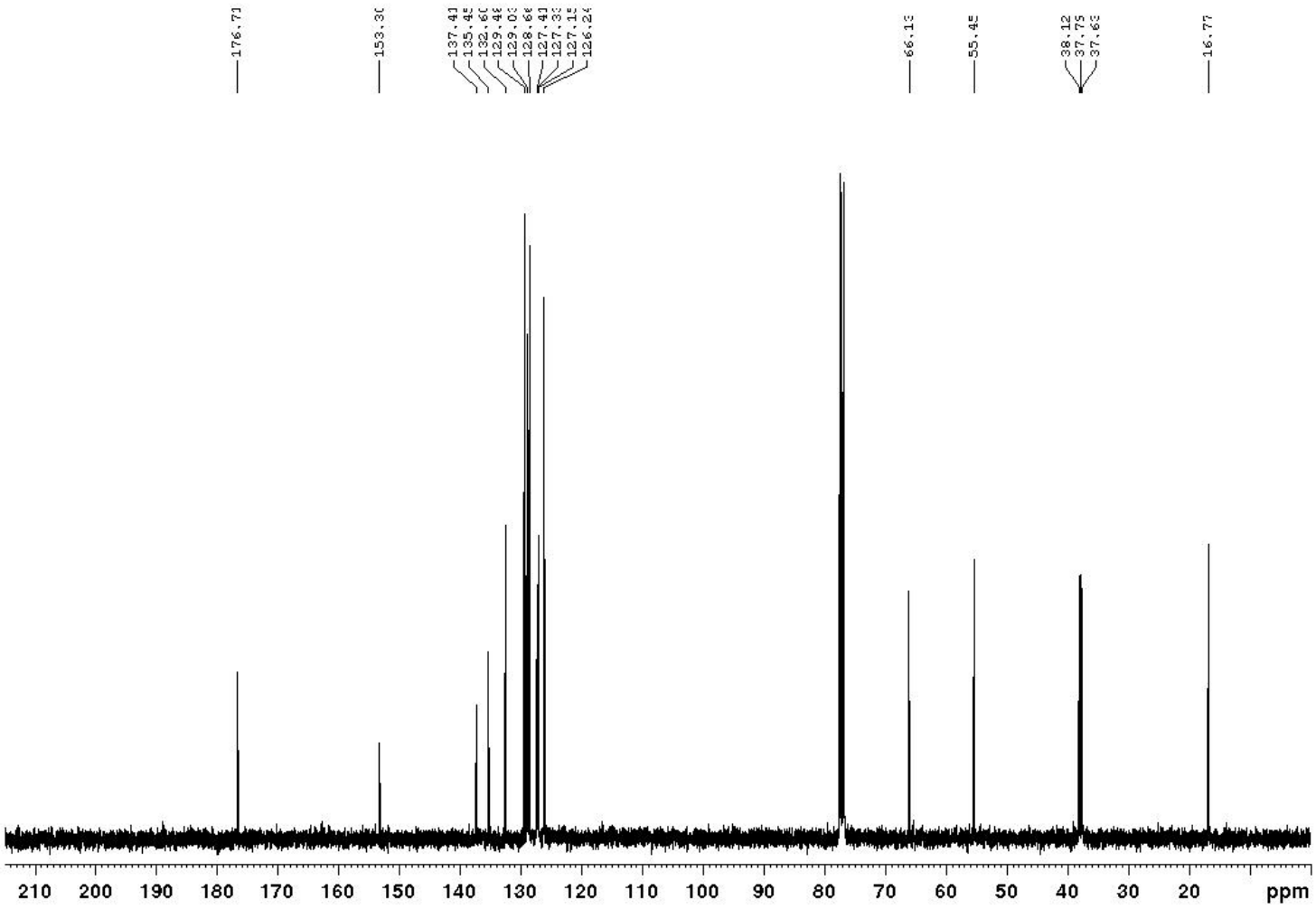
S5

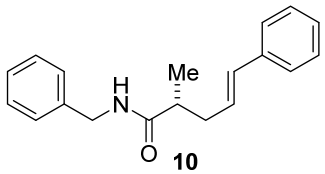
$[\alpha]_D^{20}$	+14.6º (*c* 1.0, $CHCl_3$)
m.p.	98.5–99.5 ºC
TLC analysis	R_f 0.4 (70:30 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.10–7.50 (10H, m, aryl), 6.49 (1H, d, *J* = 15.8 Hz, n), 6.28 (1H, dd, *J* = 15.5 and 7.3 Hz, m), 4.65–4.75 (1H, m, f), 4.20 (1H, dd, *J* = 8.7 and 8.7 Hz, g), 4.14 (1H, dd, *J* = 9.1 and 3.0 Hz, g), 3.95–4.05 (1H, m, e), 3.29 (1H, dd, *J* = 13.4 and 3.3 Hz, e), 2.60–2.75 (2H, m, j,l), 2.35–2.50 (1H, m, l), 1.27 (3H, d, *J* = 6.8 Hz, k)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 176.71 (j), 153.30 (h), 137.41 (d), 135.45 (o), 132.60 (n), 129.48 (aryl), 129.03 (aryl), 128.66 (aryl), 127.41 (aryl), 127.33 (aryl), 127.15 (m), 126.24 (aryl), 66.13 (g), 55.45 (f), 38.12 (e), 37.79 (j), 37.63 (l), 16.77 (k)
IR (neat)	3025, 2971, 1780 (C=O stretch), 1692 (C=O stretch), 1384, 1348, 1247, 1215, 1097, 967, 744, 697 cm^{-1}
HRMS (ESI)	$C_{22}H_{23}NNaO_3$ (M+Na): 372.1576, found 372.1582 *m/z*

^{1}H NMR

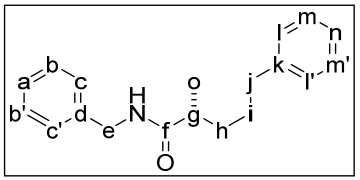

7.313
6.512
6.472
6.317
6.299
6.280
6.278
6.259
6.241
4.720
4.715
4.225
4.203
4.183
4.157
4.150
4.134
4.127
4.006
3.989
3.309
3.301
3.276
3.267
2.678
2.429
2.426
1.277
1.260
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.49
0.98
1.00
1.00
1.03
0.96
0.96
1.01
2.05
1.05
3.03

^{13}C NMR


176.71
153.30
137.41
135.45
132.60
129.48
129.03
128.68
127.41
127.33
127.15
126.24
66.13
55.45
38.12
37.75
37.63
16.77
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

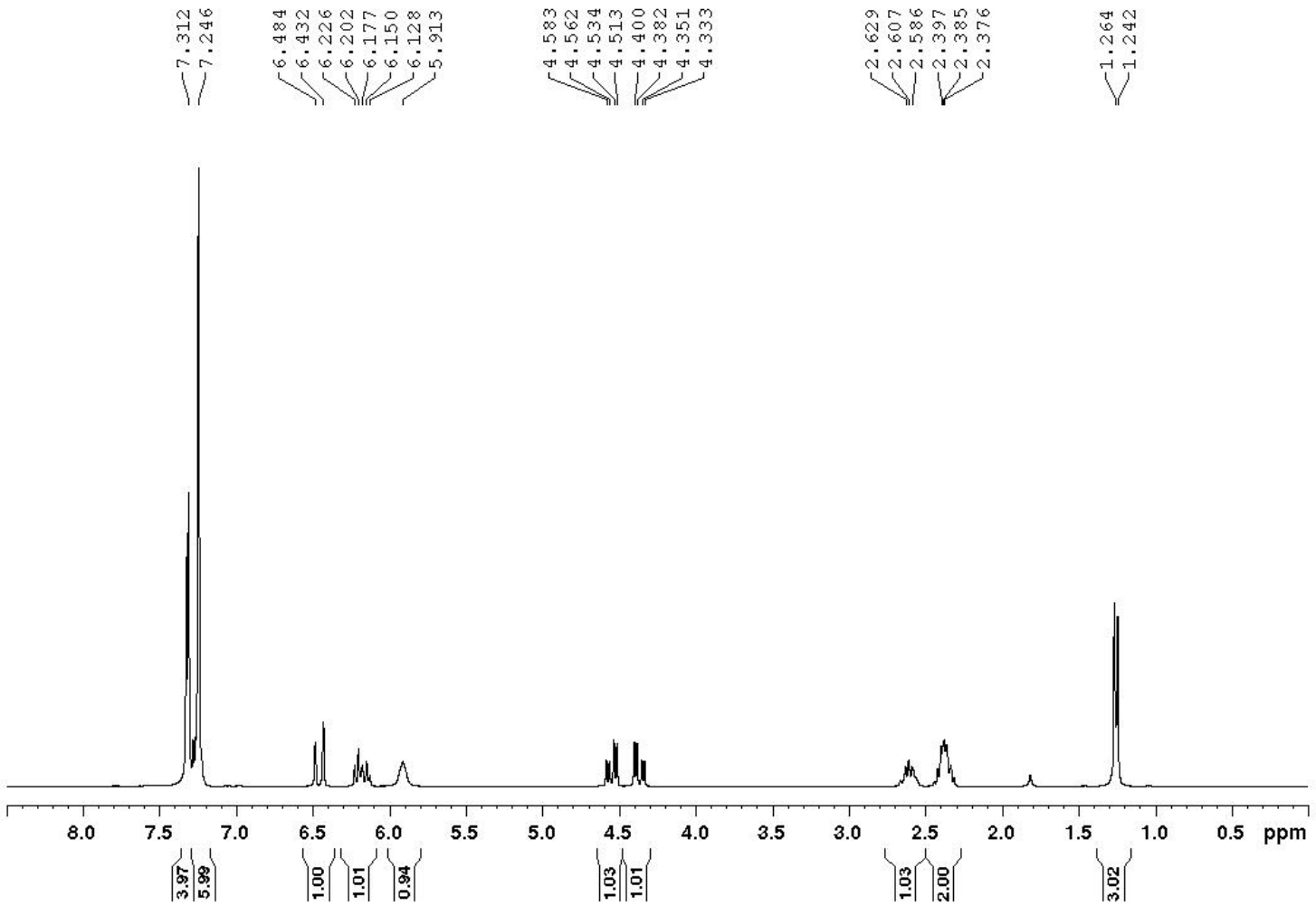


10

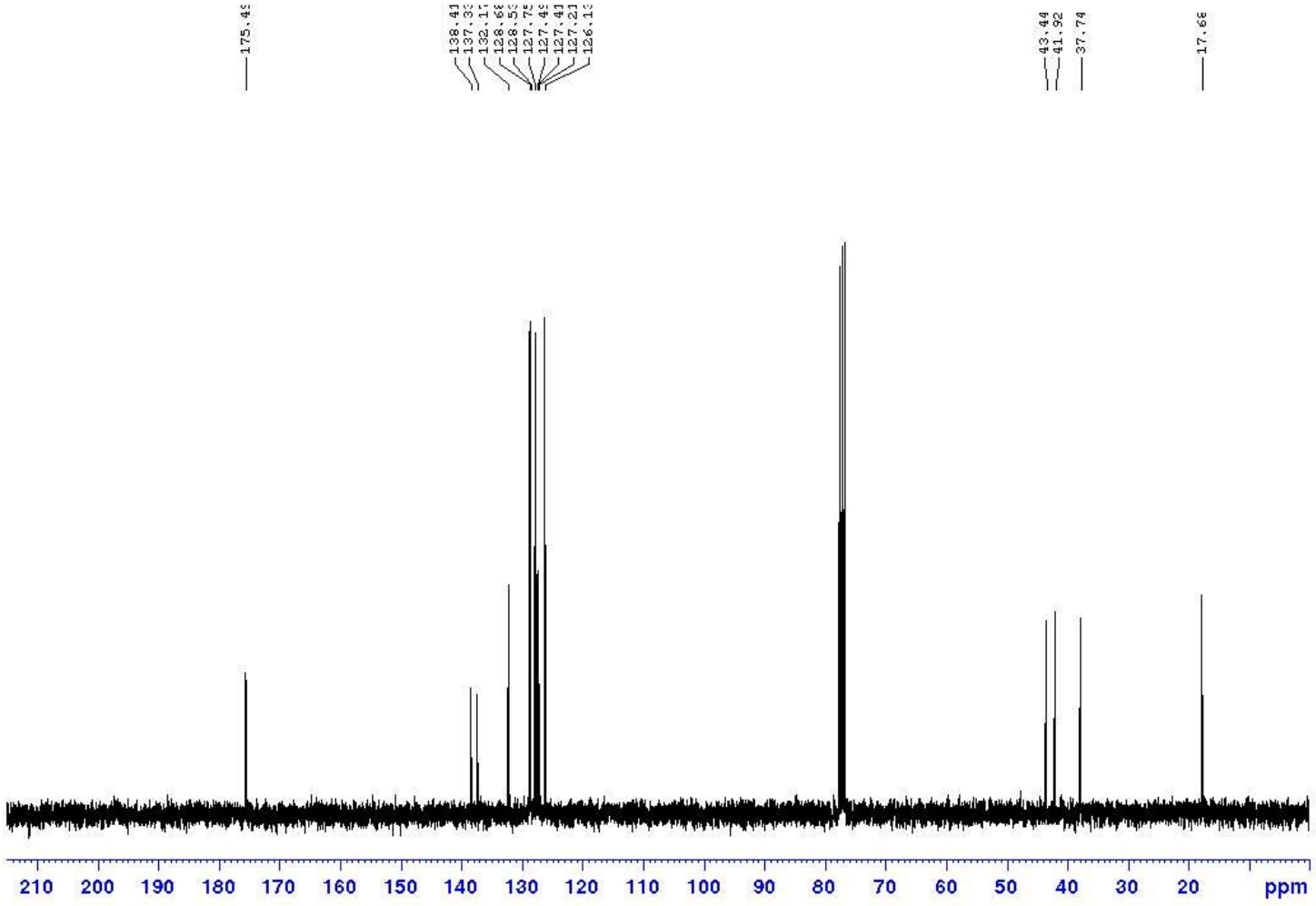


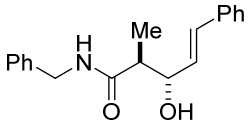
$[\alpha]_D^{20}$	+4.7° (*c* 2.0, $CHCl_3$)
m.p.	92.5–93.5 °C
TLC analysis	R_f 0.4 (60:40 hexanes:ethyl acetate)
^{1}H NMR (300 MHz, $CDCl_3$)	δ 7.20–7.35 (10H, m, a,b,b',c,c',l,l',m,m',n), 6.44 (1H, d, *J* = 15.8 Hz, j), 6.21 (1H, dt, *J* = 15.8 and 7.3 Hz, i), 5.91 (1H, br s, NH), 4.55 (1H, dd, *J* = 14.7 and 8.2 Hz, e), 4.37(1H, dd, *J* = 14.7 and 8.2 Hz, e) 2.50–2.70 (1H, m, h), 2.30–2.50 (2H, m, g,h), 1.25 (3H, d, *J* = 6.4 Hz, o)
^{13}C NMR (75 MHz, $CDCl_3$)	δ 175.49 (f), 138.41 (d), 137.33 (k), 132.17 (j), 128.68 (b,b'), 128.53 (l,l'), 127.75 (m,m'), 127.49 (i), 127.41 (a) 127.21 (n), 126.13 (c,c'), 43.44 (e), 41.92 (g), 37.74 (h), 17.68 (o)
IR (neat)	3303 (N-H stretch), 2966, 2360, 1633 (C=O stretch), 1603, 1539, 1495, 1453, 962, 739, 724, 691 cm^{-1}
HRMS (ESI)	$C_{19}H_{21}NNaO$ (M+Na): 302.1521, found 302.1526 *m/z*

^{1}H NMR

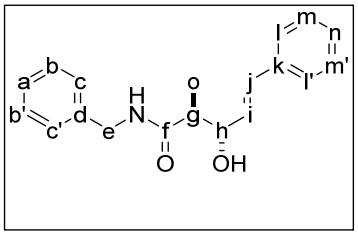

7.312
7.246
6.484
6.432
6.226
6.202
6.177
6.150
6.128
5.913
4.583
4.562
4.534
4.513
4.400
4.382
4.351
4.333
2.629
2.607
2.586
2.397
2.385
2.376
1.264
1.242
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
3.97
5.99
1.00
1.01
0.94
1.03
1.01
1.03
2.00
3.02

^{13}C NMR


43.44
41.92
37.74
17.68
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

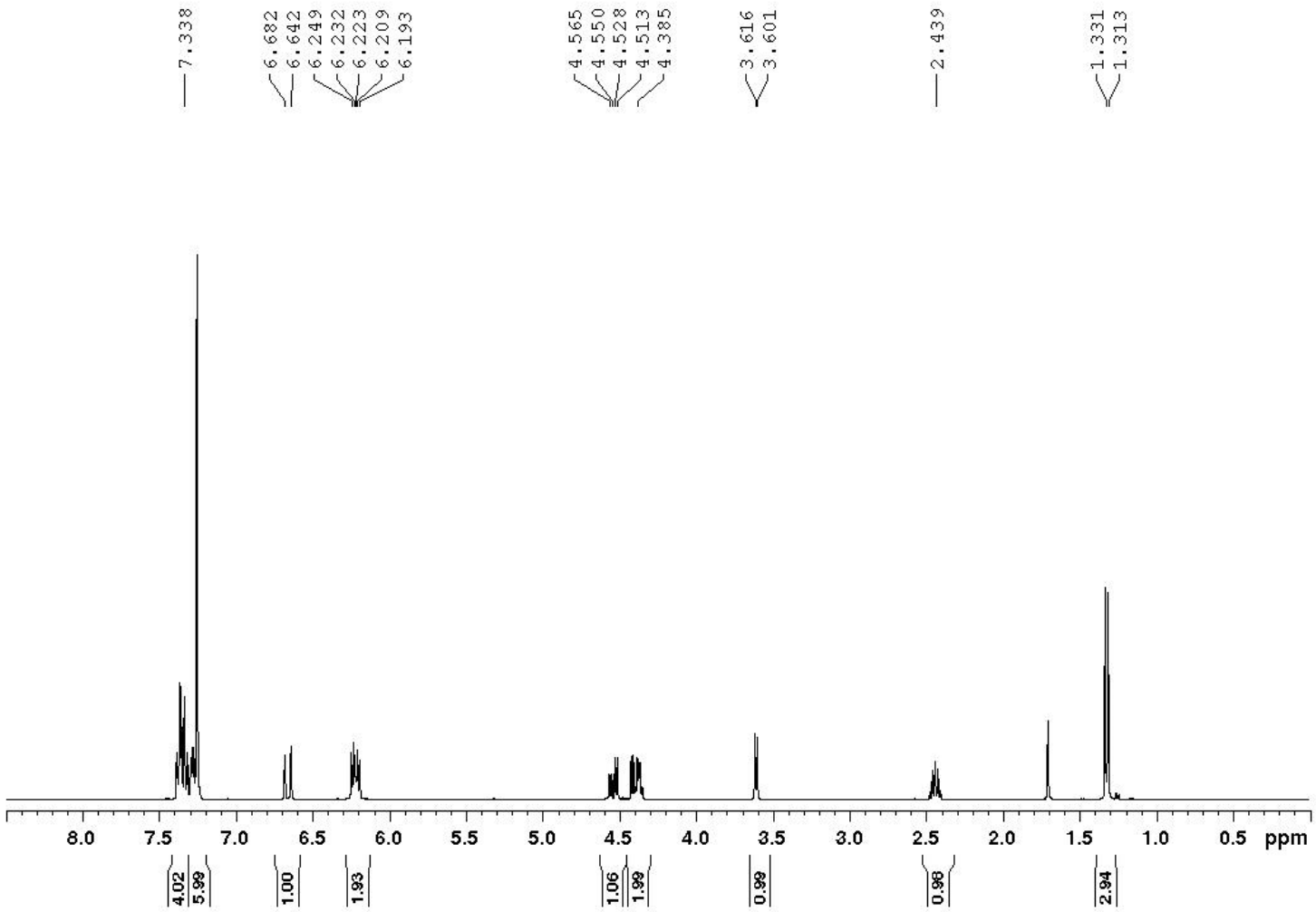


S8

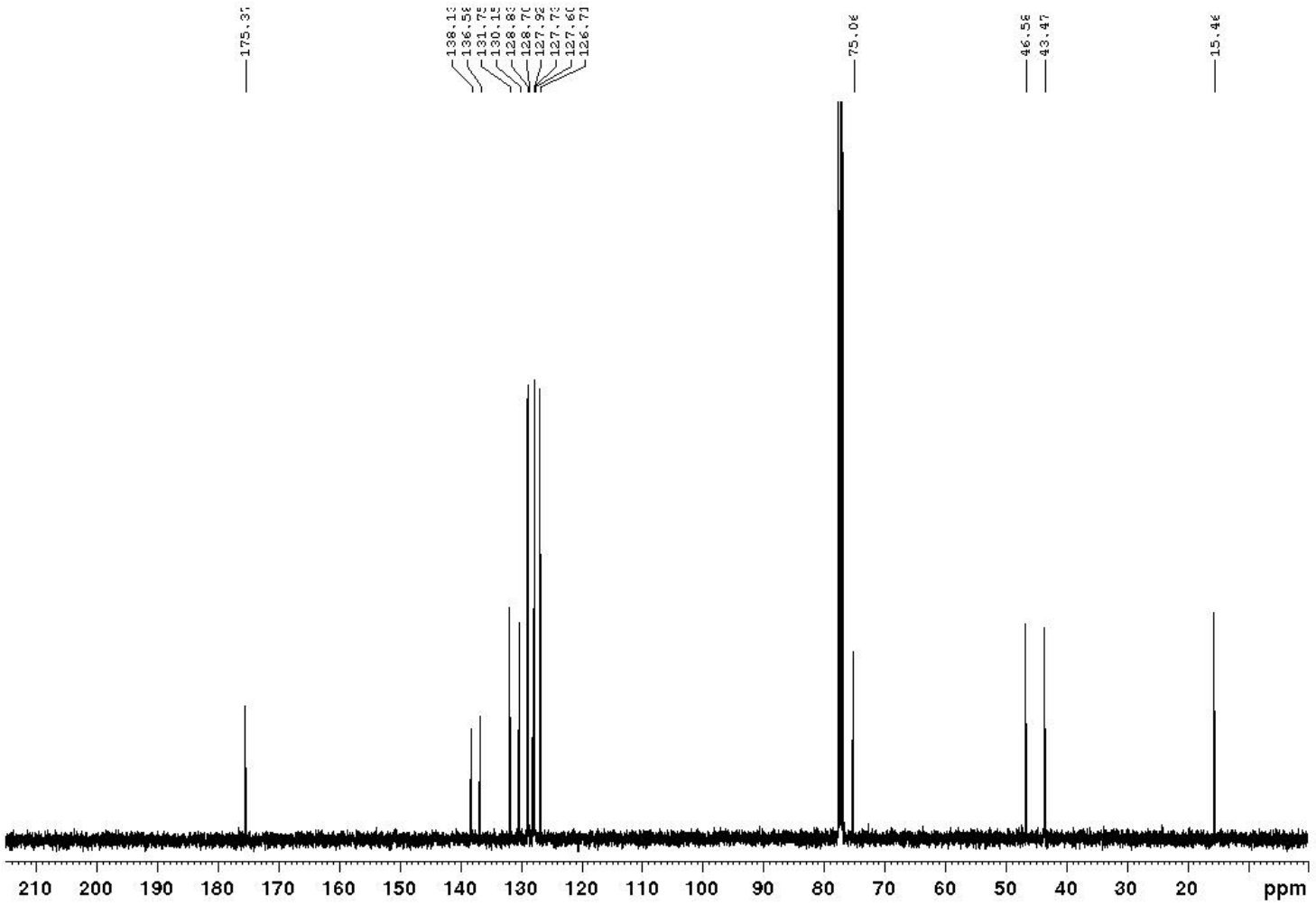


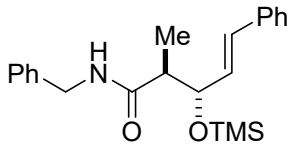
$[\alpha]_D^{20}$	$[\alpha]_D^{20}$ = +10.5° (*c* 1.0, $CHCl_3$)
m.p.	137.5–139.0 °C
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (10H, m, aryl), 6.66 (1H, d, *J* = 15.8 Hz, j), 6.22 (1H, br s, NH, *overlapped with* i), 6.22 (1H, dd, *J* = 15.9 and 6.6 Hz, i), 4.54 (1H, dd, *J* = 14.8 and 6.1 Hz, e), 4.35–4.45 (2H, m, e,h), 3.61 (1H, d, *J* = 5.9 Hz, OH), 2.40–2.50 (1H, m, g), 1.32 (3H, d, *J* = 7.1 Hz, o)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 175.37 (f), 138.12 (d), 136.58 (k), 131.75 (j), 130.15 (i), 128.83 (aryl), 128.70 (aryl), 127.92 (aryl), 127.73 (aryl), 127.60 (aryl), 126.71 (aryl), 75.06 (h), 46.58 (g), 43.47 (e), 15.46 (o)
IR (neat)	3336, 3281 (N-H stretch), 3025, 2979, 2359, 2343, 1647 (C=O stretch), 1556, 1494, 1454, 1415, 1361, 1258, 1226, 1110, 978, 965, 736, 686 cm^{-1}
HRMS (ESI)	$C_{19}H_{21}NNaO_2$ (M+Na): 318.1470, found 318.1476 *m/z*

^{1}H NMR

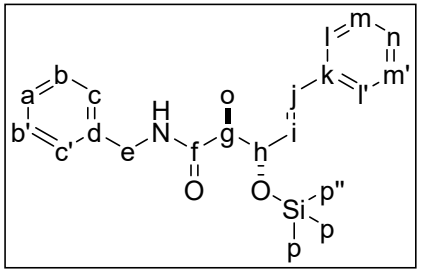

7.338
6.682
6.642
6.249
6.232
6.223
6.209
6.193
4.565
4.550
4.528
4.513
4.385
3.616
3.601
2.439
1.331
1.313
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.02
5.99
1.00
1.93
1.06
1.99
0.99
0.96
2.94

^{13}C NMR


175.37
138.13
136.58
131.75
130.15
128.83
128.70
127.92
127.73
127.60
126.71
75.06
46.58
43.47
15.46
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

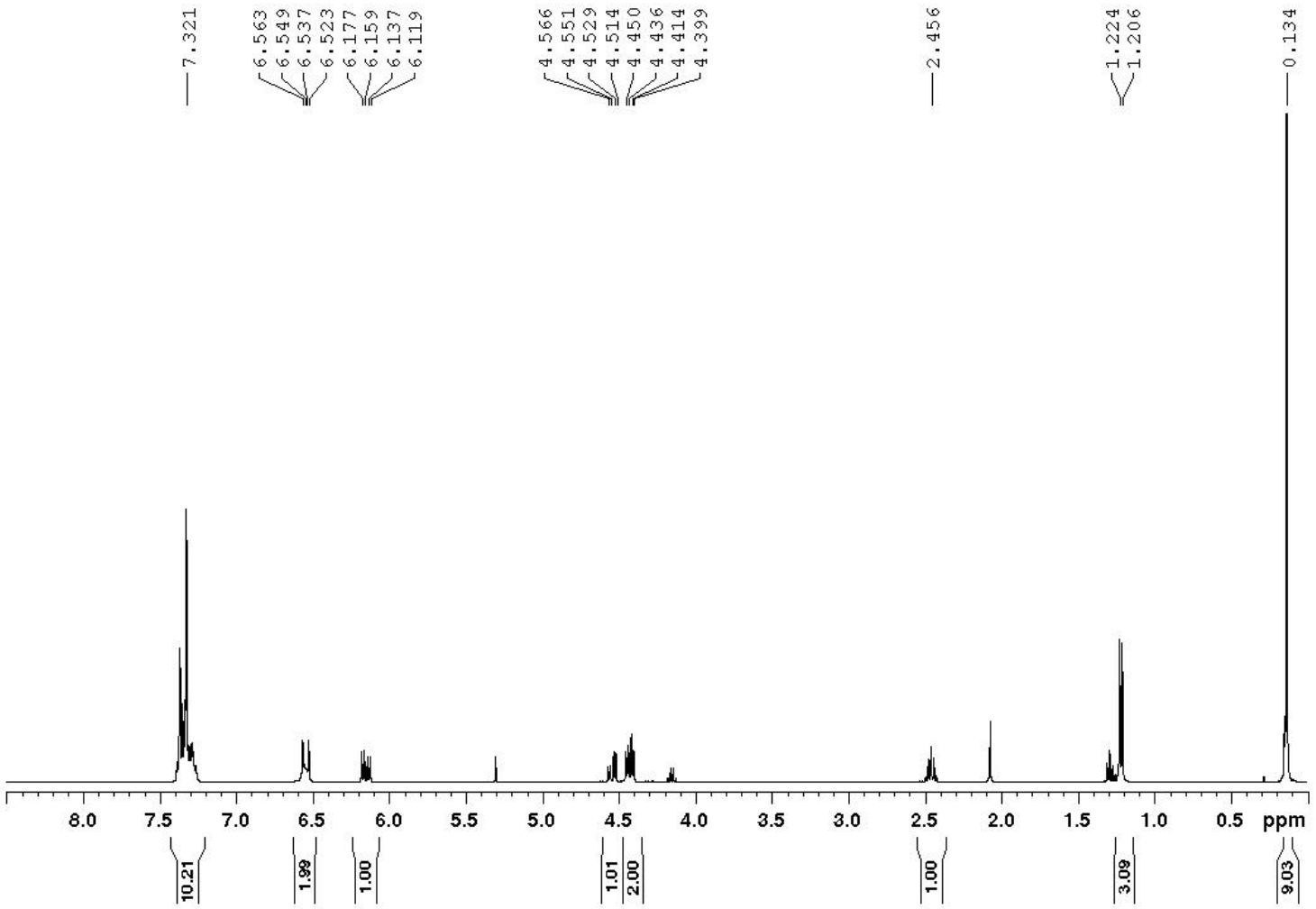


12

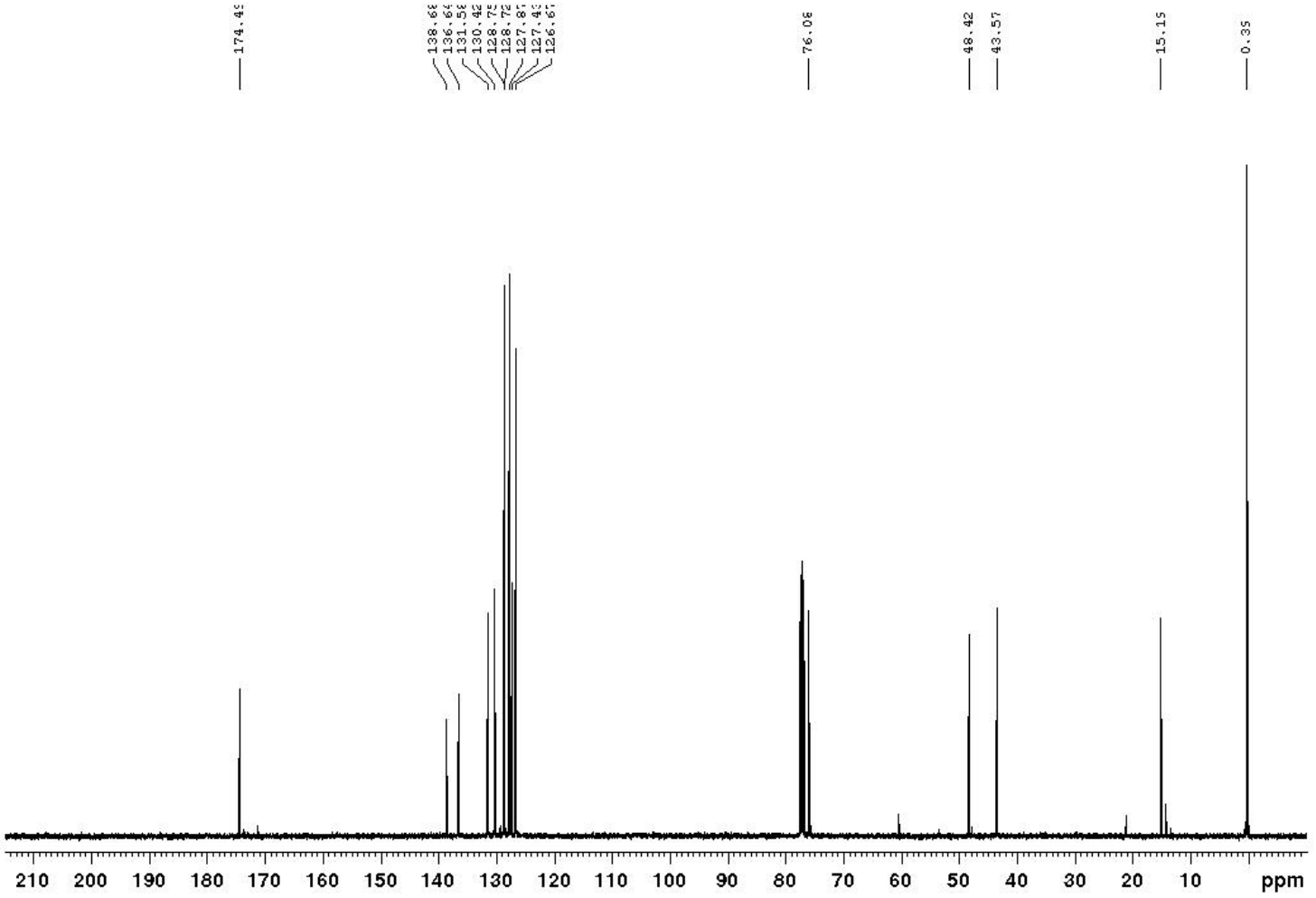


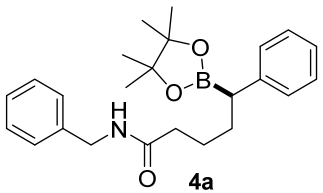
$[\alpha]_D^{20}$	$[\alpha]_D^{20}$ = +77.7º (*c* 2.0, $CHCl_3$)
m.p.	106.0–107.5 ºC
TLC analysis	R_f 0.5 (60:40 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (10H, m, aryl), 6.54 (1H, d, *J* = 4.9 Hz, NH, *overlapped with* j), 6.54 (1H, d, *J* = 15.7 Hz, j), 6.15 (1H, dd, *J* = 15.9 and 7.1 Hz, i), 4.54 (1H, dd, *J* = 14.8 and 5.9 Hz, e), 4.35–4.45 (2H, m, e,h), 2.40–2.50 (1H, m, g), 1.22 (3H, d, *J* = 7.1 Hz, o), 0.13 (9H, s, p,p',p'')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 174.49 (f), 138.68 (d), 136.64 (k), 131.58 (j), 130.42 (i), 128.75 (aryl), 128.72 (aryl), 127.87 (aryl), 127.43 (aryl), 126.67 (aryl), 76.08 (h), 48.42 (g), 43.57 (e), 15.19 (o), 0.39 (p,p',p'')
IR (neat)	3260 (N-H stretch), 2958, 1642 (C=O stretch), 1563, 1247, 1051, 970, 883, 838, 736, 692 cm^{-1}

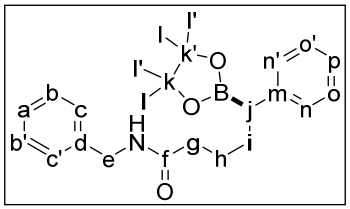
^{1}H NMR


7.321
6.563
6.549
6.537
6.523
6.177
6.159
6.137
6.119
4.566
4.551
4.529
4.514
4.450
4.436
4.414
4.399
2.456
1.224
1.206
0.134
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.21
1.99
1.00
1.01
2.00
1.00
3.09
9.03

^{13}C NMR

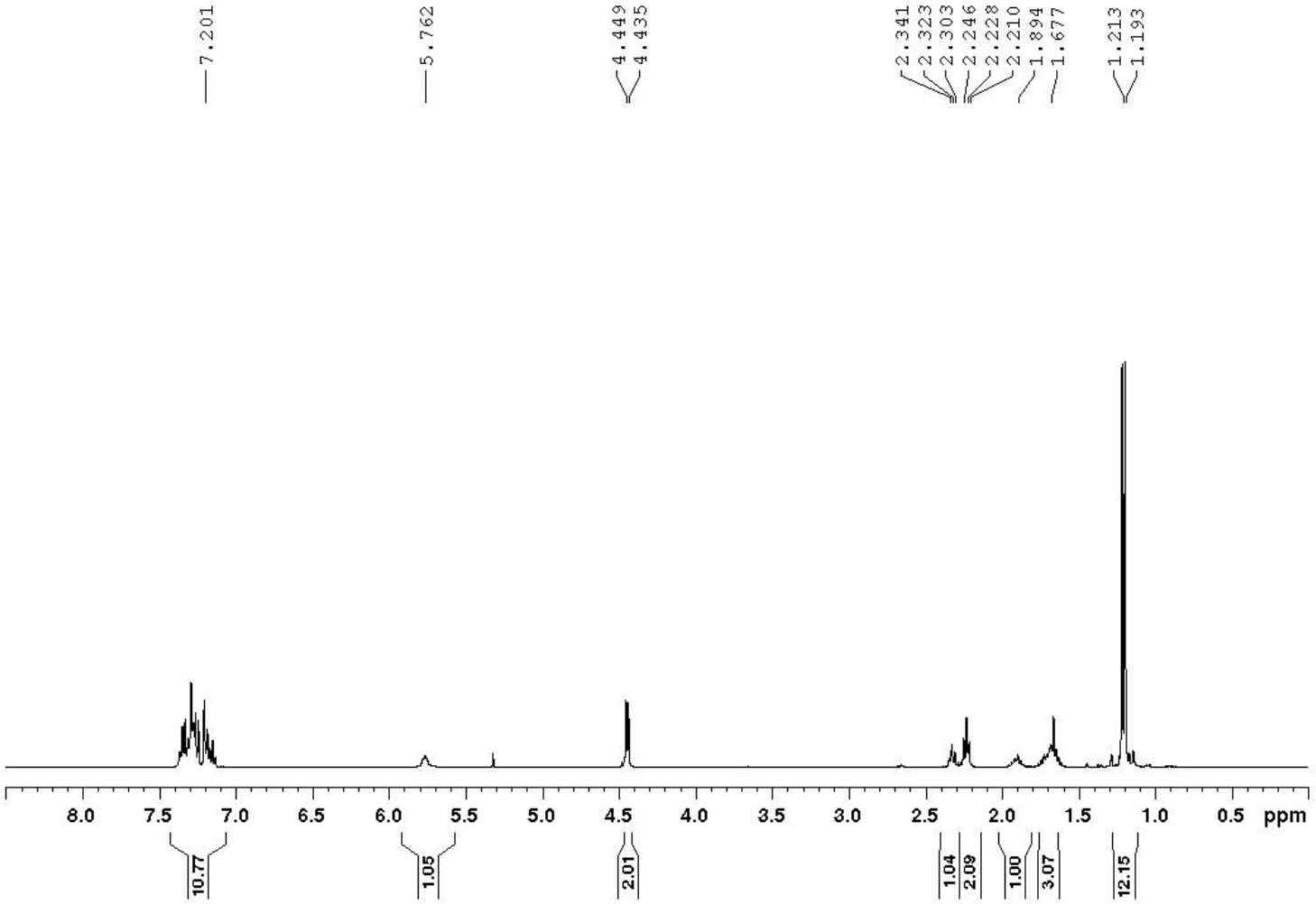

174.48
138.66
136.64
131.56
130.42
128.75
128.72
127.87
127.41
126.67
76.08
48.42
43.57
15.15
0.35
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



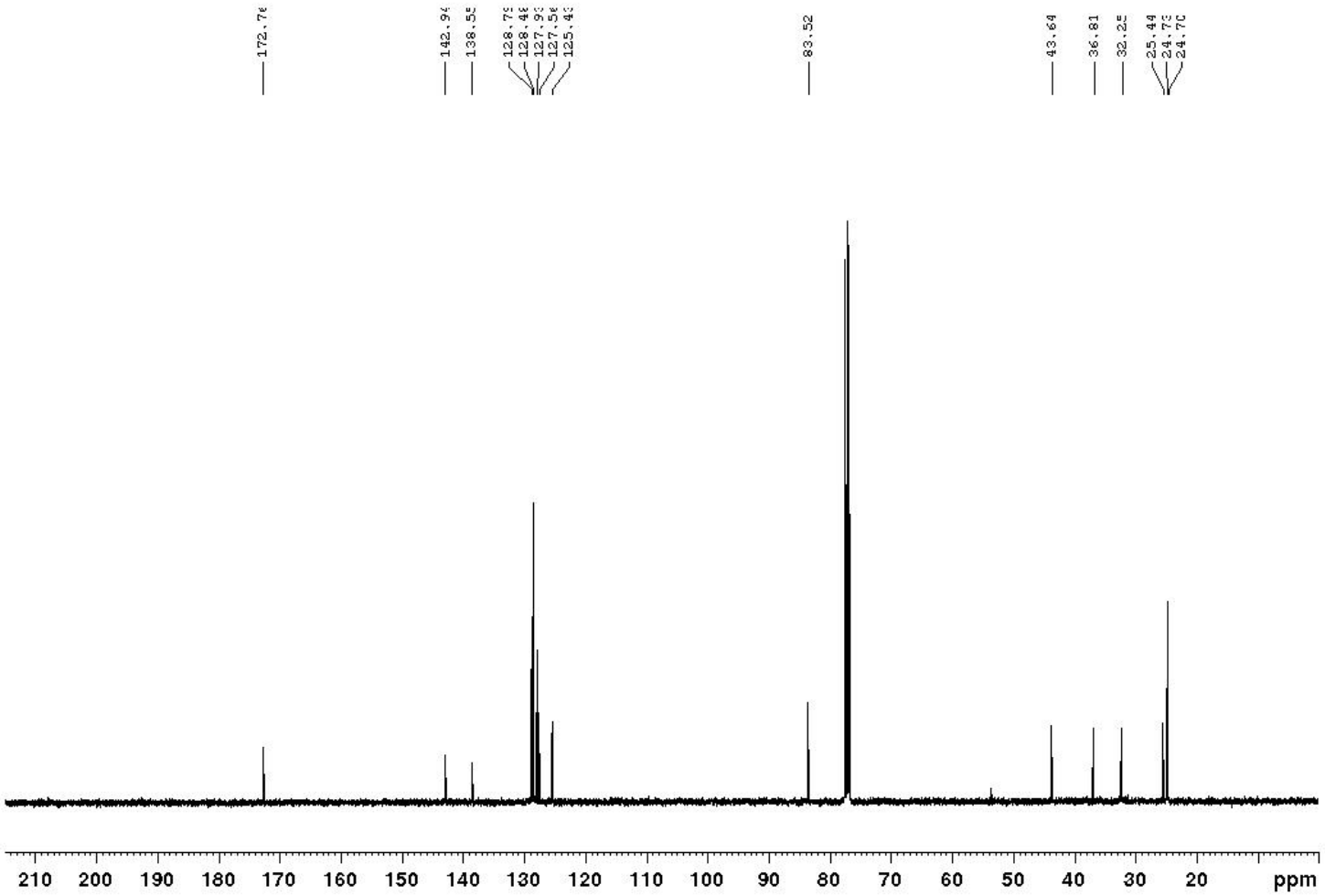


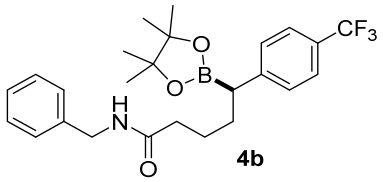
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.10–7.40 (10H, m, a,b,b',c,c',n,n',o,o',p), 5.76 (1H, br s, NH), 4.44 (2H, d, J = 5.6 Hz, e), 2.32 (1H, t, J = 7.3 Hz, j), 2.23 (2H, t, J = 7.4 Hz, g), 1.80–1.95 (1H, m, h), 1.60–1.75 (3H, m, h,i), 1.21 and 1.19 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.76 (f), 142.94 (m), 138.55 (d), 128.79 (b,b'), 128.48 (n,n',o,o'), 127.93 (c,c'), 127.56 (a), 125.43 (p), 83.52 (k,k'), 43.64 (e), 36.81 (g), 32.25 (h), 25.44 (i), 24.73 and 24.70 (l,l')

^{1}H NMR

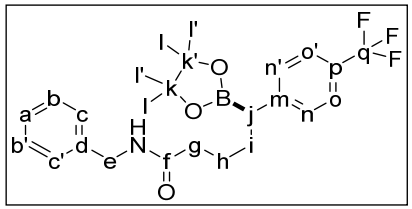

7.201
5.762
4.449
4.435
2.341
2.323
2.303
2.246
2.228
2.210
1.894
1.677
1.213
1.193
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.77
1.05
2.01
1.04
2.09
1.00
3.07
12.15

^{13}C NMR


172.7
142.9
138.5
128.7
128.4
127.9
127.5
125.4
83.52
43.64
36.81
32.25
25.44
24.7
24.7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

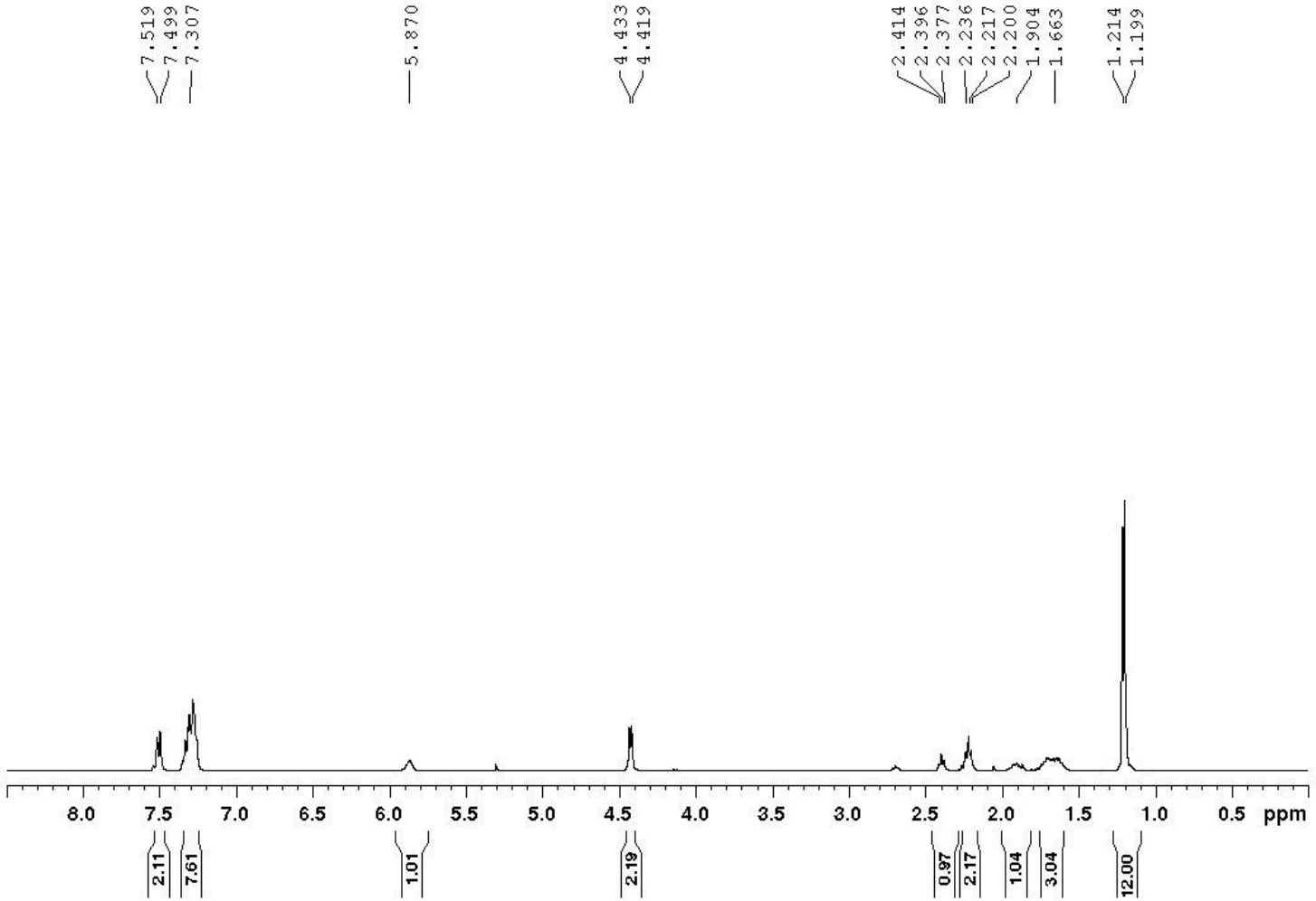


4b

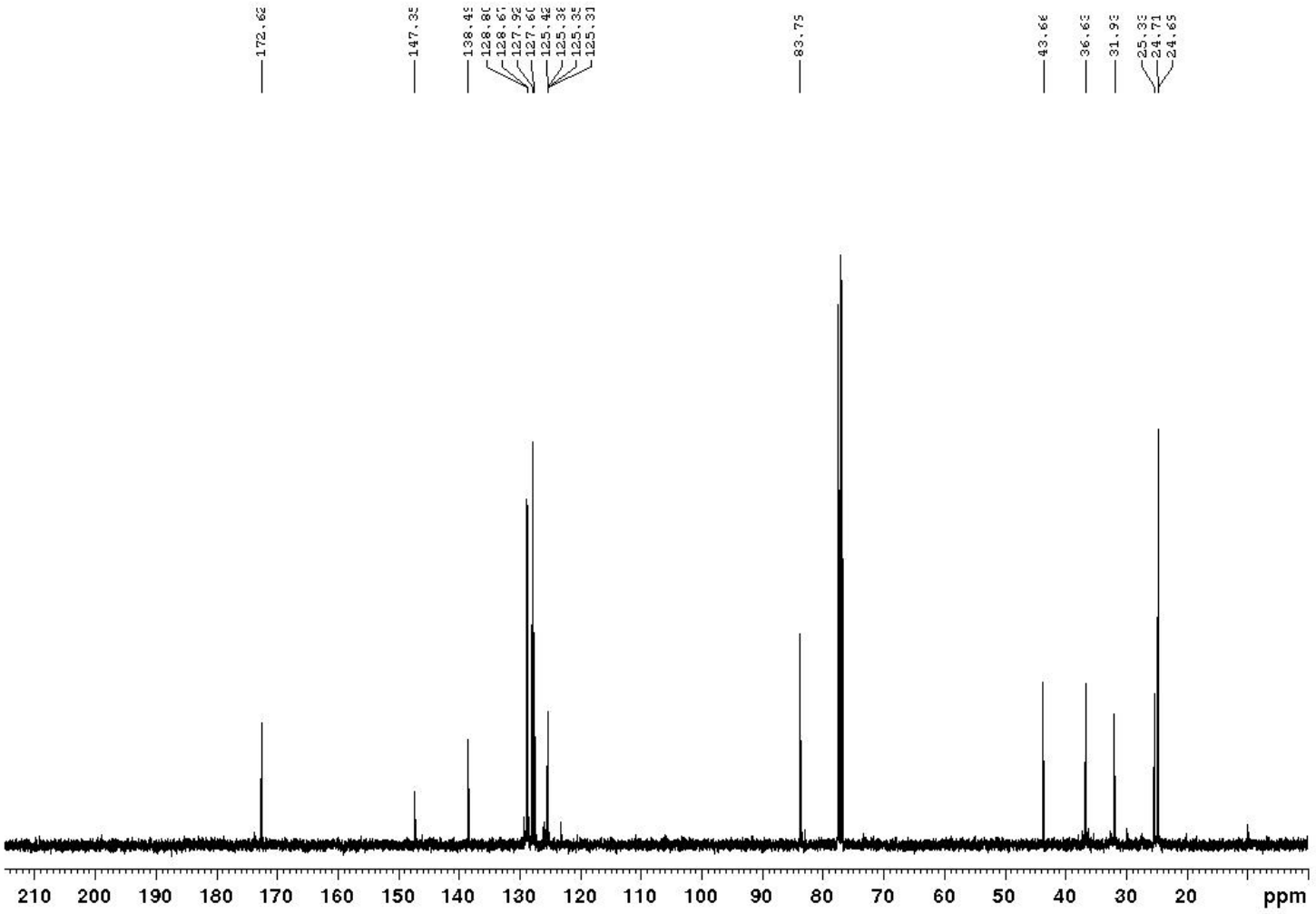


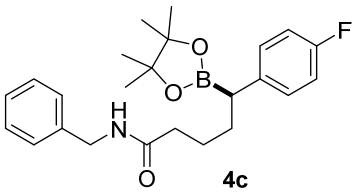
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.51 (2H, d, *J* = 8.1 Hz, o,o'), 7.25–7.35 (7H, m, a,b,b',c'c',n,n'), 5.87 (1H, br s, NH), 4.43 (2H, d, *J* = 5.5 Hz, e), 2.40 (1H, t, *J* = 7.2 Hz, j), 2.22 (2H, t, *J* = 7.4 Hz, g), 1.80–2.00 (1H, m, h), 1.60–1.80 (3H, m, h,i), 1.21 and 1.20 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.62 (f), 147.35 (m), 138.49 (d), 128.80 (aryl), 128.67 (aryl), 127.92 (aryl), 127.60 (aryl), 125.37 (q, *J* = 3.7 Hz, aryl), 83.79 (k,k'), 43.66 (e), 36.63 (g), 31.93 (h), 25.33 (i), 24.71 and 24.69 (l,l')

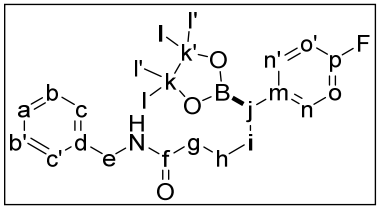
^{1}H NMR


7.519
7.499
7.307
5.870
4.433
4.419
2.414
2.396
2.377
2.236
2.217
2.200
1.904
1.663
1.214
1.199
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.11
7.61
1.01
2.19
0.97
2.17
1.04
3.04
12.00

^{13}C NMR

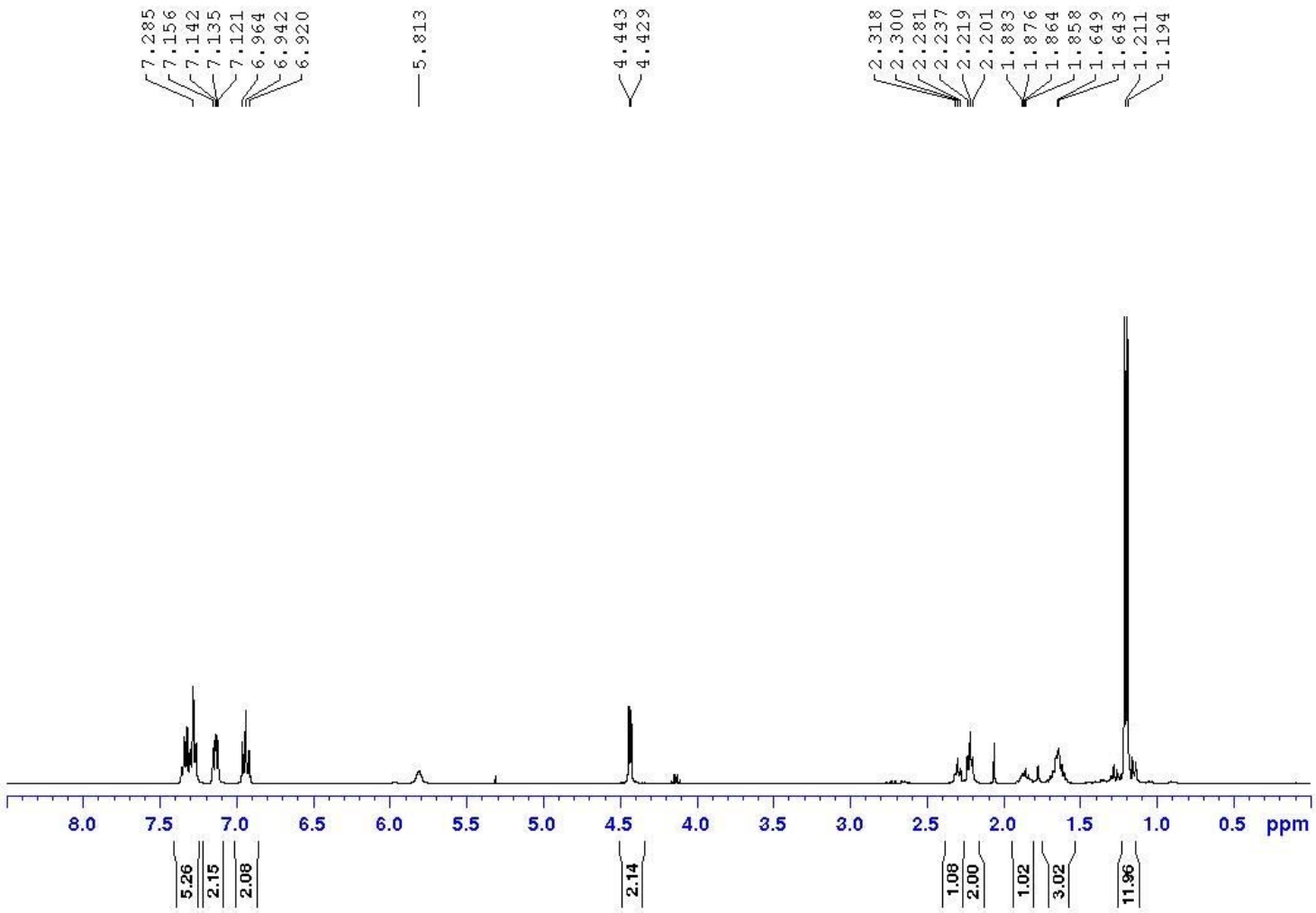

172.62
147.35
138.45
128.80
128.67
127.92
127.60
125.46
125.38
125.32
125.31
83.75
43.66
36.63
31.93
25.33
24.71
24.65
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



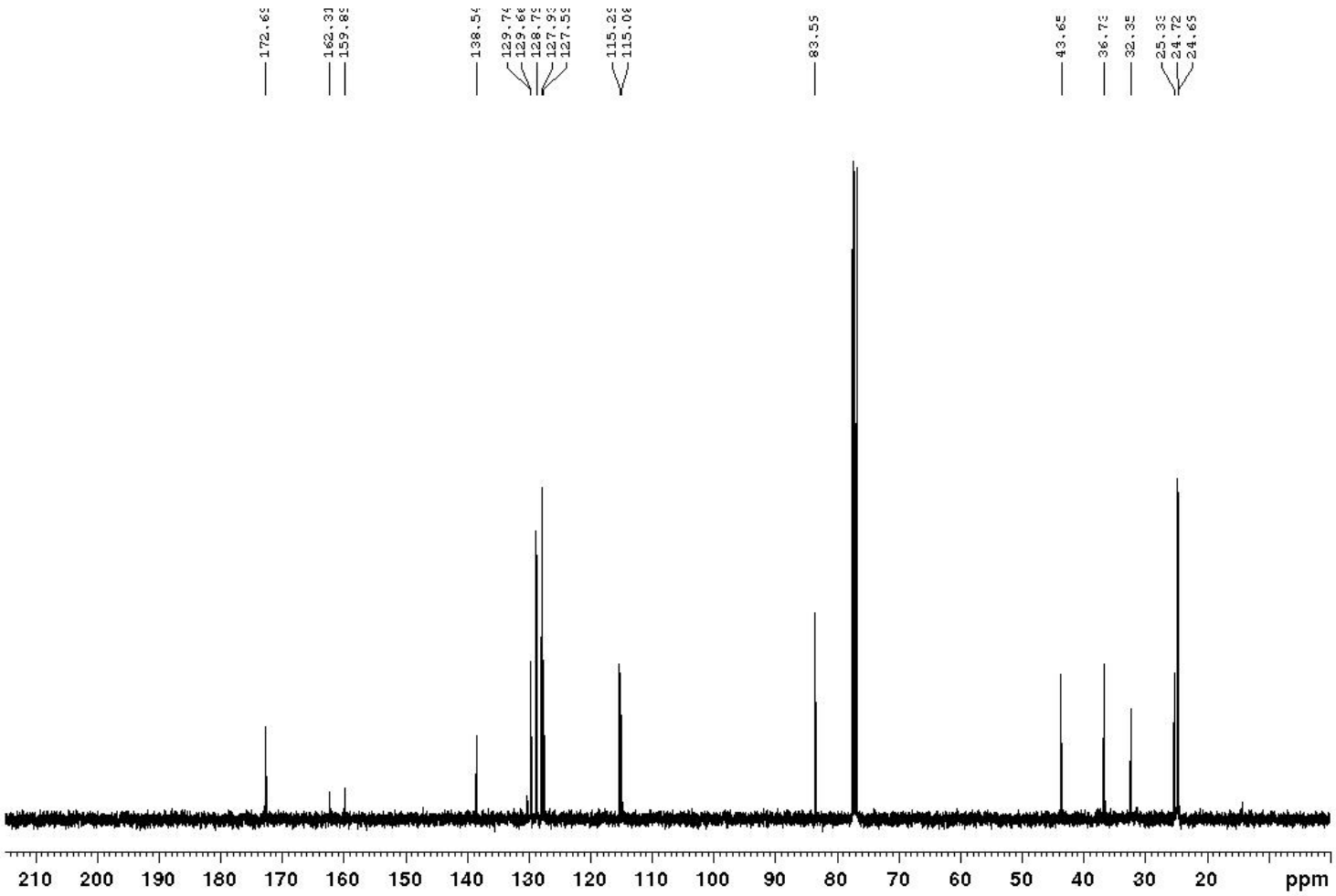


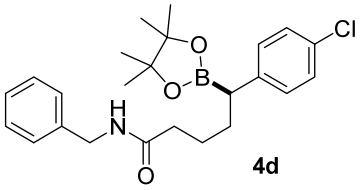
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.40 (5H, m, aryl), 7.14 (2H, dd, *J* = 8.6 and 5.5 Hz, aryl), 6.94 (2H, *J* = 8.8 Hz, aryl), 5.81 (1H, br s, NH), 4.44 (2H, d, *J* = 5.9 Hz, e), 2.30 (1H, t, *J* = 7.3 Hz, j), 2.22 (2H, t, *J* = 7.4 Hz, g), 1.80–1.90 (1H, m, h), 1.60–1.75 (3H, m, f,h), 1.21 and 1.19 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.69 (f), 161.10 (d, *J* = 242 Hz, p), 138.54 (d), 129.74 (aryl), 129.66 (aryl), 128.79 (aryl), 127.83 (aryl), 127.59 (aryl), 115.19 (d, *J* = 21 Hz, o,o'), 83.59 (k,k'), 43.65 (e), 36.73 (g), 32.35 (h), 25.33 (i), 24.72 and 24.69 (l,l')

^{1}H NMR

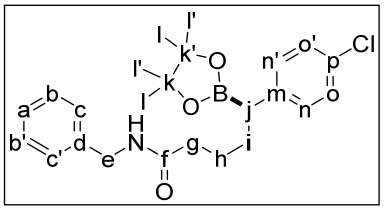

7.285
7.156
7.142
7.135
7.121
6.964
6.942
6.920
5.813
4.443
4.429
2.318
2.300
2.281
2.237
2.219
2.201
1.883
1.876
1.864
1.858
1.649
1.643
1.211
1.194
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.26
2.15
2.08
2.14
1.08
2.00
1.02
3.02
11.96

^{13}C NMR


172.69
162.31
159.89
138.54
129.74
129.68
128.79
127.99
127.59
115.29
115.08
83.55
43.65
36.73
32.35
25.33
24.72
24.65
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

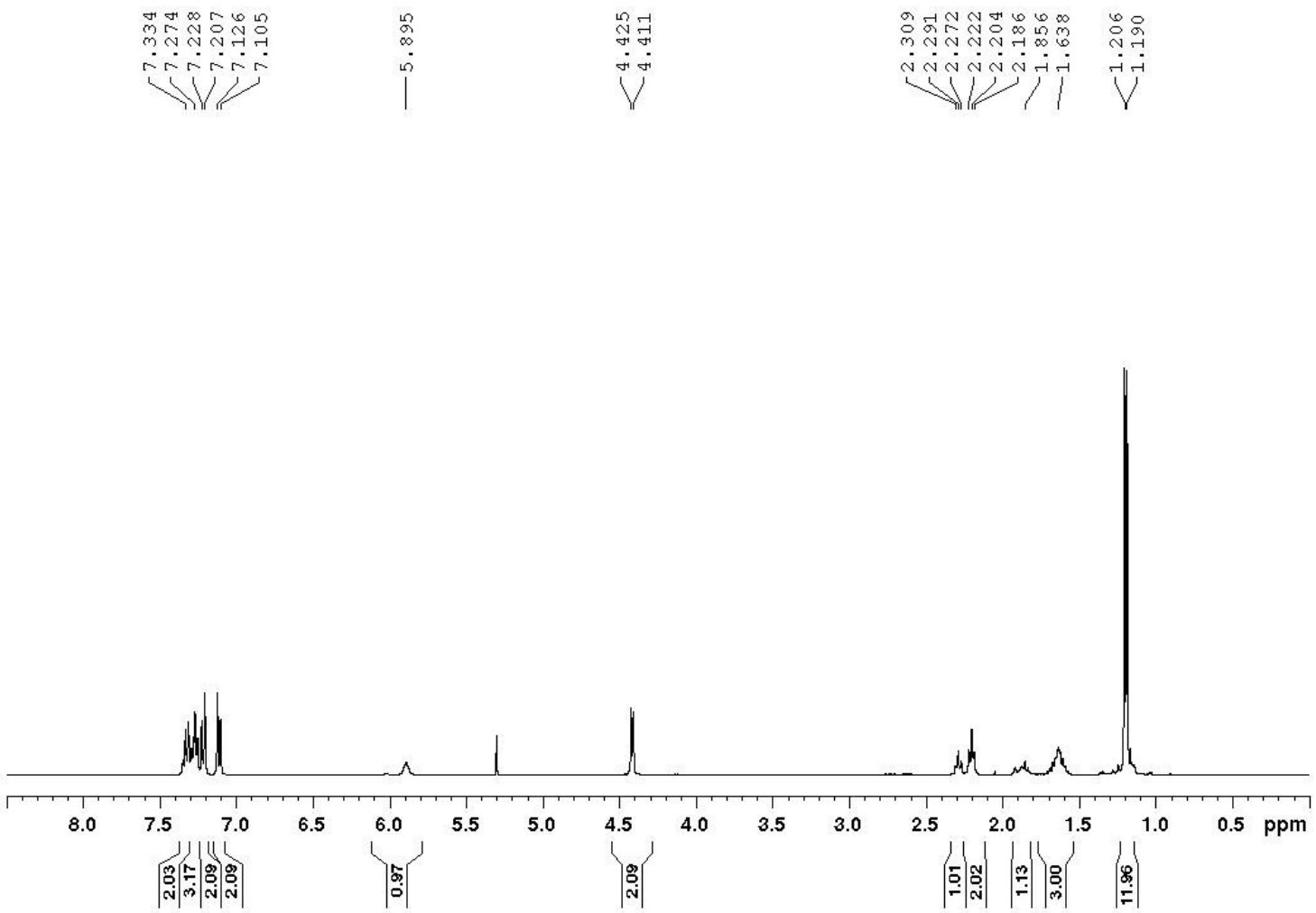


4d

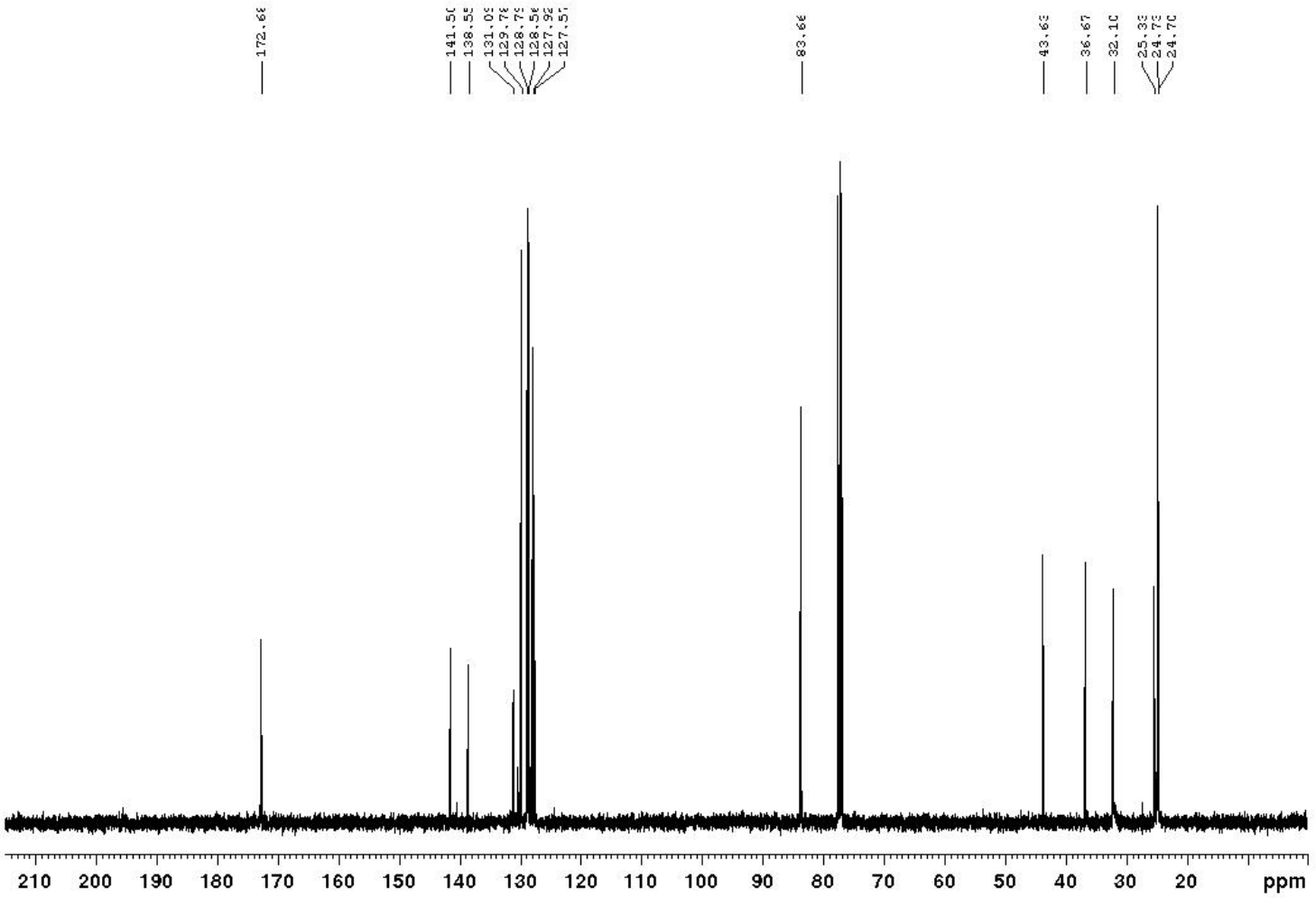


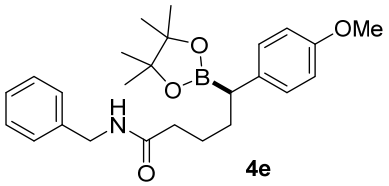
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.35 (5H, m, a,b,b',o,o',p), 7.22 (2H, d, *J* = 8.4 Hz, c,c'), 7.12 (2H, d, *J* = 8.4 Hz, n,n'), 5.90 (1H, br s, NH), 4.42 (2H, d, *J* = 5.7 Hz, e), 2.29 (1H, t, *J* = 7.3 Hz, j), 2.20 (2H, t, *J* = 7.4 Hz, g), 1.80–1.90 (1H, m, h), 1.50–1.70 (3H, m, f,h), 1.21 and 1.19 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.68 (f), 141.50 (m), 138.55 (d), 131.09 (p). 129.78 (n,n'), 128.79 (b,b'), 128.56 (o,o'), 127.92 (c,c'), 127.57 (a), 83.66 (k,k'), 43.63 (e), 36.67 (g), 32.10 (h), 25.33 (i), 24.73 and 24.70 (l,l')

^{1}H NMR

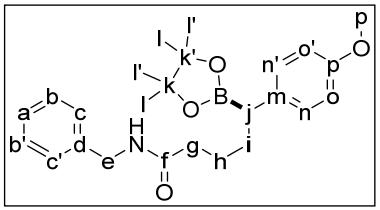

7.334
7.274
7.228
7.207
7.126
7.105
5.895
4.425
4.411
2.309
2.291
2.272
2.222
2.204
2.186
1.856
1.638
1.206
1.190
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.03
3.17
2.09
2.09
0.97
2.09
1.01
2.02
1.13
3.00
11.96

^{13}C NMR


172.68
141.50
138.55
131.05
129.78
128.75
128.54
127.98
127.57
83.66
43.68
36.67
32.10
25.38
24.75
24.70
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

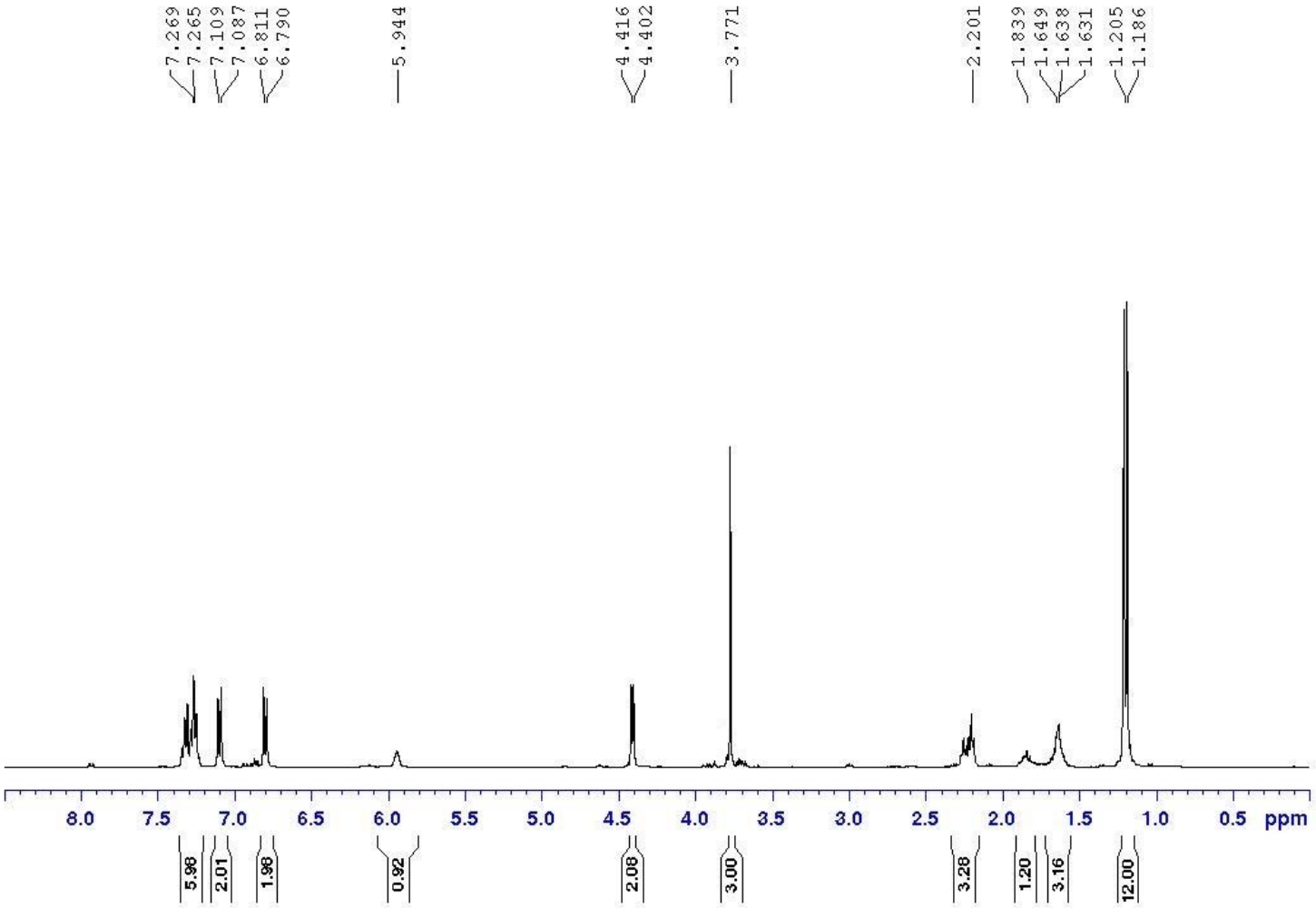


4e

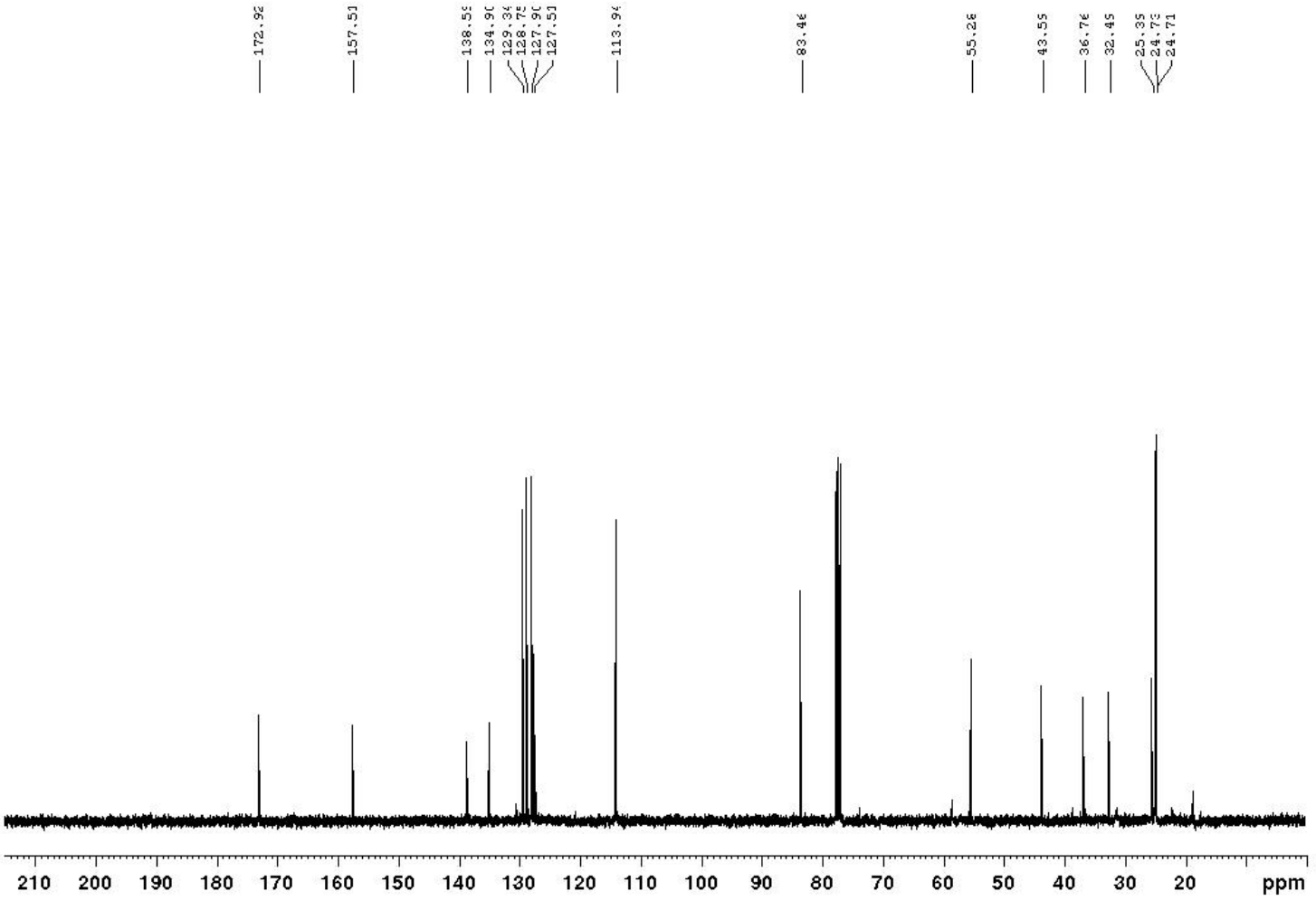


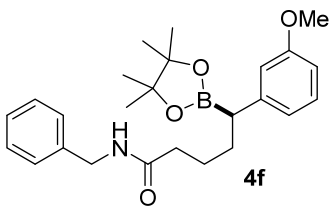
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (5H, m, a,b,b',c,c'), 7.10 (2H, d, *J* = 8.6 Hz, n,n'), 6.80 (2H, d, *J* = 8.6 Hz, o,o'), 5.94 (1H, br s, NH)), 4.41 (2H, d, *J* = 5.7 Hz, e), 3.77 (3H, s, s), 2.15–2.30 (3H, m, g,j), 1.80–1.90 (1H, m, h), 1.55–1.70 (3H, m, h,i), 1.21 and 1.19 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.92 (f), 157.51 (q), 138.59 (d), 134.90 (m), 129.34 (n,n'), 128.75 (b,b'), 127.90 (c,c,'), 127.51 (a), 113.94 (o,o'), 83.46 (k,k'), 55.28 (s), 43.59 (e), 36.76 (g), 32.49 (h), 25.39 (i), 24.73 and 24.71 (l,l')

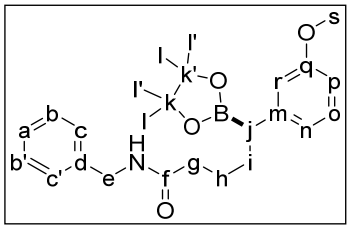
^{1}H NMR


7.269
7.265
7.109
7.087
6.811
6.790
5.944
4.416
4.402
3.771
2.201
1.839
1.649
1.638
1.631
1.205
1.186
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.98
2.01
1.98
0.92
2.08
3.00
3.28
1.20
3.16
12.00

^{13}C NMR

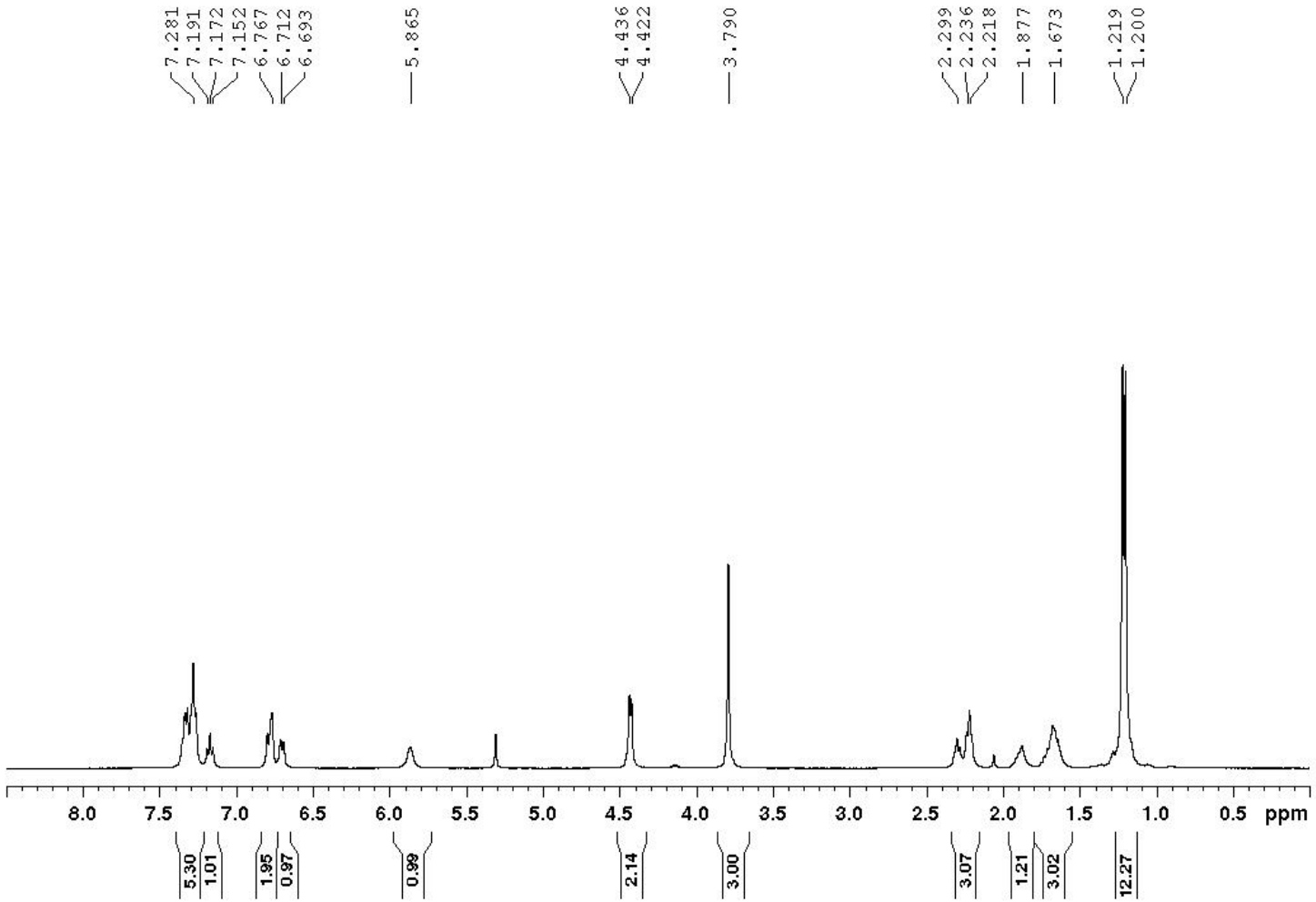

172.92
157.51
138.55
134.90
129.34
128.75
127.90
127.51
113.94
83.46
55.26
43.55
36.76
32.45
25.35
24.73
24.71
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



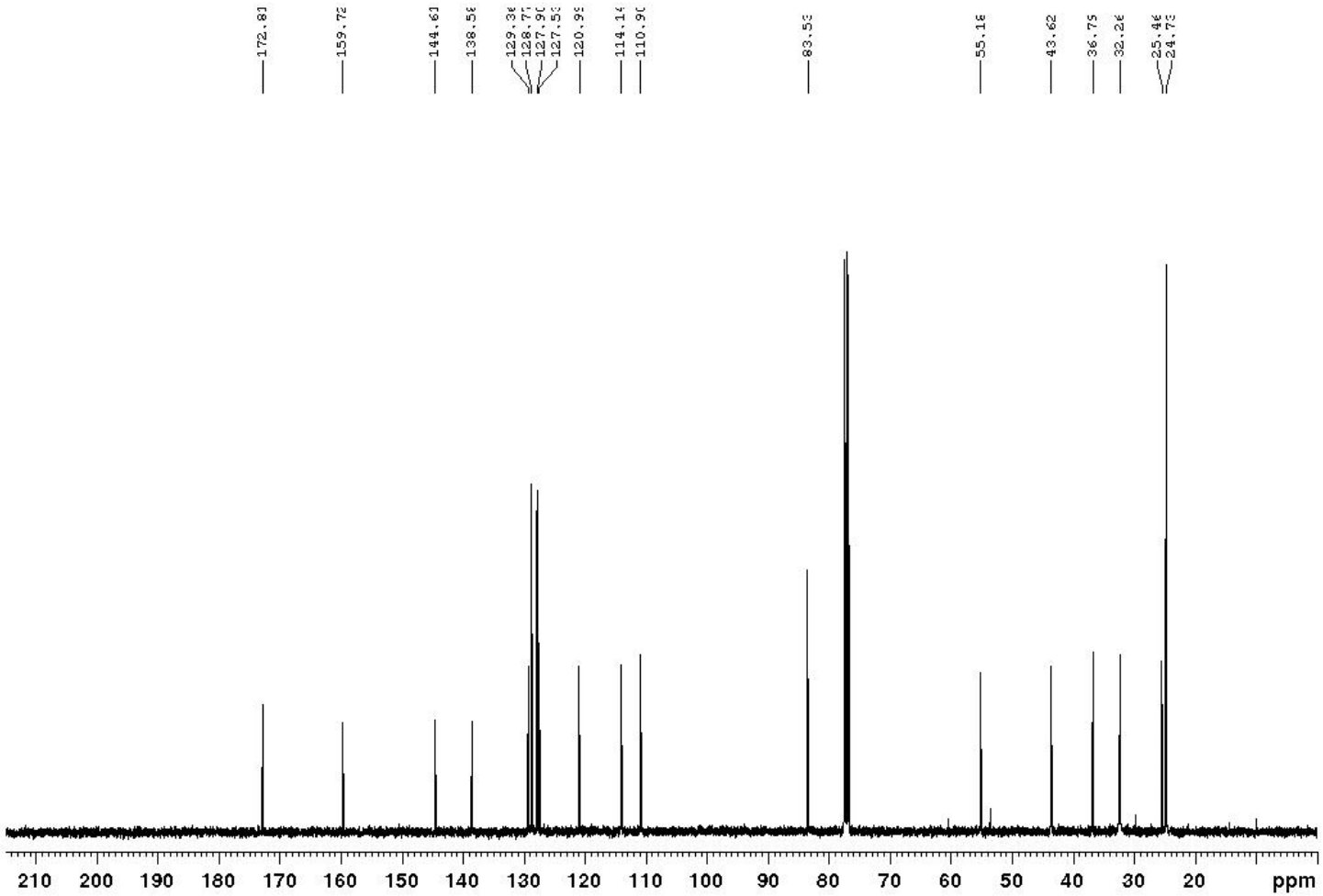


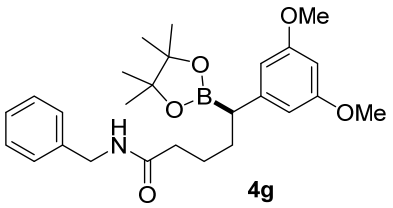
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (5H, m, aryl), 7.17 (1H, t, *J* = 7.7 Hz, aryl), 6.75–6.85 (2H, m, aryl), 6.71 (1H, d, *J* = 7.8 Hz, aryl), 5.87 (1H, br s, NH)), 4.43 (2H, d, *J* = 5.5 Hz, e), 3.79 (3H, s, s), 2.15–2.35 (3H, m, g,j), 1.80–1.95 (1H, m, h), 1.55–1.80 (3H, m, h,i), 1.22 and 1.20 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.81 (f), 159.72 (q), 144.61 (m), 138.85 (d), 129.35 (o), 128.77 (b,b'), 127.90 (c,c,'), 127.53 (a), 120.99 (n), 114.14 (p), 110.90 (r), 83.53 (k,k'), 55.18 (s), 43.62 (e), 36.79 (g), 32.26 (h), 25.46 (i), 24.73 (l,l')

^{1}H NMR

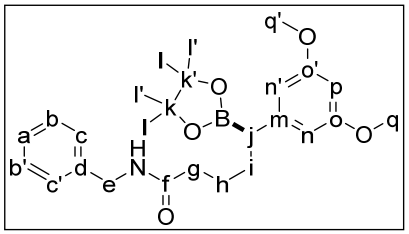

7.281
7.191
7.172
7.152
6.767
6.712
6.693
5.865
4.436
4.422
3.790
2.299
2.236
2.218
1.877
1.673
1.219
1.200
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.30
1.01
1.95
0.97
0.99
2.14
3.00
3.07
1.21
3.02
12.27

^{13}C NMR


172.83
159.72
144.63
138.58
129.38
128.77
127.90
127.55
120.95
114.14
110.90
83.53
55.18
43.62
36.75
32.26
25.46
24.73
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

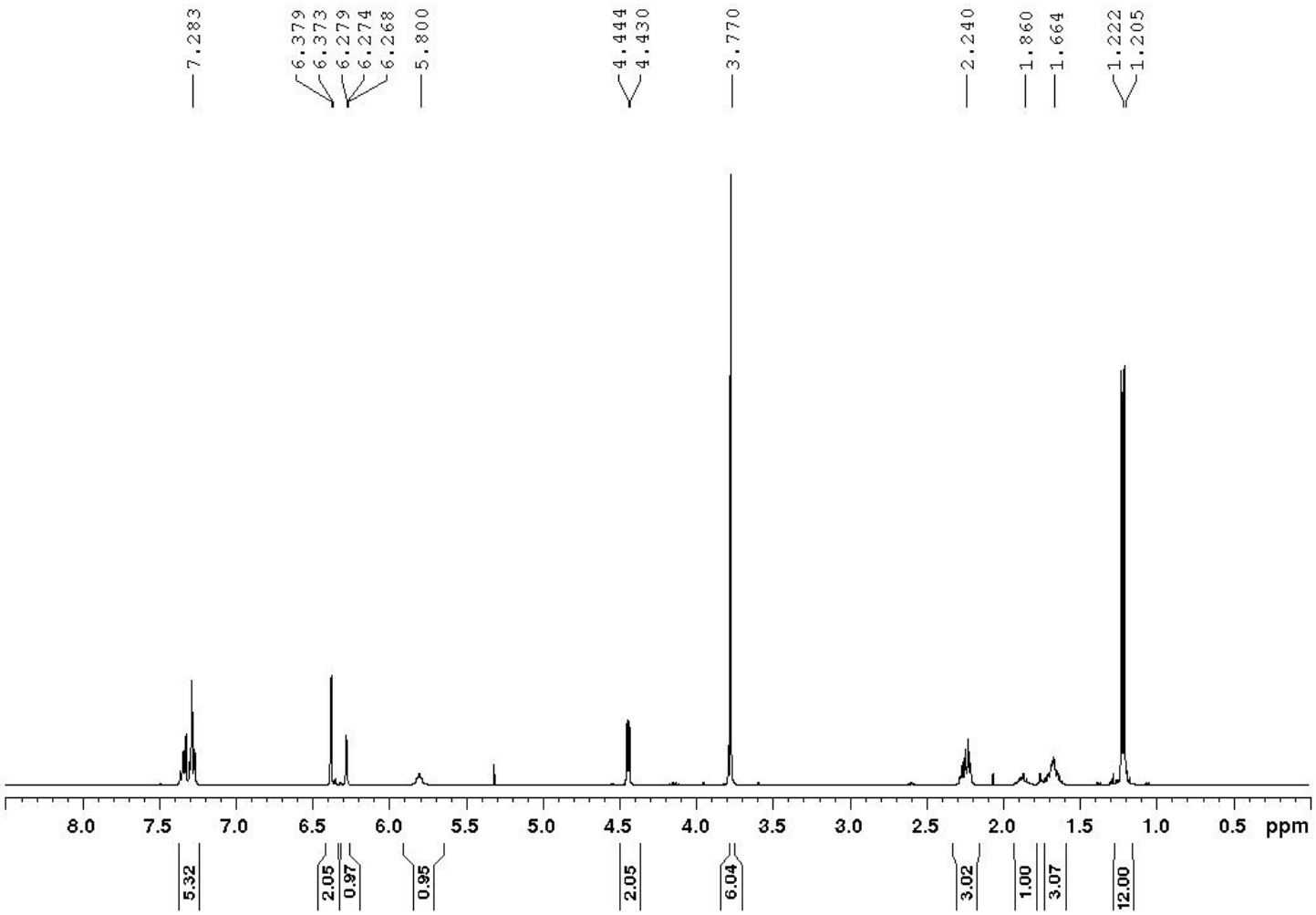


4g

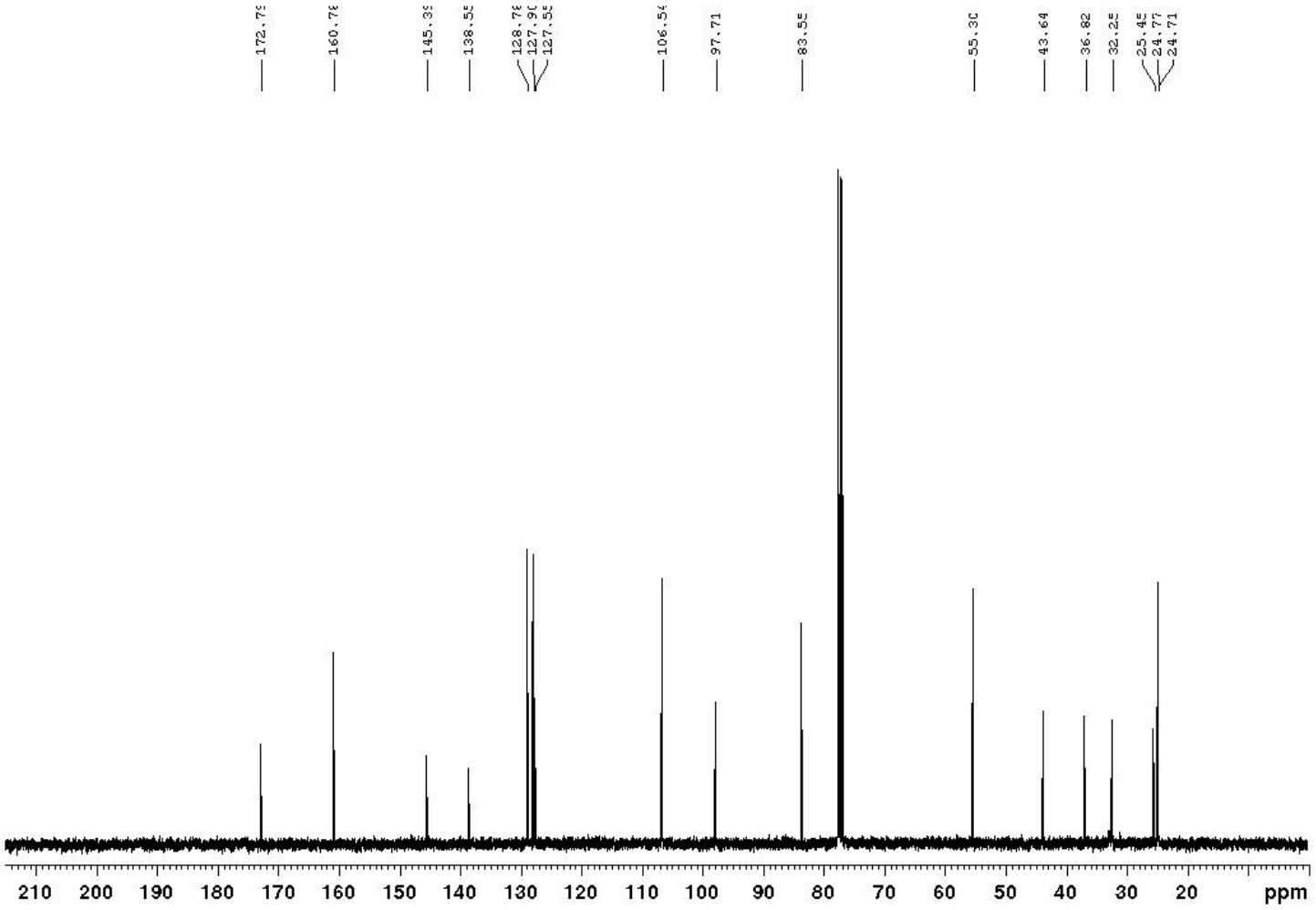


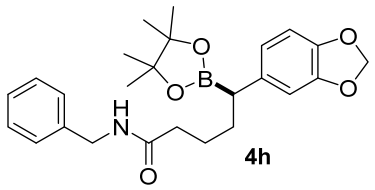
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.35 (5H, m, a,b,b',c,c'), 6.38 (2H, d, *J* = 2.2 Hz, n,n'), 6.37 (1H, t, *J* = 2.2 Hz, p), 5.80 (1H, br s, NH), 4.44 (2H, d, *J* = 5.7 Hz, e), 3.77 (6H, s, q,q'), 2.15–2.30 (3H, m, g,j), 1.80–1.90 (1H, m, h), 1.60–1.75 (3H, m, h,i), 1.22 and 1.21 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.79 (f), 160.78 (o,o'), 145.39 (m), 138.55 (d), 128.78 (b,b'), 127.90 (c,c,'), 127.55 (a), 106.54 (n,n'), 97.71 (p), 83.55 (k,k'), 55.30 (s), 43.64 (e), 36.82 (g), 32.25 (h), 25.45 (i), 24.77 and 24.71 (l,l')

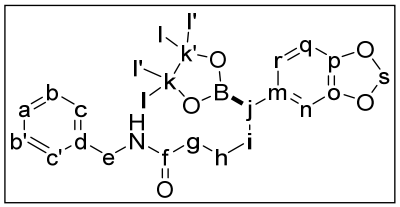
^{1}H NMR


7.283
6.379
6.373
6.279
6.274
6.268
5.800
4.444
4.430
3.770
2.240
1.860
1.664
1.222
1.205
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.32
2.05
0.97
0.95
2.05
6.04
3.02
1.00
3.07
12.00

^{13}C NMR

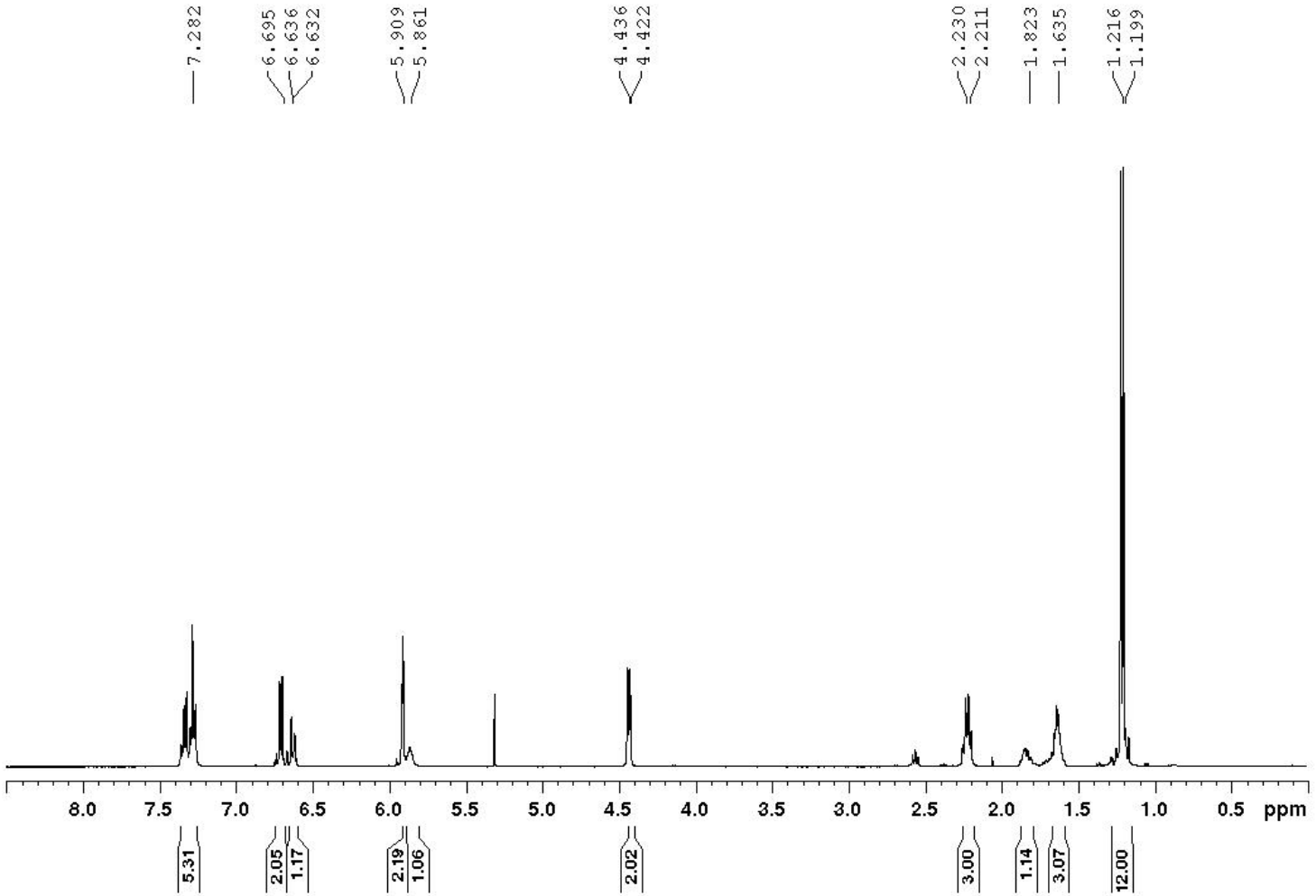

172.7
160.7
145.3
138.5
128.7
127.9
127.5
106.5
97.71
83.5
55.30
43.64
36.82
32.2
25.4
24.7
24.71
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



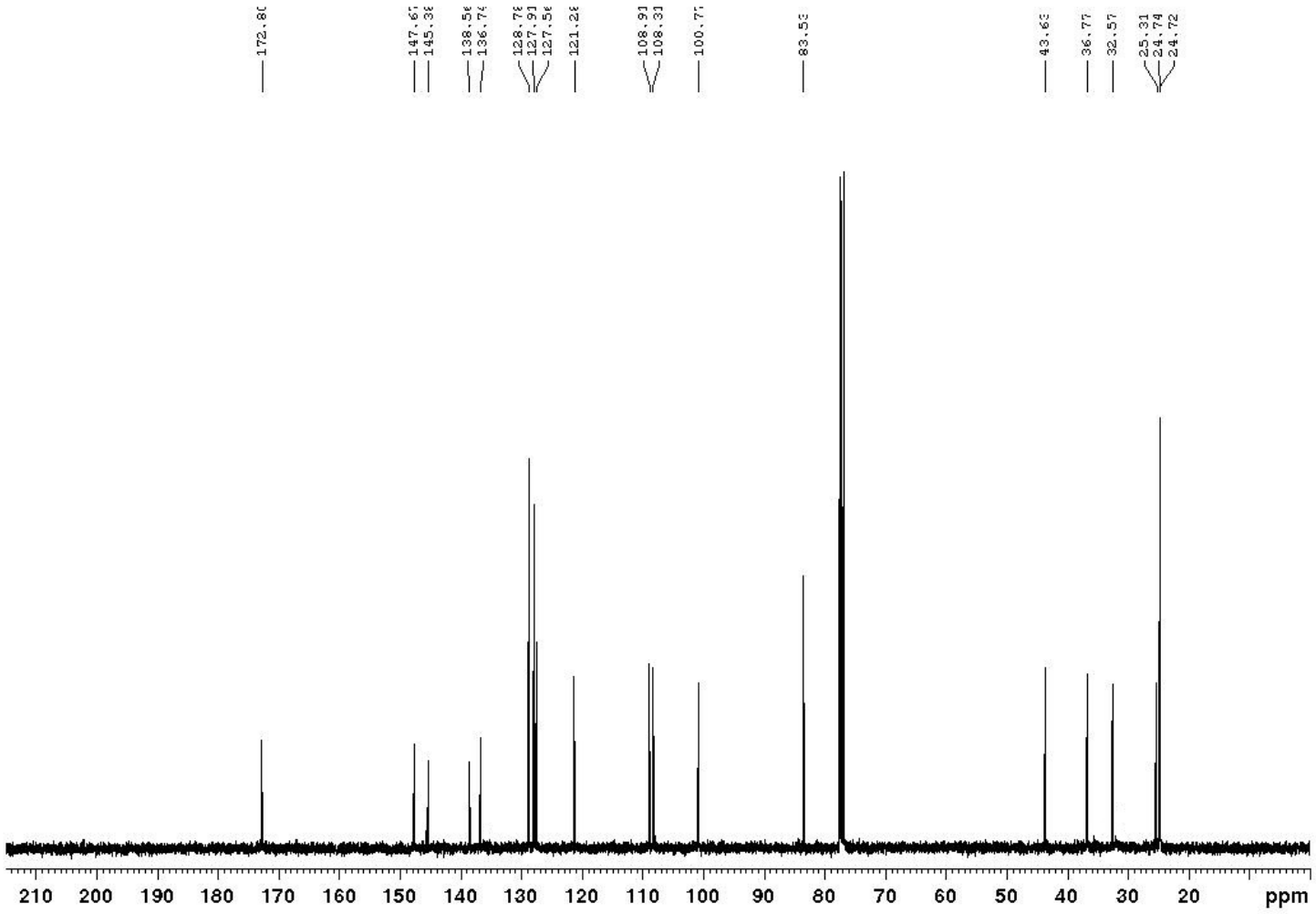


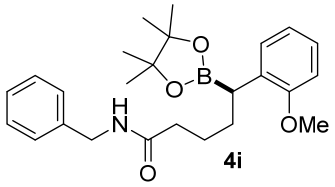
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.35 (5H, m, a,b,b',c,c'), 6.70–6.75 (2H, m, aryl), 6.60–6.65 (1H, m, aryl), 5.90–5.95 (2H, m, s), 5.86 (1H, br s, NH), 4.43 (2H, d, J = 5.7 Hz, e), 2.15–2.25 (3H, m, g,j), 1.80–1.90 (1H, m, h), 1.60–1.70 (3H, m, h,i), 1.22 and 1.20 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.80 (f), 147.67 (o), 145.38 (p), 138.56 (d), 136.74 (m), 128.78 (b,b'), 127.91 (c,c,'), 127.56 (a), 121.28 (aryl), 108.91 (aryl), 108.31 (aryl), 100.77 (s), 83.53 (k,k'), 43.63 (e), 36.77 (g), 32.57 (h), 25.31 (i), 24.74 and 24.72 (l,l')

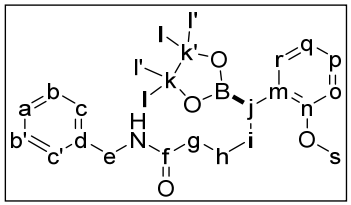
^{1}H NMR


7.282
6.695
6.636
6.632
5.909
5.861
4.436
4.422
2.230
2.211
1.823
1.635
1.216
1.199
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.31
2.05
1.17
2.19
1.06
2.02
3.00
1.14
3.07
12.00

^{13}C NMR

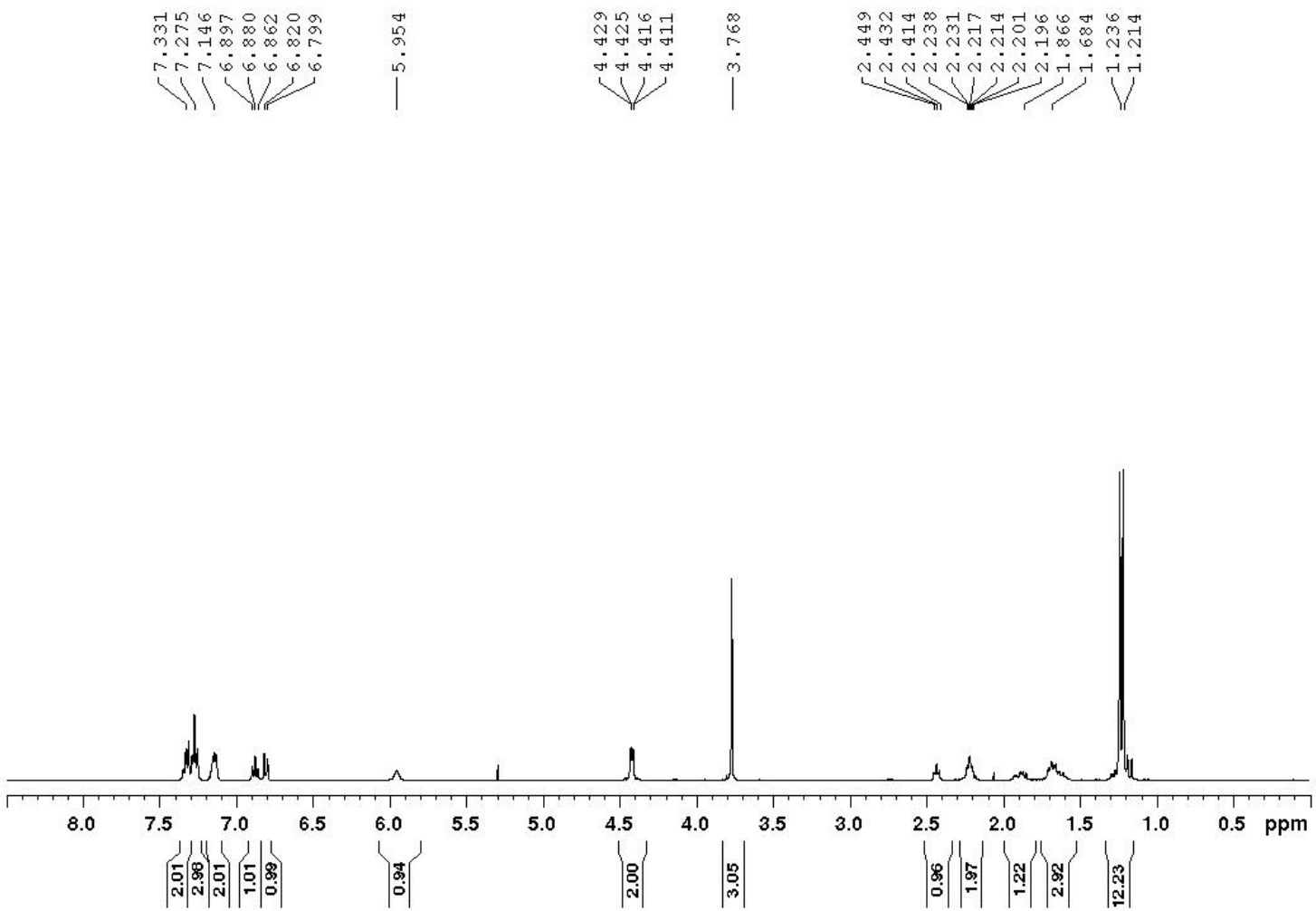

172.8
147.6
145.3
138.5
136.7
128.7
127.9
127.5
121.2
108.9
108.3
100.7
83.5
43.6
36.7
32.5
25.3
24.7
24.7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



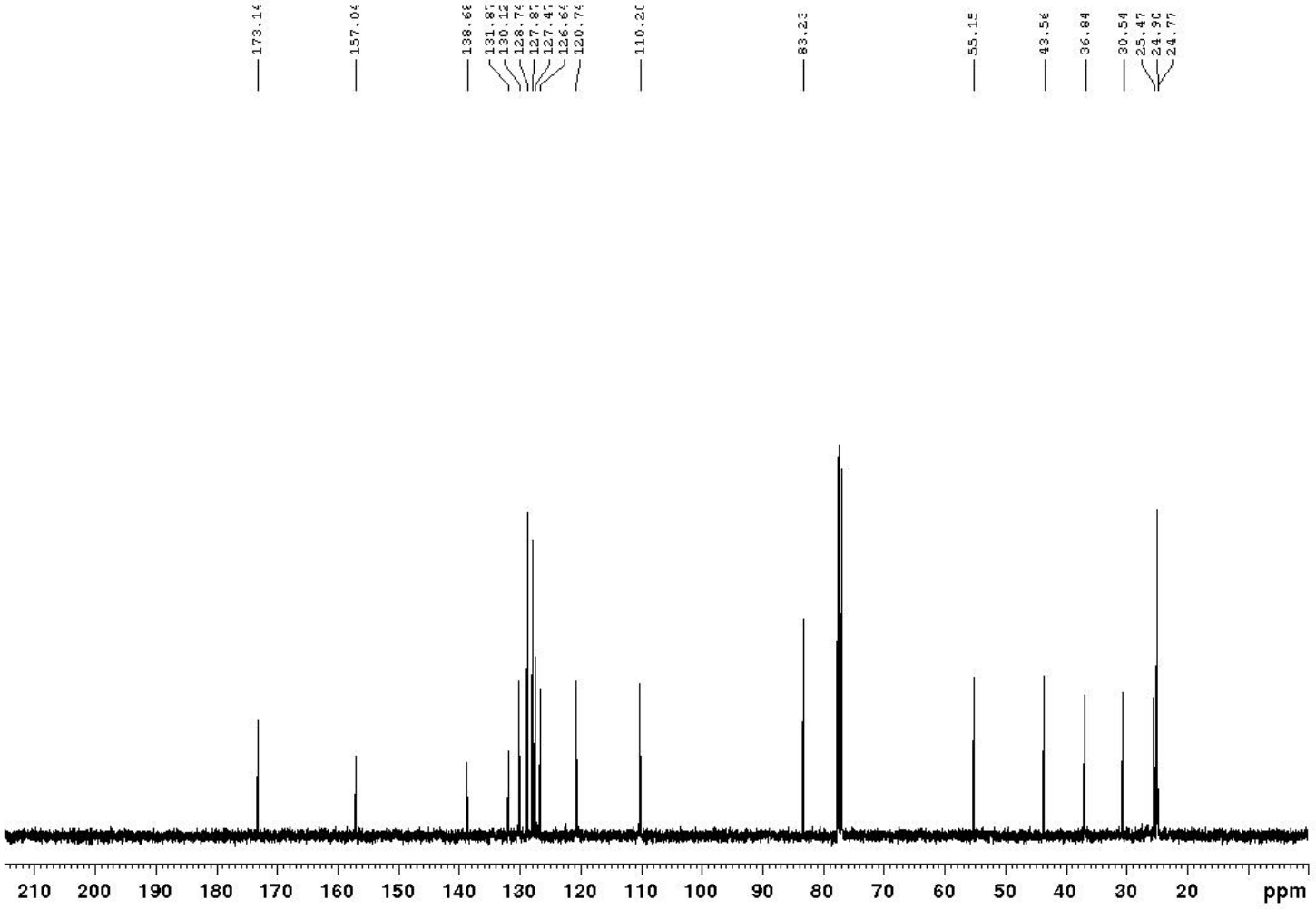


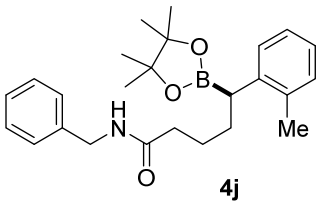
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.30–7.35 (2H, m, b,b'), 7.25–7.30 (3H, m, a,c,c'), 7.10–7.20 (2H, m, q,r), 6.88 (1H, t, *J* = 7.0 Hz, p), 6.82 (1H, d, *J* = 8.3 Hz, o), 5.95 (1H, br s, NH), 4.42 (2H, dd, *J* = 5.5 and 1.8 Hz, e), 3.77 (3H, s, s), 2.43 (1H, t, *J* = 6.9 Hz, j), 2.22 (2H, td, *J* = 8.1 and 2.8 Hz, g), 1.80–1.95 (1H, m, h), 1.55–1.75 (3H, m, h,i), 1.24 and 1.21 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.14 (f), 157.04 (n), 138.68 (d), 131.87 (m), 130.12 (r), 128.74 (b,b'), 127.87 (c,c,'), 127.47 (a), 126.64 (q), 120.74 (p), 110.20 (o), 83.23 (k,k'), 55.15 (s), 43.56 (e), 36.84 (g), 30.54 (h), 25.47 (i), 24.90 and 24.77 (l,l')

^{1}H NMR

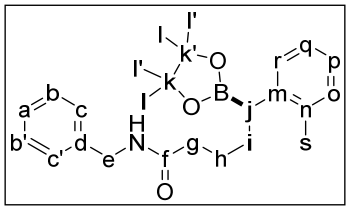

7.331
7.275
7.146
6.897
6.880
6.862
6.820
6.799
5.954
4.429
4.425
4.416
4.411
3.768
2.449
2.432
2.414
2.238
2.231
2.217
2.214
2.201
2.196
1.866
1.684
1.236
1.214
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.01
2.98
2.01
1.01
0.99
0.94
2.00
3.05
0.96
1.97
1.22
2.92
12.23

^{13}C NMR


173.14
157.04
138.68
131.87
130.12
128.74
127.87
127.41
126.64
120.74
110.20
83.23
55.15
43.56
36.84
30.54
25.47
24.90
24.77
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

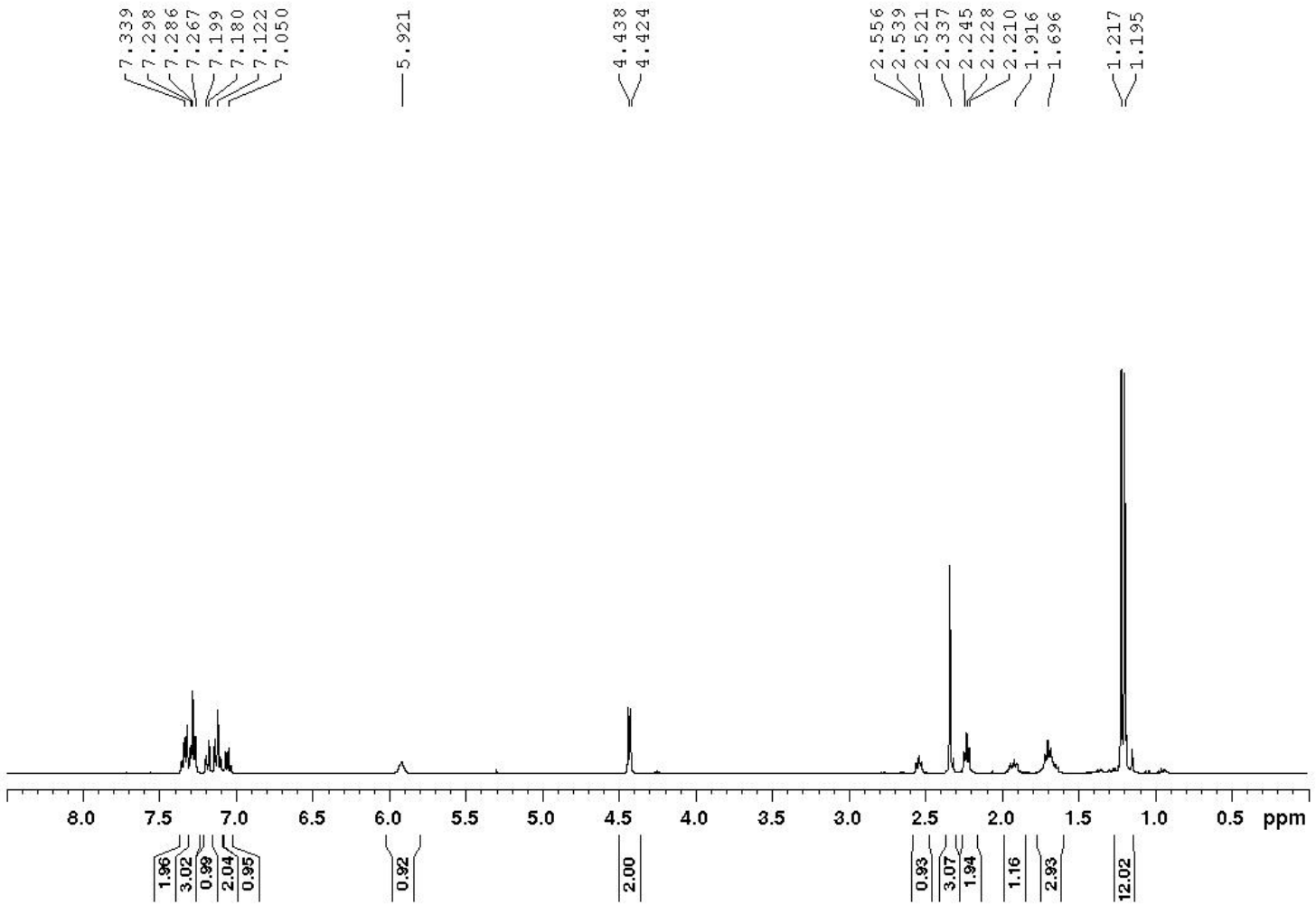


4j

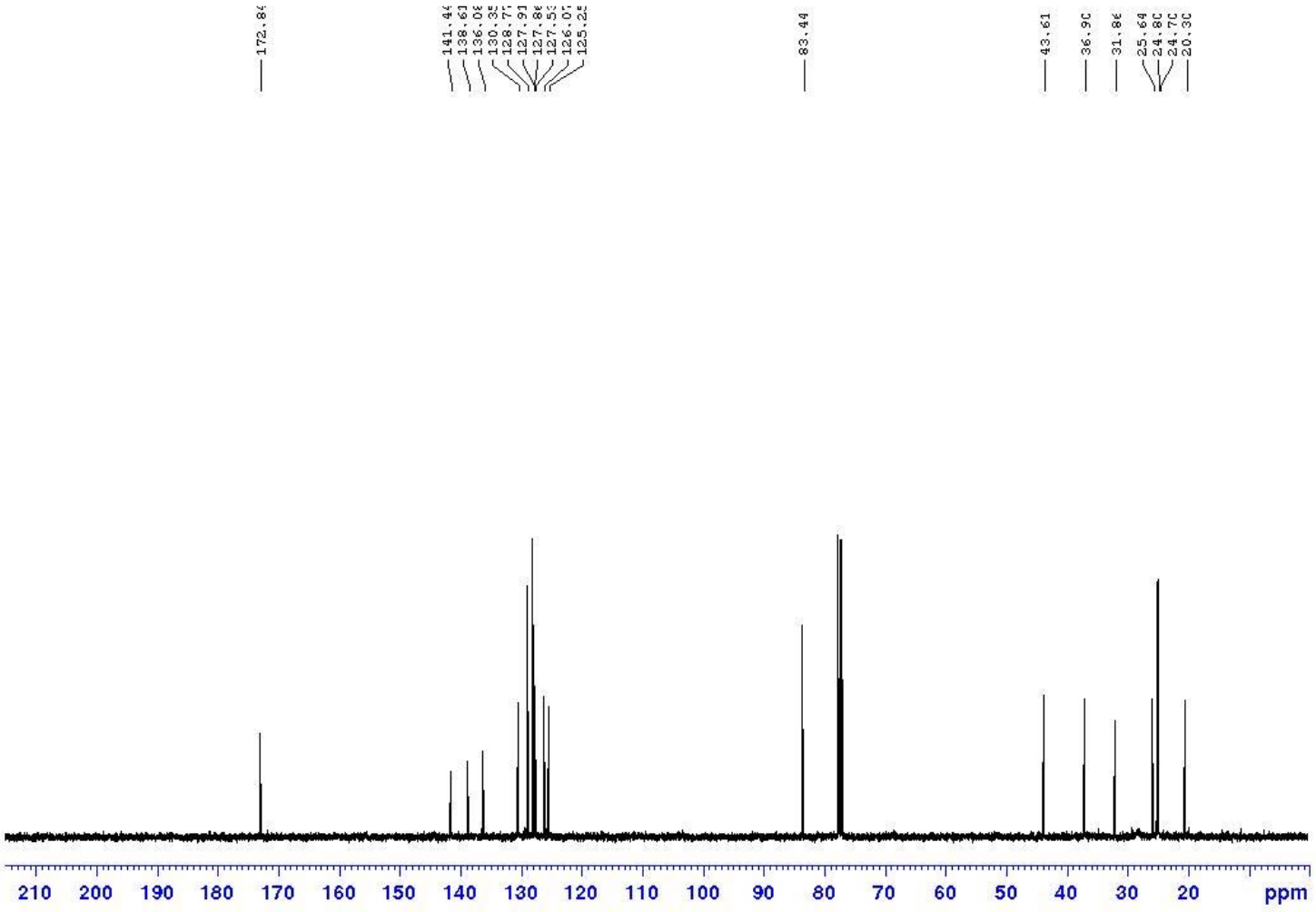


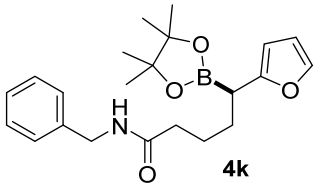
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.30–7.35 (2H, m, b,b'), 7.25–7.30 (3H, m, c,c',o), 7.19 (1H, d, *J* = 7.5 Hz, a), 7.10–7.15 (2H, m, q,r), 7.00–7.10 (1H, m, p), 5.92 (1H, br s, NH), 4.43 (2H, d, *J* = 5.9 Hz, e), 2.54 (1H, t, *J* = 7.0 Hz, j), 2.34 (3H, s, s), 2.23 (2H, t, *J* = 6.8 Hz, g), 1.85–2.00 (1H, m, h), 1.60–1.80 (3H, m, h,i), 1.22 and 1.20 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.84 (f), 141.44 (m), 138.61 (d), 136.08 (n), 130.35 (o), 128.77 (b,b'), 127.91 (c,c,'), 127.86 (r), 127.53 (a), 126.07 (p), 125.25 (q), 83.44 (k,k'), 43.61 (e), 36.90 (g), 31.86 (h), 25.64 (i), 24.80 and 24.70 (l,l'), 20.30 (s)

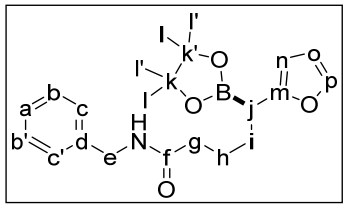
^{1}H NMR


7.339
7.298
7.286
7.267
7.199
7.180
7.122
7.050
5.921
4.438
4.424
2.556
2.539
2.521
2.337
2.245
2.228
2.210
1.916
1.696
1.217
1.195
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.96
3.02
0.99
2.04
0.95
0.92
2.00
0.93
3.07
1.94
1.16
2.93
12.02

^{13}C NMR

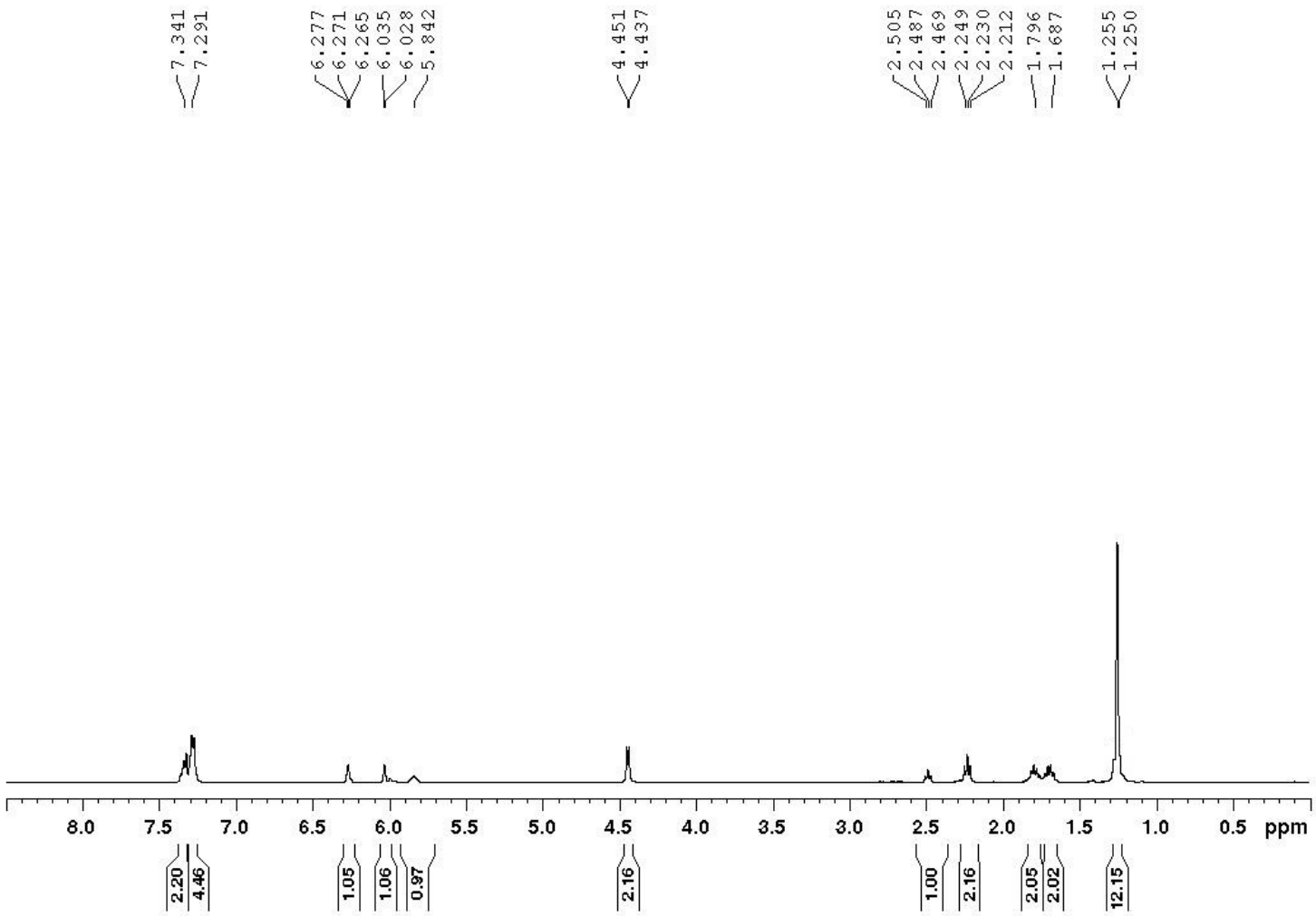

172.84
141.44
138.63
136.06
130.32
128.77
127.93
127.88
127.53
126.07
125.25
83.44
43.61
36.90
31.86
25.64
24.80
24.70
20.30
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



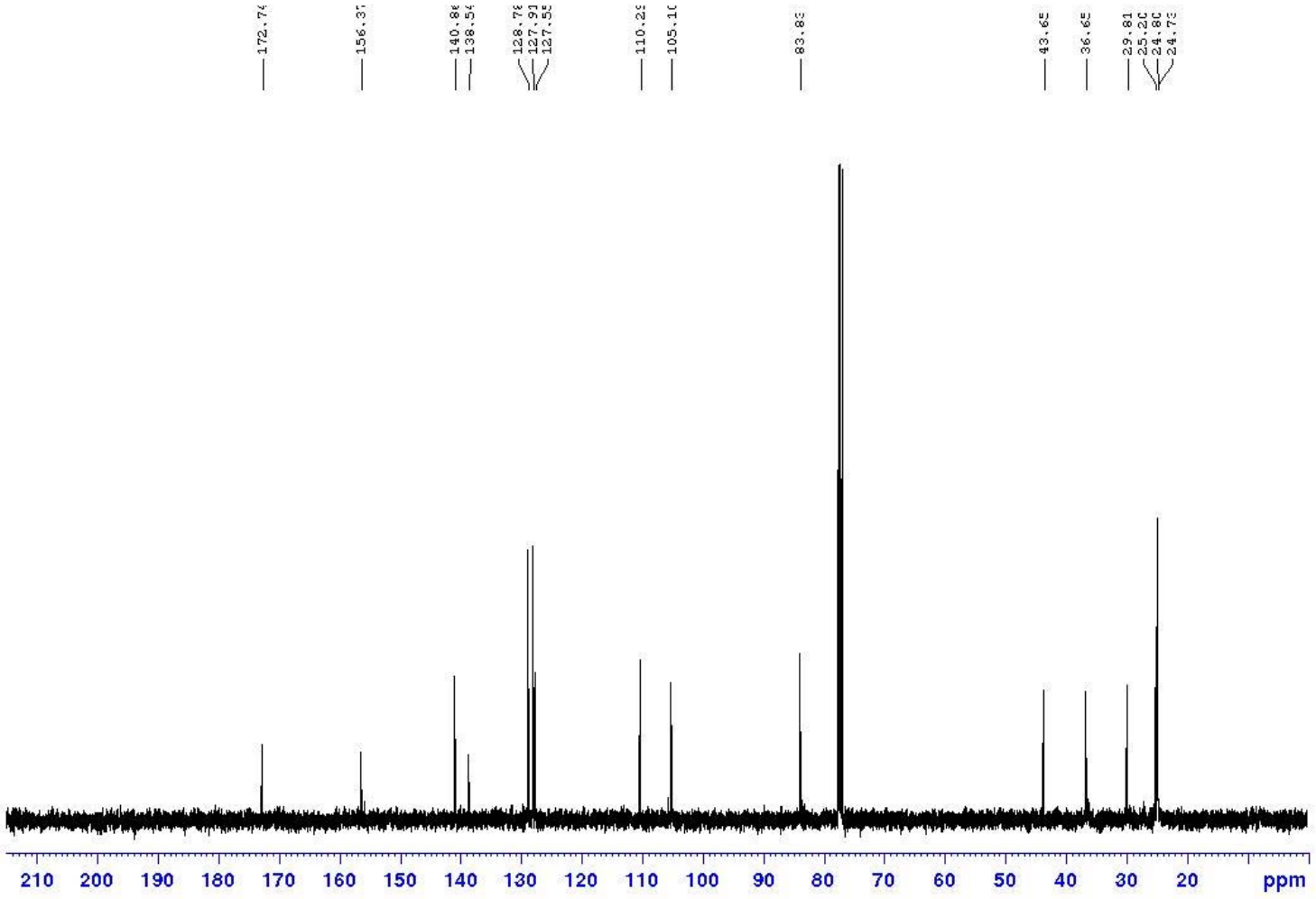


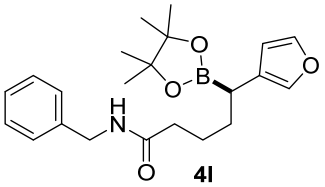
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.30–7.40 (2H, m, b,b'), 7.25–7.30 (4H, m, a,c,c',p), 6.27 (1H, t, *J* = 2.5 Hz, o), 6.03 (1H, d, *J* = 2.9 Hz, n), 5.84 (1H, br s, NH), 4.45 (2H, d, *J* = 5.6 Hz, e), 2.49 (1H, t, *J* = 7.1 Hz, j), 2.23 (2H, t, *J* = 7.4 Hz, g), 1.75–1.85 (2H, m, h), 1.65–1.75 (2H, m, i), 1.26 and 1.25 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.74 (f), 156.37 (m), 140.86 (p), 138.54 (d), 128.78 (b,b'), 127.91 (c,c,'), 127.55 (a), 110.29 (o), 105.10 (n), 83.83 (k,k'), 43.85 (e), 36.65 (g), 29.81 (h), 25.20 (i), 24.80 and 24.73 (l,l')

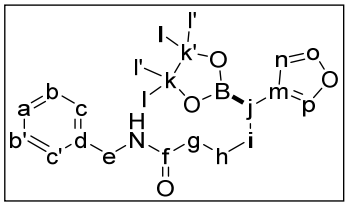
^{1}H NMR


7.341
7.291
6.277
6.271
6.265
6.035
6.028
5.842
4.451
4.437
2.505
2.487
2.469
2.249
2.230
2.212
1.796
1.687
1.255
1.250
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.20
4.46
1.05
1.06
0.97
2.16
1.00
2.16
2.05
2.02
12.15

^{13}C NMR

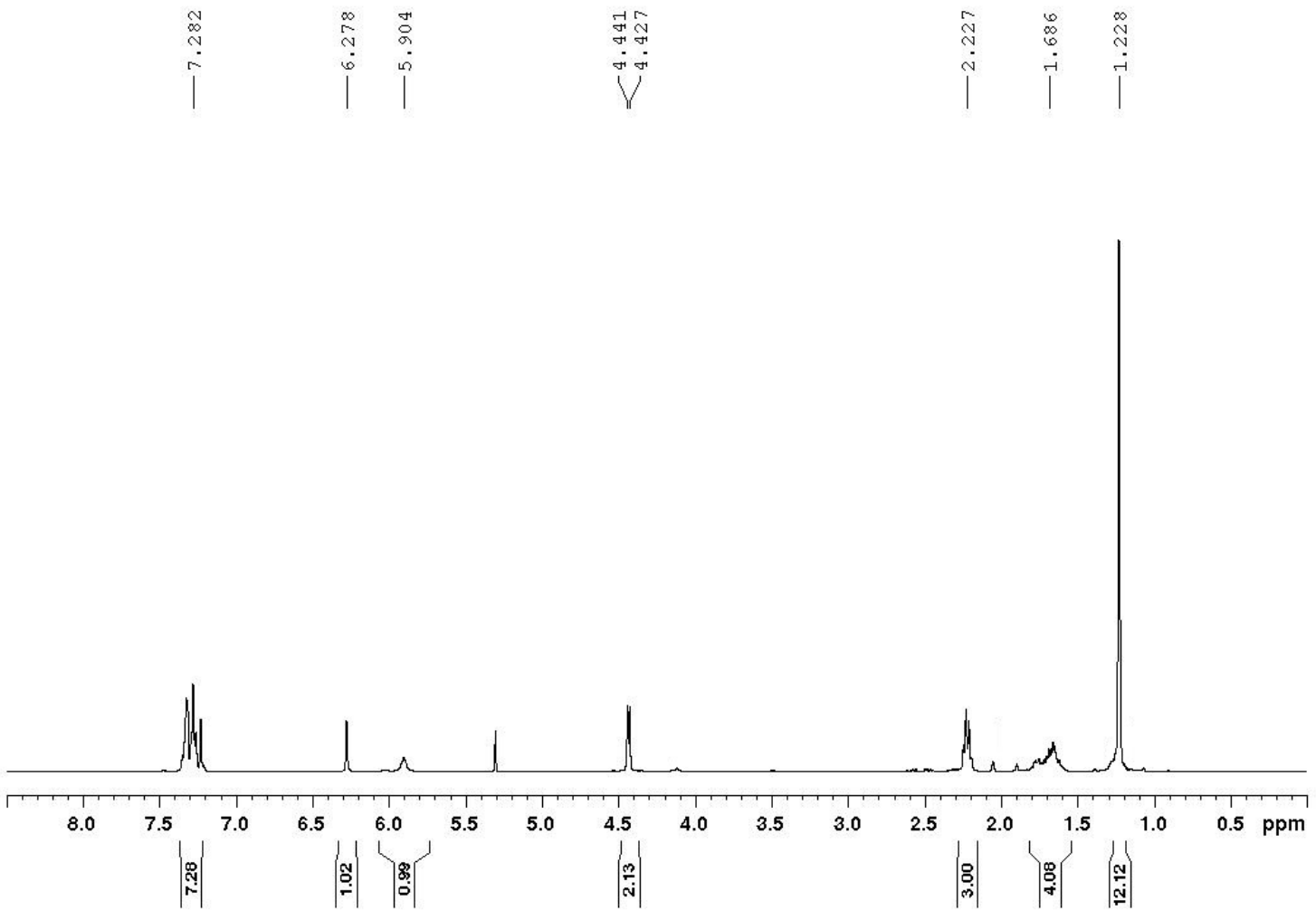

172.74
156.37
140.88
138.54
128.78
127.93
127.55
110.25
105.10
83.83
43.65
36.65
29.81
25.20
24.80
24.73
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



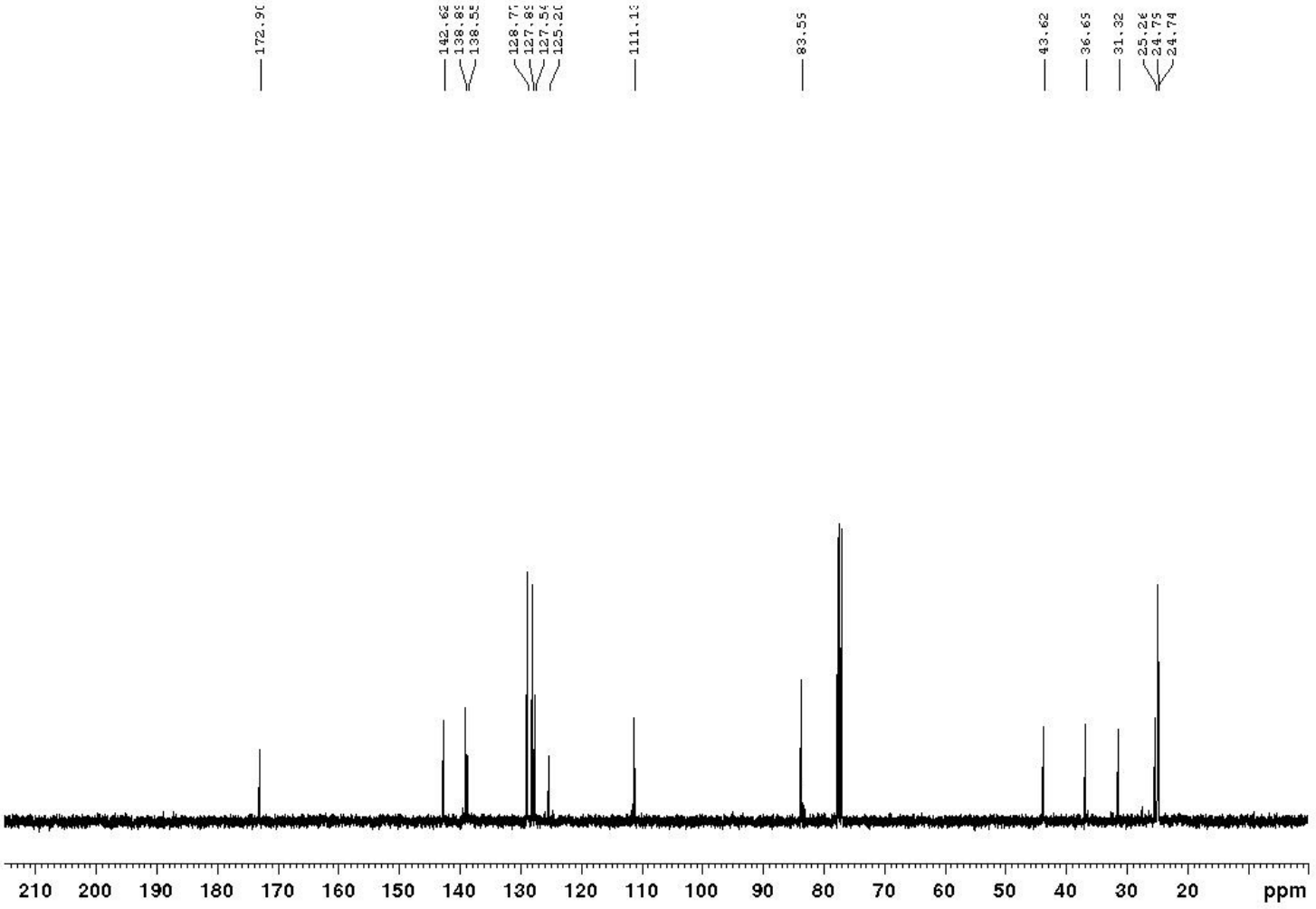


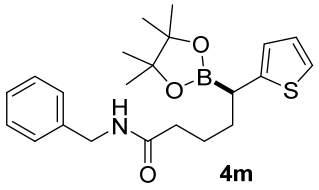
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (7H, m, a,b,b',c,c',o,p), 6.28 (1H, d, J = 0.0 Hz, n), 5.90 (1H, br s, NH), 4.43 (2H, d, J = 5.6 Hz, e), 2.15–2.30 (3H, m, g,j), 1.60–1.80 (2H, m, h,i), 1.23 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.90 (f), 142.62 (o), 138.89 (p), 138.55 (d), 128.77 (b,b'), 127.89 (c,c,'), 127.54 (a), 125.20 (m), 111.13 (n), 83.59 (k,k'), 43.62 (e), 36.69 (g), 31.32 (h), 25.26 (i), 24.79 and 24.74 (l,l')

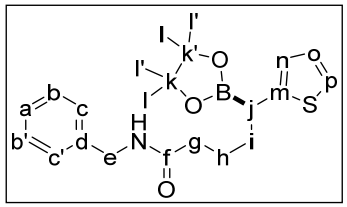
^{1}H NMR


7.282
6.278
5.904
4.441
4.427
2.227
1.686
1.228
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
7.28
1.02
0.99
2.13
3.00
4.08
12.12

^{13}C NMR

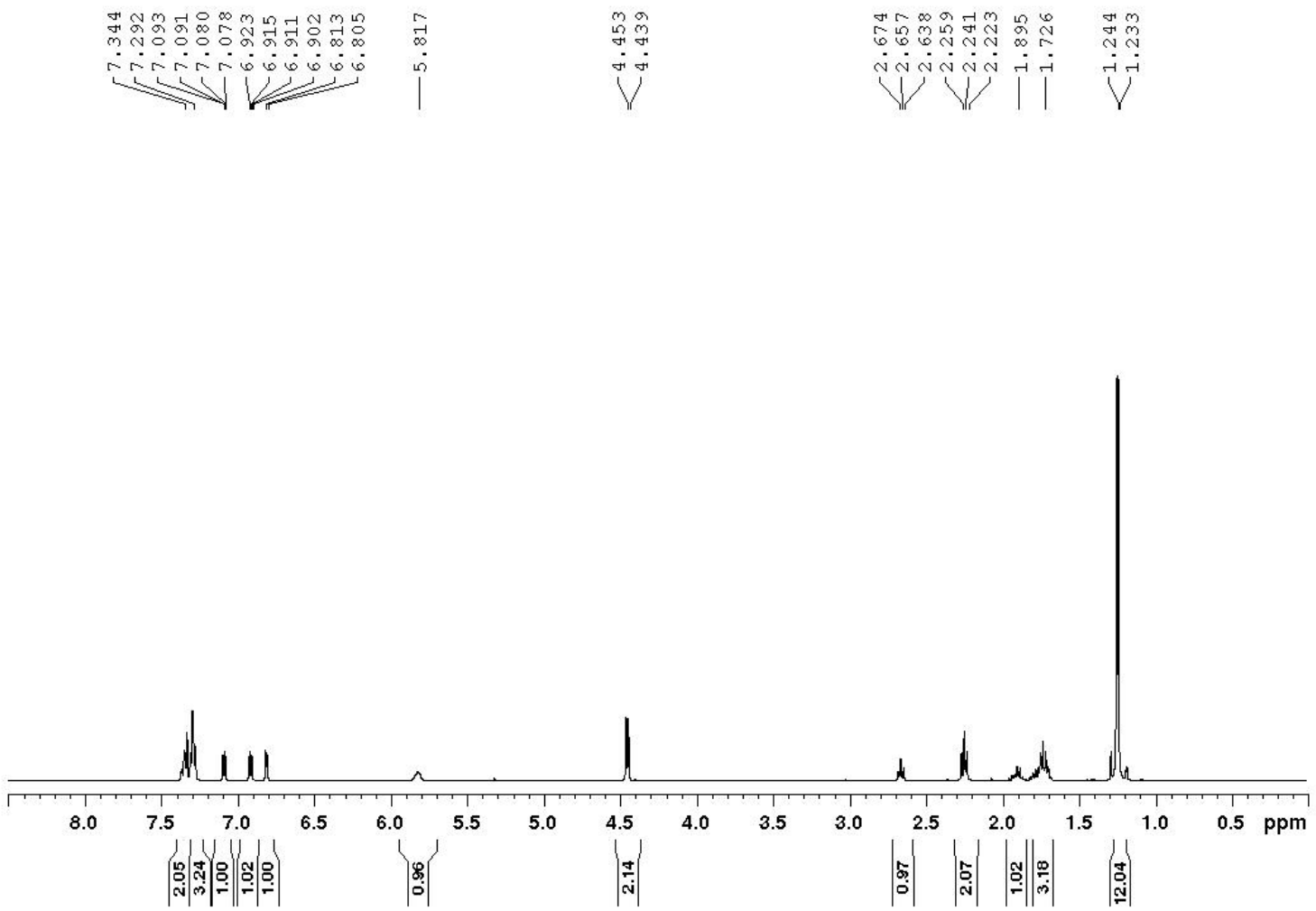

172.90
142.62
138.85
138.55
128.77
127.85
127.54
125.20
111.13
83.55
43.62
36.65
31.32
25.26
24.75
24.74
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



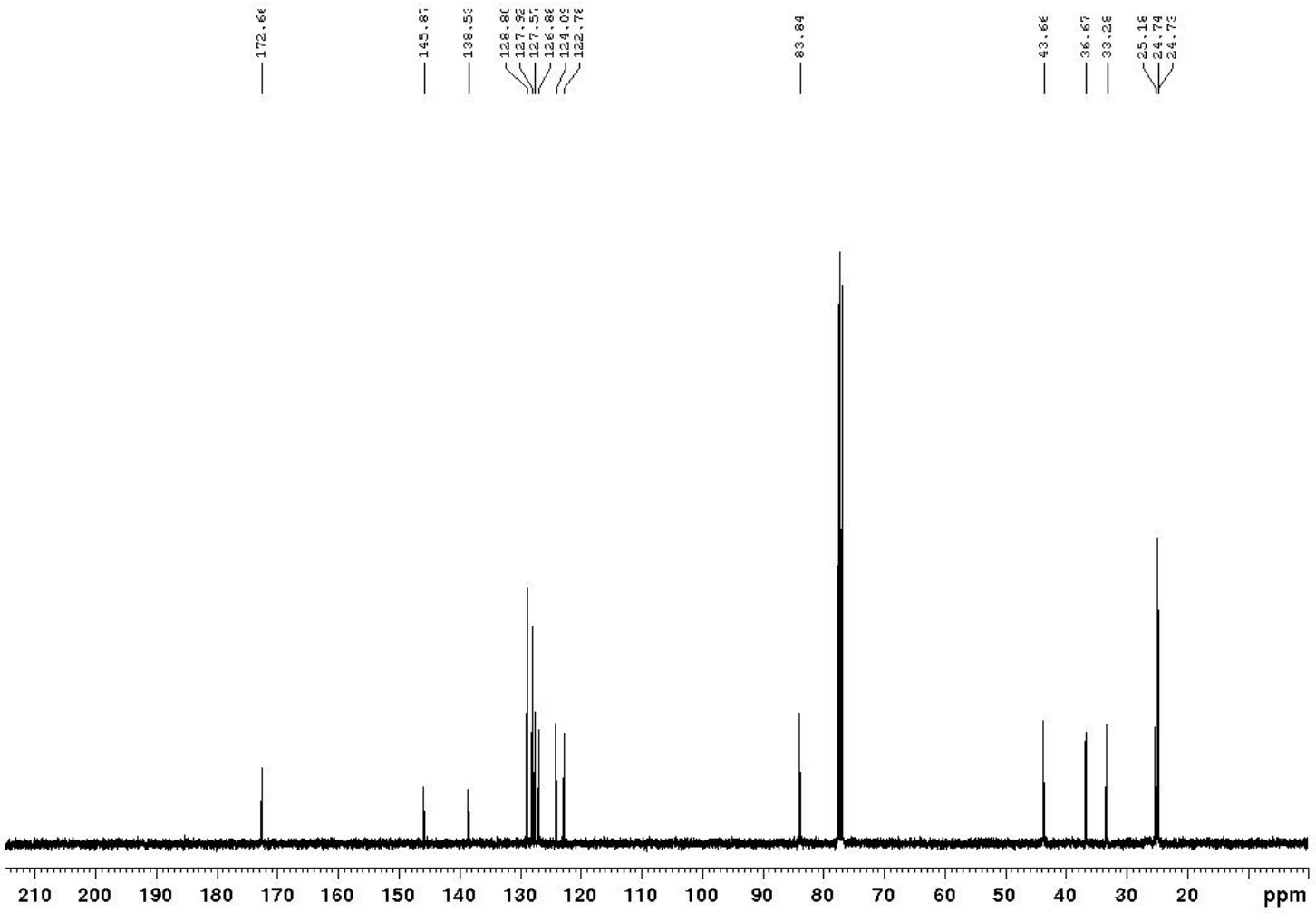


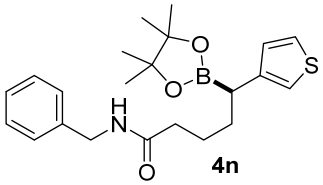
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.30–7.40 (2H, m, b,b'), 7.25–7.30 (3H, m, a,c,c'), 7.09 (1H, dd, *J* = 5.1 and 0.9 Hz, p), 6.91 (1H, dd, *J* = 5.1 and 3.4 Hz, o), 6.81 (1H, d, *J* = 3.4 Hz, n), 5.82 (1H, br s, NH), 4.44 (2H, d, *J* = 5.7 Hz, e), 2.66 (1H, t, *J* = 7.1 Hz, j), 2.24 (2H, t, *J* = 7.3 Hz, g), 1.85–1.95 (1H, m, h), 1.65–1.80 (3H, m, h,i), 1.24 and 1.23 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.66 (f), 145.87 (m), 138.53 (d), 128.80 (b,b'), 127.92 (c,c,'), 127.57 (a), 126.88 (o), 124.09 (n), 122.78 (p), 83.84 (k,k'), 43.66 (e), 36.67 (g), 33.28 (h), 25.18 (i), 24.74 and 24.73 (l,l')

^{1}H NMR

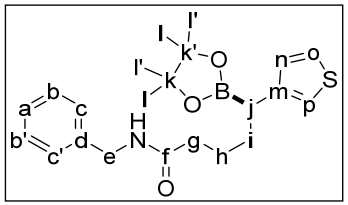

7.344
7.292
7.093
7.091
7.080
7.078
6.923
6.915
6.911
6.902
6.813
6.805
5.817
4.453
4.439
2.674
2.657
2.638
2.259
2.241
2.223
1.895
1.726
1.244
1.233
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.05
3.24
1.00
1.02
1.00
0.96
2.14
0.97
2.07
1.02
3.18
12.04

^{13}C NMR


172.6
145.8
138.5
128.8
127.9
127.5
126.8
124.0
122.7
83.84
43.66
36.67
33.28
25.18
24.79
24.73
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

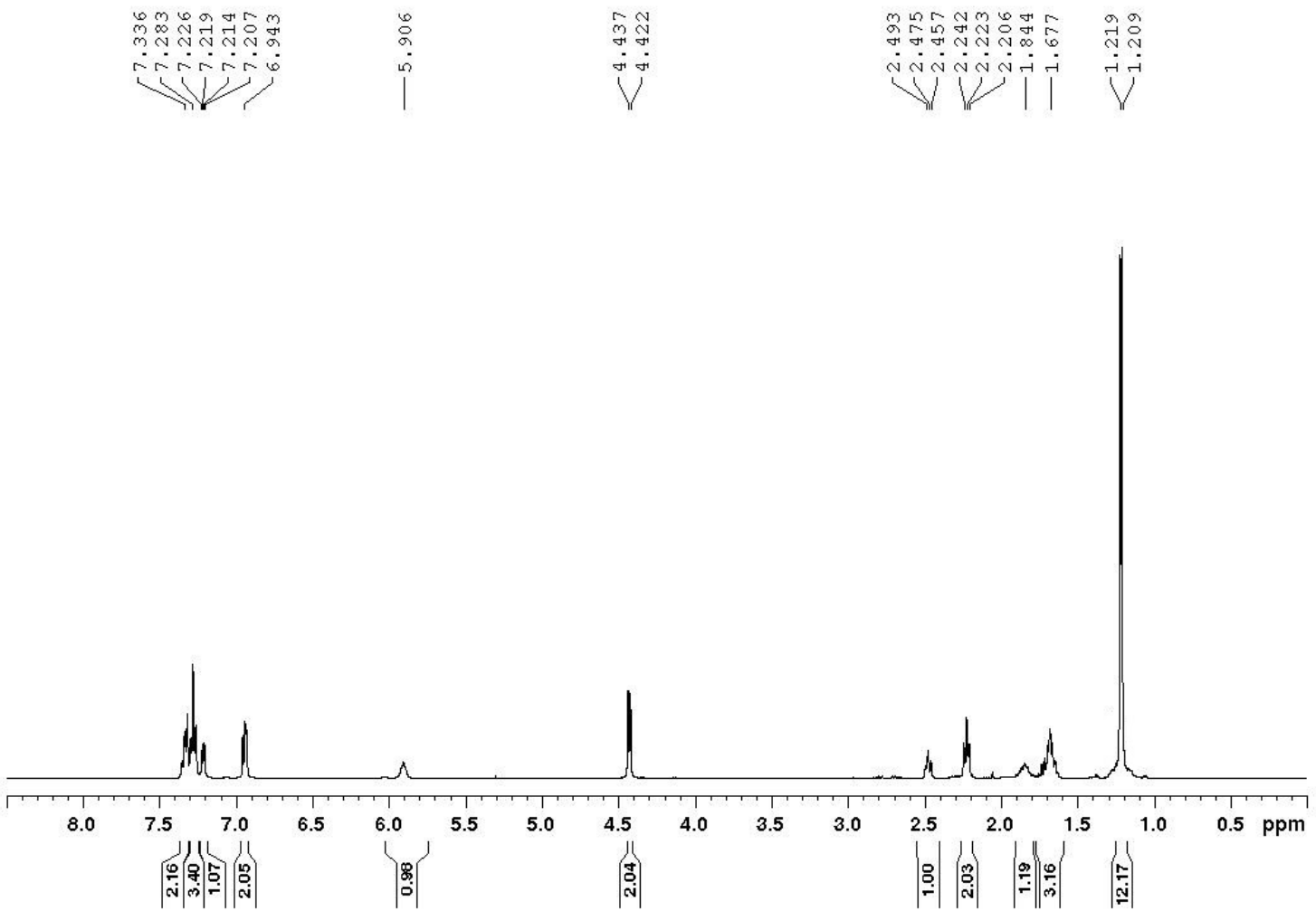


4n

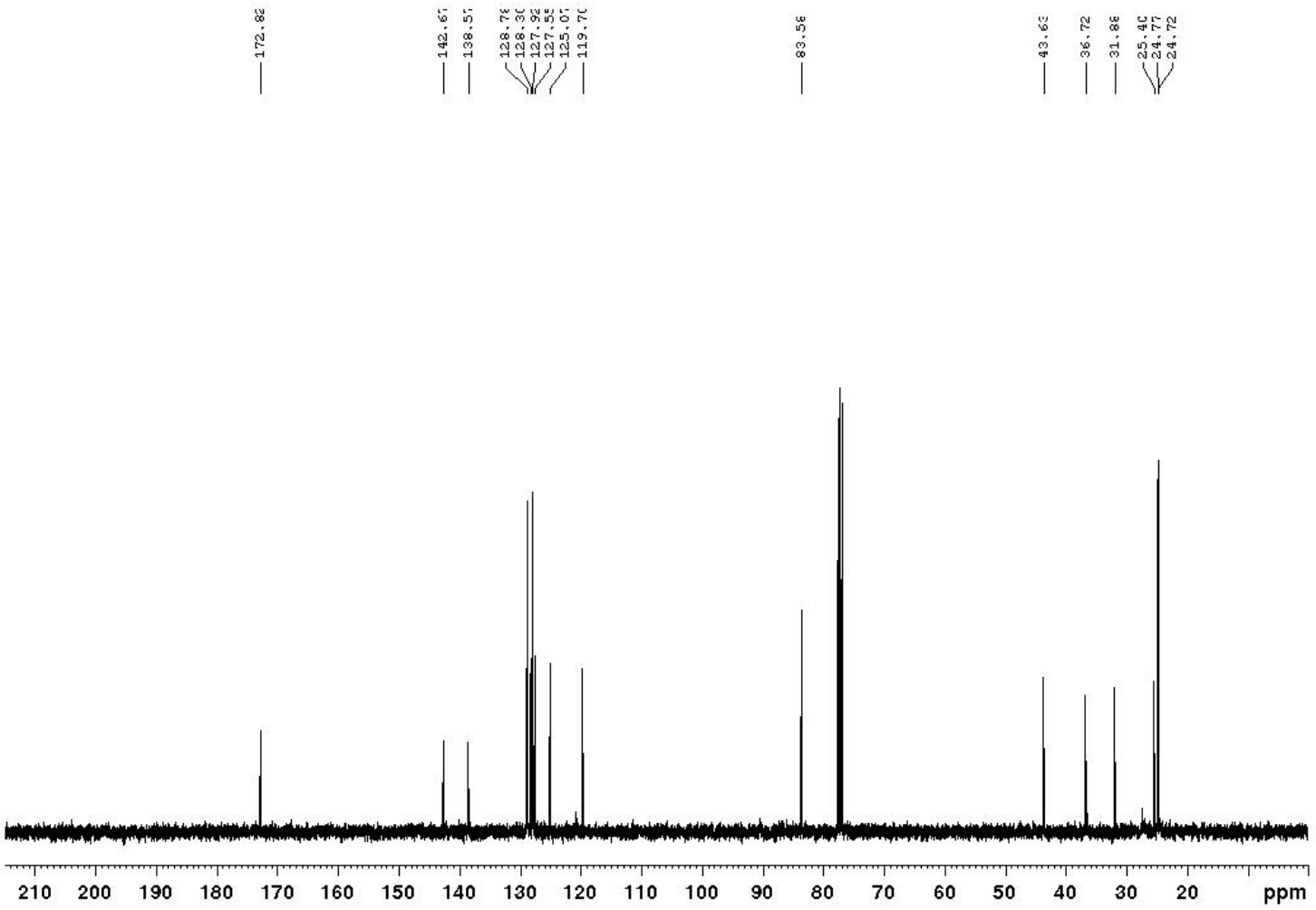


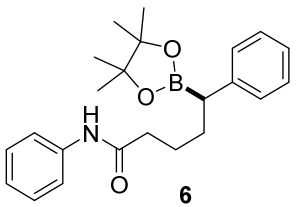
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.30–7.35 (2H, m, b,b'), 7.25–7.30 (3H, m, a,c,c'), 7.22 (1H, dd, *J* = 4.8 and 3.0 Hz, o), 6.90–7.00 (2H, m, n,p), 5.91 (1H, br s, NH), 4.43 (2H, d, *J* = 5.7 Hz, e), 2.48 (1H, t, *J* = 7.1 Hz, j), 2.22 (2H, t, *J* = 7.4 Hz, g), 1.80–1.90 (1H, m, h), 1.60–1.80 (3H, m, h,i), 1.22 and 1.21 (12H, s, l,l')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.82 (f), 142.67 (m), 138.57 (d), 128.78 (b,b'), 128.30 (n), 127.92 (c,c,'), 127.55 (a), 125.07 (o), 119.70 (p), 83.58 (k,k'), 43.63 (e), 36.72 (g), 31.88 (h), 25.40 (i), 24.77 and 24.72 (l,l')

^{1}H NMR

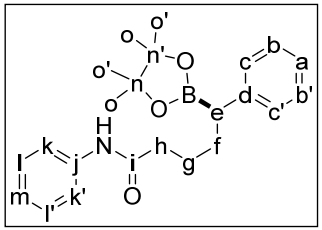

7.336
7.283
7.226
7.219
7.214
7.207
6.943
5.906
4.437
4.422
2.493
2.475
2.457
2.242
2.223
2.206
1.844
1.677
1.219
1.209
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.16
3.40
1.07
2.05
0.98
2.04
1.00
2.03
1.19
3.16
12.17

^{13}C NMR


172.82
142.67
138.57
128.78
128.30
127.98
127.58
125.07
119.70
83.58
43.68
36.72
31.88
25.40
24.77
24.72
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

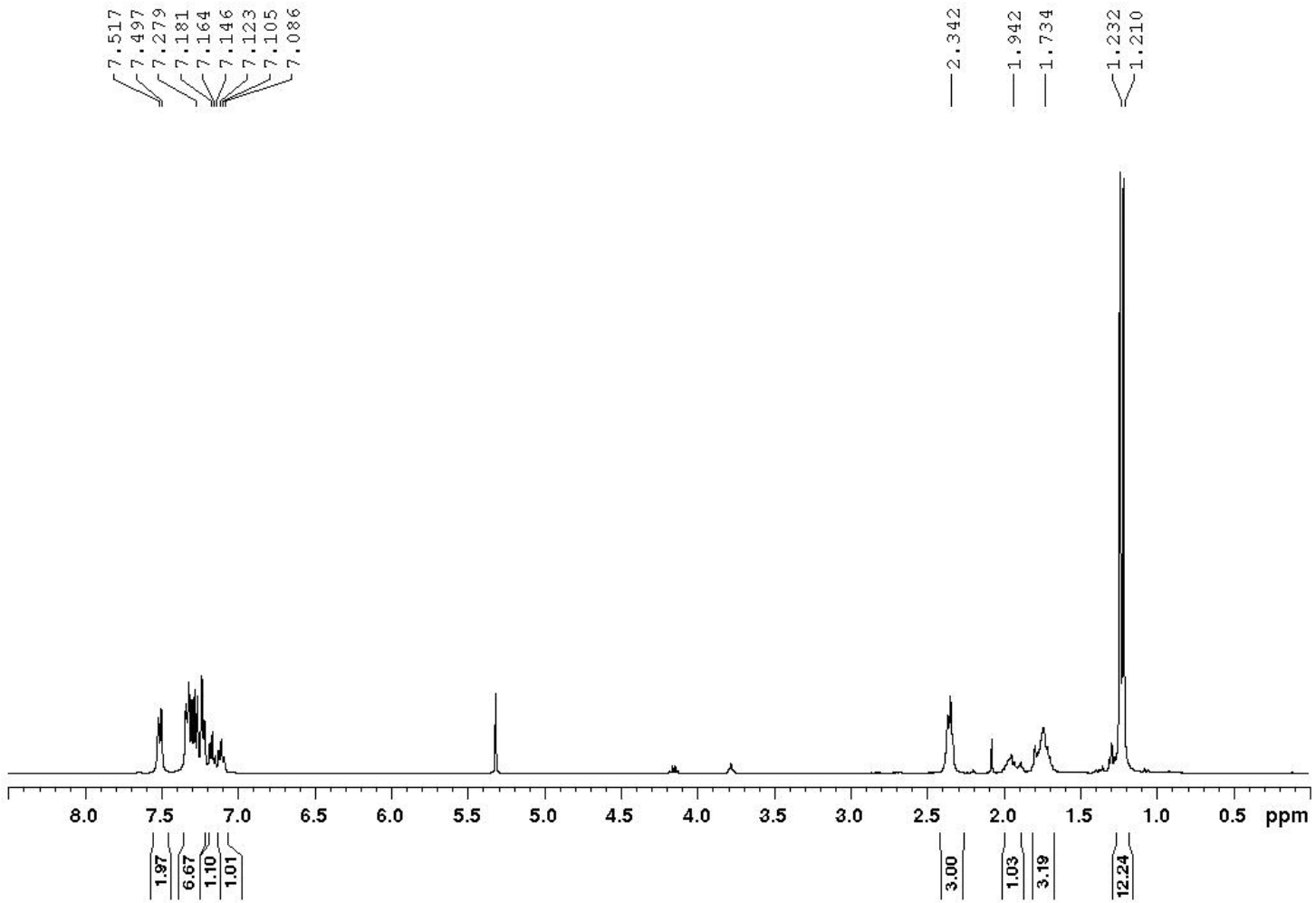


6

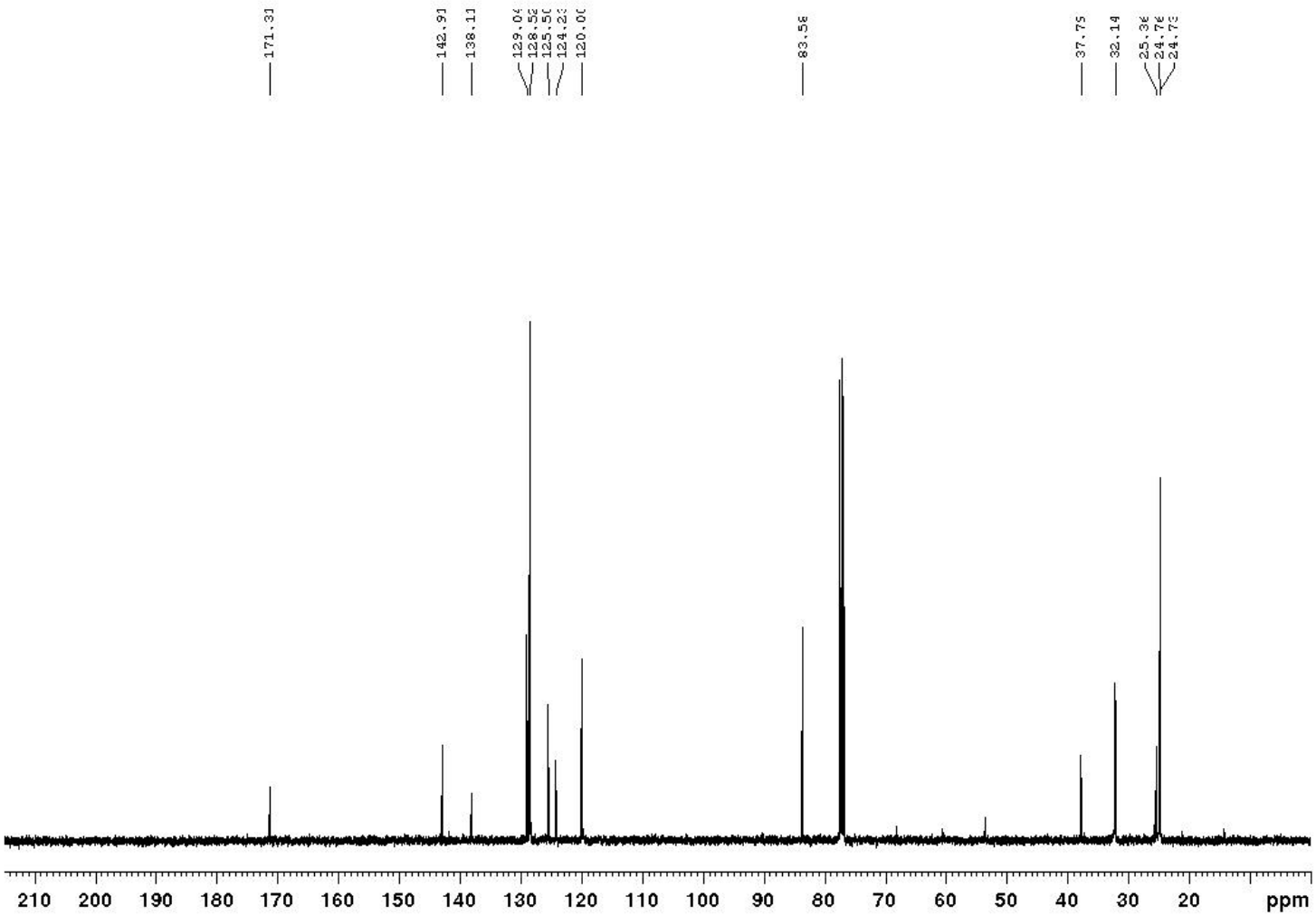


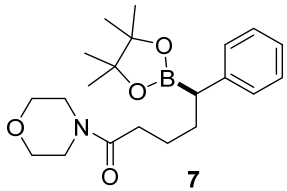
TLC analysis	R_f 0.6 (50:50 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.51 (2H, d, *J* = 7.8 Hz, k,k'), 7.20–7.35 (6H, m, b,b'c,c',l,l'), 7.16 (1H, t, *J* = 7.1 Hz, a), 7.11 (1H, t, *J* = 7.1 Hz, m), 2.30–2.40 (3H, e,h), 1.85–2.00 (1H, m, g), 1.65–1.80 (3H, m, f,g), 1.23 and 1.21 (12H, s, o,o')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.31 (i), 142.91 (d), 138.11 (j), 129.04 (l,l'), 128.52 (b,b',c,c'), 125.49 (a), 124.23 (m), 120.00 (k,k'), 83.58 (n,n'), 37.79 (g), 32.14 (h), 25.36 (f), 24.76 and 24.73 (o,o')

^{1}H NMR

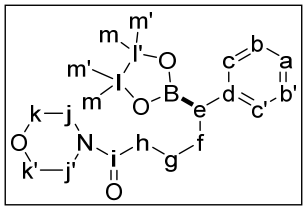

7.517
7.497
7.279
7.181
7.164
7.146
7.123
7.105
7.086
2.342
1.942
1.734
1.232
1.210
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.97
6.67
1.10
1.01
3.00
1.03
3.19
12.24

^{13}C NMR


171.31
142.93
138.11
129.04
128.56
125.50
124.23
120.00
83.56
37.75
32.14
25.36
24.78
24.73
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

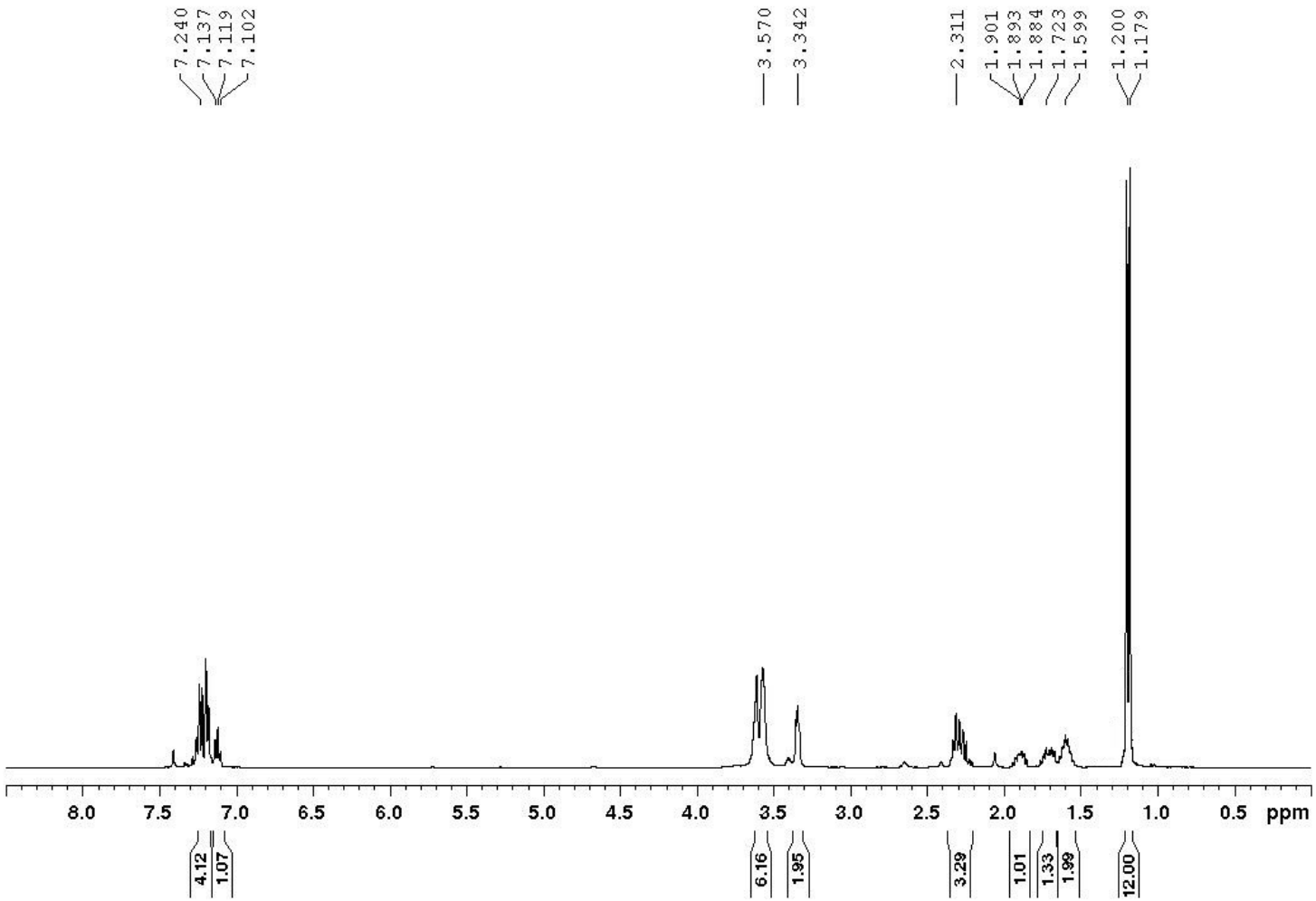


7

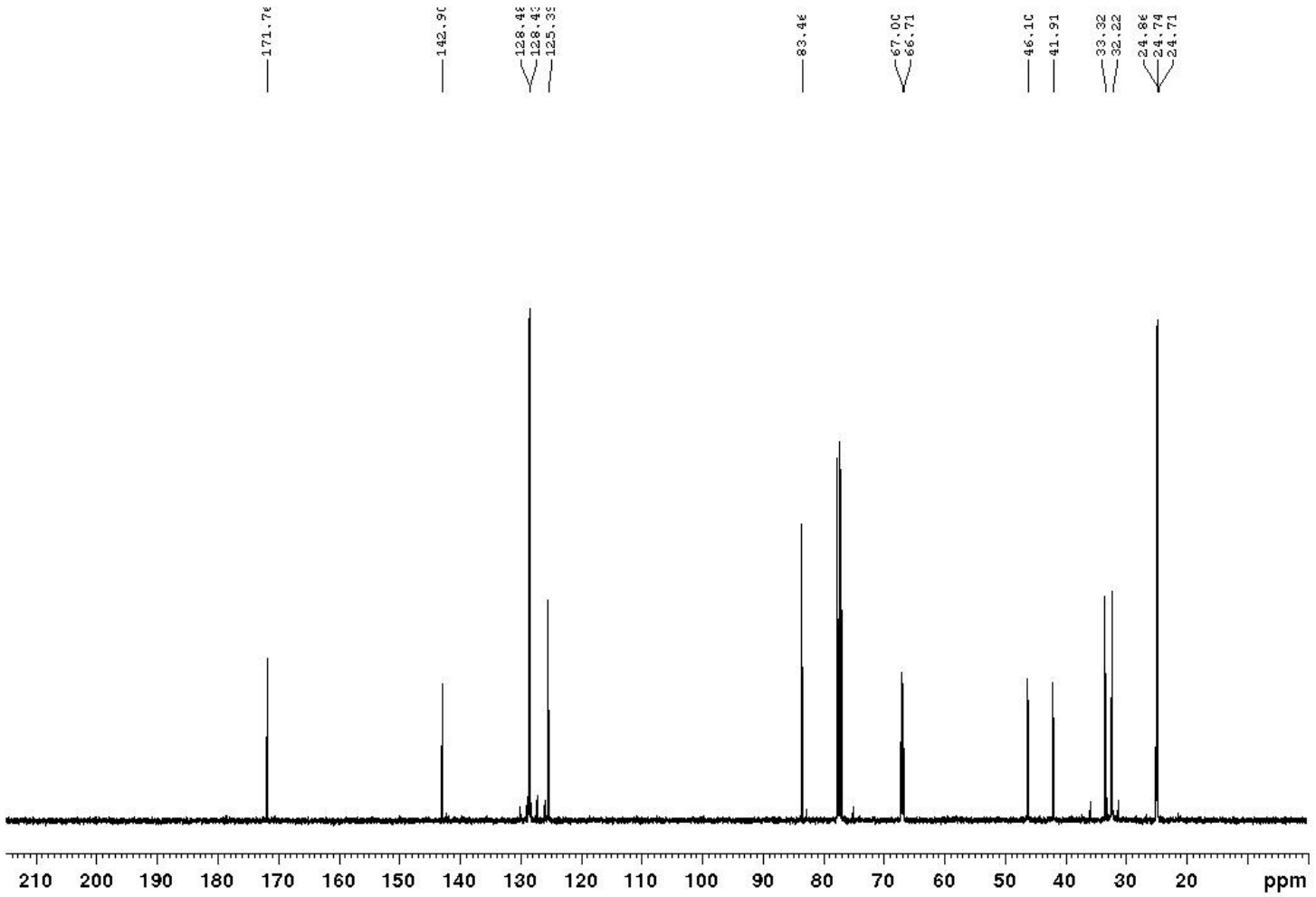


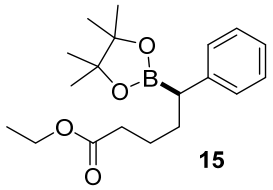
TLC analysis	R_f 0.4 (30:70 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.15–7.25 (4H, m, b,b',c,c'), 7.12 (1H, t, *J* = 7.0 Hz, a), 3.55–3.65 (6H, m, j,j',k,k'), 3.30–3.35 (2H, m, j,j'), 2.20–2.35 (3H, m, e,h), 1.85–1.95 (1H, m, g), 1.65–1.75 (1H, m, g), 1.55–1.65 (2H, m, f), 1.20 and 1.18 (12H, s, m,m')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.76 (i), 142.90 (d), 128.48 (b,b'), 128.43 (c,c'), 125.39 (a), 83.46 (l,l'), 67.00 and 67.71 (k,k'), 46.10 and 41.91 (j, j'), 33.32 (g), 32.22 (h), 24.86 (f), 24.74 and 24.71 (m,m')

^{1}H NMR

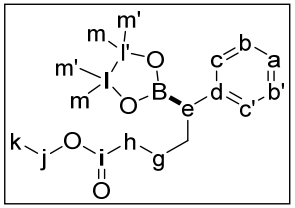

7.240
7.137
7.119
7.102
3.570
3.342
2.311
1.901
1.893
1.884
1.723
1.599
1.200
1.179
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.12
1.07
6.16
1.95
3.29
1.01
1.33
1.99
12.00

^{13}C NMR


171.7
142.9
128.4
128.4
125.3
83.4
67.0
66.7
46.1
41.9
33.3
32.2
24.8
24.7
24.7
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

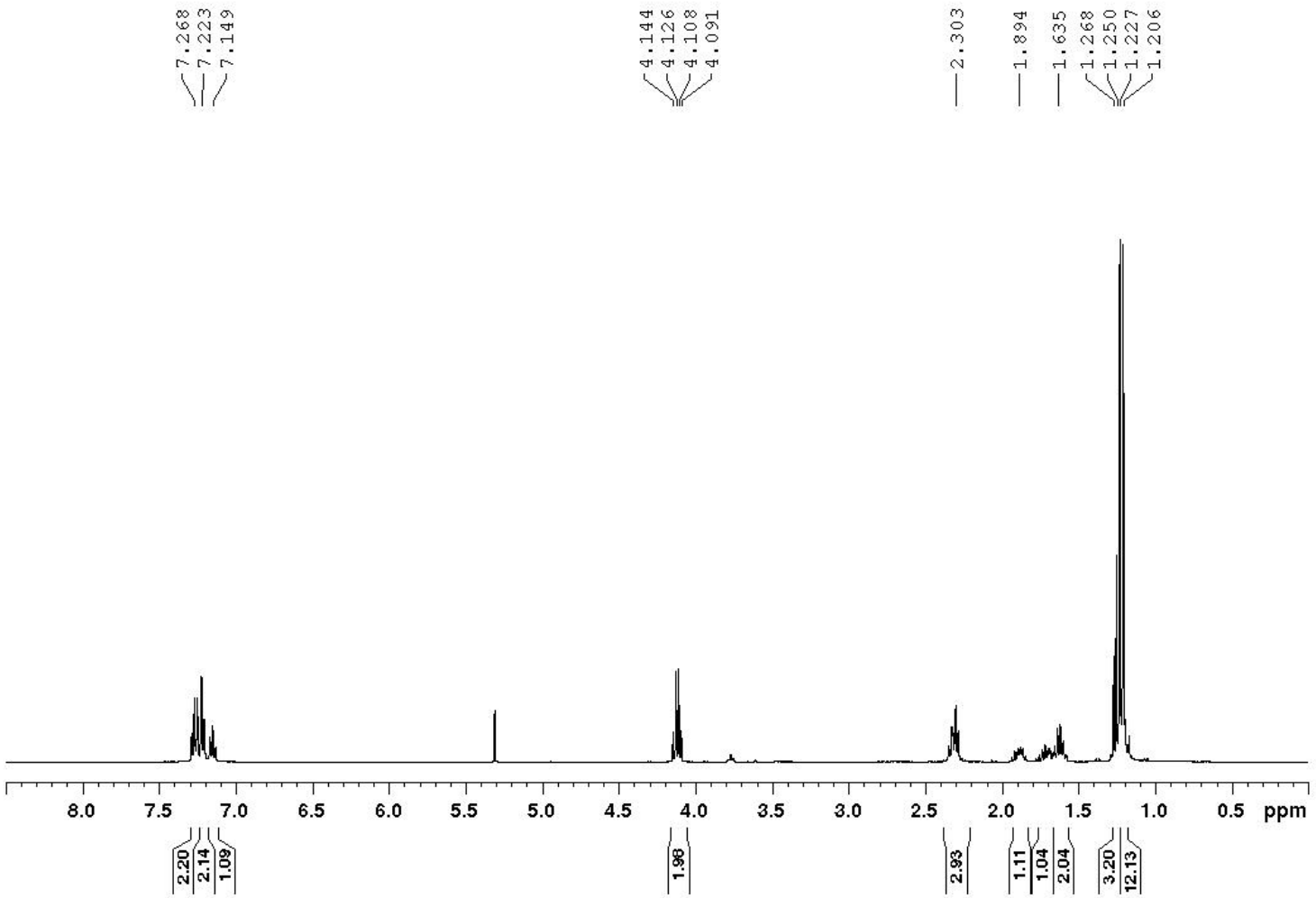


15

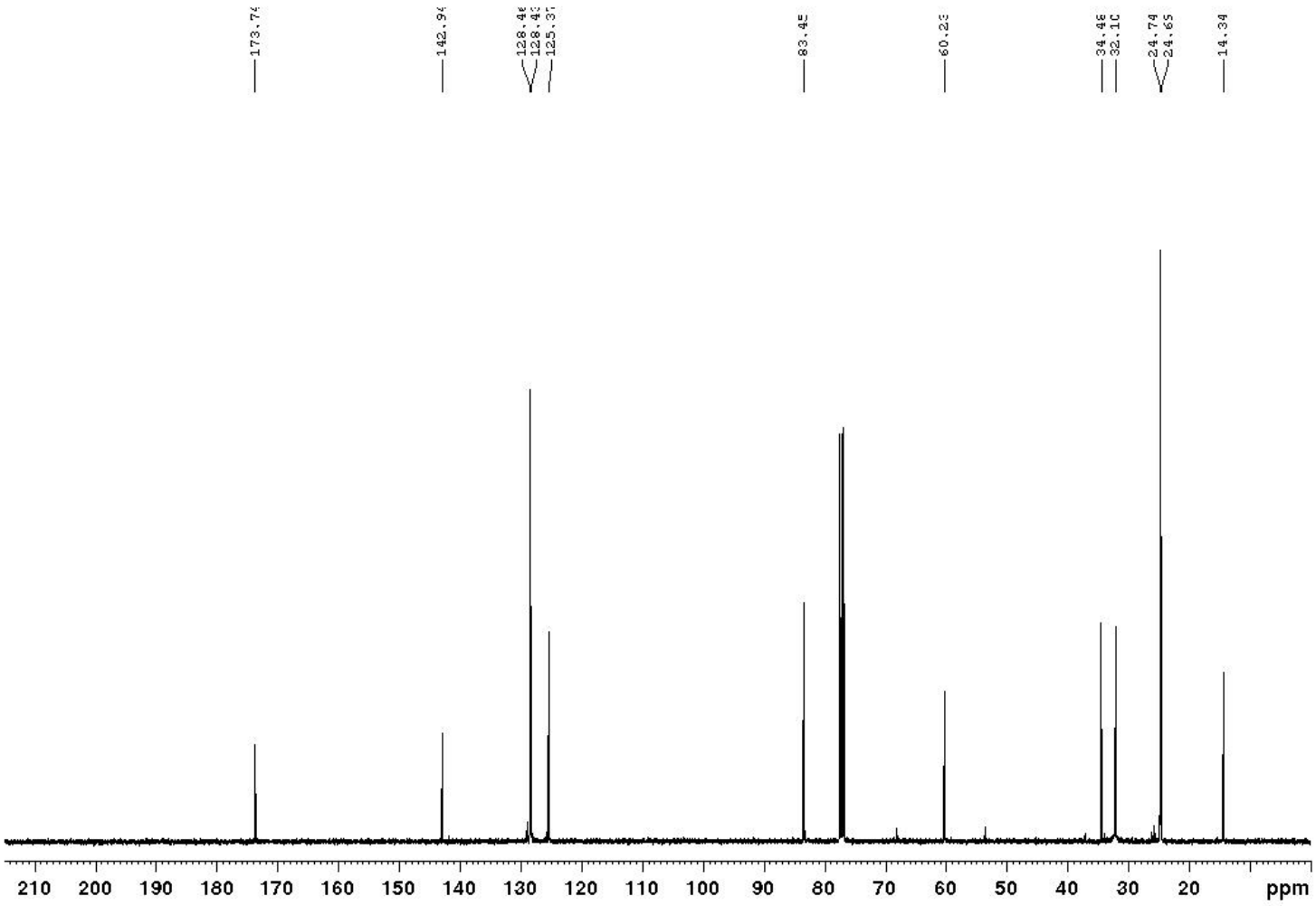


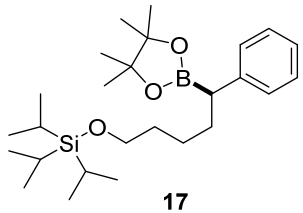
TLC analysis	R_f 0.7 (80:20 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.30 (2H, m, b,b'), 7.15–7.25 (2H, m, c,c'), 7.10–7.15 (1H, m, a), 4.12 (2H, q, *J* = 7.1 Hz, j), 2.20–2.40 (3H, m, e,h), 1.85–1.95 (1H, m, g), 1.65–1.75 (1H, m, g), 1.55–1.65 (2H, m, f), 1.25 (3H, t, *J* = 7.2 Hz, k), 1.23 and 1.21 (12H, s, m,m')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.74 (i), 142.94 (d), 128.46 (b,b'), 128.43 (c,c'), 125.37 (a), 83.45 (l,l'), 60.23 (j), 34.48 (g), 32.10 (h), 24.74 and 24.69 (f,m,m'), 14.34 (k)

^{1}H NMR

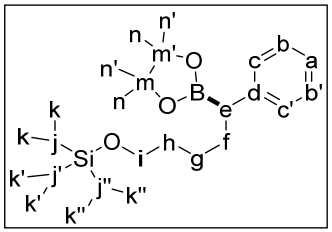

7.268
7.223
7.149
4.144
4.126
4.108
4.091
2.303
1.894
1.635
1.268
1.250
1.227
1.206
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.20
2.14
1.09
1.98
2.93
1.11
1.04
2.04
3.20
12.13

^{13}C NMR


173.74
142.94
128.46
128.41
125.31
83.45
60.23
34.46
32.10
24.74
24.65
14.34
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

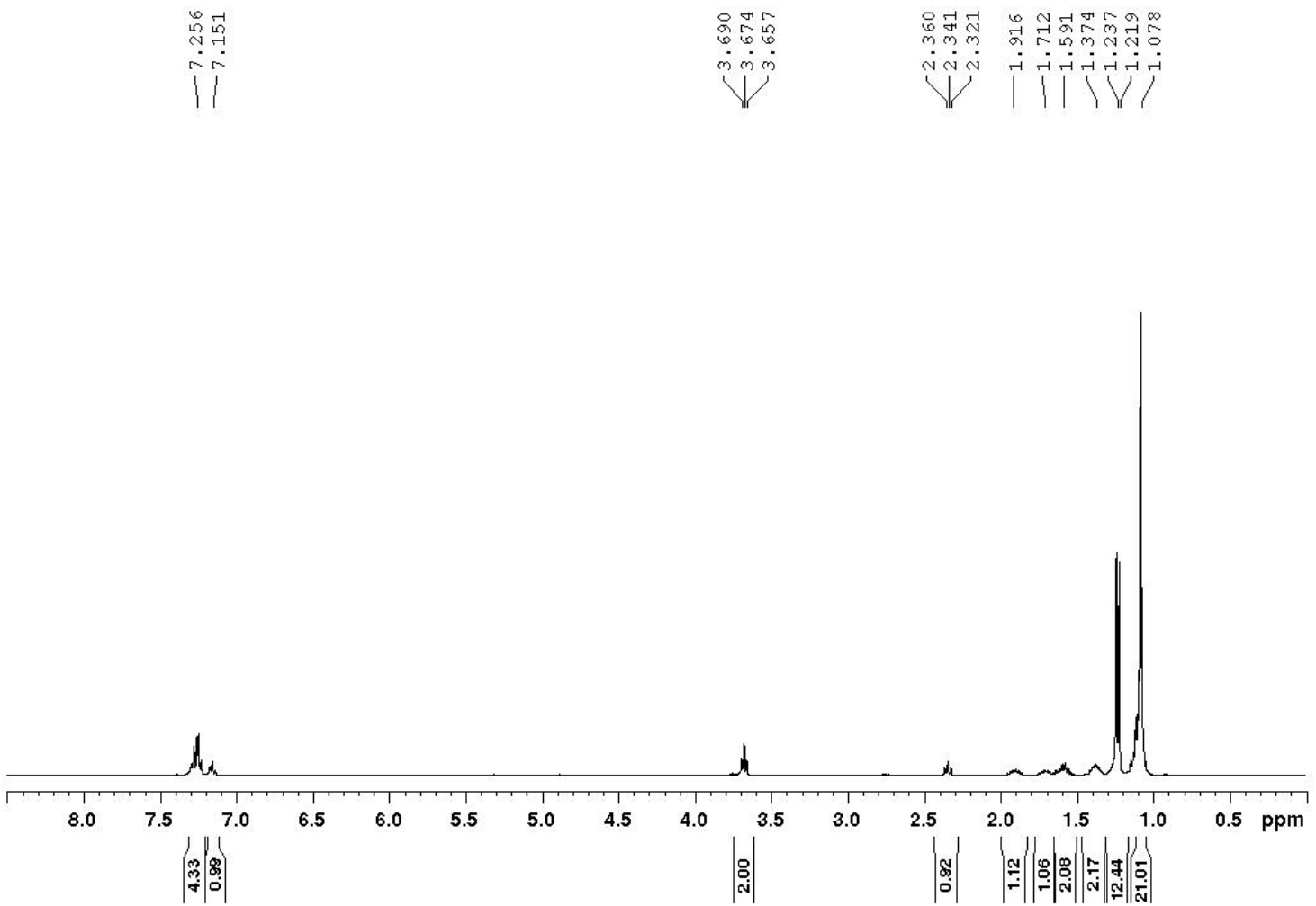


17

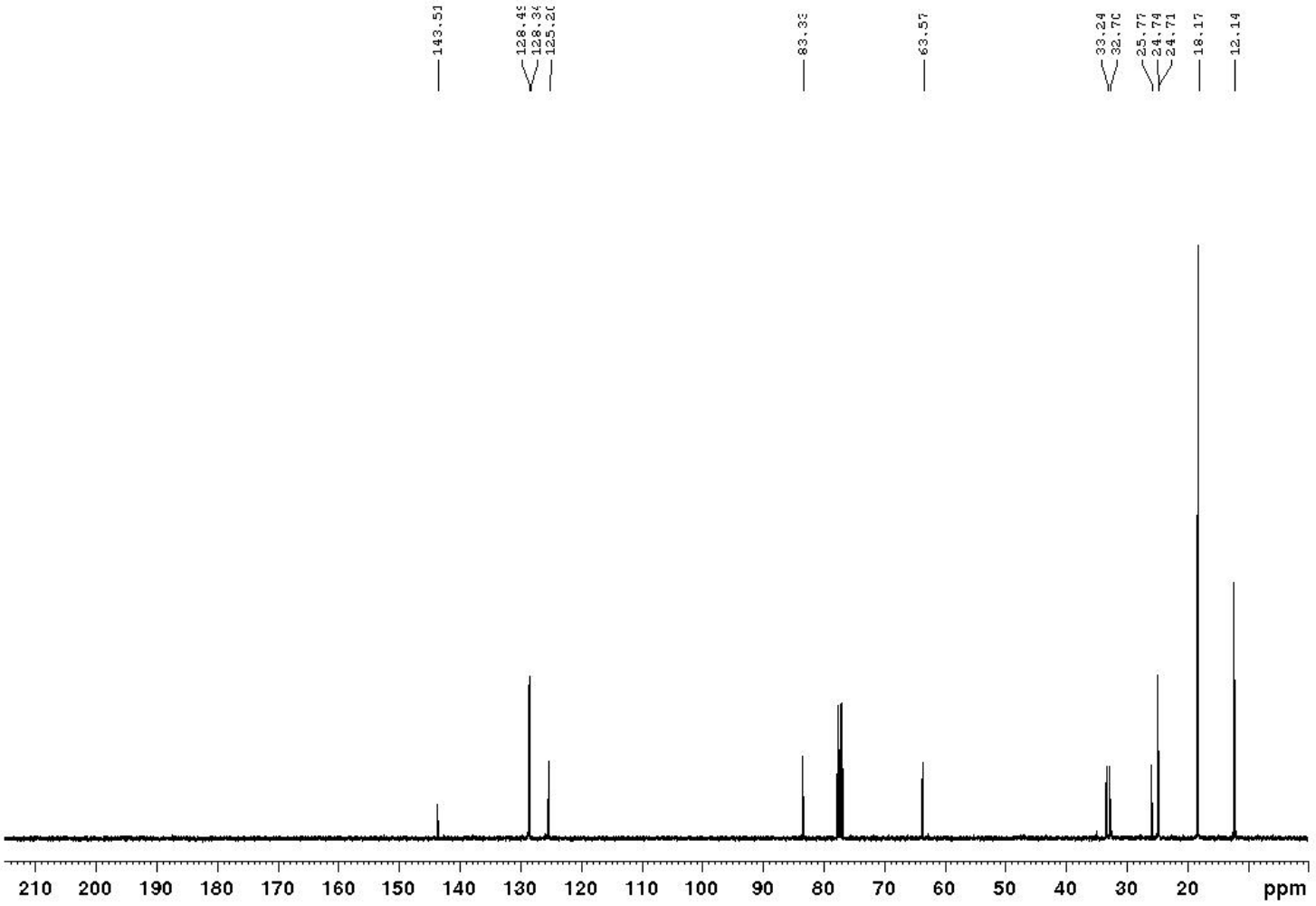


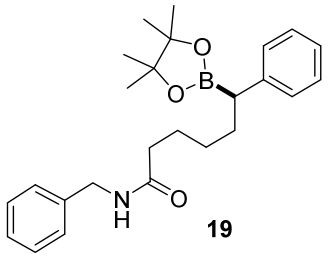
TLC analysis	R_f 0.7 (80:20 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.30 (4H, m, b,b’,c,c’), 7.10–7.20 (1H, m, a), 3.67 (2H, t, *J* = 6.6 Hz, l), 2.34 (1H, t, *J* = 7.9 Hz, e), 1.85–2.00 (1H, m, h), 1.65–1.75 (1H, m, h), 1.50–1.65 (2H, m, g), 1.30–1.45 (2H, m, f), 1.24 and 1.22 (12H, s, n,n’), 1.05–1.10 (21H, m, j,j’,j’’,k,k’,k’’)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 143.51 (d), 128.49 (b,b’), 128.34 (c,c’), 125.20 (a), 83.33 (m,m’), 63.57 (l), 33.24 (g), 32.70 (h), 25.77 (f), 24.74 and 24.71 (n,n’), 18.17 (k,k’,k’’), 12.14 (j,j’,j’’)

^{1}H NMR

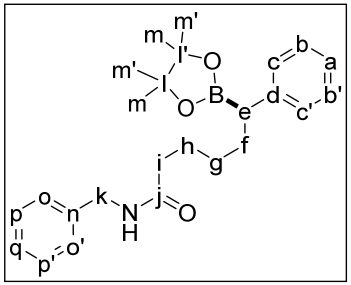

7.256
7.151
3.690
3.674
3.657
2.360
2.341
2.321
1.916
1.712
1.591
1.374
1.237
1.219
1.078
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.33
0.99
2.00
0.92
1.12
1.06
2.08
2.17
12.44
21.01

^{13}C NMR


143.51
128.45
128.34
125.20
83.38
63.57
33.24
32.70
25.77
24.79
24.71
18.17
12.14
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

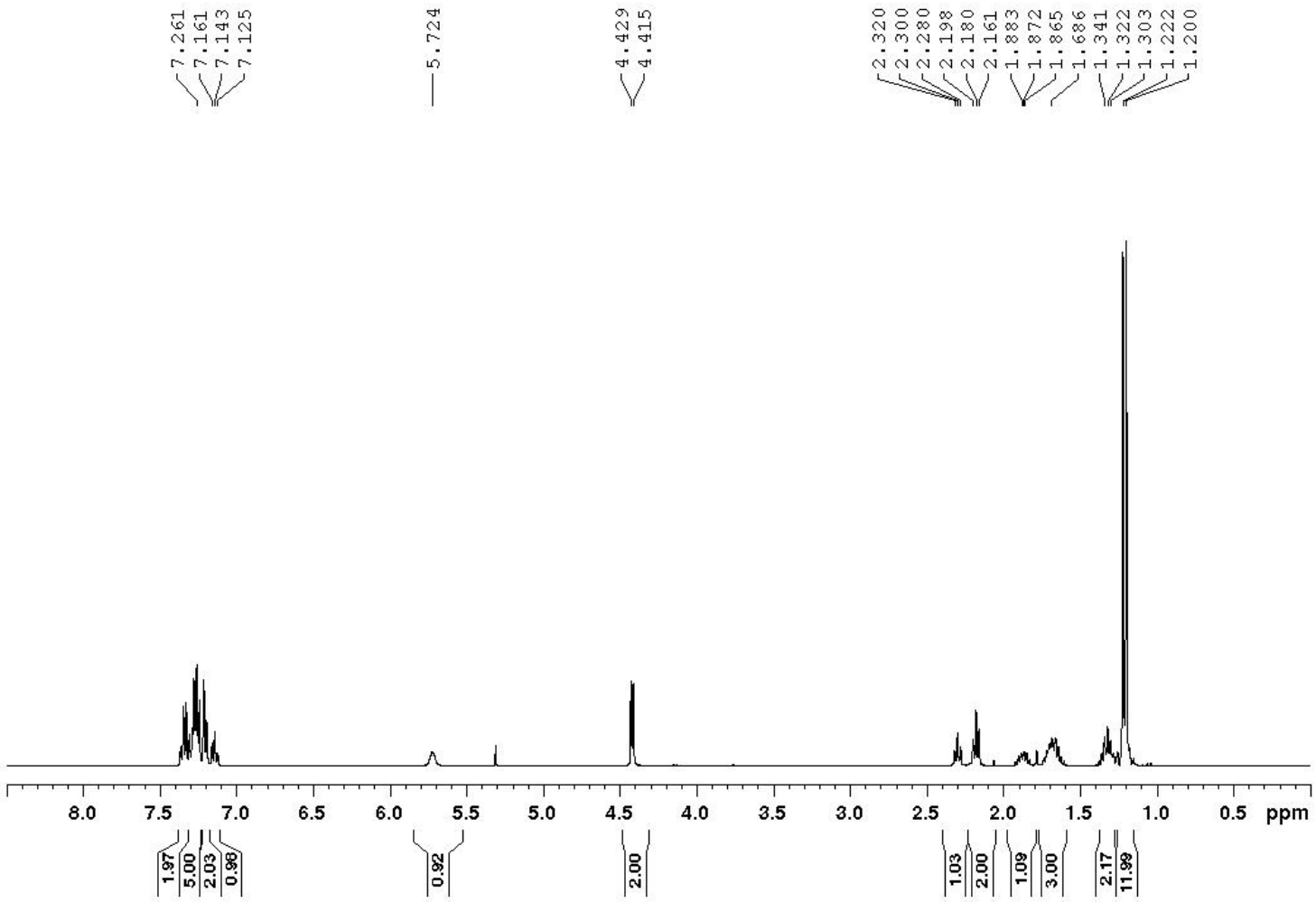


19

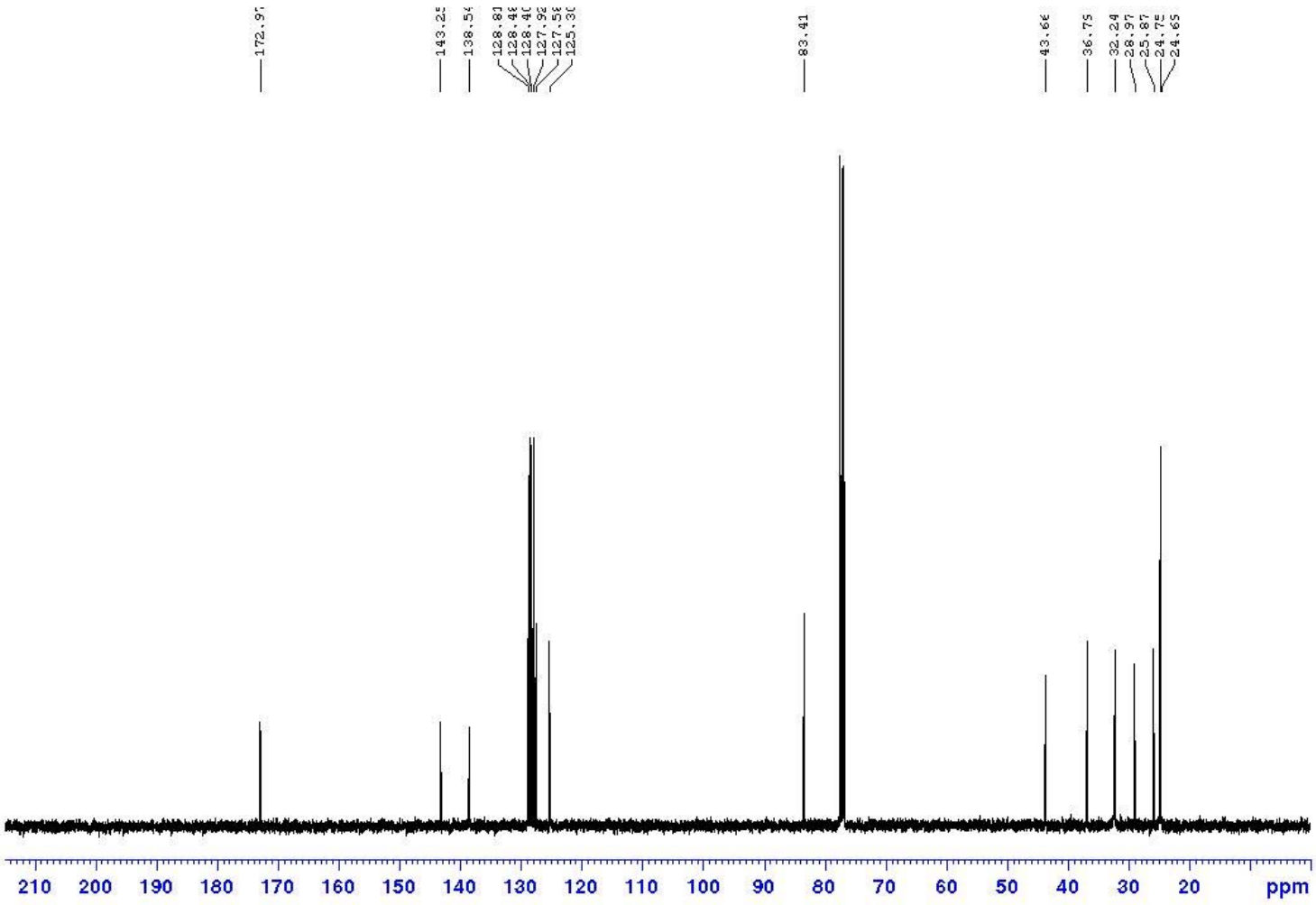


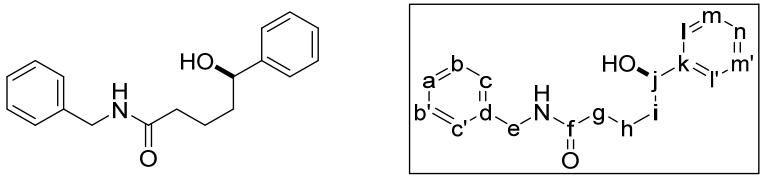
TLC analysis	R_f 0.4 (40:60 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.15–7.40 (9H, m, b,b',c,c',o,o',p,p',q), 7.14 (1H, t, *J* = 7.1 Hz, a), 5.72 (1H, br s, NH), 4.42 (2H, d, *J* = 5.7 Hz, k), 2.30 (1H, t J = 7.8 Hz, e), 2.18 (2H, t, *J* = 7.2 Hz, i), 1.80–1.90 (1H, m, h), 1.60–1.80 (3H, m, f,h), 1.25–1.40 (2H, m, g), 1.22 and 1.20 (12H, s, m,m')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.97 (j), 143.25 (n), 138.54 (d), 128.81 (b,b'), 128.48 (p,p'), 128.44 (c,c'), 127.92 (q), 125.30 (a), 83.41 (l,l'), 43.66 (k), 36.79 (i), 32.24 (h), 28.97 (g), 25.87 (f), 24.75 and 24.69 (m,m')

^{1}H NMR


7.261
7.161
7.143
7.125
5.724
4.429
4.415
2.320
2.300
2.280
2.198
2.180
2.161
1.883
1.872
1.865
1.686
1.341
1.322
1.303
1.222
1.200
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.97
5.00
2.03
0.98
0.92
2.00
1.03
2.00
1.09
3.00
2.17
11.99

^{13}C NMR

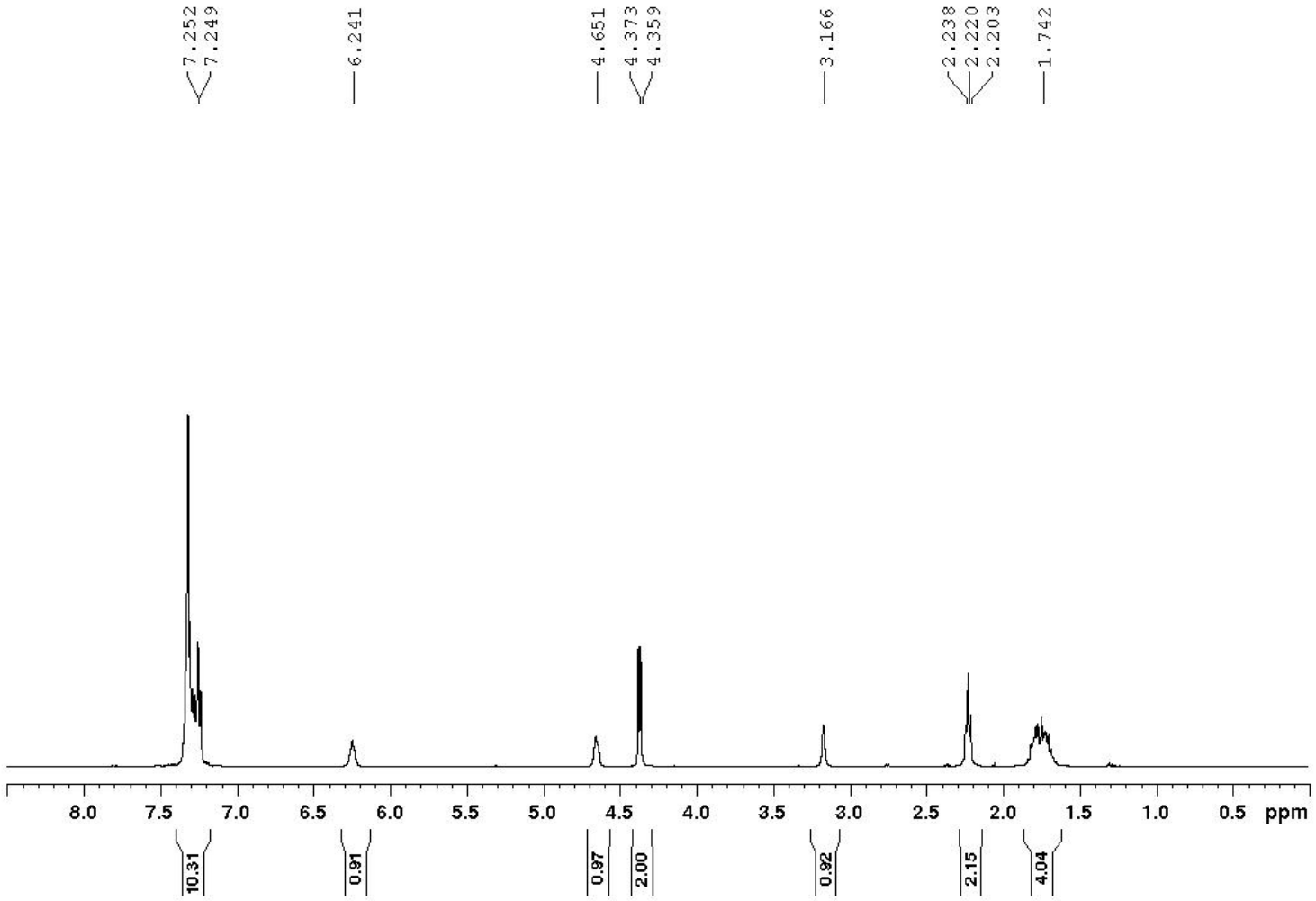

172.97
143.25
138.54
128.83
128.48
128.40
127.92
127.58
125.30
83.41
43.66
36.75
32.24
28.97
25.87
24.75
24.65
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm



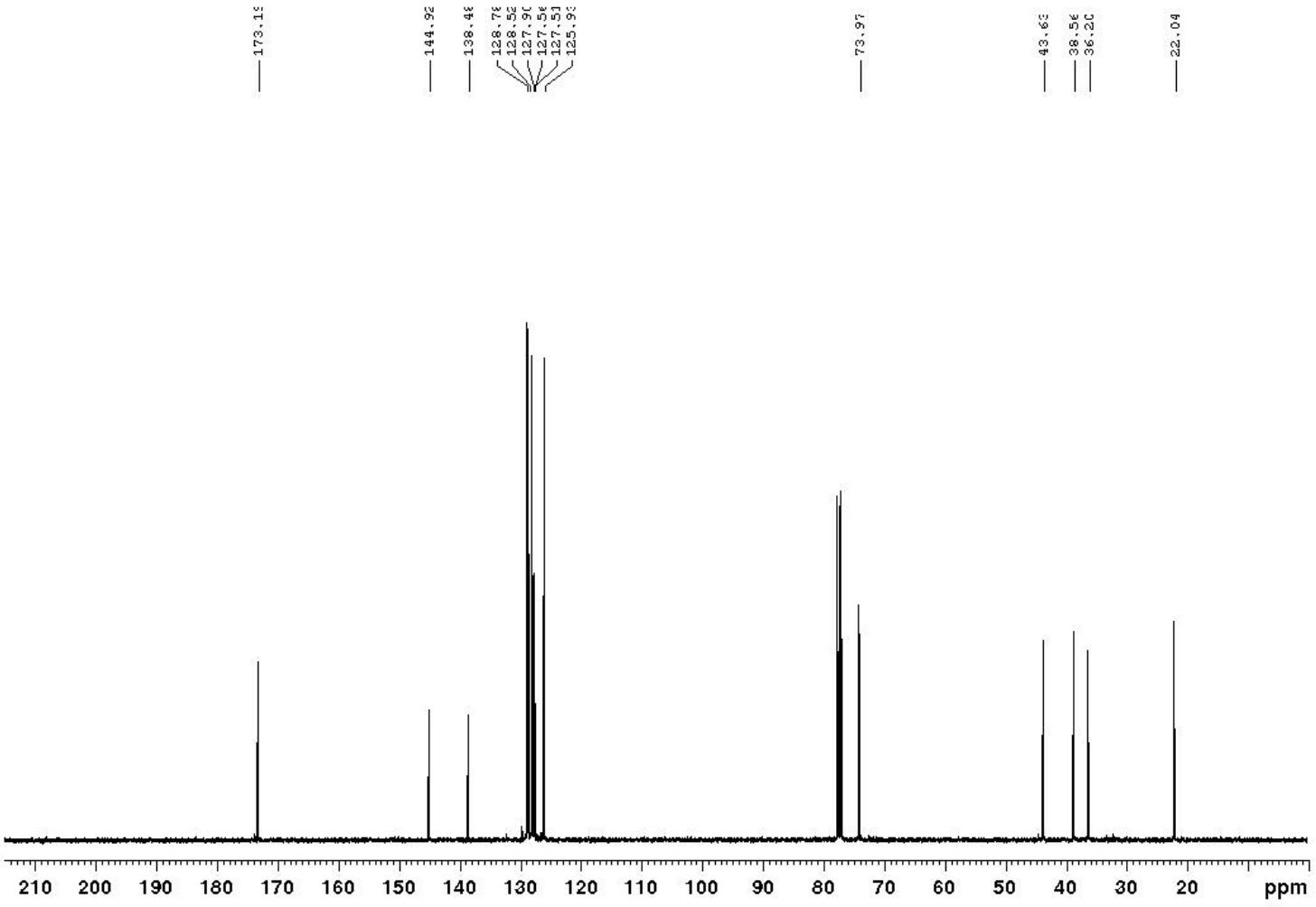
4a-OH

$[\alpha]_D^{20}$	–16.3° (*c* 2.0, $CHCl_3$)
Enantiomeric ratio analysis	Enantiomeric ratio was checked by converting to the corresponding Mosher's ester **4a-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.36 (major 95%) and –71.61 (minor 5%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (10H, m, a,b,b',c,c',l,l',m,m',n), 6.24 (1H, br s, NH), 4.60–4.70 (1H, m, j), 4.37 (2H, d, *J* = 5.7 Hz, e), 3.17 (1H, br s, OH), 2.22 (2H, t, *J* = 6.8 Hz, g), 1.65–1.85 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.19 (f), 144.92 (k), 138.48 (d), 128.78 (m,m'), 128.52 (b,b'), 127.90 (l,l'), 127.56 (n), 127.51 (a), 125.93 (c,c'), 73.97 (j), 43.63 (e), 38.56 (i), 36.20 (g), 22.04 (h)
IR (neat)	3285 (O–H stretch), 3029 (N–H stretch), 2925, 1642 (C=O stretch), 1544, 1494, 1453, 1247, 1027, 749, 696 cm^{-1}
HRMS (EI)	$C_{18}H_{21}NO_2$ (M): 283.1572, found 283.1578 *m/z*

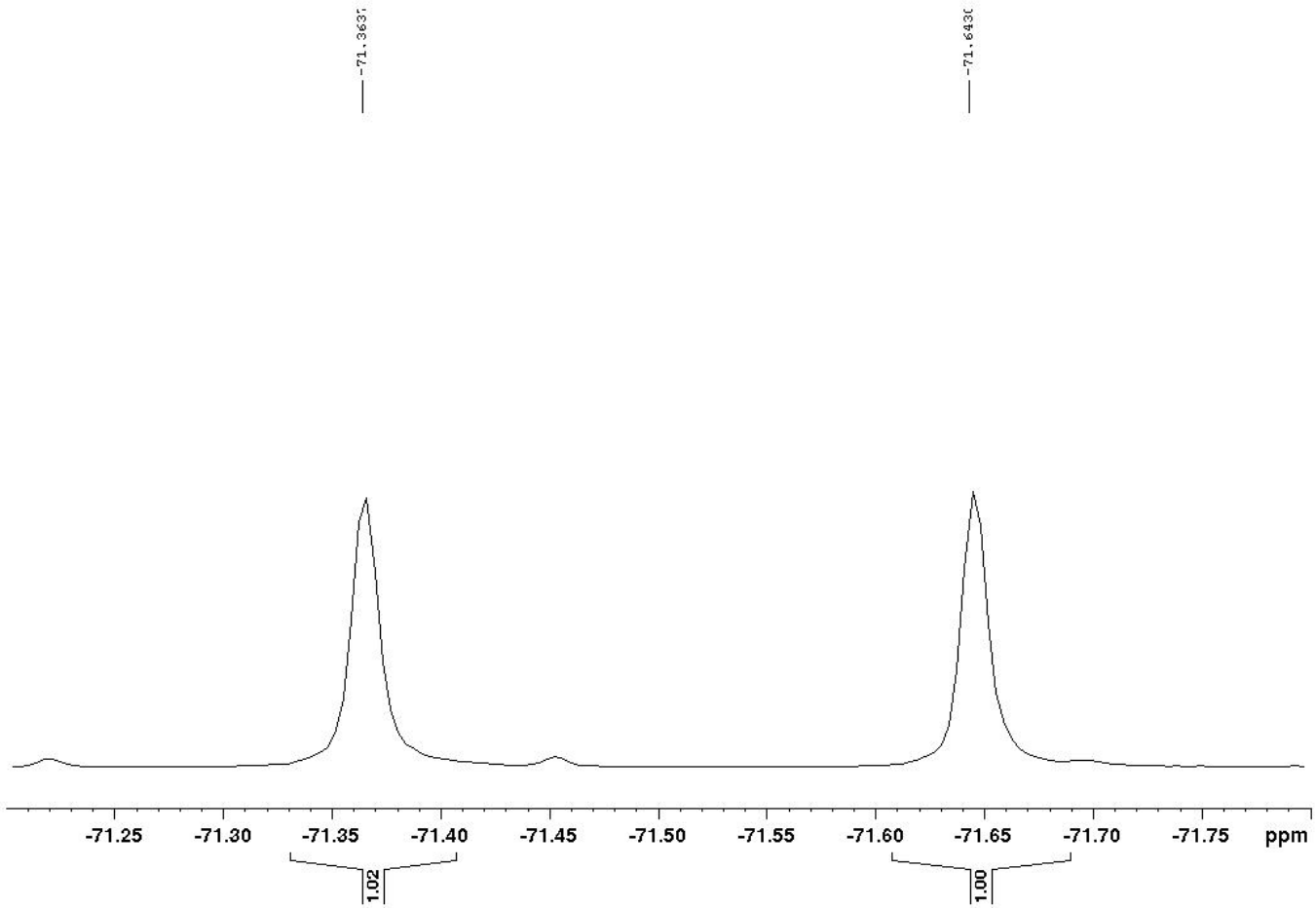
^{1}H NMR


7.252
7.249
6.241
4.651
4.373
4.359
3.166
2.238
2.220
2.203
1.742
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.31
0.91
0.97
2.00
0.92
2.15
4.04

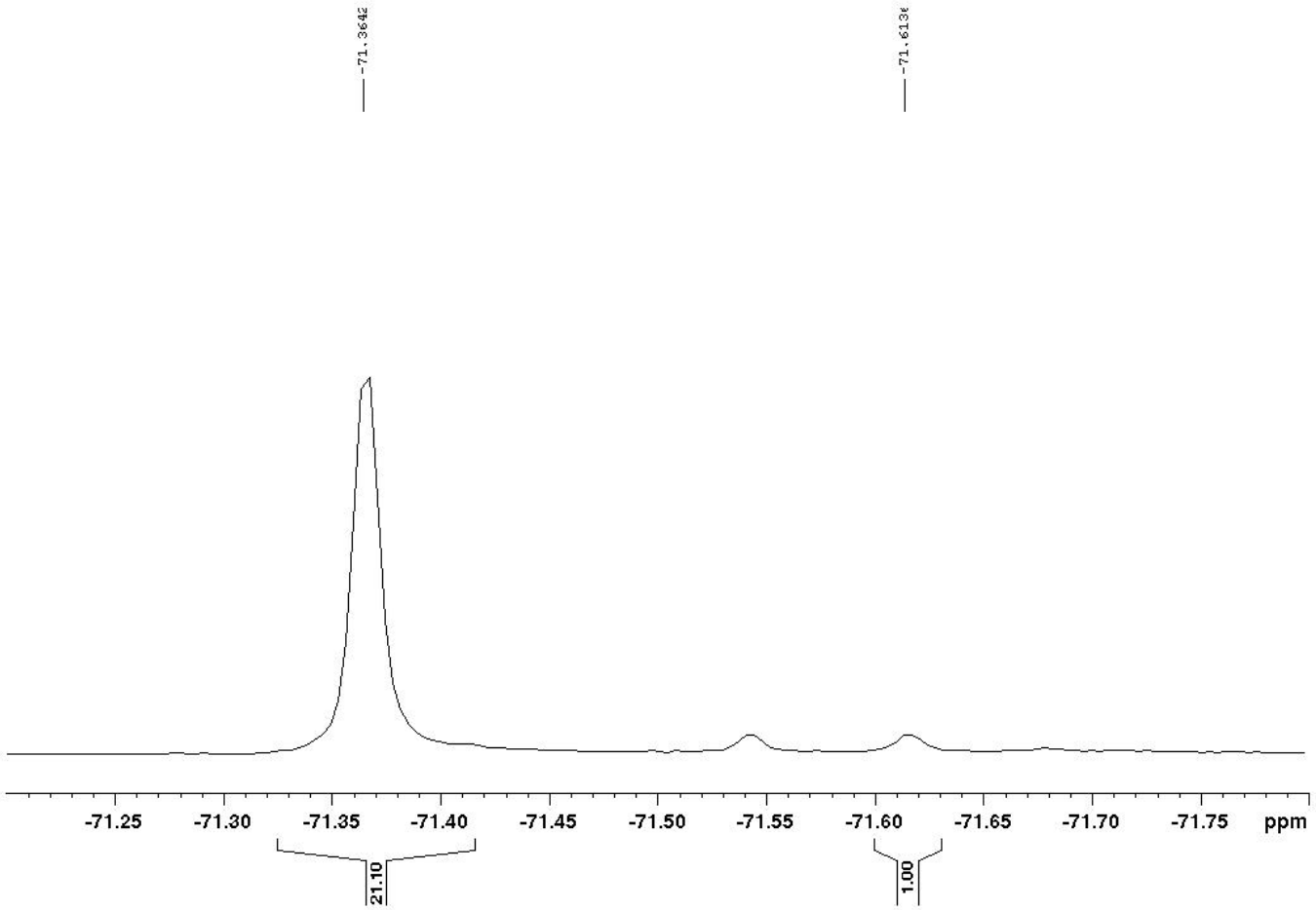
^{13}C NMR


173.1
144.92
138.48
128.78
128.52
127.90
127.58
127.51
125.93
73.97
43.63
38.58
36.20
22.04
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

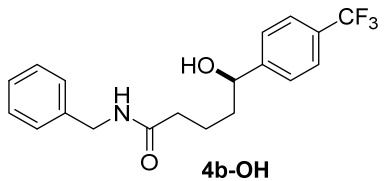
^{19}F NMR of Mosher's ester 4a-Mosher



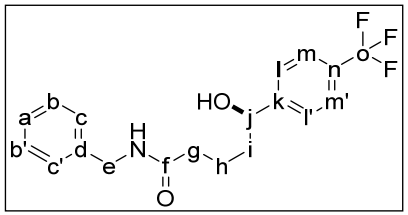
Mixture of diastereomeric 4a-Mosher


-71.3642
-71.6138
-71.25
-71.30
-71.35
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
ppm
21.10
1.00

(*R*,*S*)-**4a-Mosher**

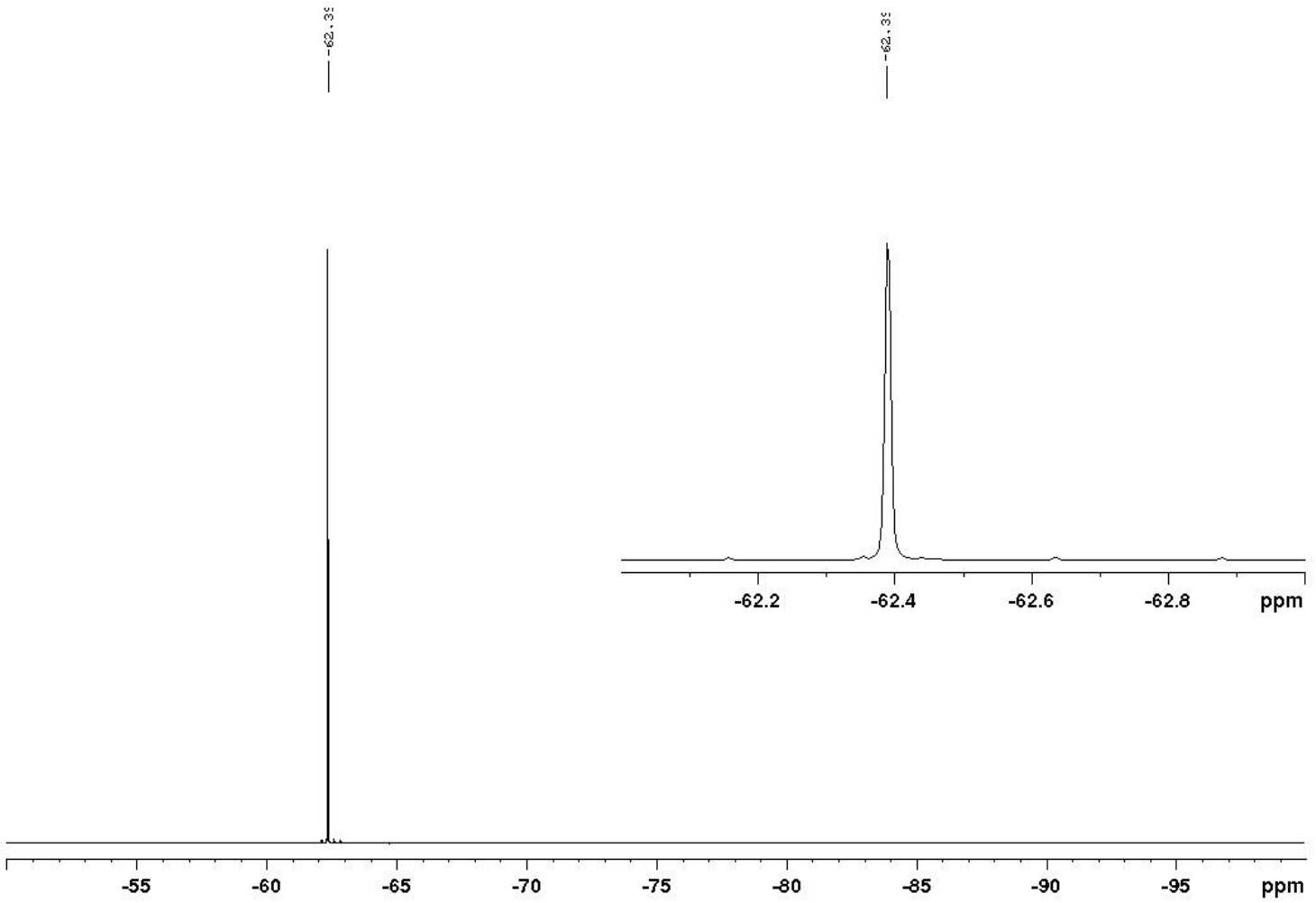


4b-OH

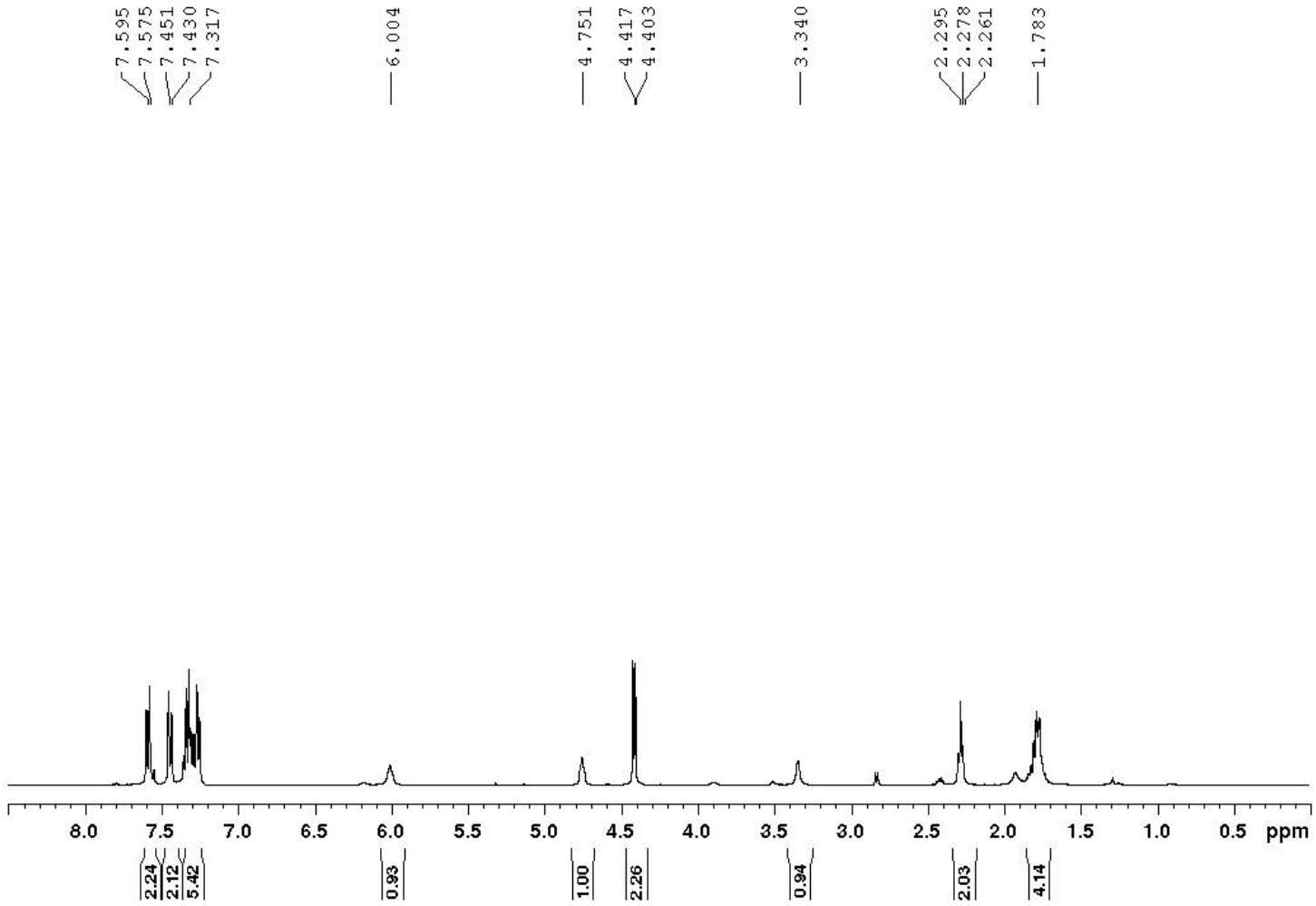


$[\alpha]_D^{20}$	–22.5° (*c* 1.0, $CHCl_3$)
m.p.	71.5–72.5 °C
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4b-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.28 (major 95%) and –71.47 (minor 5%)
^{19}F NMR (376 MHz, $CDCl_3$)	δ –62.39 (CF_3)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.58 (2H, d, *J* = 8.1 Hz, m,m'), 7.44 (2H, d, *J* = 8.1 Hz, c,c'), 7.20–7.35 (5H, m, a,b,b',l,l'), 6.00 (1H, br s, NH), 4.70–4.80 (1H, m, j), 4.41 (2H, d, *J* = 5.7 Hz, e), 3.34 (1H, br s, OH), 2.28 (2H, t, *J* = 6.7 Hz, g), 1.70–1.85 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.00 (f), 148.91 (k), 138.25 (d), 129.86 (n), 129.60 (q, *J* = 32.1 Hz, o), 128.85 (b,b'), 127.91 (l,l'), 127.70 (a), 126.15 (c,c'), 125.43 (q, *J* = 3.8 Hz, m,m'), 73.23 (j), 43.76 (e), 38.63 (i), 35.97 (g), 21.59 (h)
IR (neat)	3296 (O–H stretch), 2924, 1644 (C=O stretch), 1545, 1323, 1161, 1110, 1066, 1016, 840, 730, 697, 603 cm^{-1}
HRMS (ESI)	$C_{19}H_{20}F_3NNaO_2$ (M+Na): 374.1344, found 374.1351 *m/z*

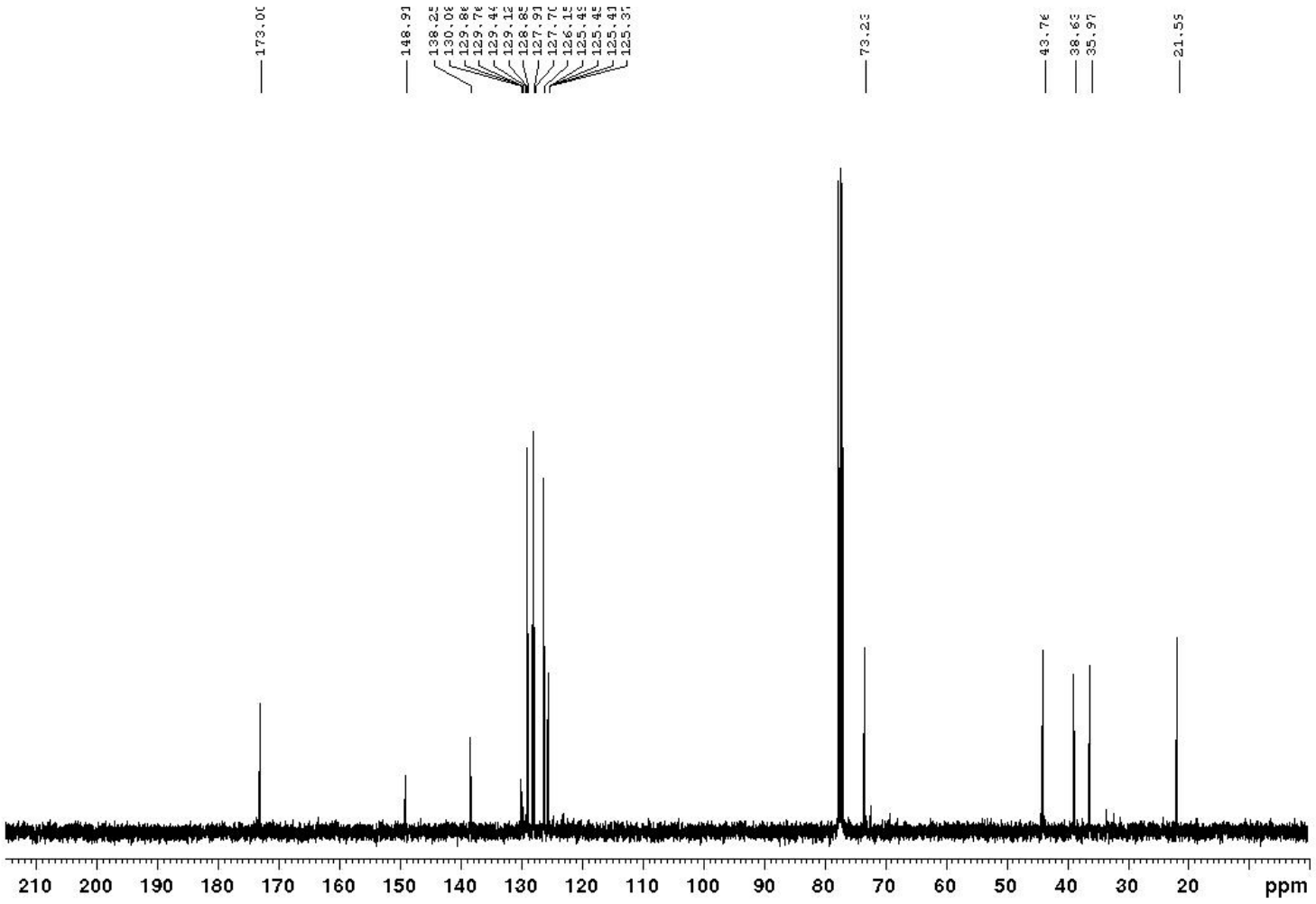
^{19}F NMR



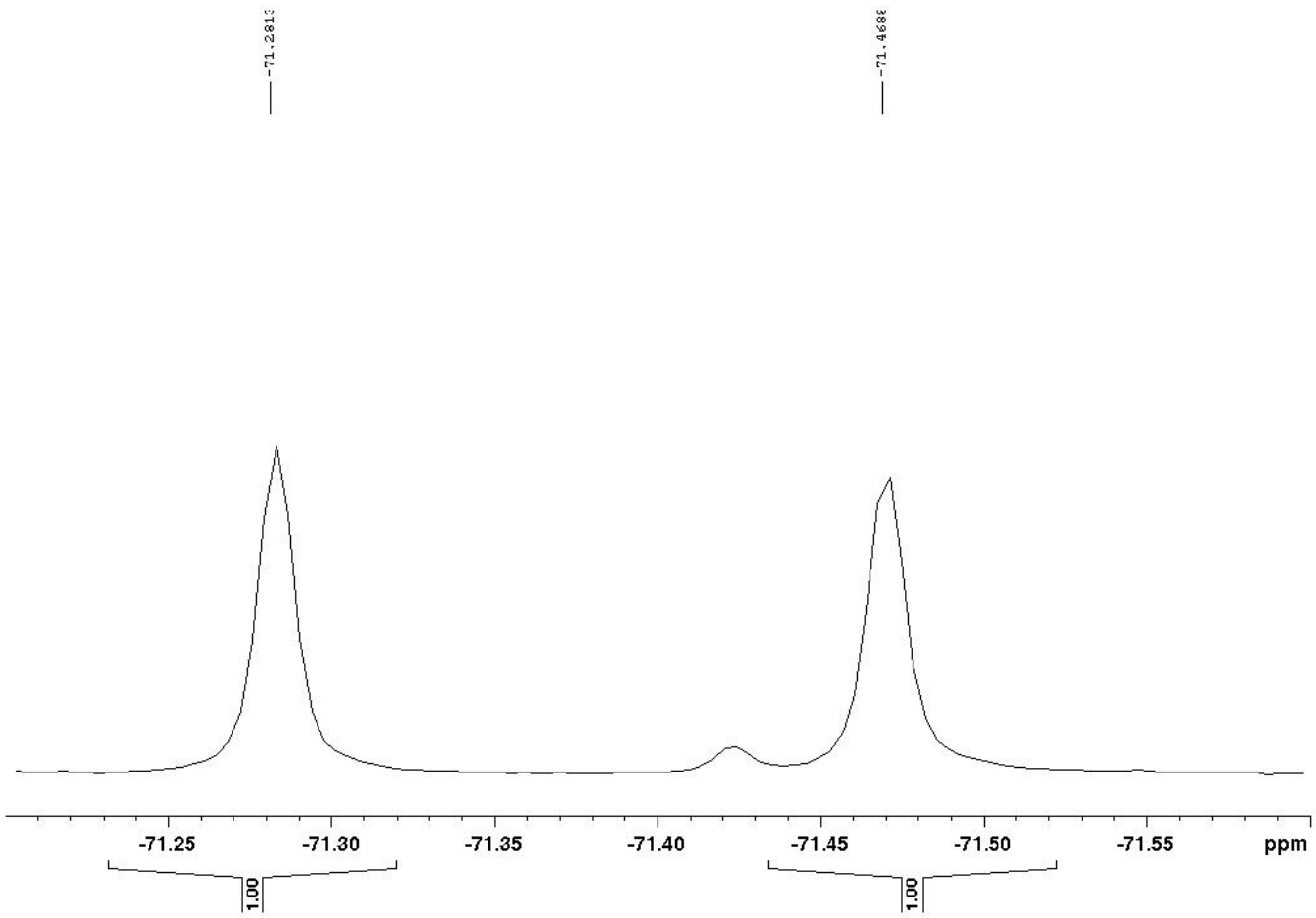
^{1}H NMR


7.595
7.575
7.451
7.430
7.317
6.004
4.751
4.417
4.403
3.340
2.295
2.278
2.261
1.783
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.24
2.12
5.42
0.93
1.00
2.26
0.94
2.03
4.14

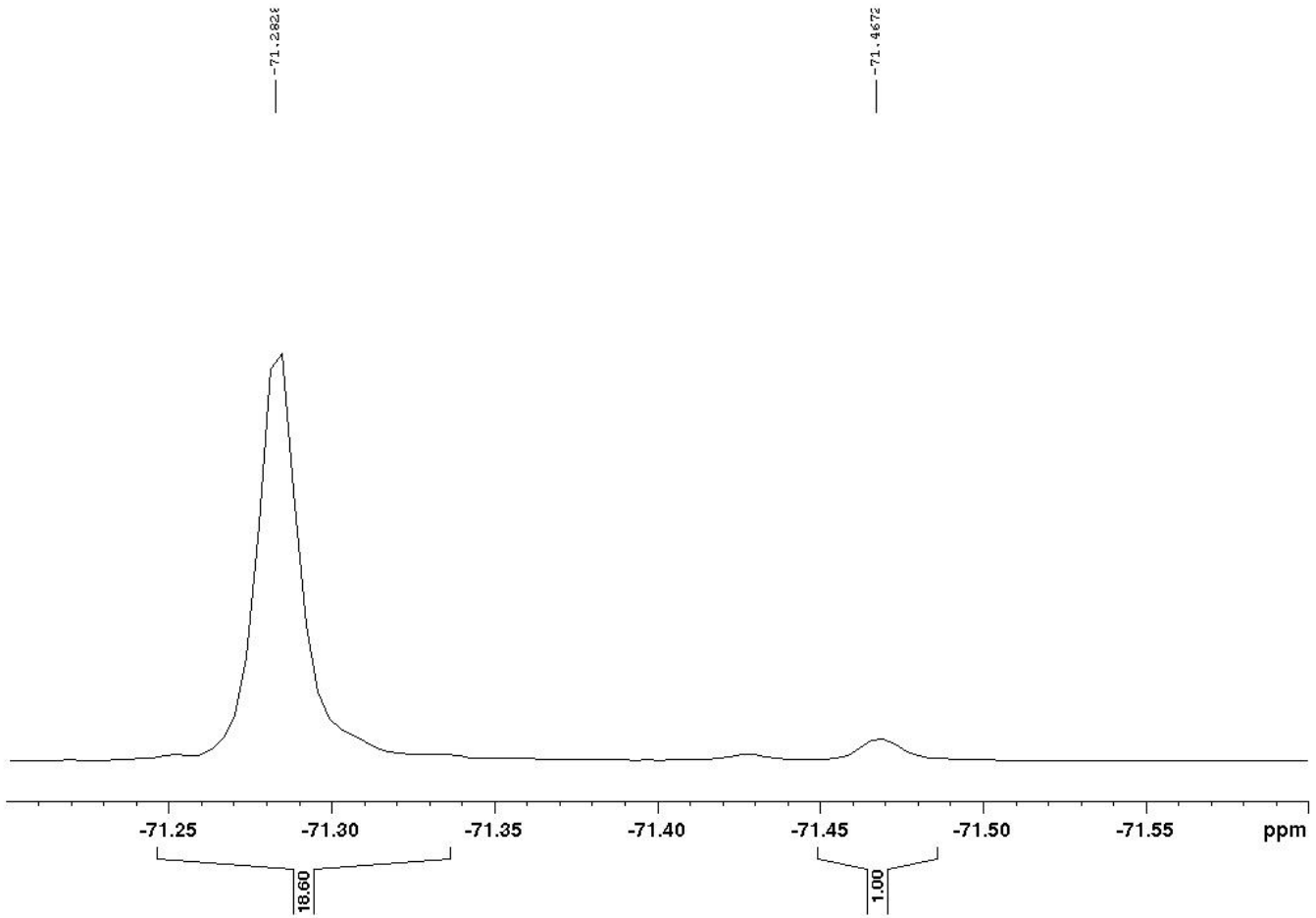
^{13}C NMR


173.0
148.9
138.2
130.0
129.8
129.7
129.4
129.1
128.8
127.9
127.7
126.1
125.4
125.4
125.4
125.3
73.2
43.7
38.6
35.9
21.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

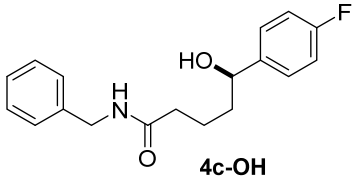
^{19}F NMR of Mosher's ester 4b-Mosher



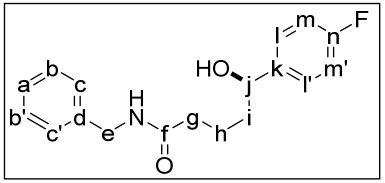
Mixture of diastereomeric 4b-Mosher


-71.2828
-71.4672
-71.25
-71.30
-71.35
-71.40
-71.45
-71.50
-71.55
ppm
18.60
1.00

(*R*,*S*)-**4b-Mosher**

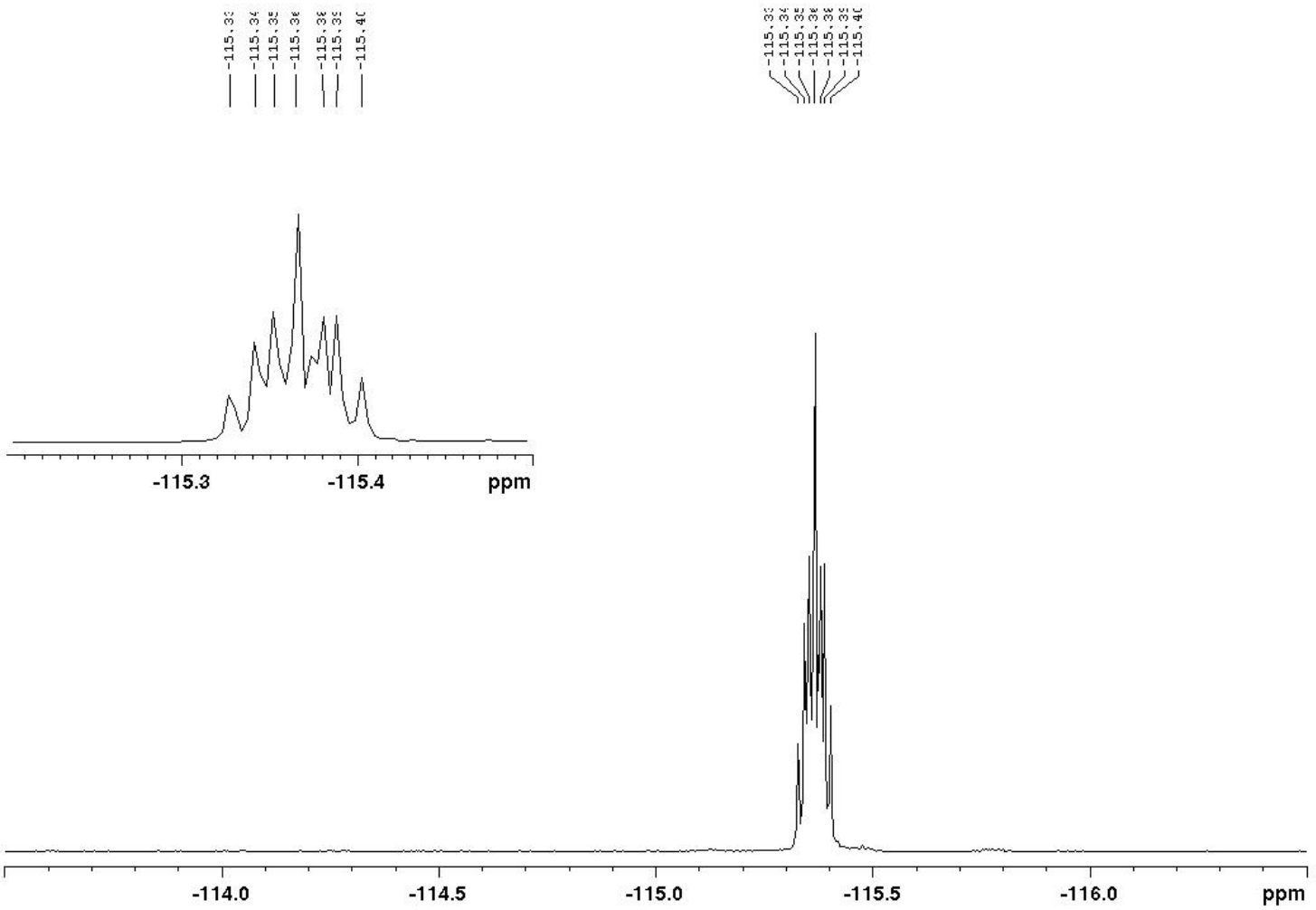


4c-OH

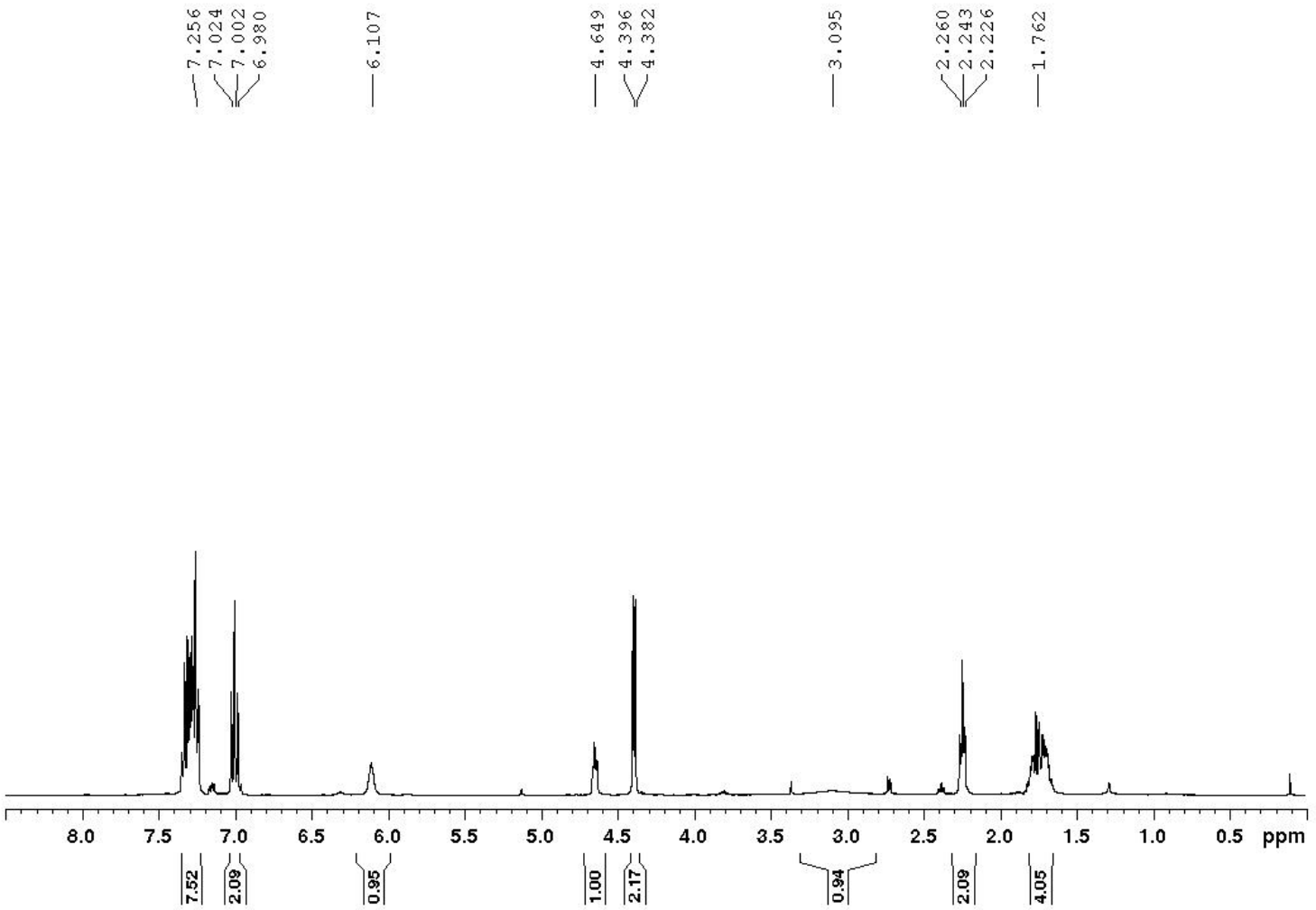


$[\alpha]_D^{20}$	–20.1° (*c* 2.0, $CHCl_3$)
m.p.	64.5–65.5 °C
HPLC analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4c-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.25 (major 95%) and –71.51 (minor 5%)
^{19}F NMR (376 MHz, $CDCl_3$)	δ –115.30 to –115.40 ($C(sp^2)$–F)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.35 (7H, m, a,b,b',c,c',l,l'), 7.00 (2H, t, *J* = 8.7 Hz, m,m'), 6.11 (1H, br s, NH), 4.60–4.70 (1H, m, j), 4.39 (2H, d, *J* = 5.7 Hz, e), 3.09 (1H, br s, OH), 2.24 (2H, t, *J* = 6.9 Hz, g), 1.65–1.80 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.07 (f), 162.17 (d, *J* = 243 Hz, n), 140.65 and 140.62 (k), 138.36 (d), 128.82 (b,b'), 127.90 (l,l'), 127.63 and 127.56 and 127.48 (a,c,c'), 115.28 (d, *J* = 21 Hz, m,m'), 73.30 (j), 43.69 (e), 38.64 (i), 36.11 (g), 21.87 (h)
IR (neat)	3283 (O–H stretch), 2921, 1643 (C=O stretch), 1548, 1508, 1219, 1155, 1076, 1014, 835, 730, 697 cm^{-1}
HRMS (ESI)	$C_{18}H_{20}FNNaO_2$ (M+Na): 324.1376, found 324.1385 *m/z*

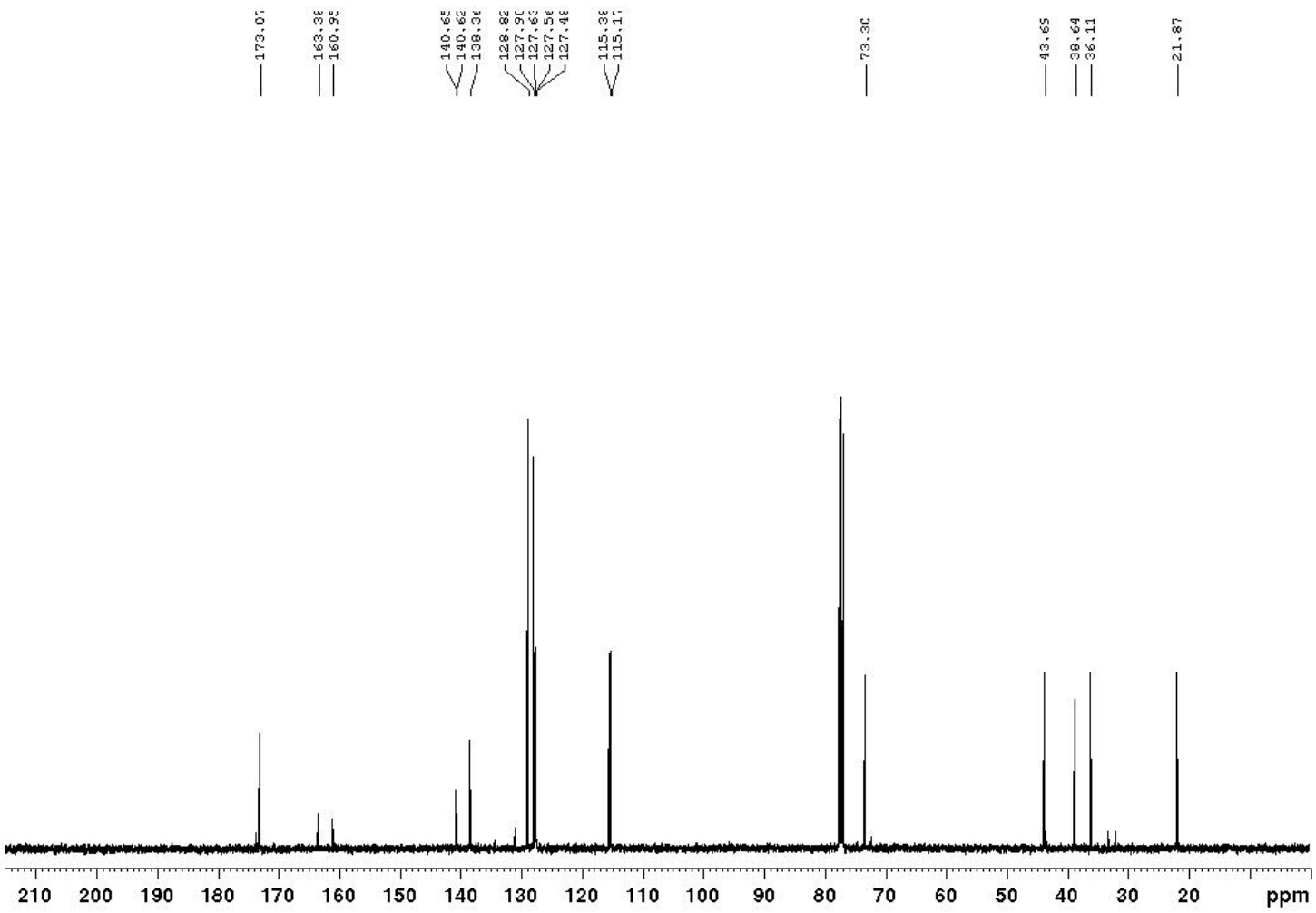
^{19}F NMR



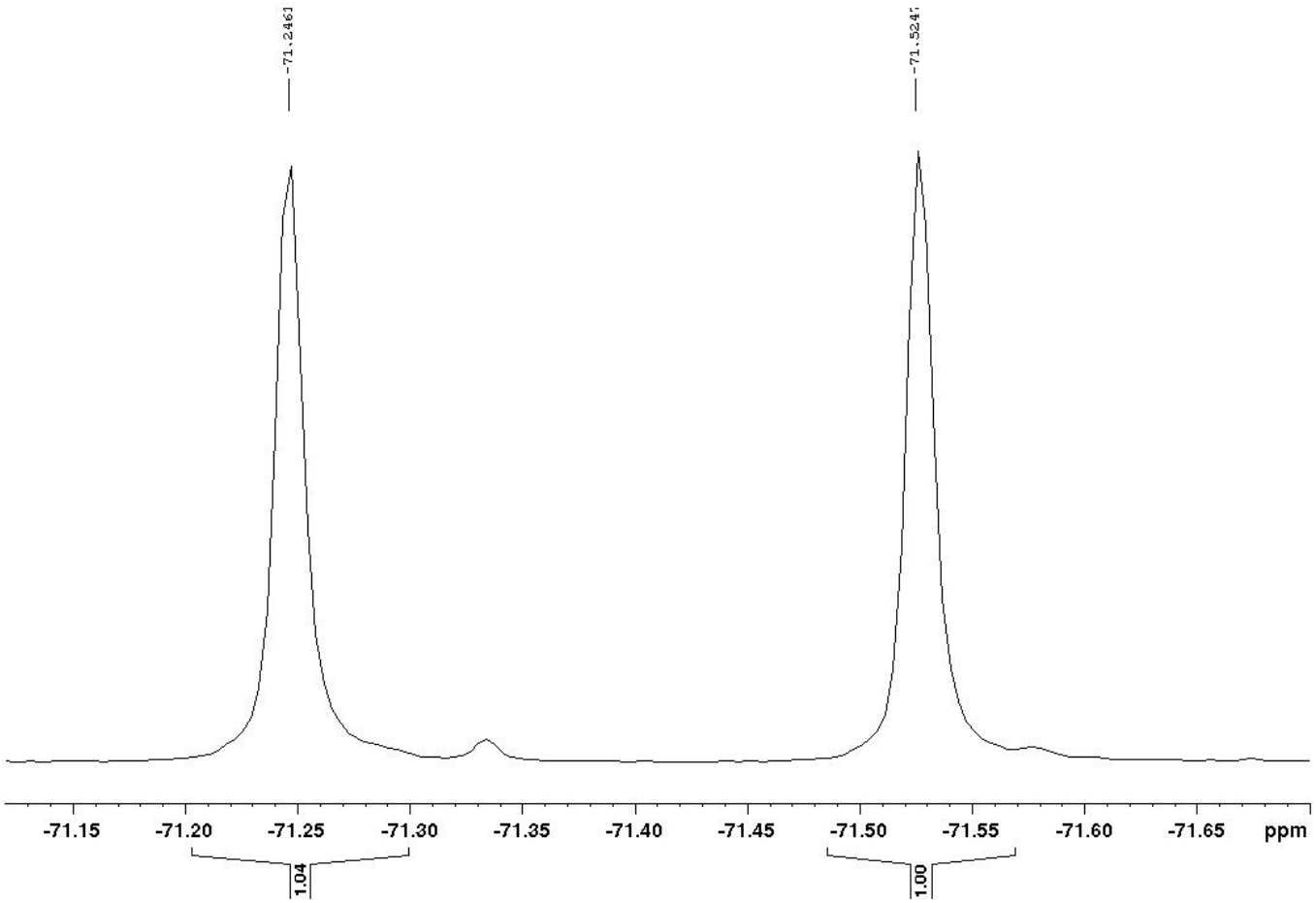
^{1}H NMR


7.256
7.024
7.002
6.980
6.107
4.649
4.396
4.382
3.095
2.260
2.243
2.226
1.762
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
7.52
2.09
0.95
1.00
2.17
0.94
2.09
4.05

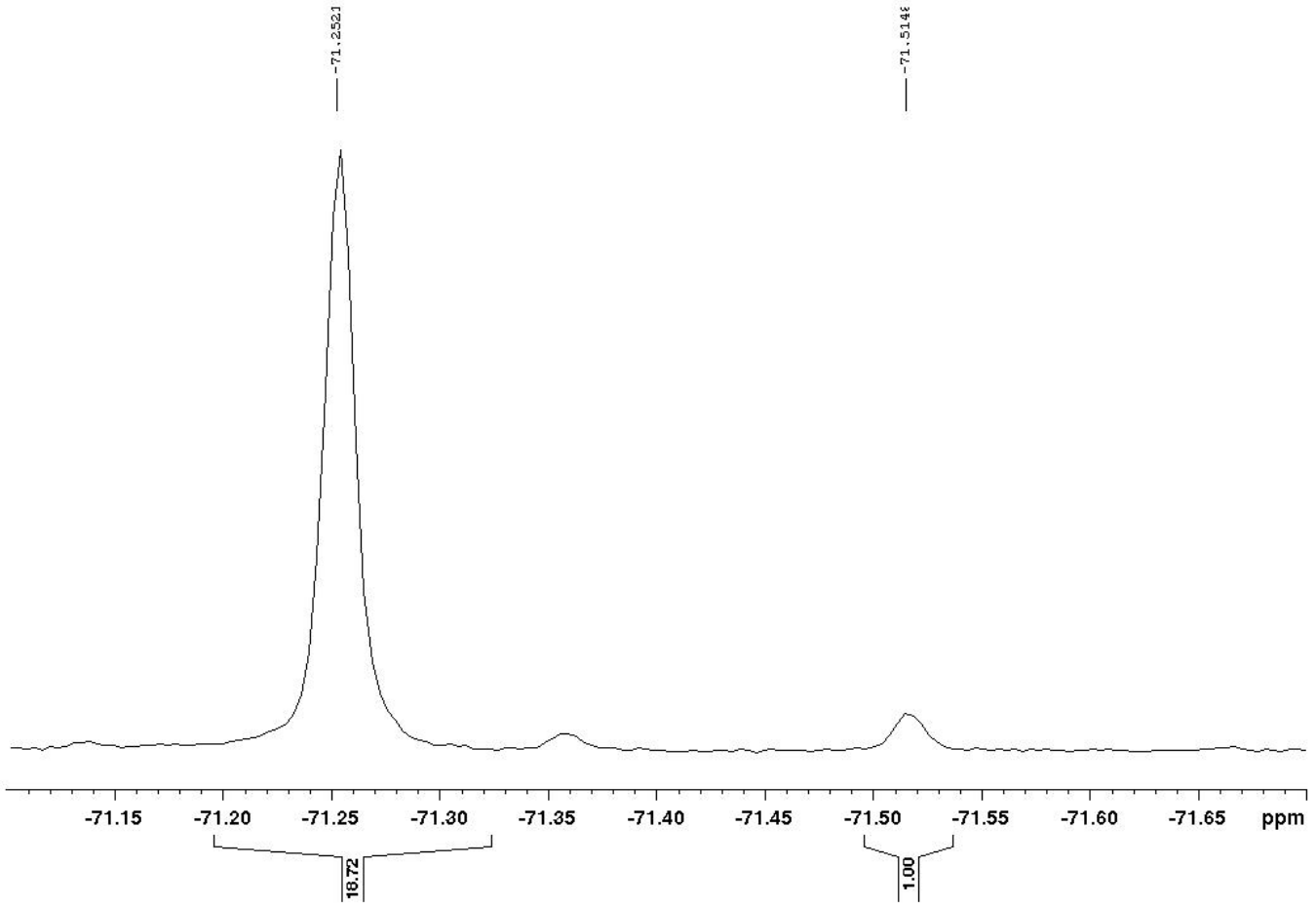
^{13}C NMR


173.07
163.38
160.95
140.65
140.62
138.38
128.82
127.90
127.63
127.58
127.48
115.38
115.11
73.30
43.65
38.64
36.11
21.87
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

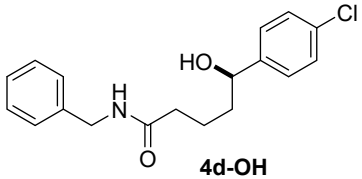
^{19}F NMR of Mosher's ester 4c-Mosher



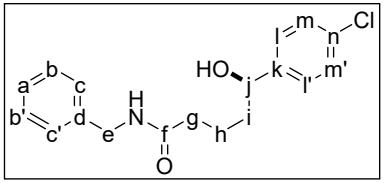
Mixture of diastereomeric 4c-Mosher


-71.2521
-71.5148
-71.15
-71.20
-71.25
-71.30
-71.35
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
ppm
18.72
1.00

(*R*,*S*)-**4c-Mosher**

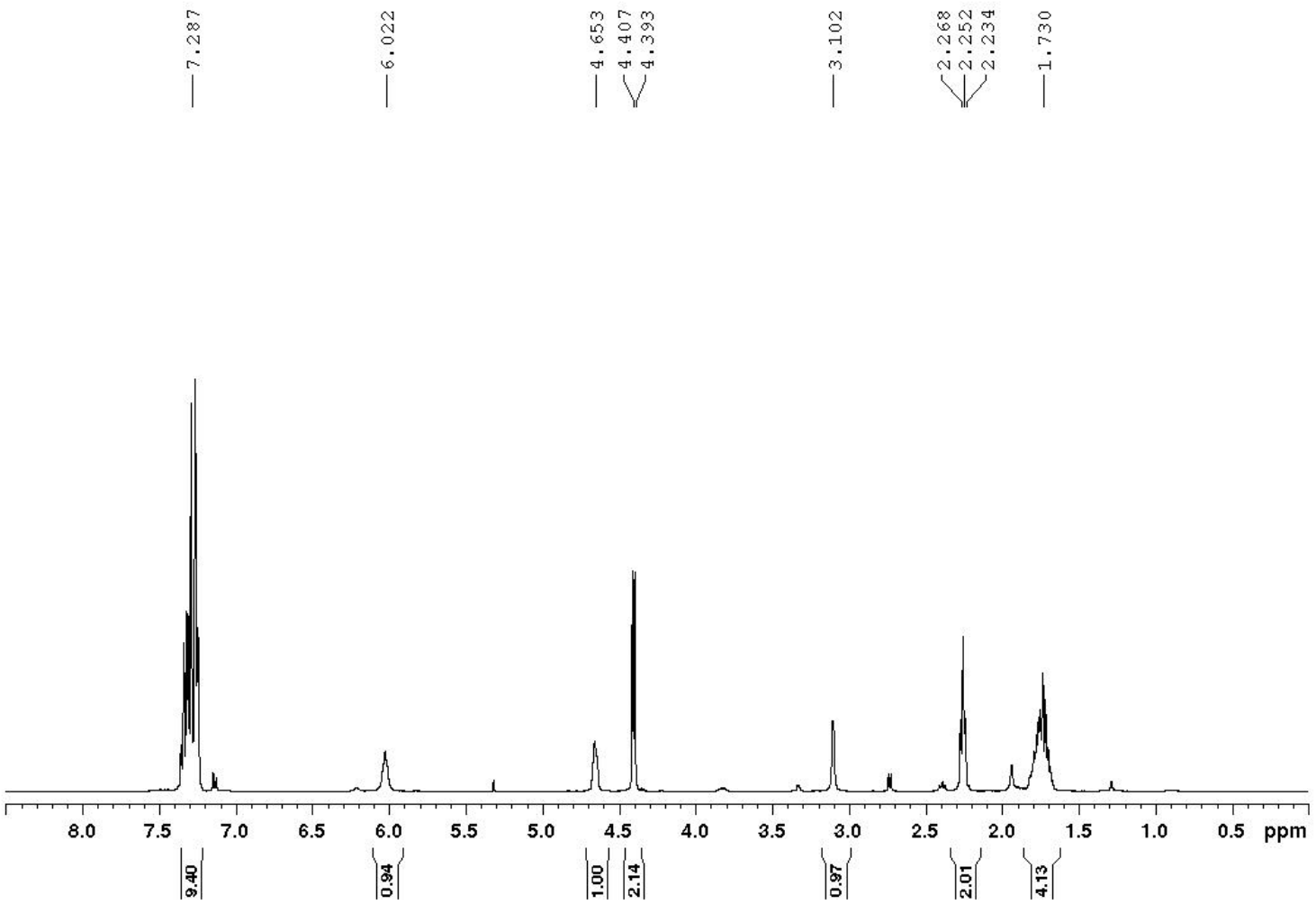


4d-OH

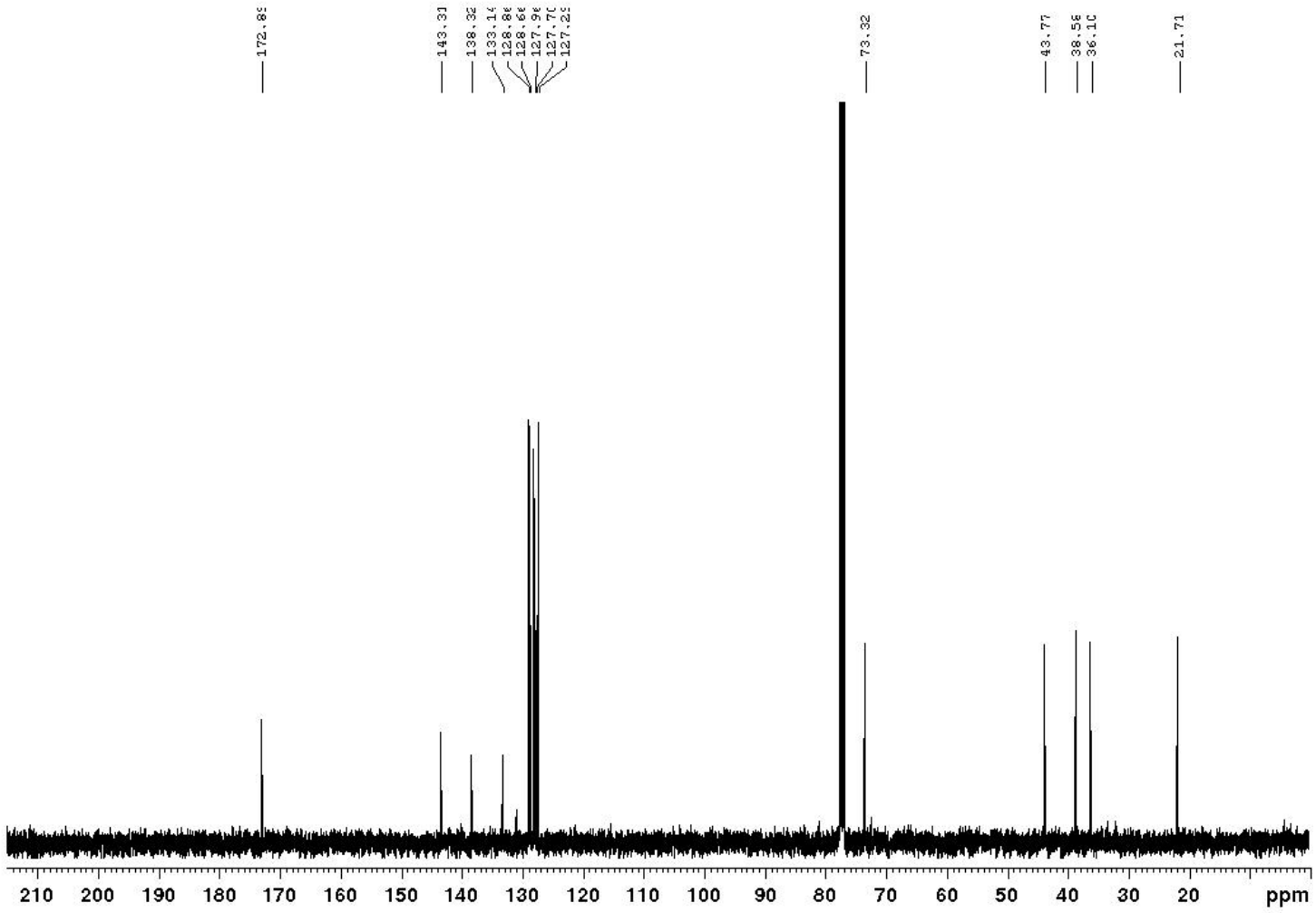


$[\alpha]_D^{20}$	–21.1° (*c* 2.0, $CHCl_3$)
m.p.	108.5–110.0 °C
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4d-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.35 (major 95%) and –71.57 (minor 5%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (9H, m, a,b,b',c,c',l,l',m,m'), 6.02 (1H, br s, NH), 4.60–4.80 (1H, m, j), 4.40 (2H, d, *J* = 5.7 Hz, e), 3.10 (1H, br s, OH), 2.25 (2H, t, *J* = 6.4 Hz, g), 1.65–1.85 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.89 (f), 143.31 (k), 138.32 (d), 133.14 (n), 128.86 (b,b'), 128.66 (m,m'), 127.96 (l,l'), 127.70 (a), 127.29 (c,c'), 73.32 (j), 43.77 (e), 38.58 (i), 36.10 (g), 21.71 (h)
IR (neat)	3281 (O–H stretch), 2915, 2360, 1629 (C=O stretch), 1531, 1486, 1454, 1077, 1009, 936, 831, 799, 749, 698 cm^{-1}
HRMS (EI)	$C_{18}H_{20}ClNO_2$ (M): 317.1183, found 317.1189 *m/z*

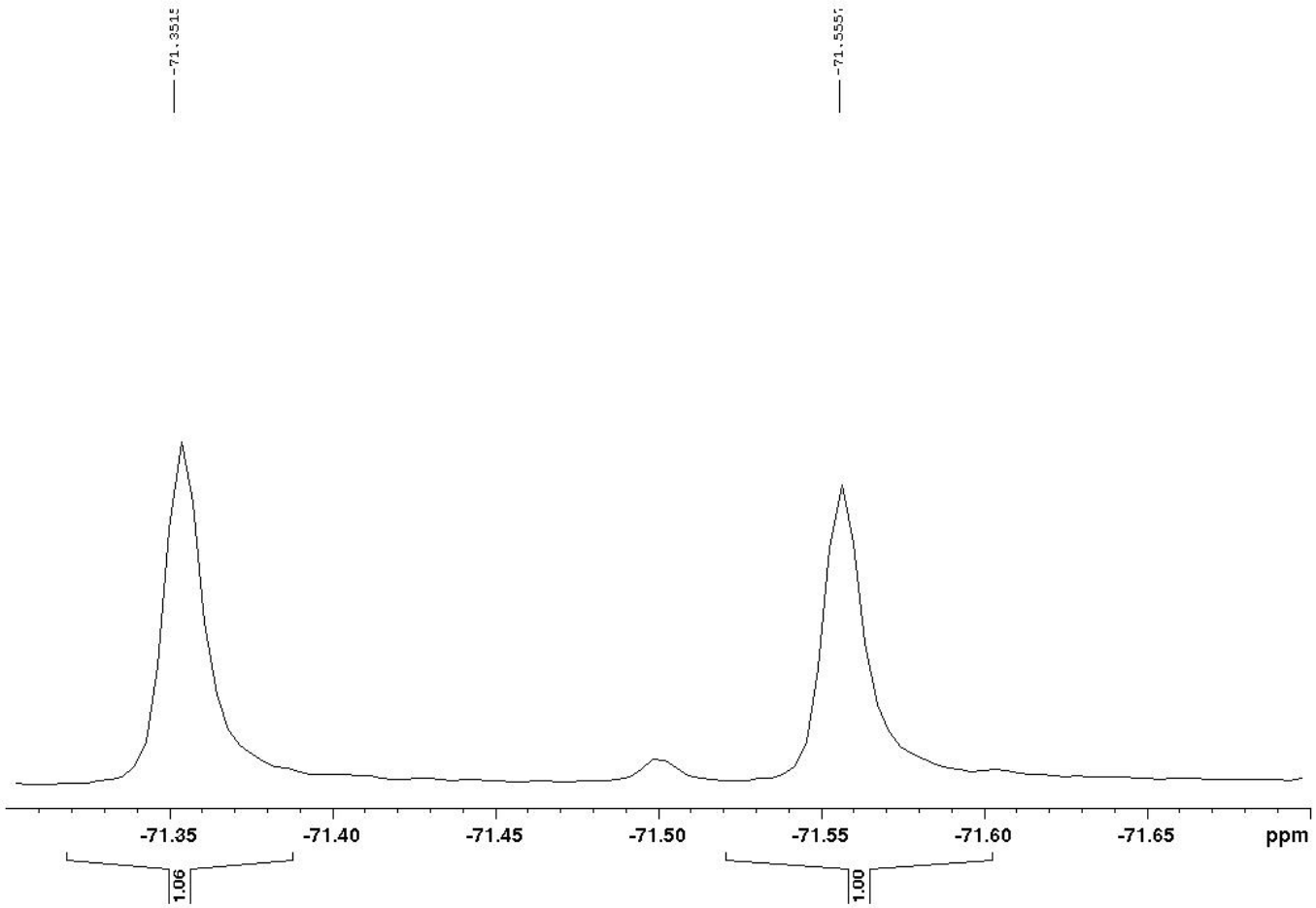
^{1}H NMR


7.287
6.022
4.653
4.407
4.393
3.102
2.268
2.252
2.234
1.730
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
9.40
0.94
1.00
2.14
0.97
2.01
4.13

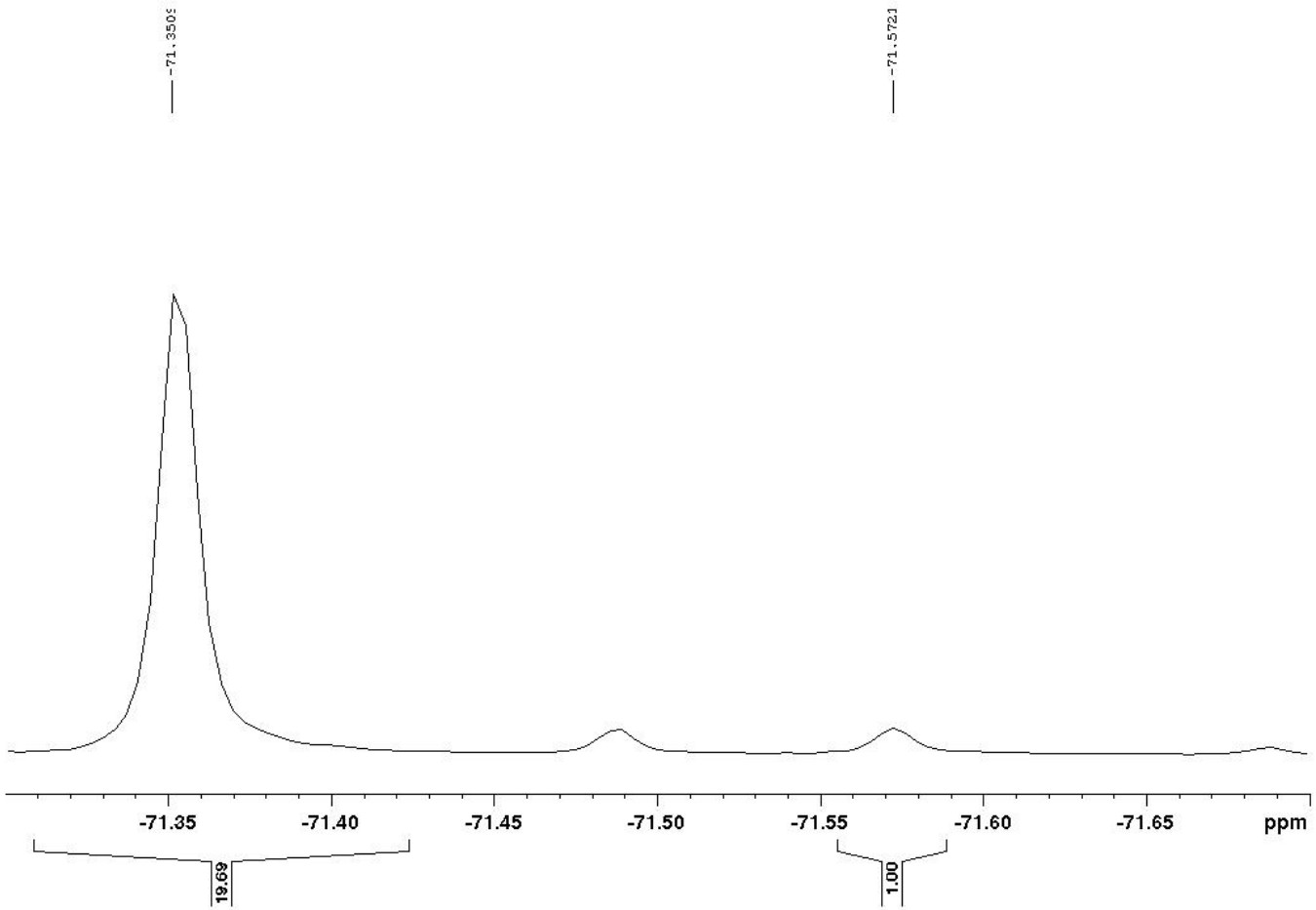
^{13}C NMR


172.8
143.31
138.32
133.14
128.8
128.6
127.9
127.7
127.2
73.32
43.77
38.56
36.10
21.71
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

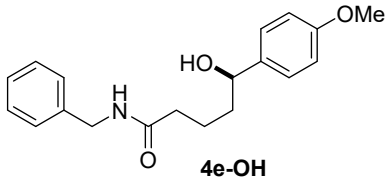
^{19}F NMR of Mosher's ester 4d-Mosher



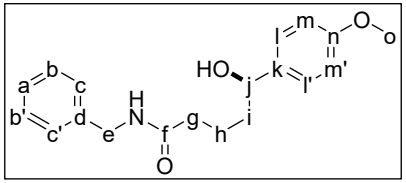
Mixture of diastereomeric 4d-Mosher


-71.3505
-71.5721
-71.35
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
ppm
19.69
1.00

(*R*,*S*)-**4d-Mosher**

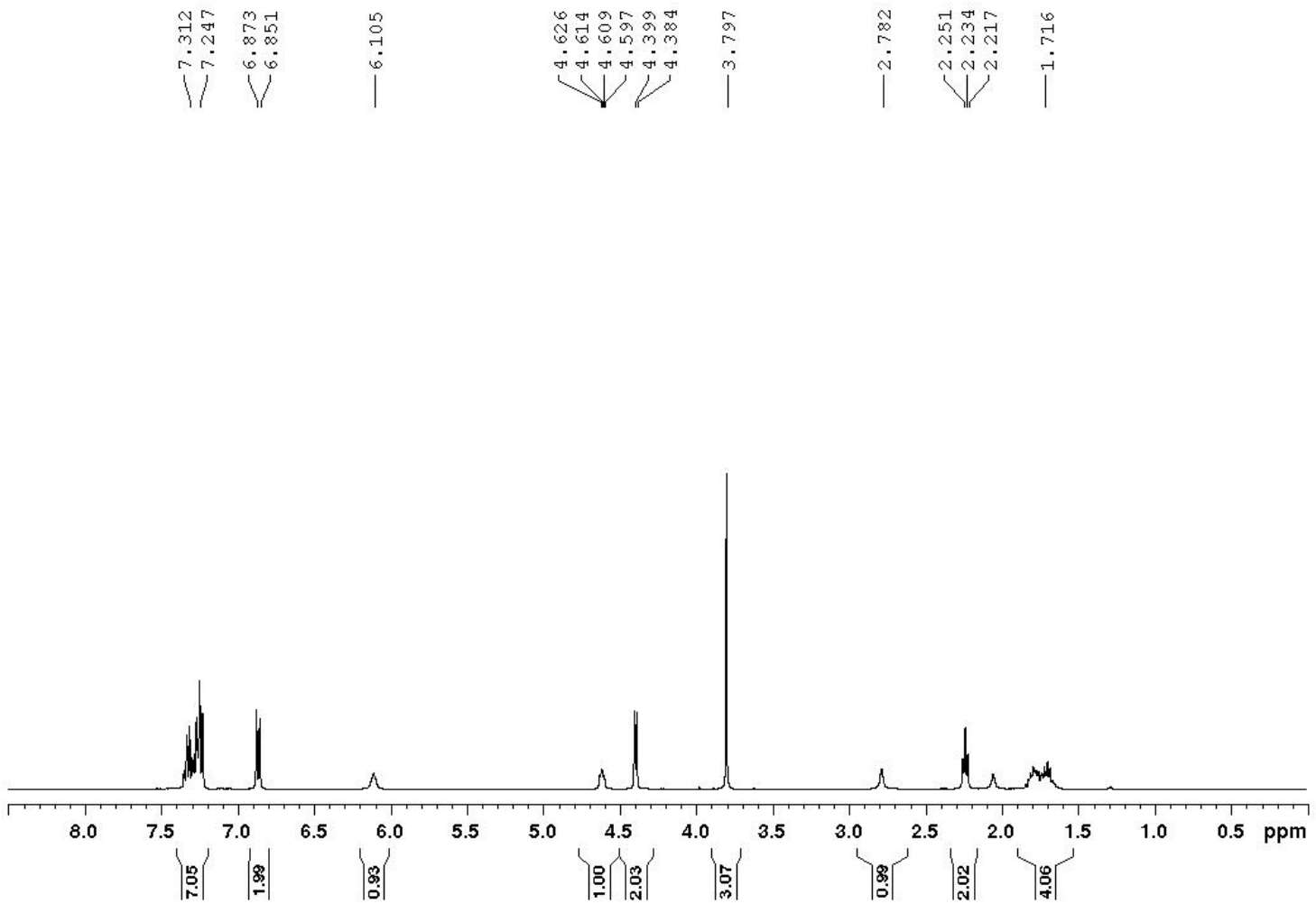


4e-OH

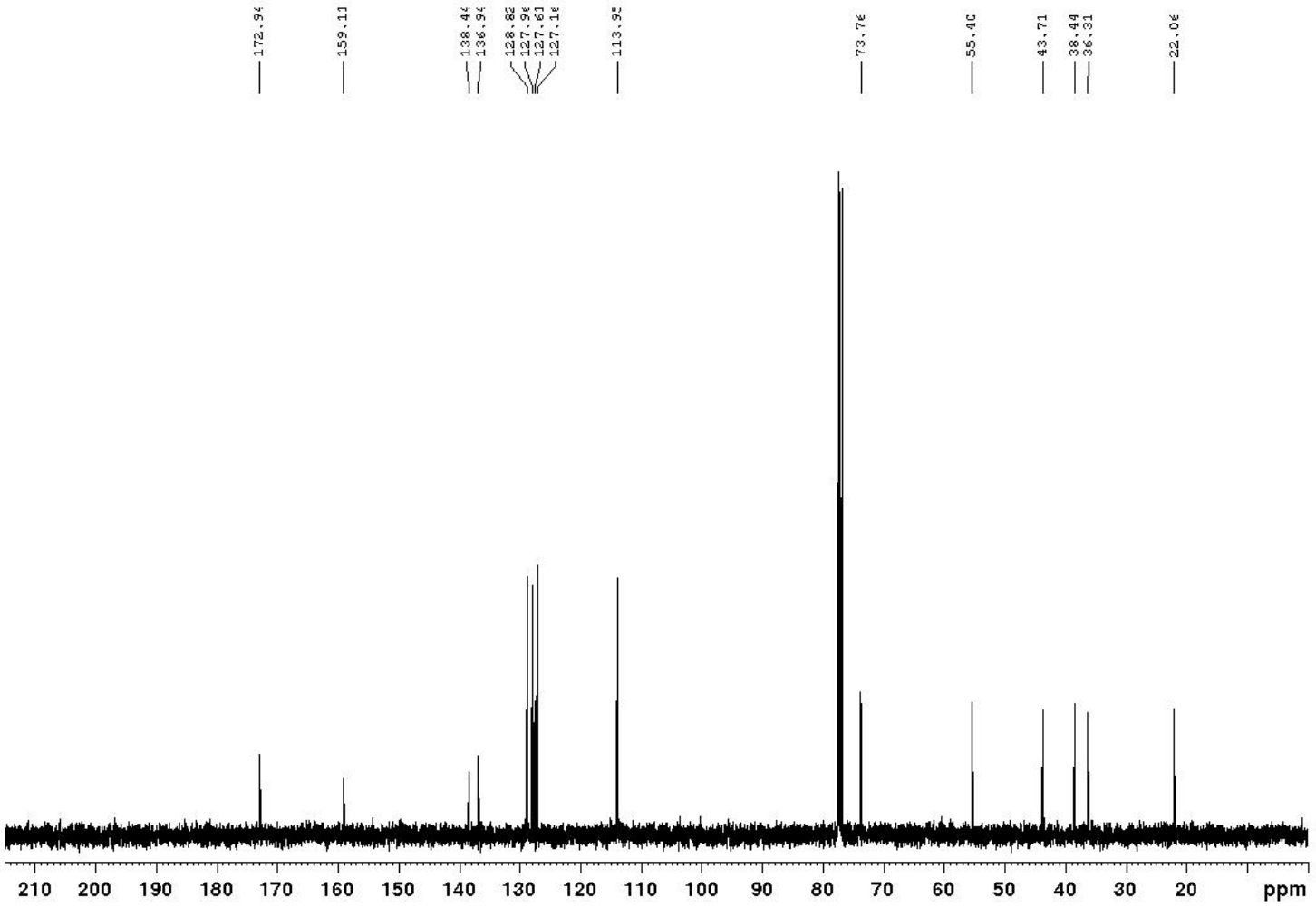


$[\alpha]_D^{20}$	–21.0° (*c* 2.0, $CHCl_3$)
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4e-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.40 (major 96%) and –71.68 (minor 4%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (7H, m, a,b,b',c,c',l,l'), 6.86 (2H, d, *J* = 8.6 Hz, m,m'), 6.11 (1H, br s, NH), 4.61 (1H, dd, *J* = 6.8 and 4.7 Hz, j), 4.39 (2H, d, *J* = 5.7 Hz, e), 3.80 (3H, s, o), 2.78 (1H, br s, OH), 2.23 (2H, t, *J* = 6.8 Hz, g), 1.65–1.85 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.94 (f), 159.11 (n), 138.44 (d), 136.94 (k), 128.82 (b,b'), 127.95 (l,l'), 127.61 (a), 127.16 (c,c'), 113.95 (m,m'), 73.76 (j), 55.40 (o), 43.71 (e), 38.44 (i), 36.31 (g), 22.06 (h)
IR (neat)	3287 (O–H stretch), 2924, 1643 (C=O stretch), 1611, 1543, 1510, 1454, 1242, 1174, 1029, 831, 732, 697 cm^{-1}
HRMS (ESI)	$C_{19}H_{23}NNaO_3$ (M+Na): 336.1576, found 336.1588 *m/z*

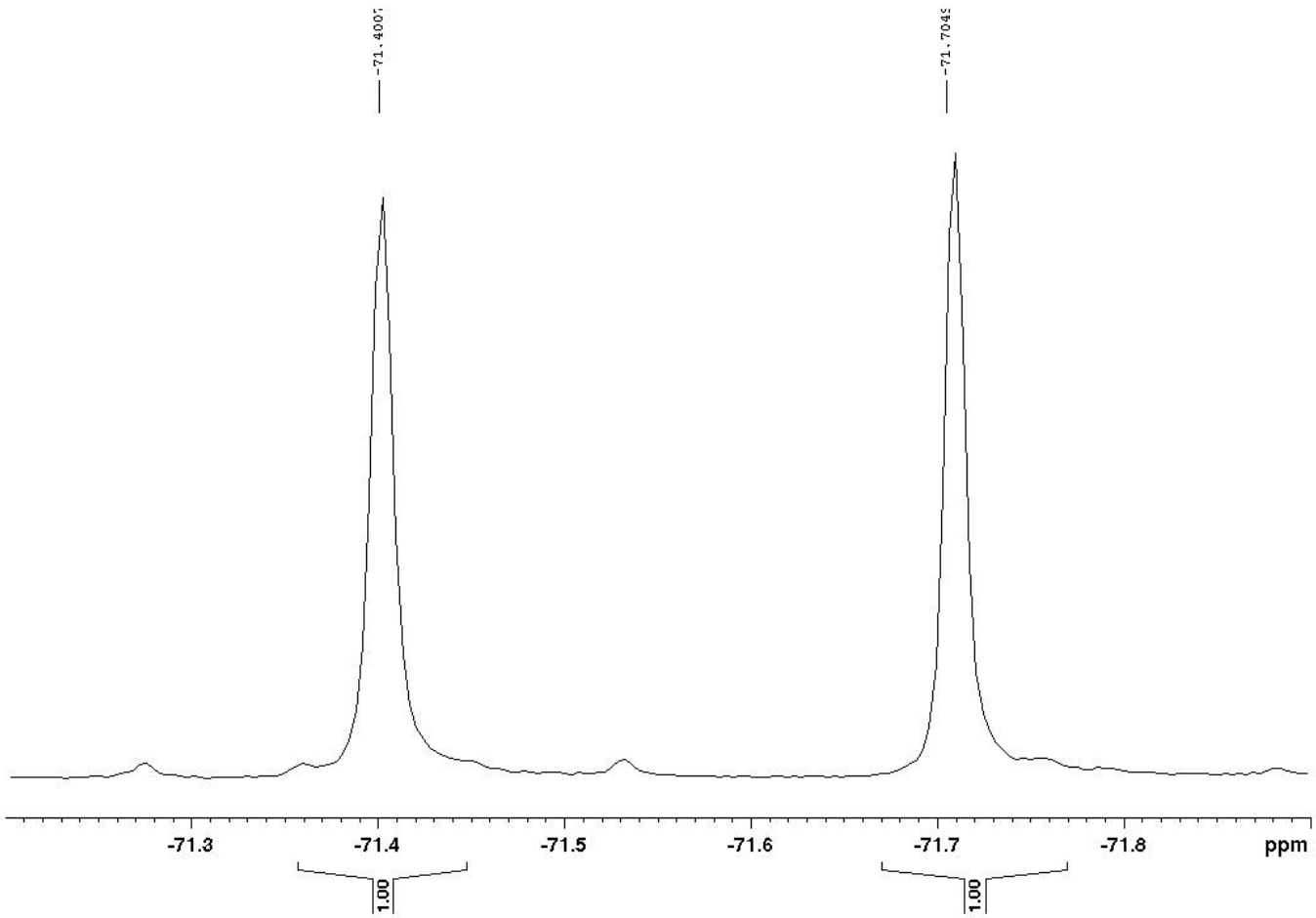
^{1}H NMR


7.312
7.247
6.873
6.851
6.105
4.626
4.614
4.609
4.597
4.399
4.384
3.797
2.782
2.251
2.234
2.217
1.716
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
7.05
1.99
0.93
1.00
2.03
3.07
0.99
2.02
4.06

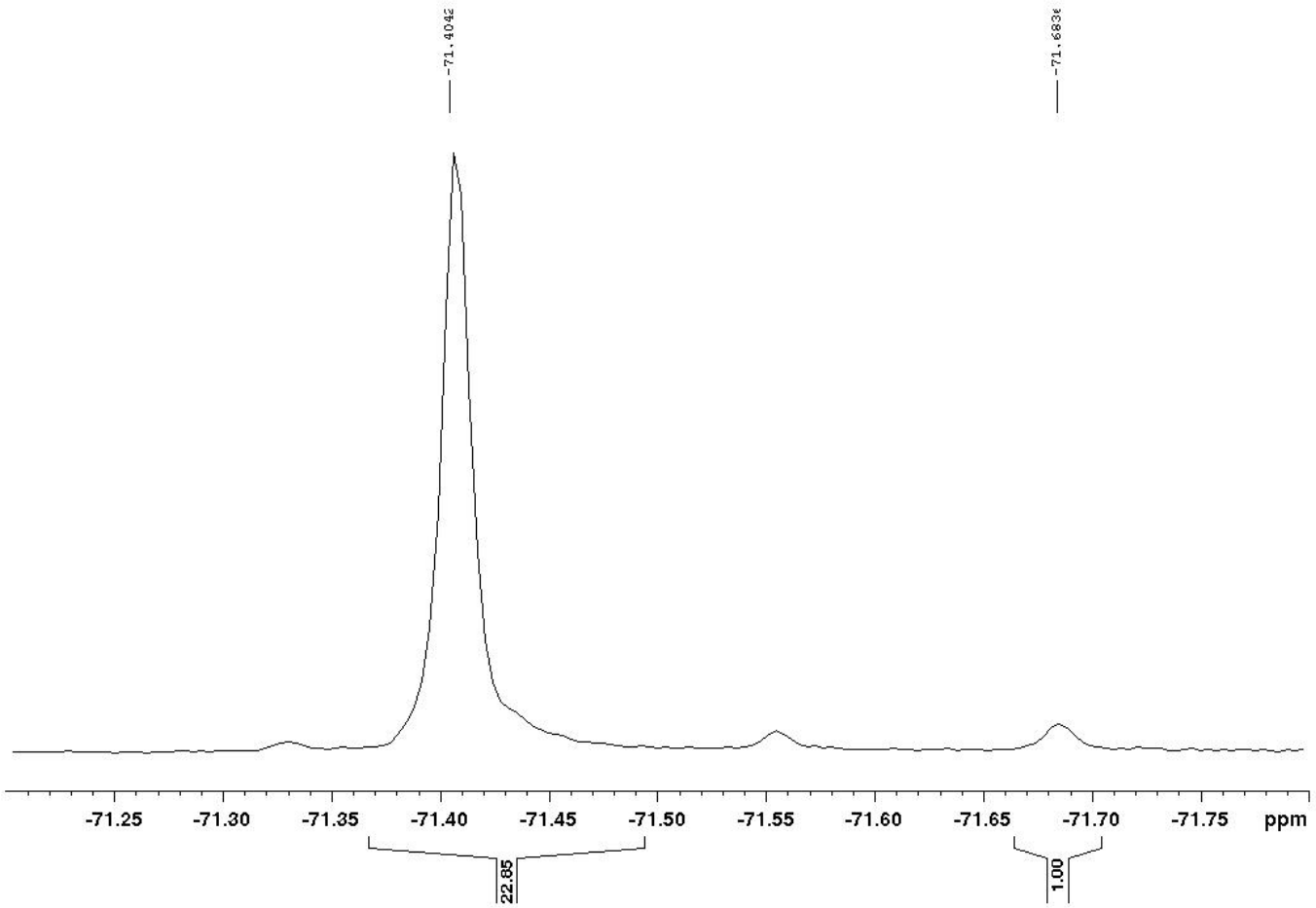
^{13}C NMR


172.94
159.11
138.44
136.94
128.86
127.96
127.61
127.18
113.95
73.76
55.40
43.71
38.44
36.31
22.06
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

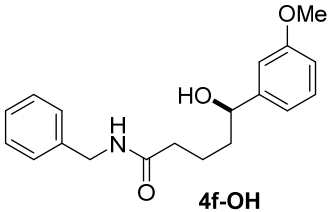
^{19}F NMR of Mosher's ester 4e-Mosher



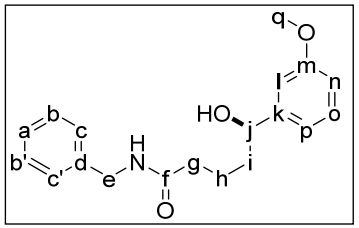
Mixture of diastereomeric 4e-Mosher


-71.4042
-71.6836
-71.25
-71.30
-71.35
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
ppm
22.85
1.00

(*R*,*S*)-**4e-Mosher**

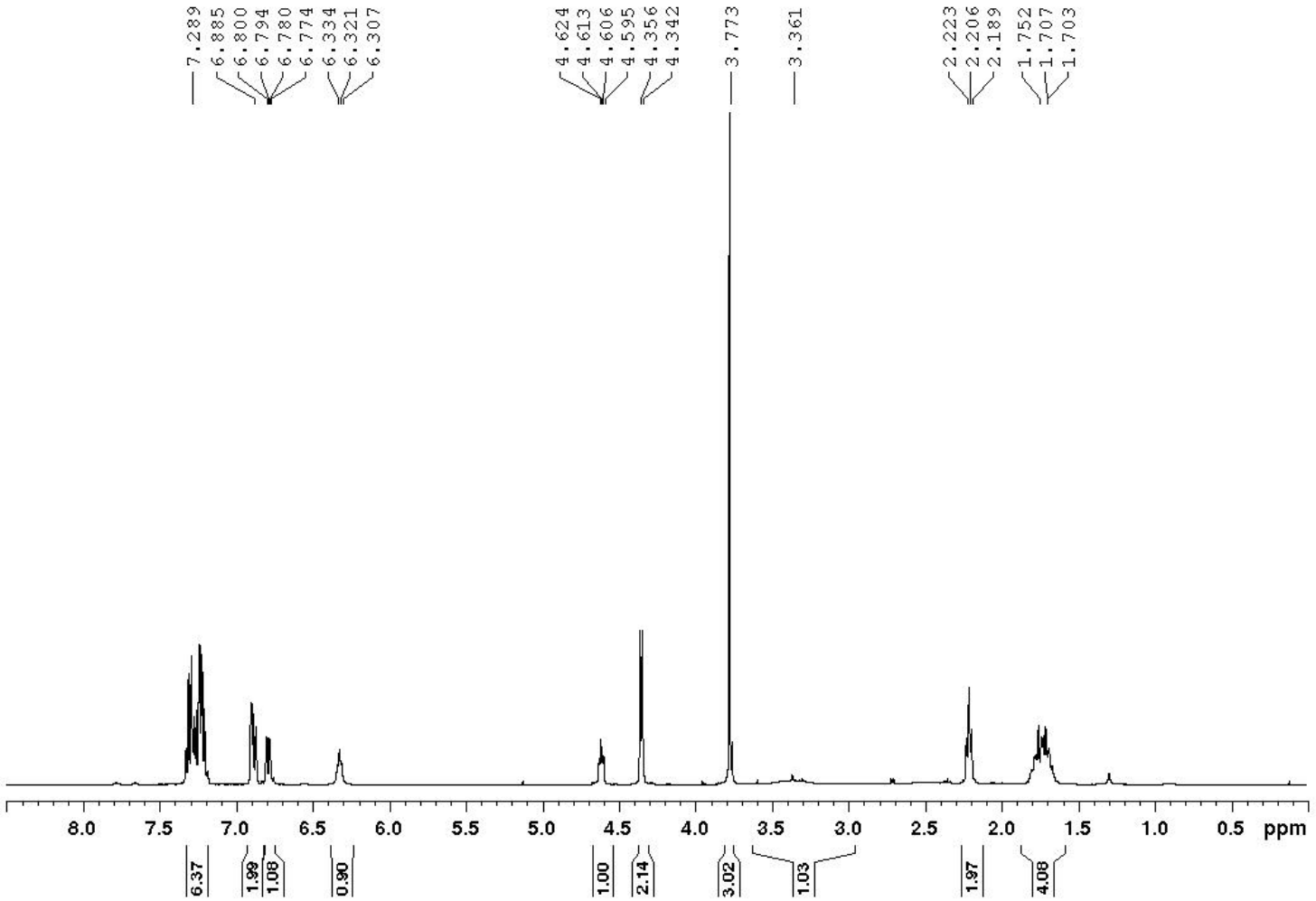


4f-OH

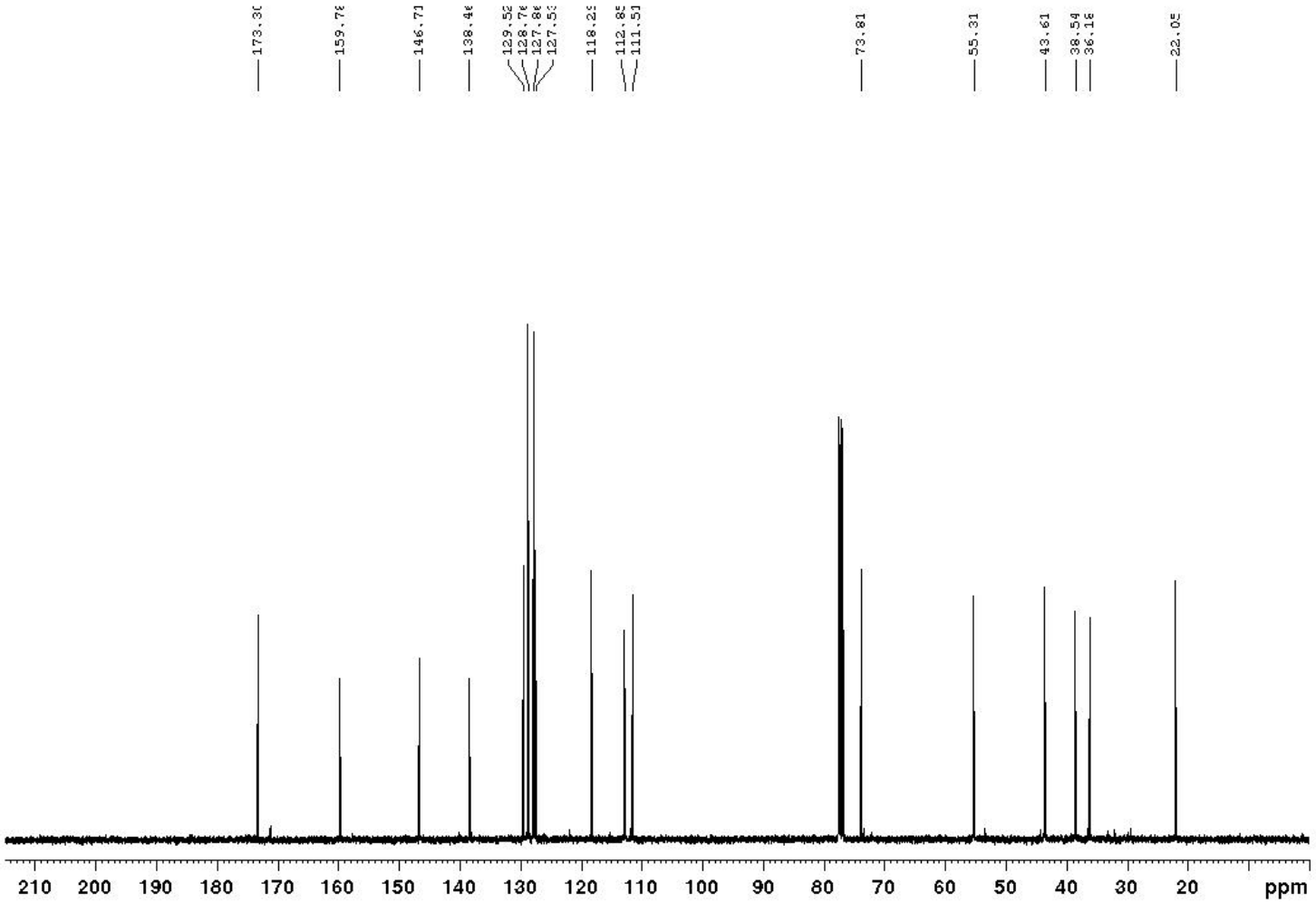


$[\alpha]_D^{20}$	–15.9° (*c* 2.0, $CHCl_3$)
Enantiomeric ratio analysis	Chiral HPLC analysis (Chiralcel-AD, 60:40 hexanes:isopropanol, flowrate = 1.0 mL/min) showed peaks at 15 minutes (97.0% (*R*)) and 19 minutes (3.0% (*S*))
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (6H, m, a,b,b',c,c',o), 6.85–6.90 (2H, m, l,p), 6.79 (1H, dd, *J* = 8.0 and 2.0 Hz, n), 6.32 (1H, dd, *J* = 5.3 and 0.0 Hz, NH), 4.61 (1H, dd, *J* = 7.4 and 4.3 Hz, j), 4.35 (2H, d, *J* = 5.7 Hz, e), 3.78 (3H, s, q), 3.36 (1H, br s, OH), 2.21 (2H, t, *J* = 6.8 Hz, g), 1.60–1.85 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.30 (f), 159.78 (m), 146.71 (k), 138.46 (d), 129.52 (o), 128.76 (b,b'), 127.86 (c,c'), 127.53 (a), 118.29 (p), 112.85 (n), 111.51 (l), 73.81 (j), 55.31 (q), 43.61 (e), 38.54 (i), 36.18 (g), 22.05 (h)
IR (neat)	3299 (O–H stretch), 2917, 1643 (C=O stretch), 1602, 1585, 1540, 1487, 1454, 1433, 1317, 1253, 1153, 1040, 783, 731, 696 cm^{-1}
HRMS (EI)	$C_{19}H_{23}NO_3$ (M): 313.1678, found 313.1680 *m/z*

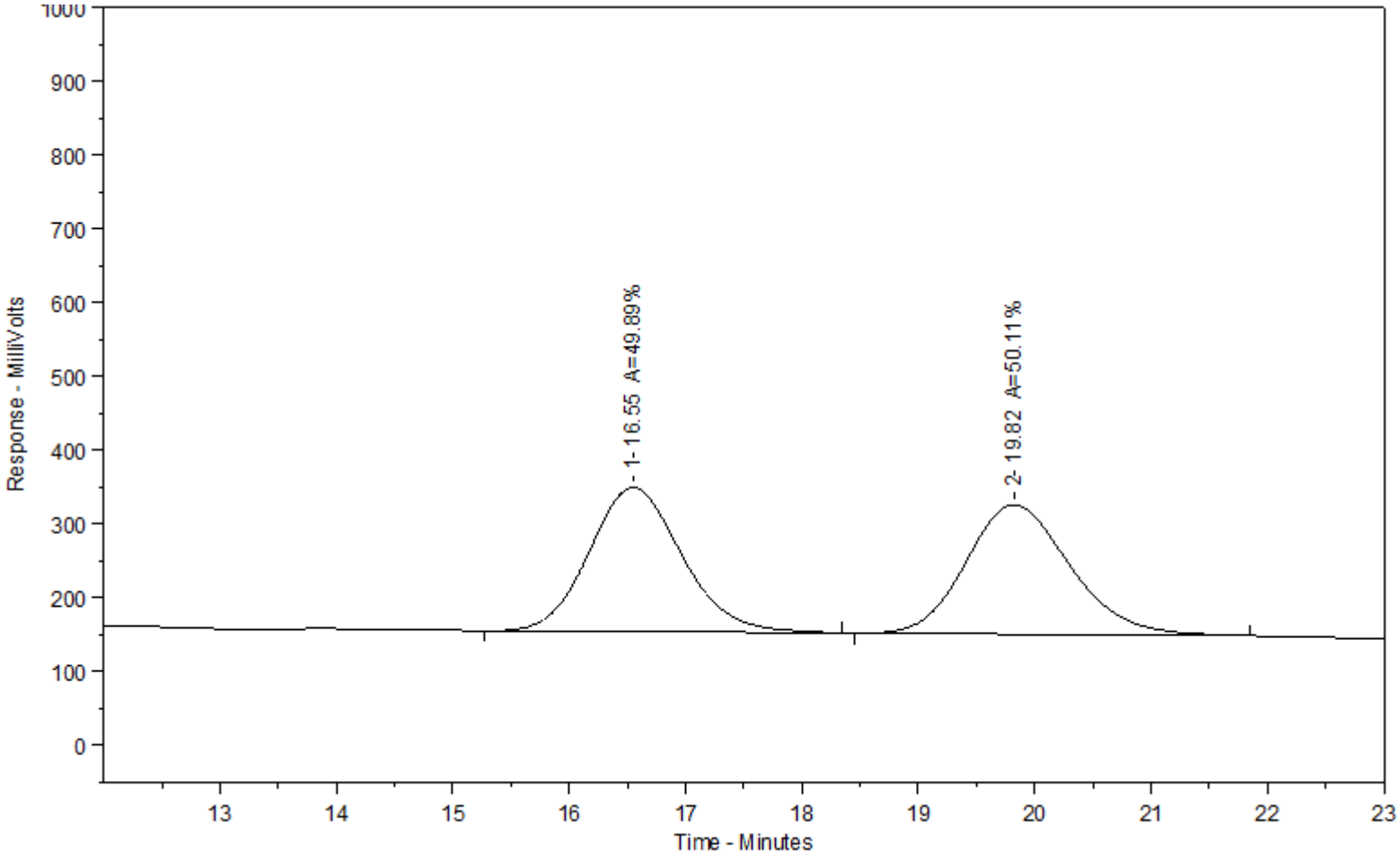
^{1}H NMR


7.289
6.885
6.800
6.794
6.780
6.774
6.334
6.321
6.307
4.624
4.613
4.606
4.595
4.356
4.342
3.773
3.361
2.223
2.206
2.189
1.752
1.707
1.703
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
6.37
1.99
1.08
0.90
1.00
2.14
3.02
1.03
1.97
4.08

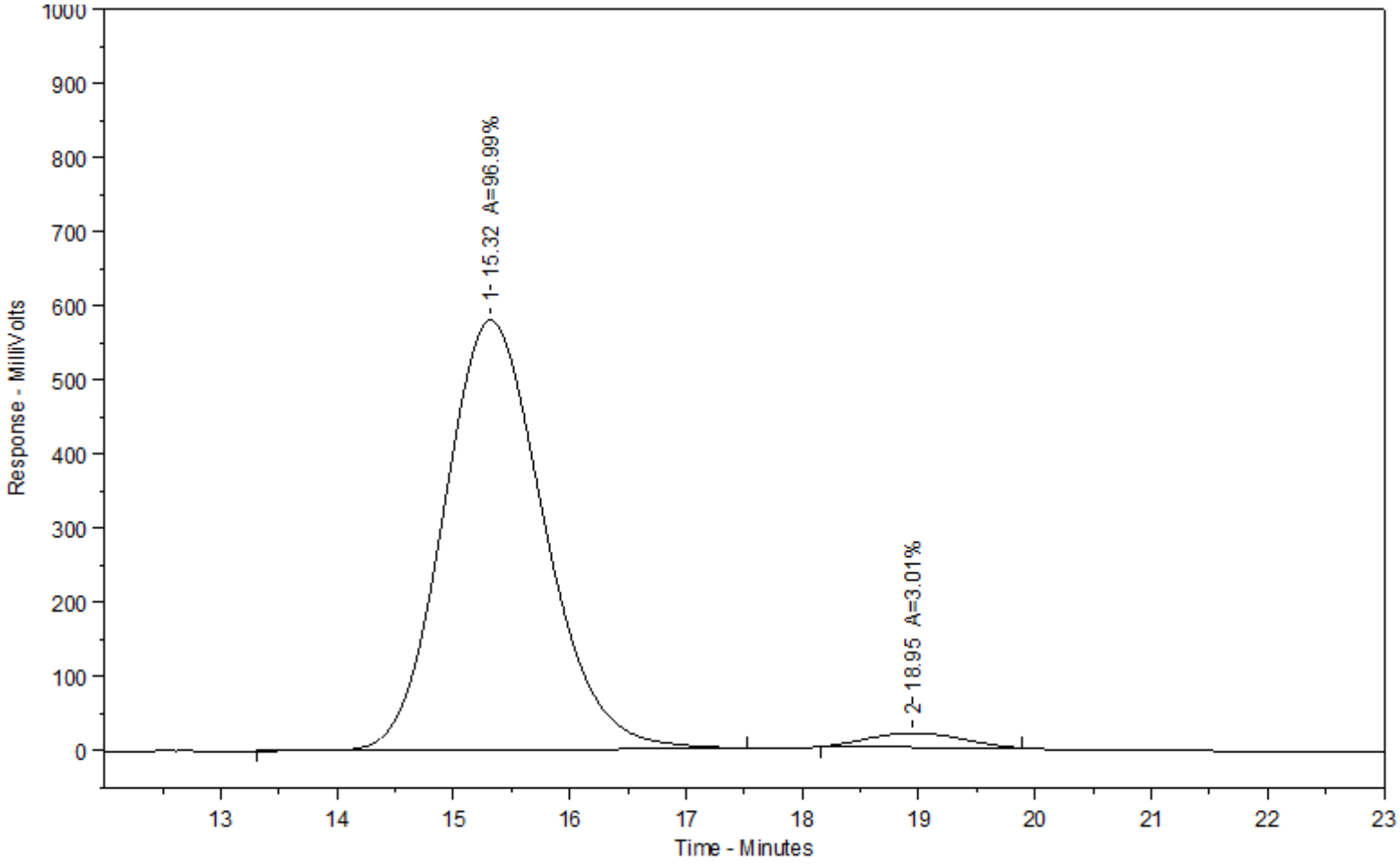
^{13}C NMR


173.30
159.78
146.71
138.48
129.52
128.78
127.88
127.53
118.25
112.85
111.53
73.81
55.31
43.61
38.54
36.18
22.05
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

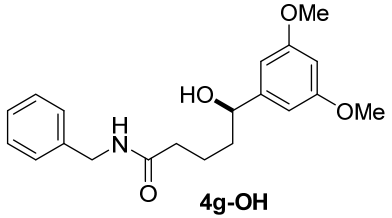
HPLC trace of 4f-OH



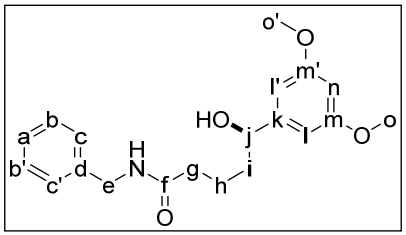
Racemic **4f-OH**


1000
900
800
700
600
500
400
300
200
100
0
Response - MilliVolts
1- 15.32 A=96.99%
2- 18.95 A=3.01%
13
14
15
16
17
18
19
20
21
22
23
Time - Minutes

(*R*)-**4f-OH**

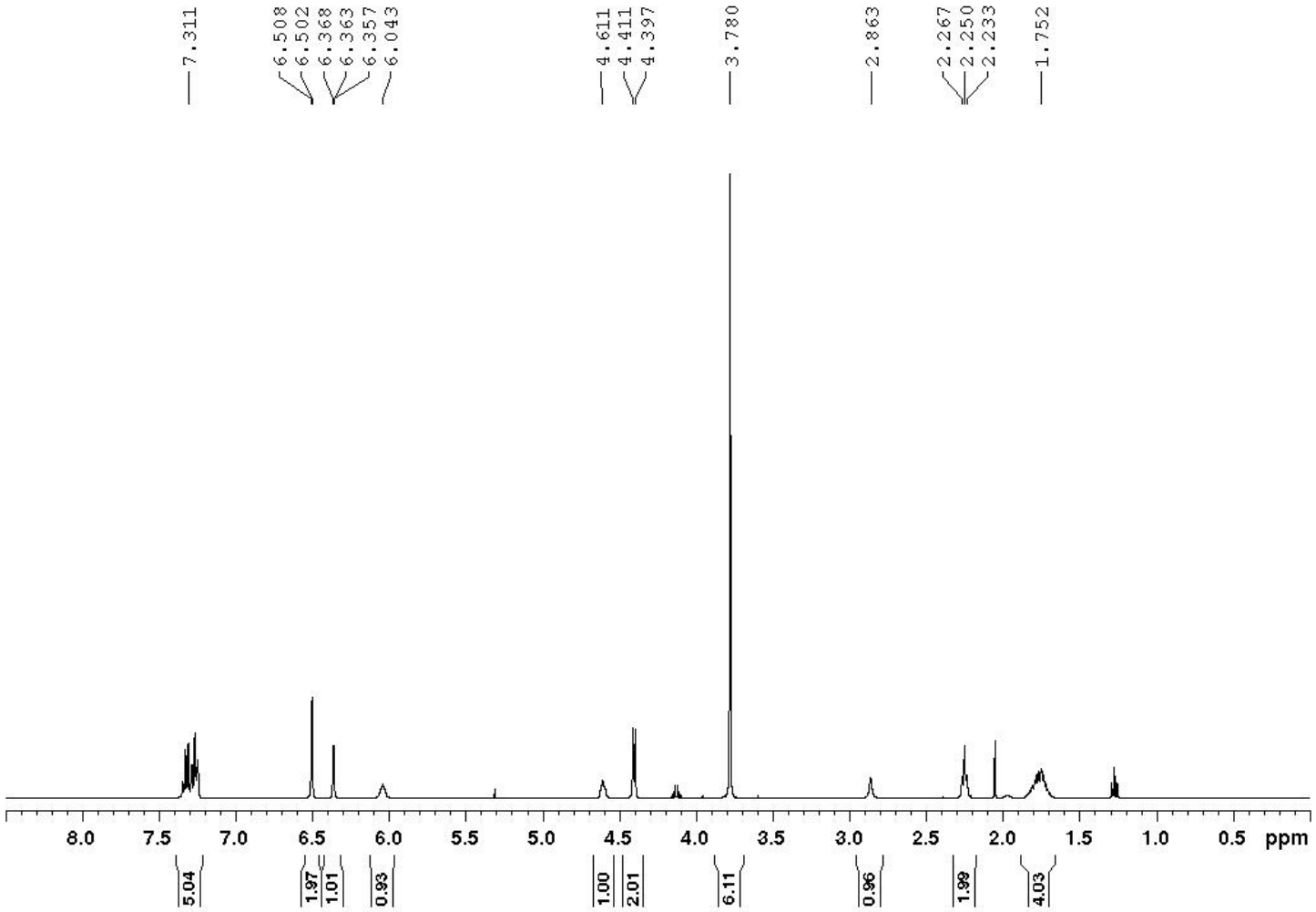


4g-OH

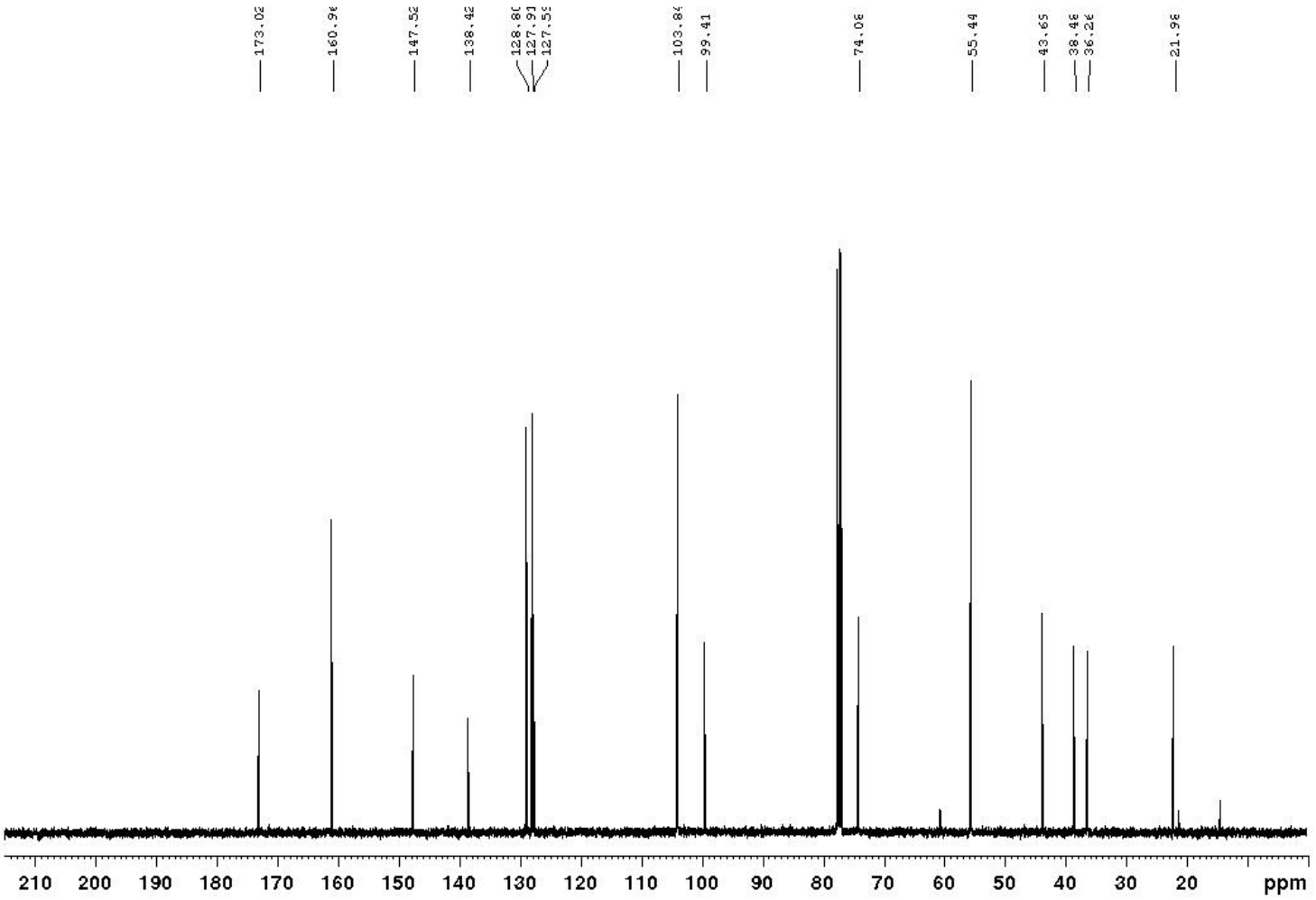


$[\alpha]_D^{20}$	–14.1° (*c* 2.0, $CHCl_3$)
m.p.	110.0–110.5 °C
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4g-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.34 (major 96.5%) and –71.50 (minor 3.5%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (5H, m, a,b,b',c,c',o), 6.50 (2H, d, *J* = 2.2 Hz, l,l'), 6.36 (1H, t, *J* = 2.2 Hz, n), 6.04 (1H, br s, NH), 4.55–4.65 (1H, m, j), 4.40 (2H, d, *J* = 5.7 Hz, e), 3.78 (6H, s, o,o'), 2.86 (1H, br s, OH), 2.25 (2H, t, *J* = 6.8 Hz, g), 1.65–1.85 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.02 (f), 160.95 (m,m'), 147.52 (k), 138.42 (d), 128.80 (b,b'), 127.91 (c,c'), 127.59 (a), 103.84 (l,l'), 99.41 (n), 74.08 (j), 55.44 (o,o'), 43.69 (e), 38.48 (i), 36.26 (g), 21.98 (h)
IR (neat)	3305, 3276 (O–H stretch), 2943, 2833, 2359, 2342, 1633 (C=O stretch), 1598, 1537, 1464, 1452, 1204, 1192, 1148, 1083, 1051, 859, 822, 747, 693 cm^{-1}
HRMS (EI)	$C_{20}H_{25}NO_4$ (M): 343.1784, found 343.1799 *m/z*

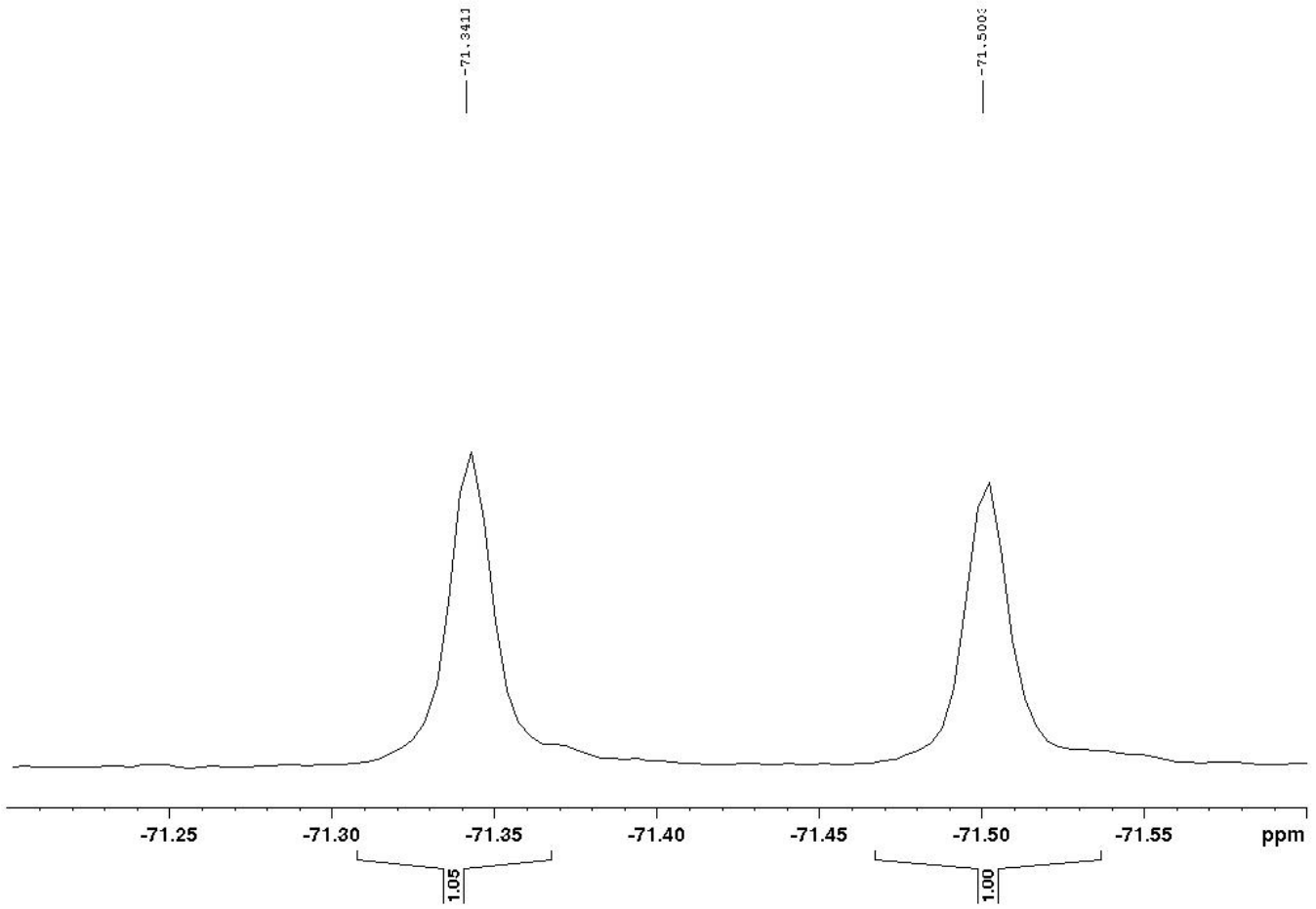
^{1}H NMR


7.311
6.508
6.502
6.368
6.363
6.357
6.043
4.611
4.411
4.397
3.780
2.863
2.267
2.250
2.233
1.752
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.04
1.97
1.01
0.93
1.00
2.01
6.11
0.96
1.99
4.03

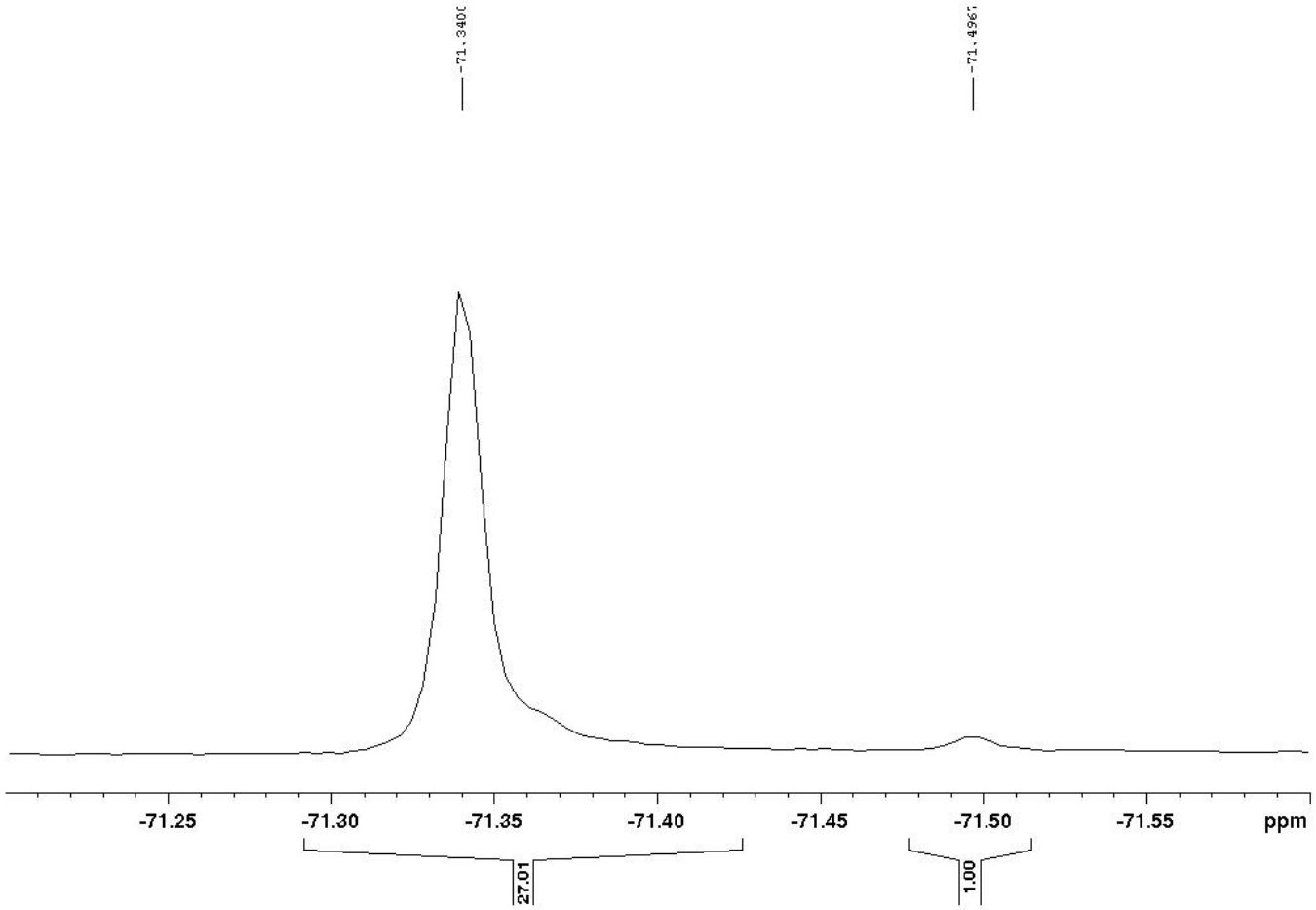
^{13}C NMR


173.02
160.98
147.52
138.42
128.80
127.91
127.55
103.84
99.41
74.08
55.44
43.65
38.48
36.28
21.98
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

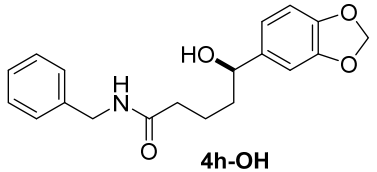
^{19}F NMR of Mosher's ester 4g-Mosher

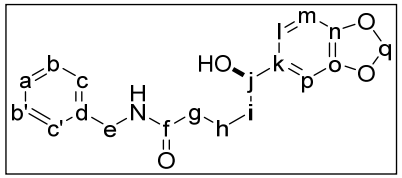


Mixture of diastereomeric 4g-Mosher


-71.3401
-71.4967
-71.25
-71.30
-71.35
-71.40
-71.45
-71.50
-71.55
ppm
27.01
1.00

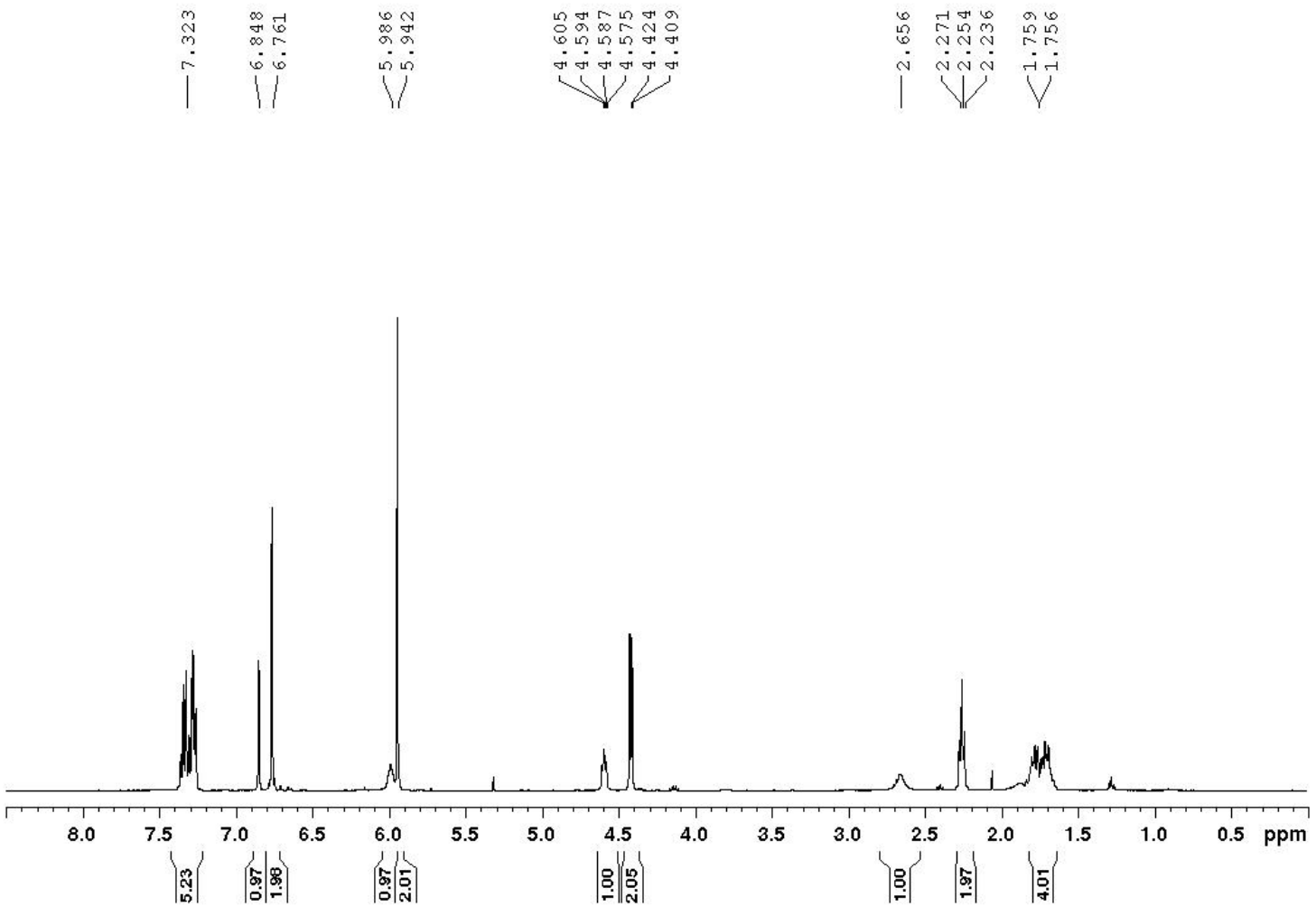
(*R*,*S*)-**4g-Mosher**



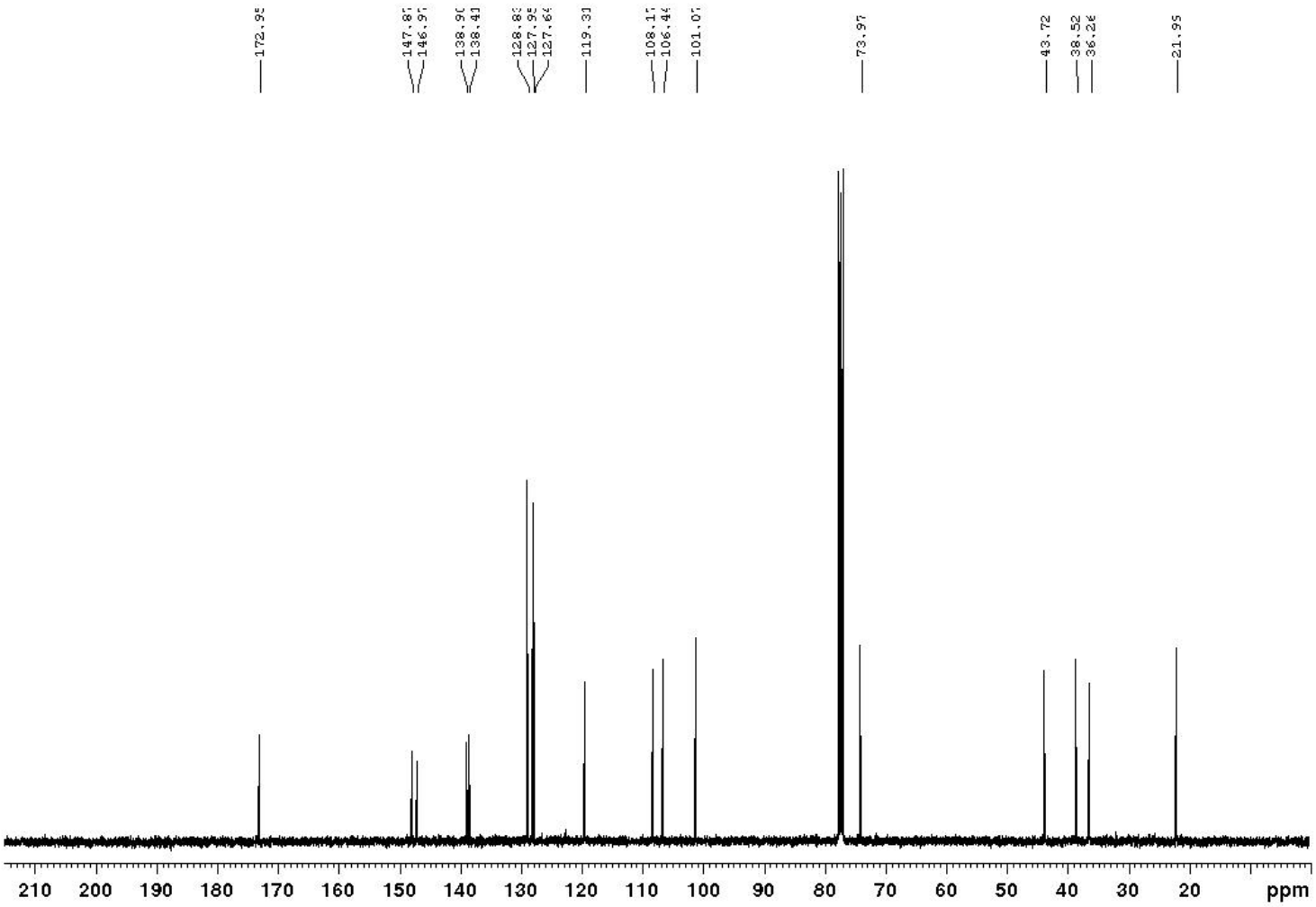


$[\alpha]_D^{20}$	–12.9° (*c* 1.0, $CHCl_3$)
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4h-Mosher;** crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.33 (major 92%) and –71.64 (minor 8%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.40 (5H, m, a,b,b',c,c'), 6.80–6.90 (1H, m, p), 6.75–6.80 (2H, m, l,m), 5.99 (1H, br s, NH), 5.94 (2H, s, q), 4.59 (1H, dd, *J* = 7.4 and 4.7 Hz, j), 4.42 (2H, d, *J* = 5.7 Hz, e), 2.66 (1H, br s, OH), 2.25 (2H, t, *J* = 6.8 Hz, g), 1.65–1.85 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.95 (f), 147.87 (o), 146.97 (n), 138.90 (k), 138.41 (d), 128.83 (b,b'), 127.95 (c,c'), 127.64 (a), 119.31 (l), 108.17 (m), 106.44 (p), 101.07 (q), 73.97 (j), 43.72 (e), 38.52 (i), 36.26 (g), 21.99 (h)
IR (neat)	3295 (O–H stretch), 2923, 1644 (C=O stretch), 1545, 1501, 1486, 1440, 1238, 1036, 933, 809, 729, 697 cm^{-1}
HRMS (ESI)	$C_{19}H_{21}NNaO_4$ (M+Na): 350.1368, found 350.1375 *m/z*

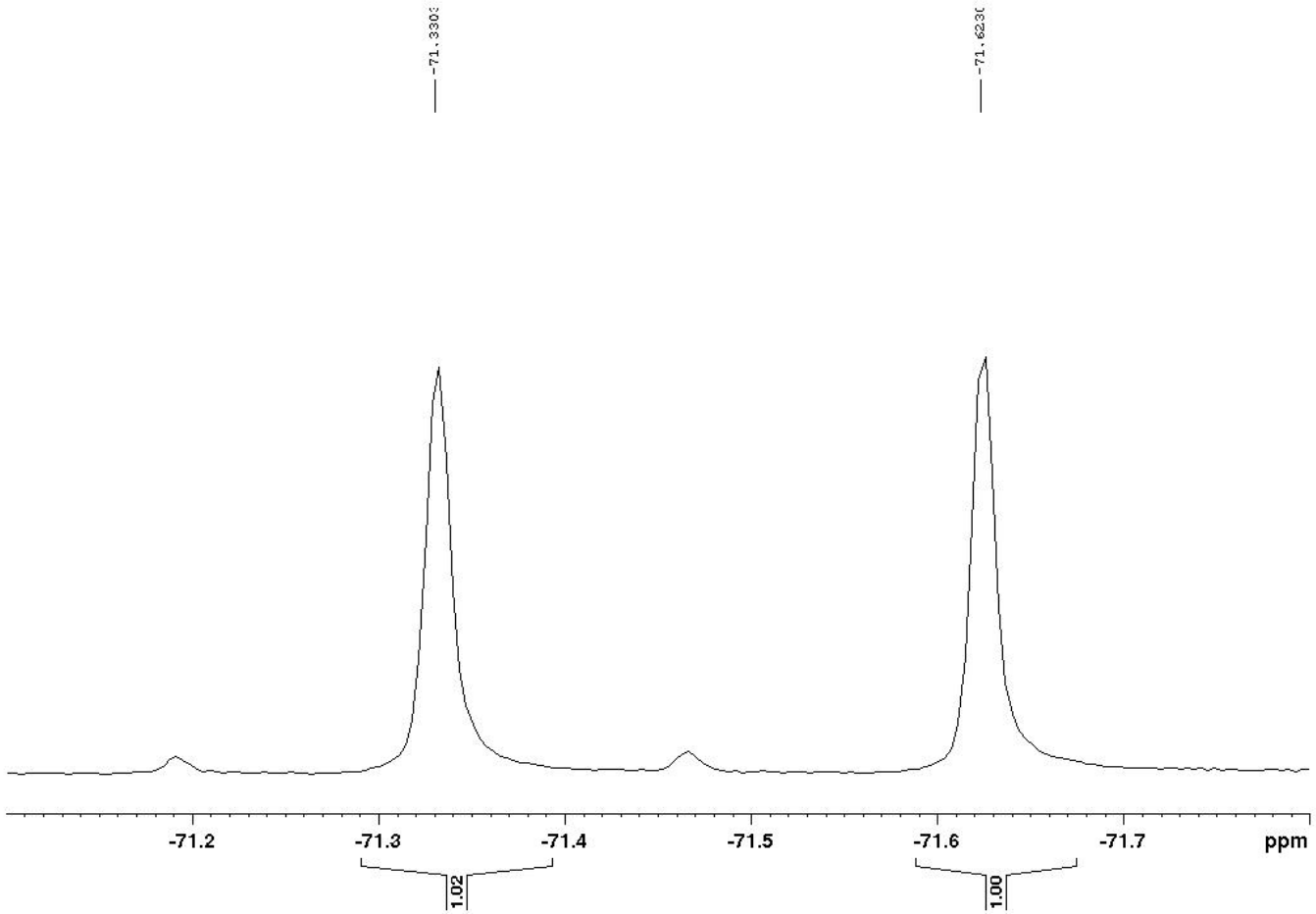
^{1}H NMR


7.323
6.848
6.761
5.986
5.942
4.605
4.594
4.587
4.575
4.424
4.409
2.656
2.271
2.254
2.236
1.759
1.756
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.23
0.97
1.98
0.97
2.01
1.00
2.05
1.00
1.97
4.01

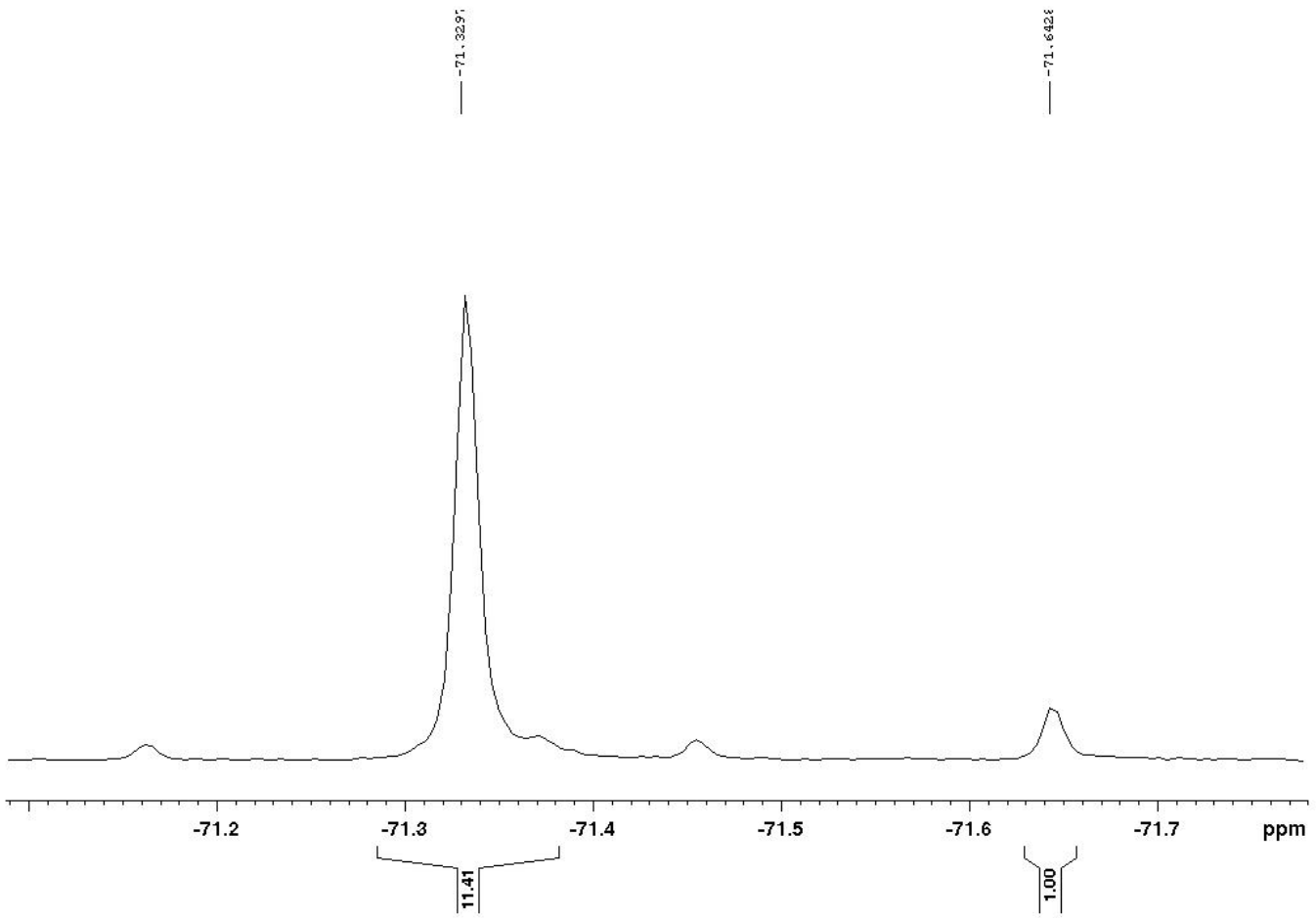
^{13}C NMR


172.95
147.87
146.97
138.90
138.41
128.83
127.95
127.64
119.30
108.17
106.46
101.07
73.97
43.72
38.52
36.26
21.95
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

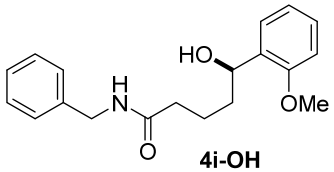
^{19}F NMR of Mosher's ester 4h-Mosher



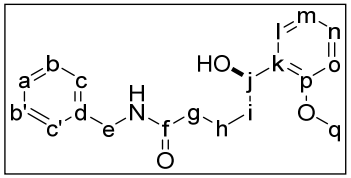
Mixture of diastereomeric 4h-Mosher



(*R*,*S*)-**4h-Mosher**

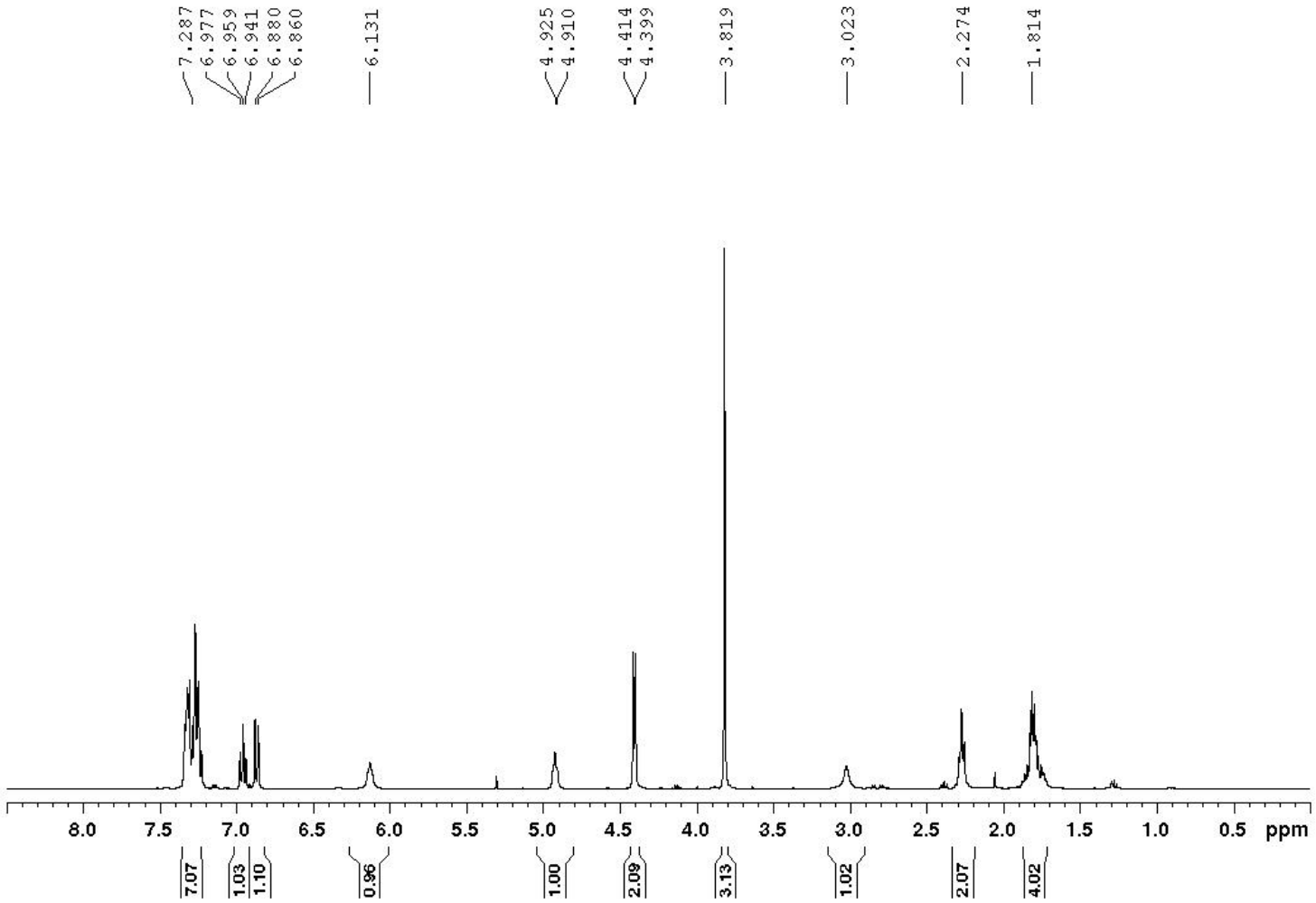


4i-OH

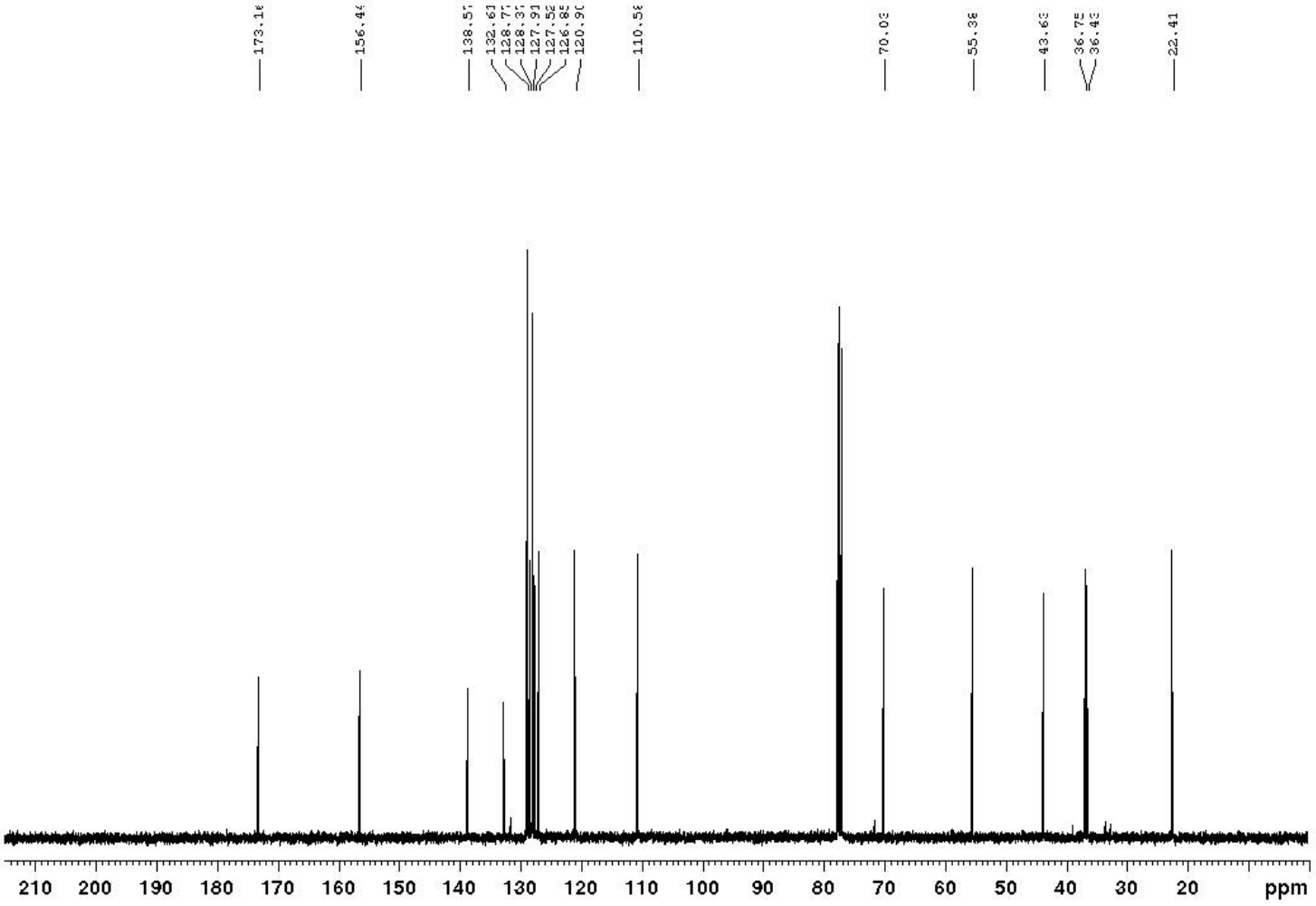


$[\alpha]_D^{20}$	–10.8º (*c* 2.0, $CHCl_3$)
m.p.	79.5–81.0 ºC
Enantiomeric ratio analysis	Chiral HPLC analysis (Chiralcel-AD, 60:40 hexanes:isopropanol, flowrate = 1.0 mL/min) showed peaks at 17 minutes (85.5% (*R*)) and 23 minutes (14.5% (*S*))
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (7H, m, a,b,b',c,c',l,m), 6.96 (1H, t, *J* = 7.1 Hz, n), 6.87 (1H, d, *J* = 8.2 Hz, o), 6.13 (1H, br s, NH), 4.85–5.00 (1H, m, j), 4.41 (2H, d, *J* = 5.7 Hz, e), 3.82 (3H, s, q), 3.02 (1H, br s, OH), 2.20–2.30 (2H, m, g), 1.70–1.90 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.16 (f), 156.44 (p), 138.57 (d), 132.61 (k), 128.77 (b,b'), 128.37 (l), 127.91 (c,c'), 127.52 (a), 126.85 (n), 120.90 (m), 110.58 (o), 70.03 (j), 55.38 (q), 43.63 (e), 36.74 (i), 36.43 (g), 22.41 (h)
IR (neat)	3294 (O–H stretch), 2923, 1643 (C=O stretch), 1601, 1586, 1544, 1490, 1454, 1438, 1285, 1237, 1049, 1027, 752, 697 cm^{-1}
HRMS (ESI)	$C_{19}H_{23}NNaO_3$ (M+Na): 336.1576, found 336.1583 *m/z*

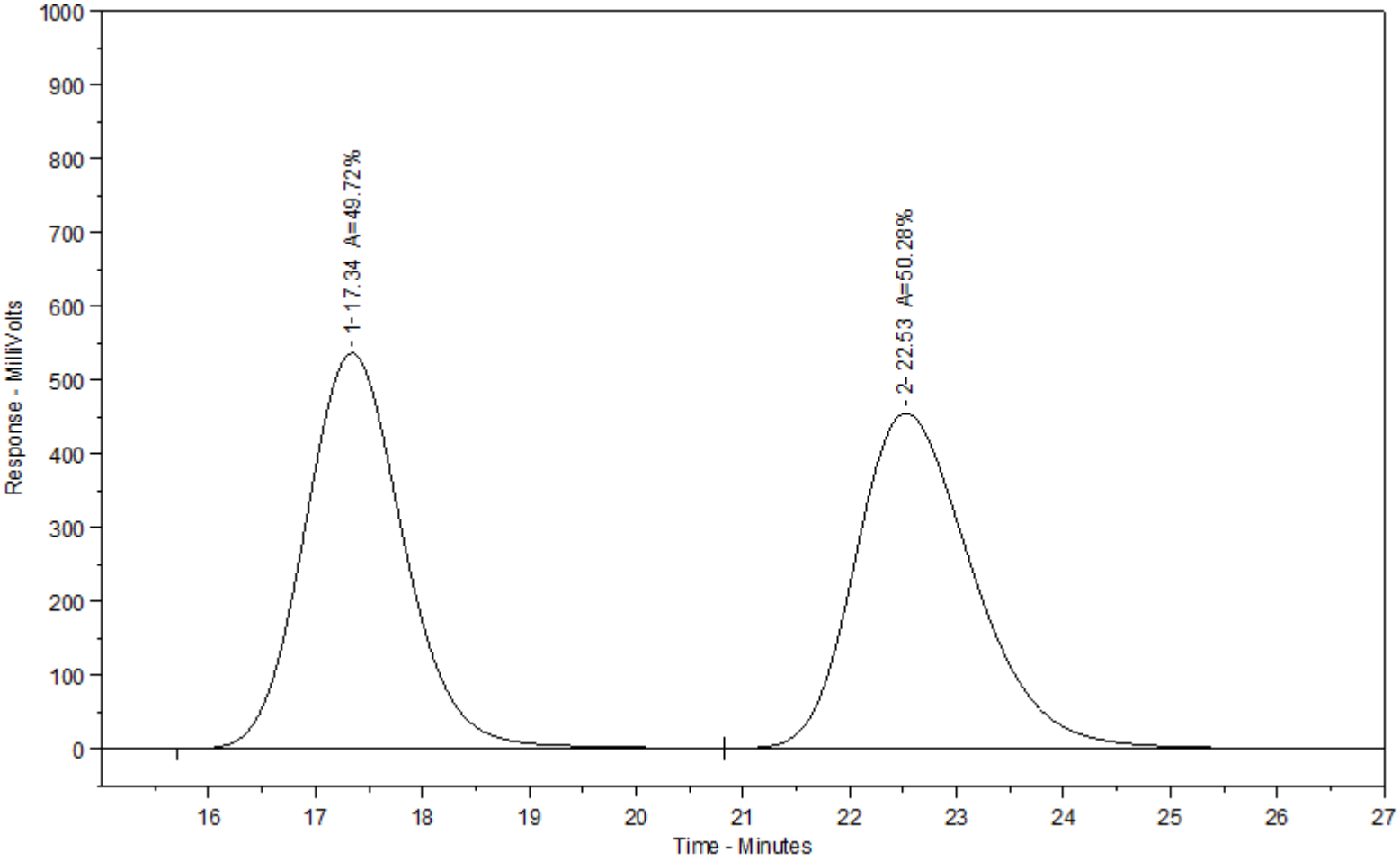
^{1}H NMR


7.287
6.977
6.959
6.941
6.880
6.860
6.131
4.925
4.910
4.414
4.399
3.819
3.023
2.274
1.814
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
7.07
1.03
1.10
0.96
1.00
2.09
3.13
1.02
2.07
4.02

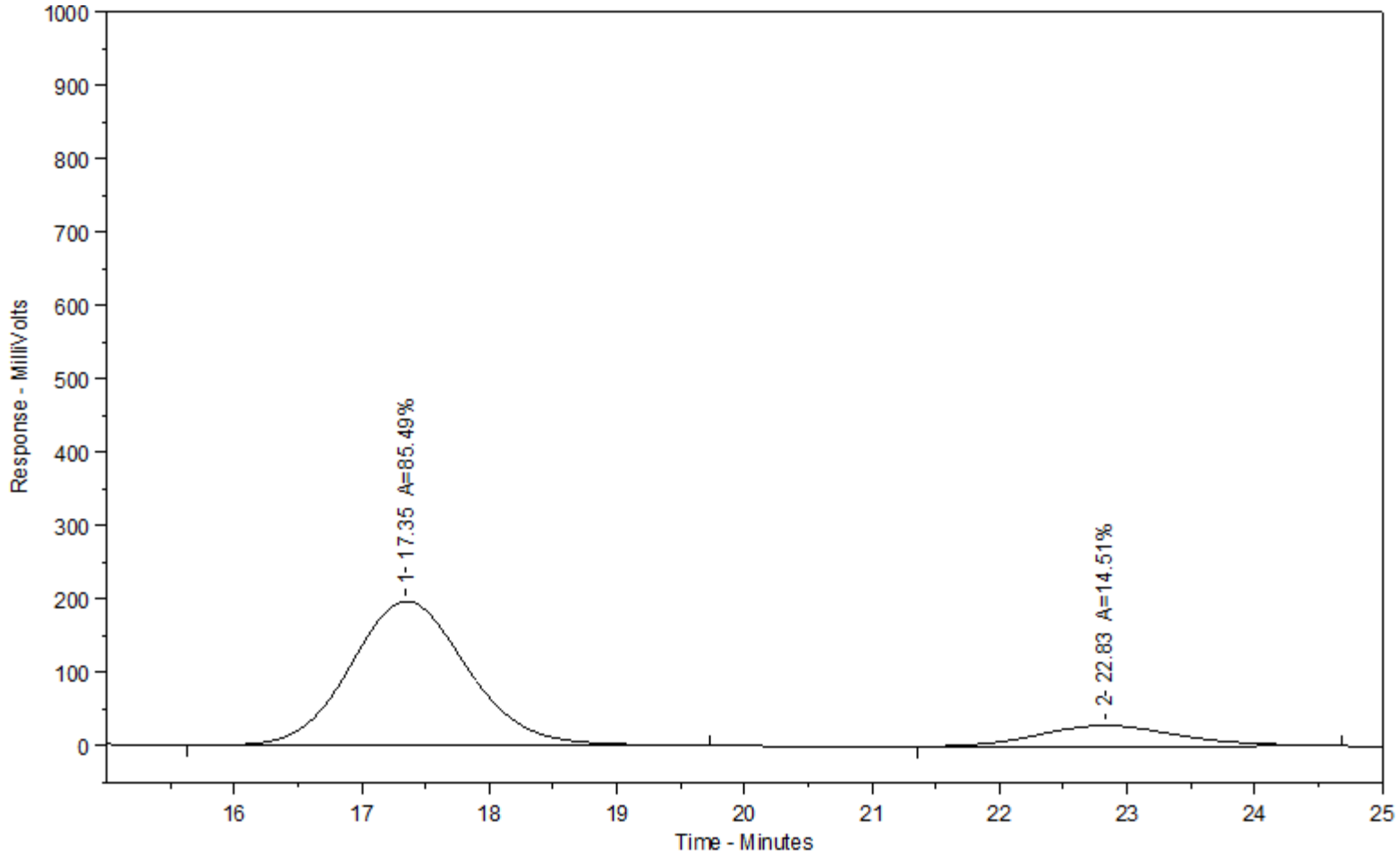
^{13}C NMR


173.1
156.4
138.5
132.6
128.7
128.3
127.9
127.5
126.8
120.9
110.5
70.0
55.3
43.6
36.7
36.4
22.4
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

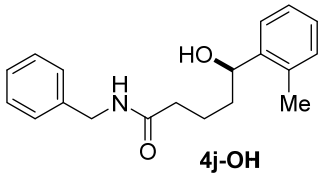
HPLC trace of 4i-OH



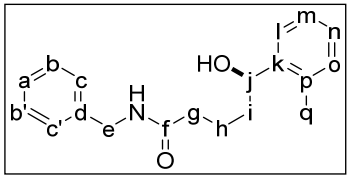
Racemic **4i-OH**


1000
900
800
700
600
500
400
300
200
100
0
Response - MilliVolts
1- 17.35 A=85.49%
2- 22.83 A=14.51%
16
17
18
19
20
21
22
23
24
25
Time - Minutes

(*R*)-**4i-OH**

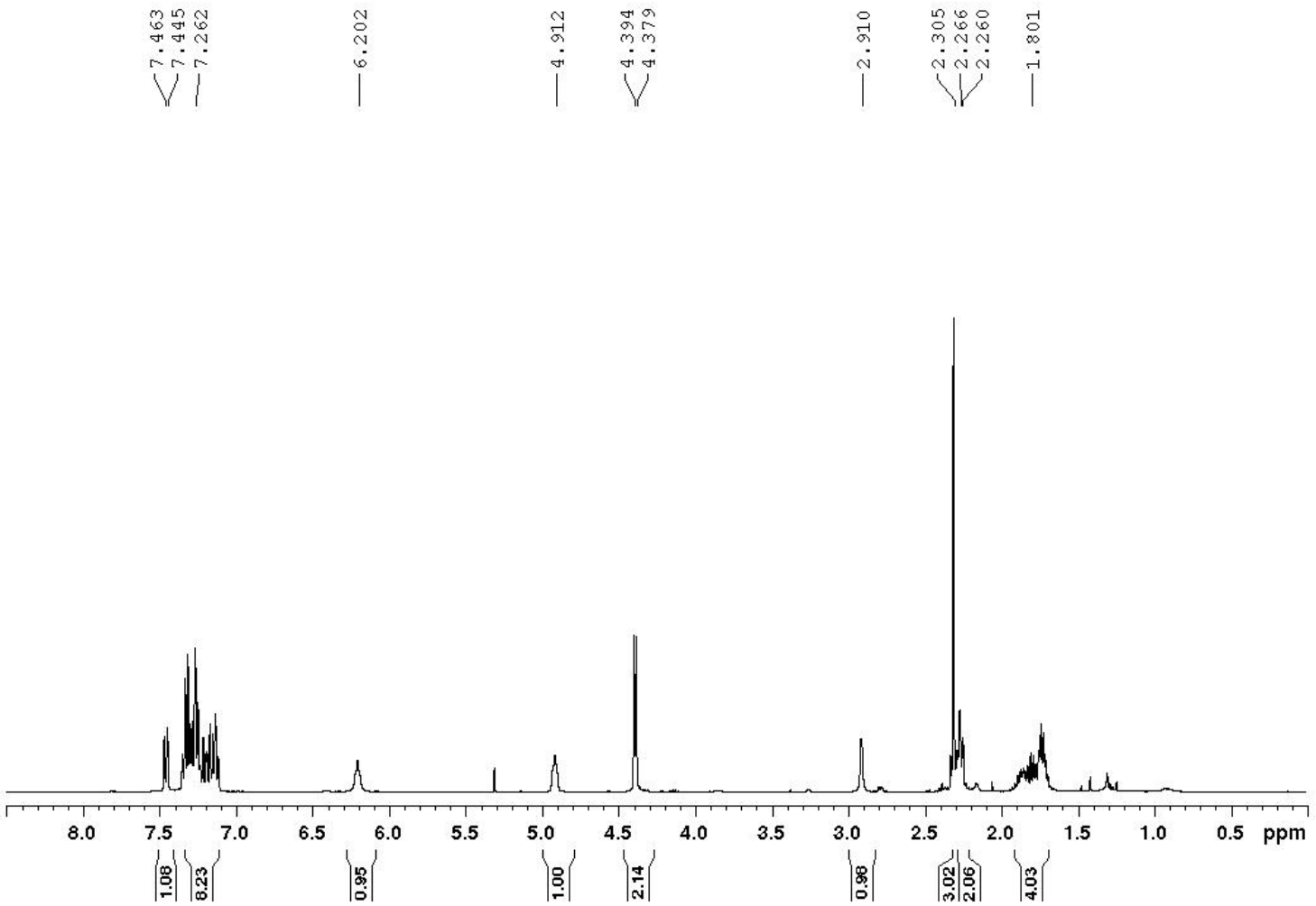


4j-OH

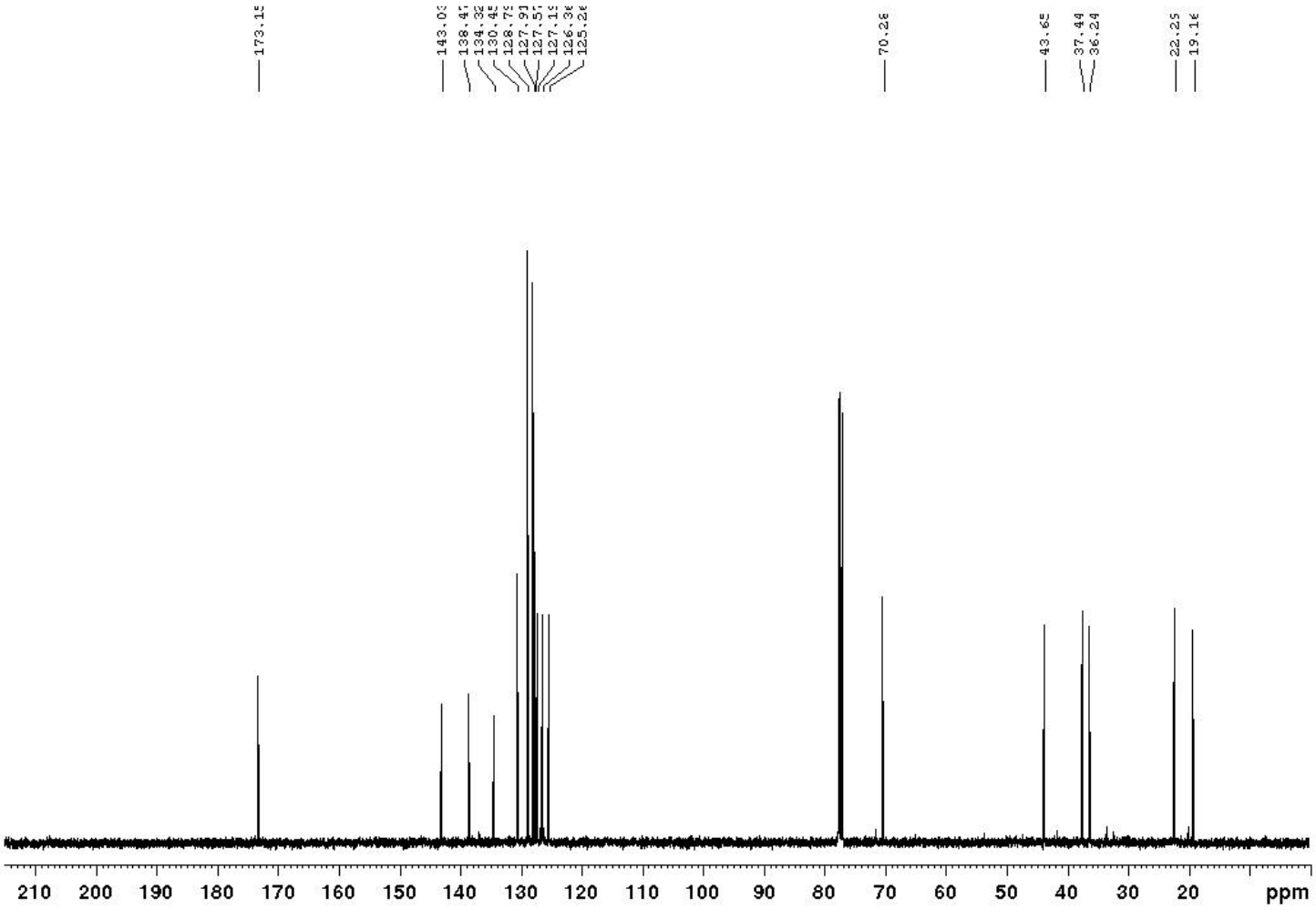


$[\alpha]_D^{20}$	–24.6° (*c* 2.0, $CHCl_3$)
HPLC analysis	Chiral HPLC analysis (Chiralpak-IC, 60:40 hexanes:isopropanol, flowrate = 1.4 mL/min) showed peaks at 14.5 minutes (93.0% (*R*)) and 16.5 minutes (7.0% (*S*))
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.45 (1H, d, *J* = 7.3 Hz, o), 7.10–7.35 (8H, m, a,b,b',c,c',l,m,n), 6.20 (1H, br s, NH), 4.85–4.95 (1H, m, j), 4.39 (2H, d, *J* = 5.7 Hz, e), 2.91 (1H, br s, OH), 2.31 (3H, s, q), 2.20–2.30 (2H, m, g), 1.70–1.90 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.15 (f), 143.03 (k), 138.47 (d), 134.32 (p), 130.45 (n), 128.79 (b,b'), 127.91 (c,c'), 127.57 (a), 127.19 (l), 126.36 (m), 125.26 (o), 70.28 (j), 43.65 (e), 37.44 (i), 36.24 (g), 22.29 (h), 19.16 (q)
IR (neat)	3292 (O–H stretch), 2920, 2360, 2342, 1635 (C=O stretch), 1538, 1495, 1453, 1059, 748, 724, 696 cm^{-1}
HRMS (EI)	$C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1725 *m/z*

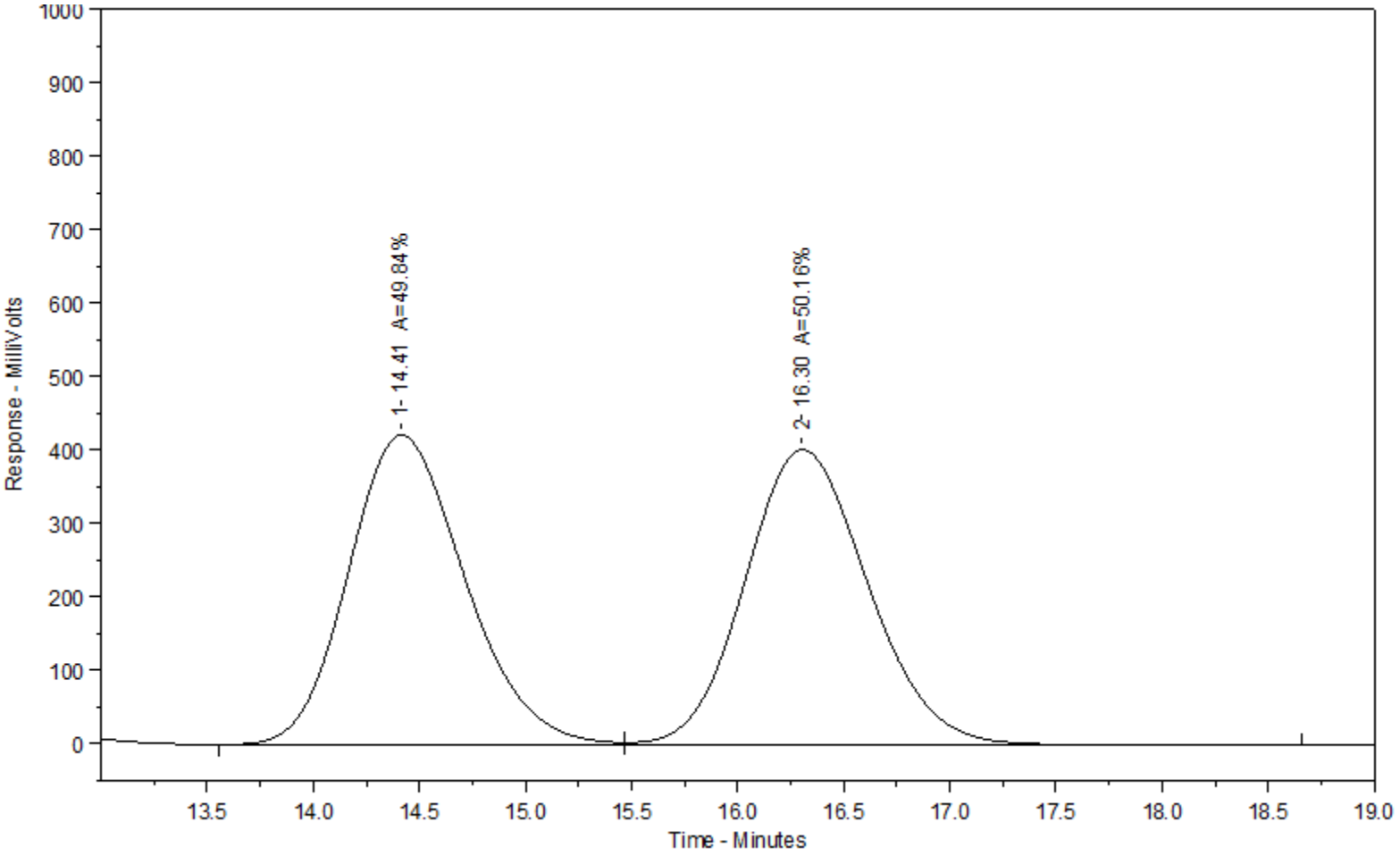
^{1}H NMR


7.463
7.445
7.262
6.202
4.912
4.394
4.379
2.910
2.305
2.266
2.260
1.801
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.08
6.23
0.95
1.00
2.14
0.96
3.02
2.06
4.03

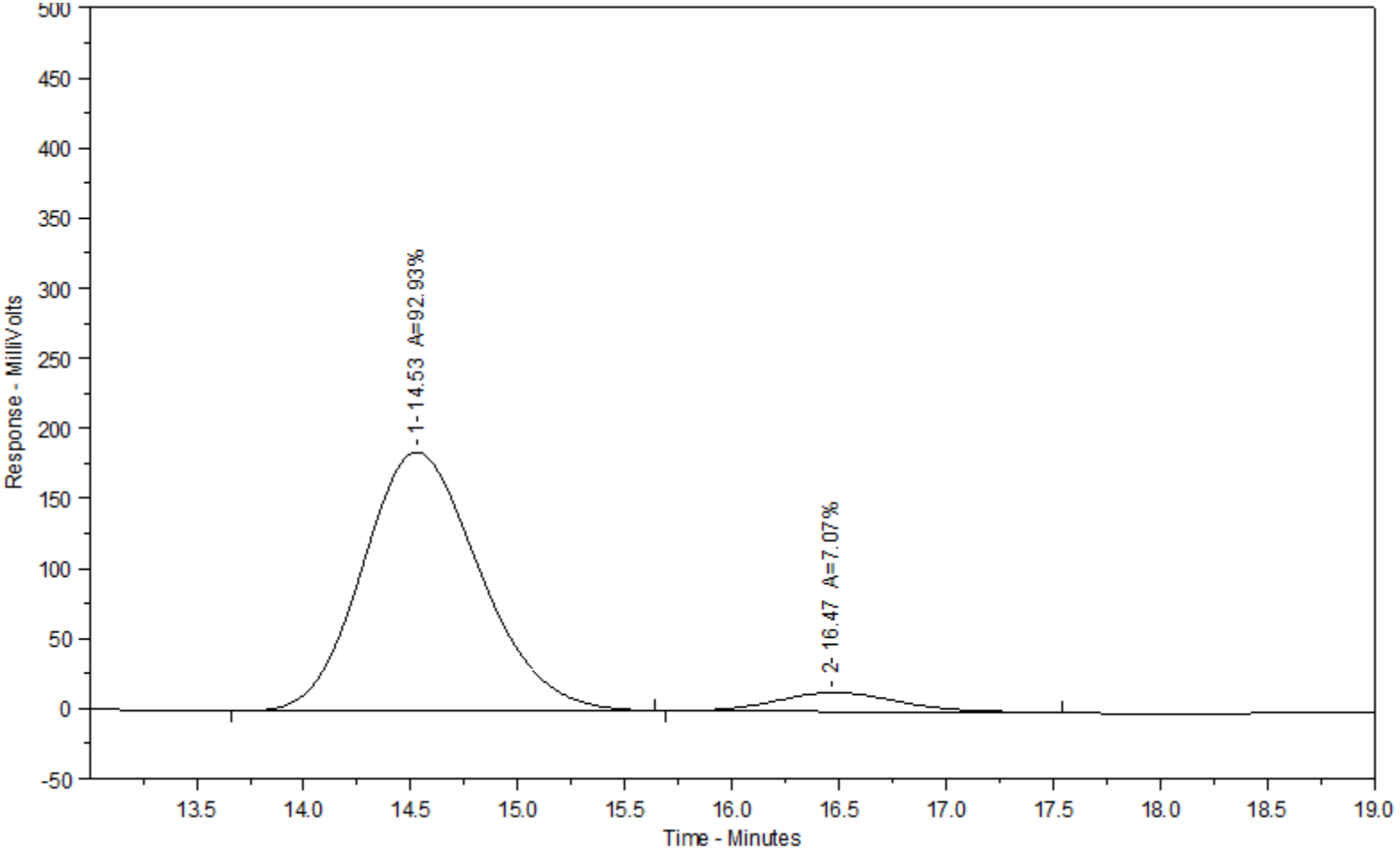
^{13}C NMR


173.15
143.03
138.47
134.32
130.45
128.75
127.91
127.57
127.15
126.38
125.24
70.28
43.65
37.44
36.24
22.25
19.16
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

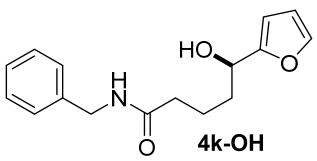
HPLC trace of 4j-OH



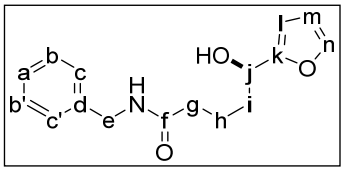
Racemic **4j-OH**


Response - MilliVolts
Time - Minutes
1- 14.53 A=92.93%
2- 16.47 A=7.07%
500
450
400
350
300
250
200
150
100
50
0
-50
13.5
14.0
14.5
15.0
15.5
16.0
16.5
17.0
17.5
18.0
18.5
19.0

(*R*)-**4j-OH**

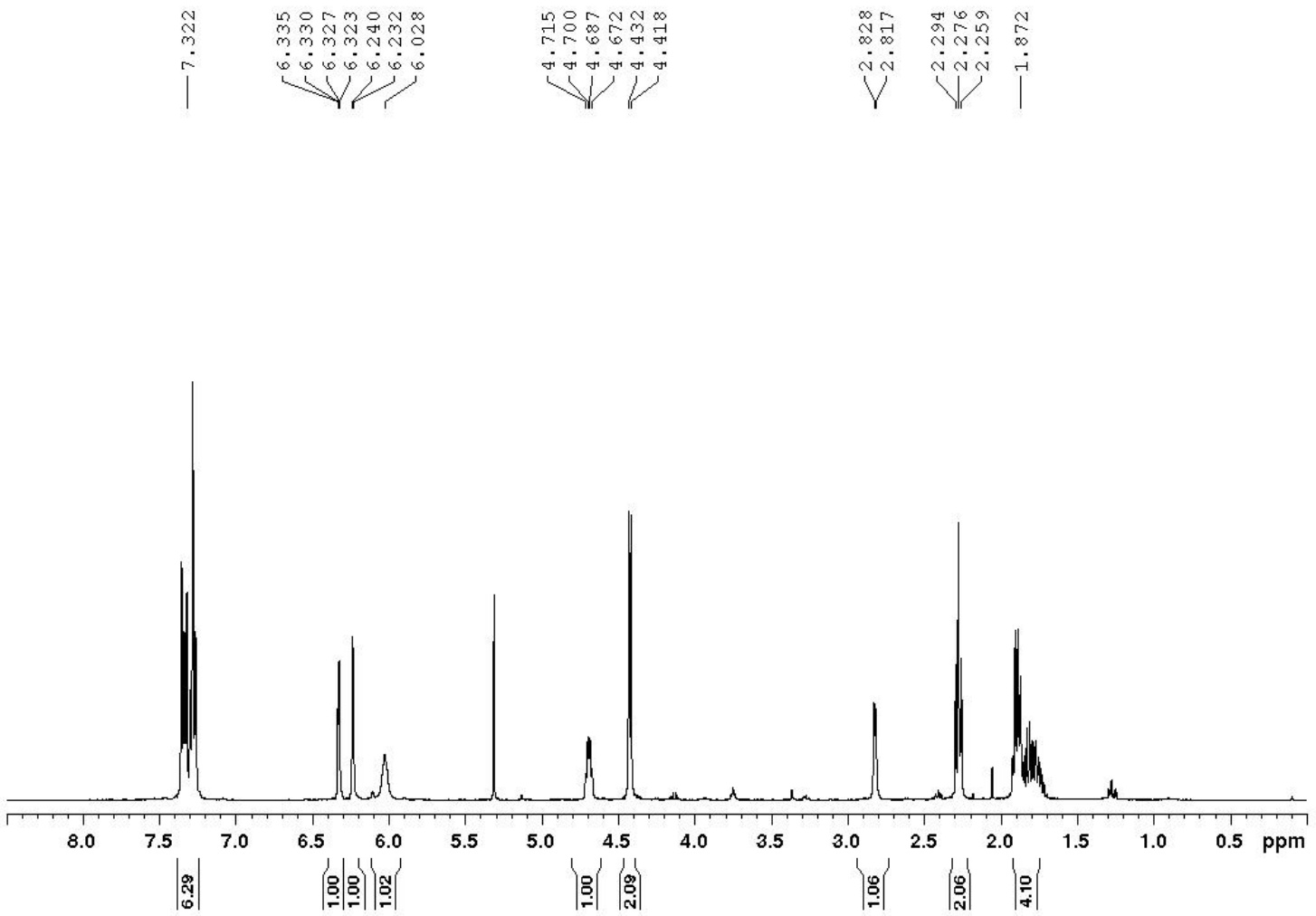


4k-OH

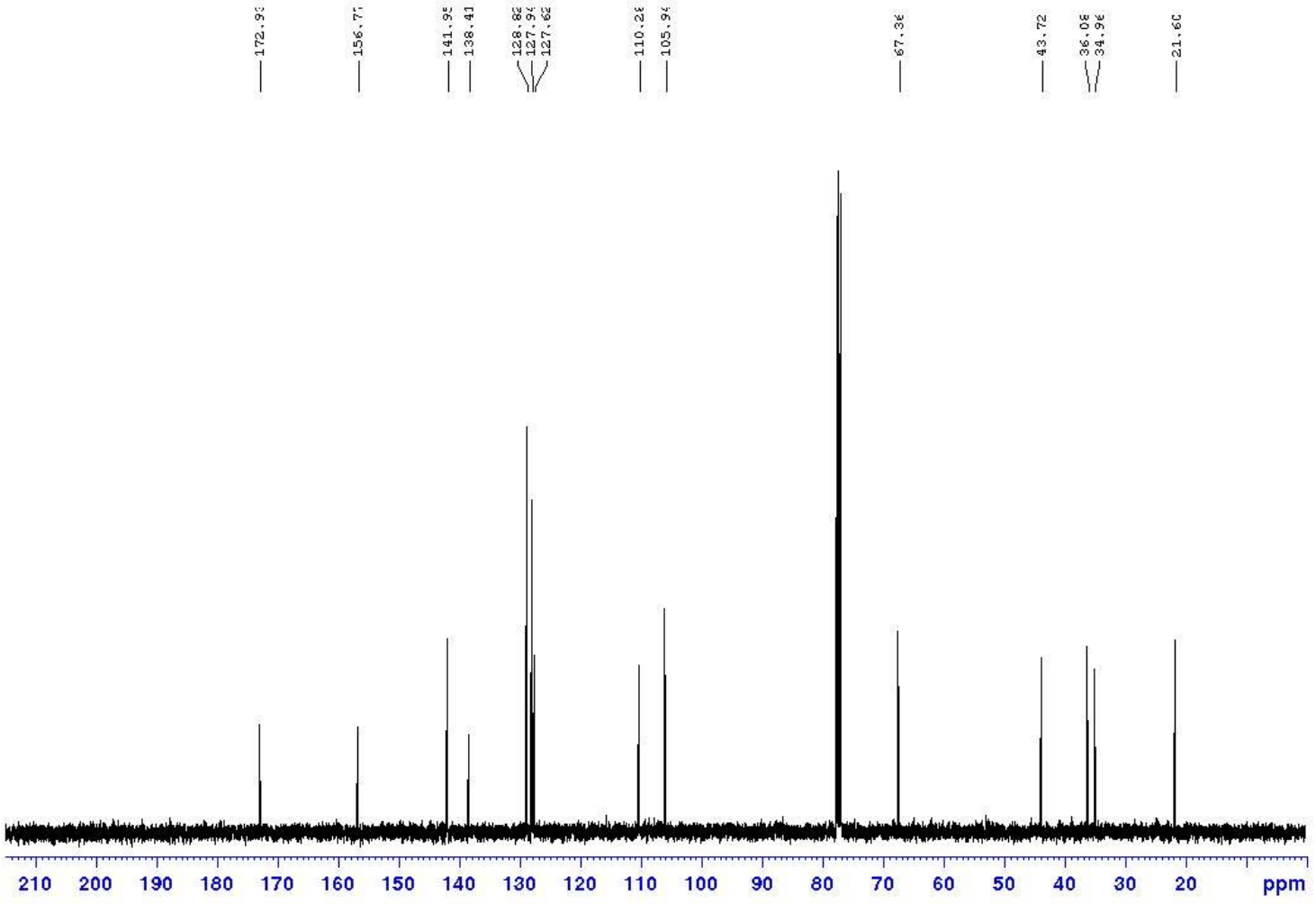


$[\alpha]_D^{20}$	–14.2° (*c* 1.0, $CHCl_3$)
HPLC analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4k-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.59 (major 97%) and –72.00 (minor 3%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.40 (6H, m, a,b,b',c,c',n), 6.33 (1H, dd, *J* = 3.2 and 1.8 Hz, m), 6.24 (1H, d, *J* = 3.2 Hz, l), 6.03 (1H, br s), 4.69 (1H, dd, *J* = 11.1 and 6.2, j), 4.43 (2H, d, *J* = 5.7 Hz, e), 2.82 (1H, d, *J* = 4.7 Hz, OH), 2.28 (2H, t, *J* = 7.2 Hz, g), 1.75–1.95 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.93 (f), 156.77 (k), 141.95 (n), 138.41 (d), 128.82 (b,b'), 127.94 (c,c'), 127.62 (a), 110.28 (m), 106.94 (l), 67.36 (j), 43.72 (e), 36.08 (g), 34.96 (i), 21.60 (h)
IR (neat)	3283 (O–H stretch), 2926, 2360, 1640 (C=O stretch), 1544, 1454, 1250, 1070, 1008, 733, 697 cm^{-1}
HRMS (ESI)	$C_{16}H_{19}NNaO_3$ (M+Na): 296.1263, found 296.1265 *m/z*

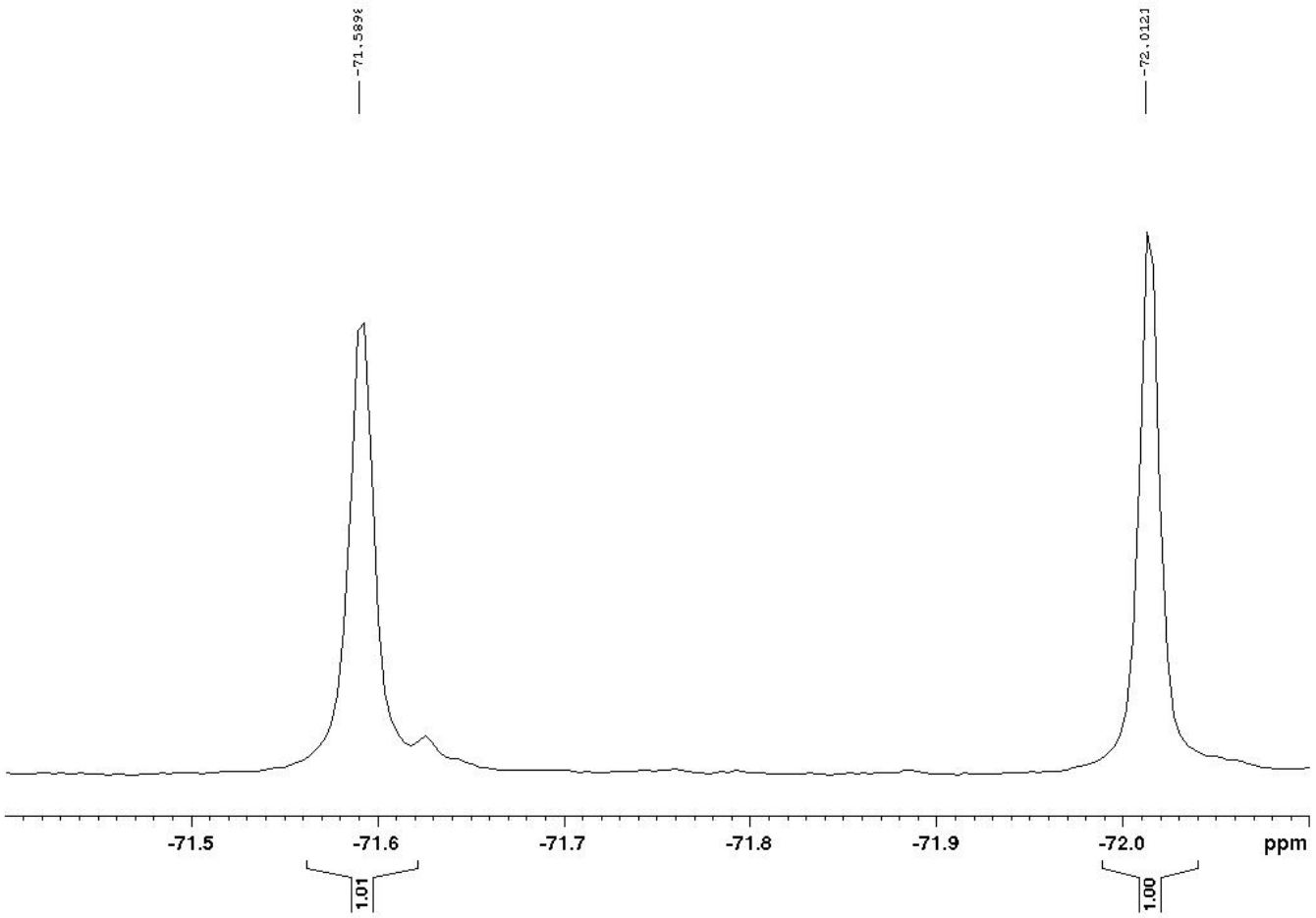
^{1}H NMR


7.322
6.335
6.330
6.327
6.323
6.240
6.232
6.028
4.715
4.700
4.687
4.672
4.432
4.418
2.828
2.817
2.294
2.276
2.259
1.872
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
6.29
1.00
1.00
1.02
1.00
2.09
1.06
2.06
4.10

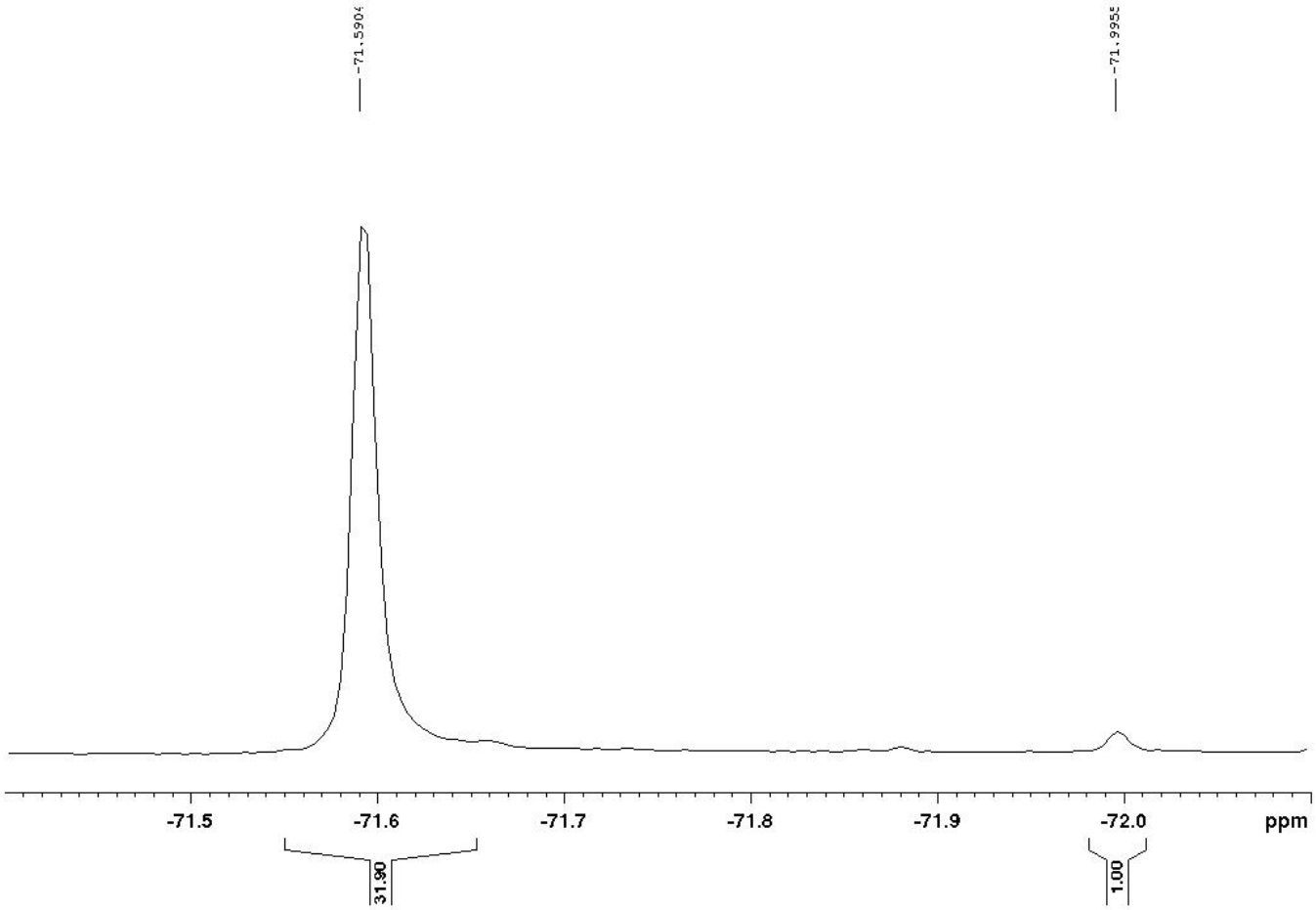
^{13}C NMR


172.9
156.7
141.9
138.4
128.8
127.9
127.6
110.2
105.9
67.3
43.72
36.0
34.9
21.6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

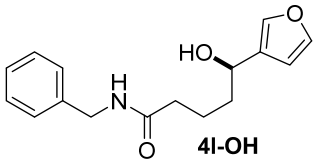
^{19}F NMR of Mosher's ester 4k-Mosher

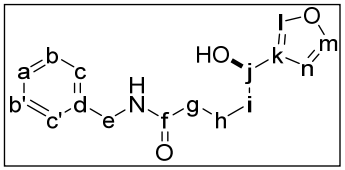


Mixture of diastereomeric 4k-Mosher



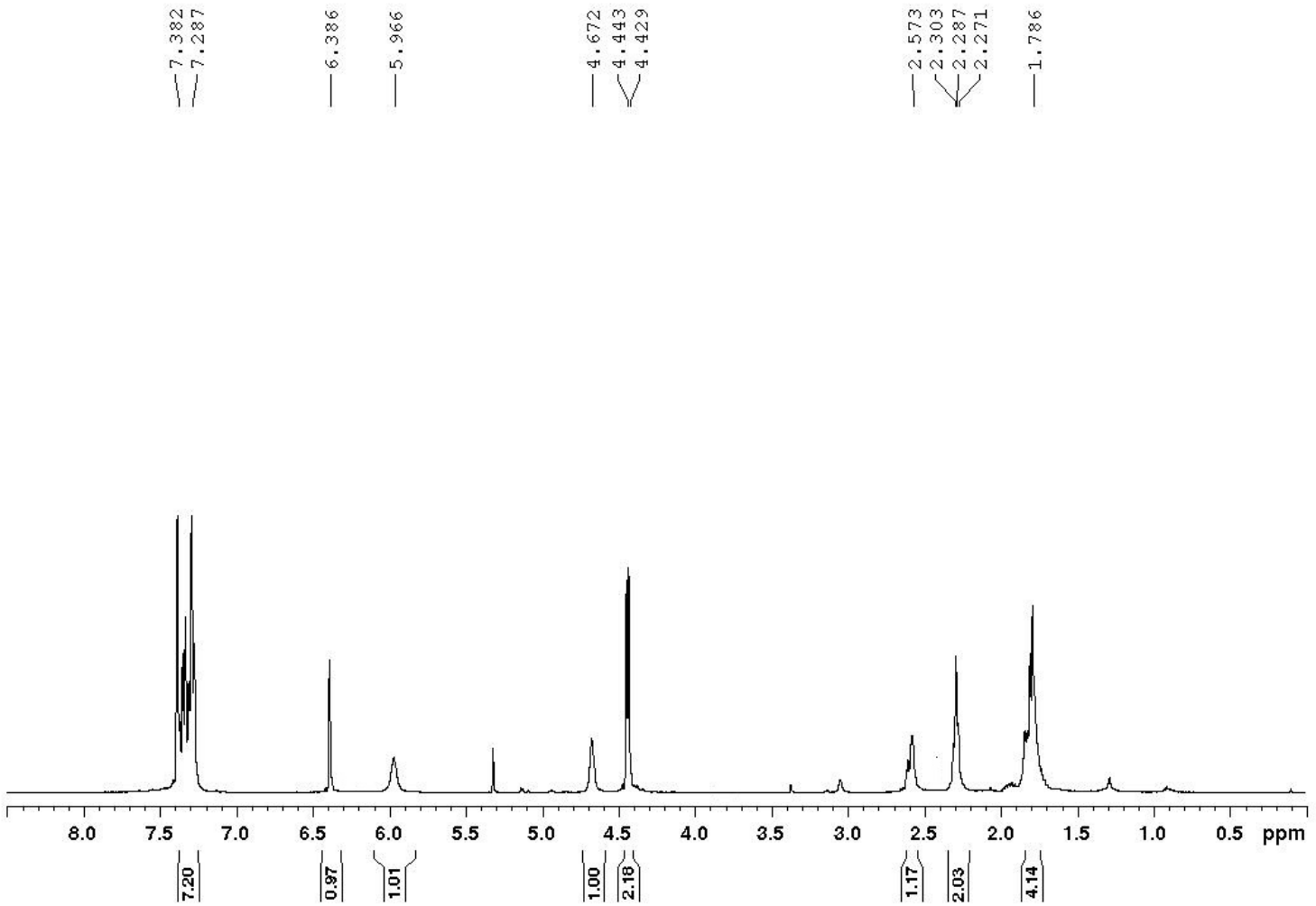
(*R*,*S*)-**4k-Mosher**



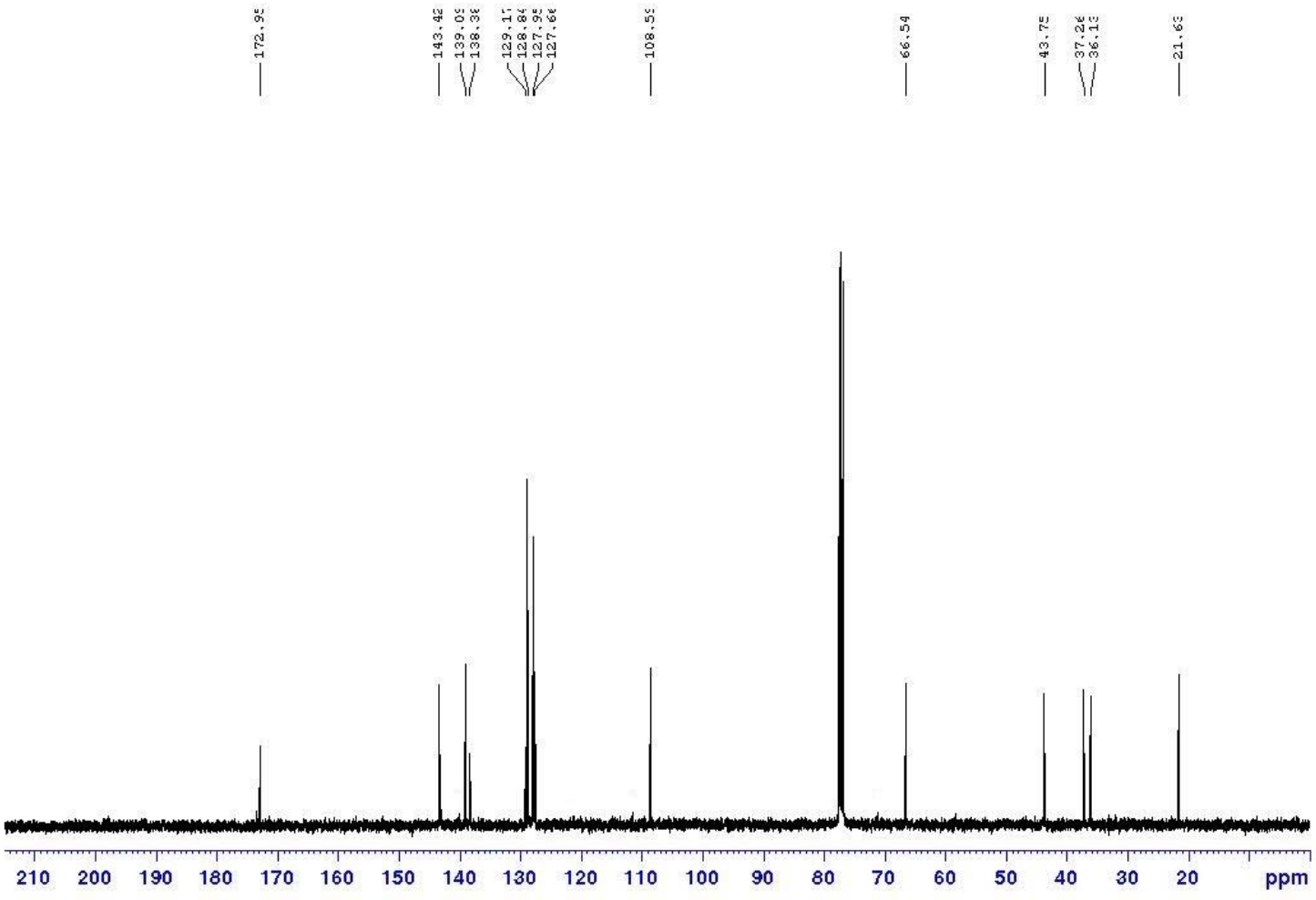


$[\alpha]_D^{20}$	–19.6° (*c* 1.0, $CHCl_3$)
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4l-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.49 (major 92%) and –71.72 (minor 8%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.40 (7H, m, a,b,b',c,c',l,m), 6.39 (1H, m, n), 5.97 (1H, br s), 4.60–4.70 (1H, m, j), 4.44 (2H, d, *J* = 5.7 Hz, e), 2.57 (1H, br s, OH), 2.29 (2H, t, *J* = 6.4 Hz, g), 1.75–1.85 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.95 (f), 143.42 (m), 139.09 (l), 138.38 (d), 129.16 (k), 128.84 (b,b'), 127.95 (c,c'), 127.66 (a), 108.59 (n), 66.54 (j), 43.75 (e), 37.26 (i), 36.13 (g), 21.63 (h)
IR (neat)	3281 (O–H stretch), 2928, 1632 (C=O stretch), 1547, 1453, 1007, 735, 696 cm^{-1}
HRMS (ESI)	$C_{16}H_{19}NNaO_3$ (M+Na): 296.1263, found 296.1263 *m/z*

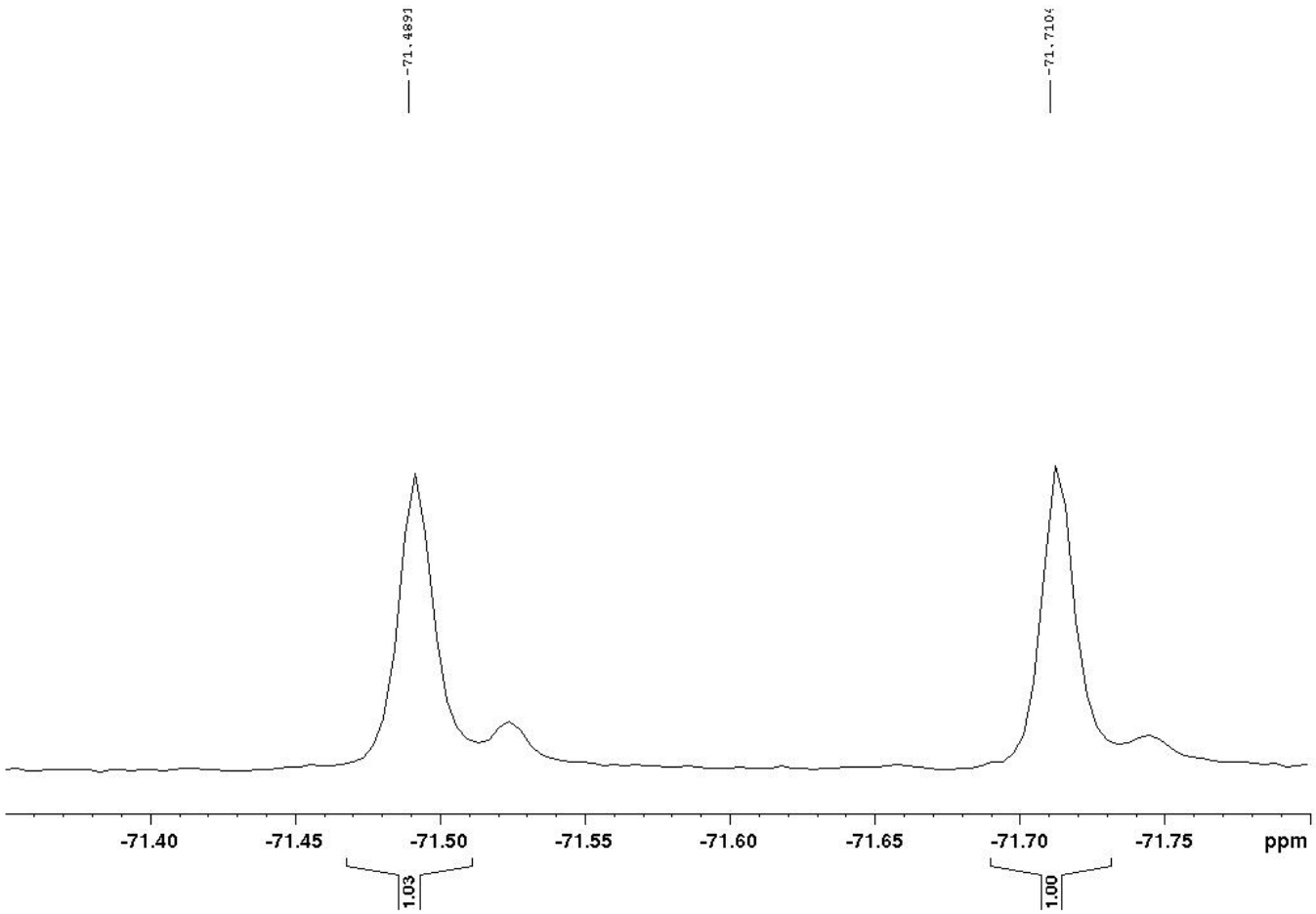
^{1}H NMR


7.382
7.287
6.386
5.966
4.672
4.443
4.429
2.573
2.303
2.287
2.271
1.786
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
7.20
0.97
1.01
1.00
2.18
1.17
2.03
4.14

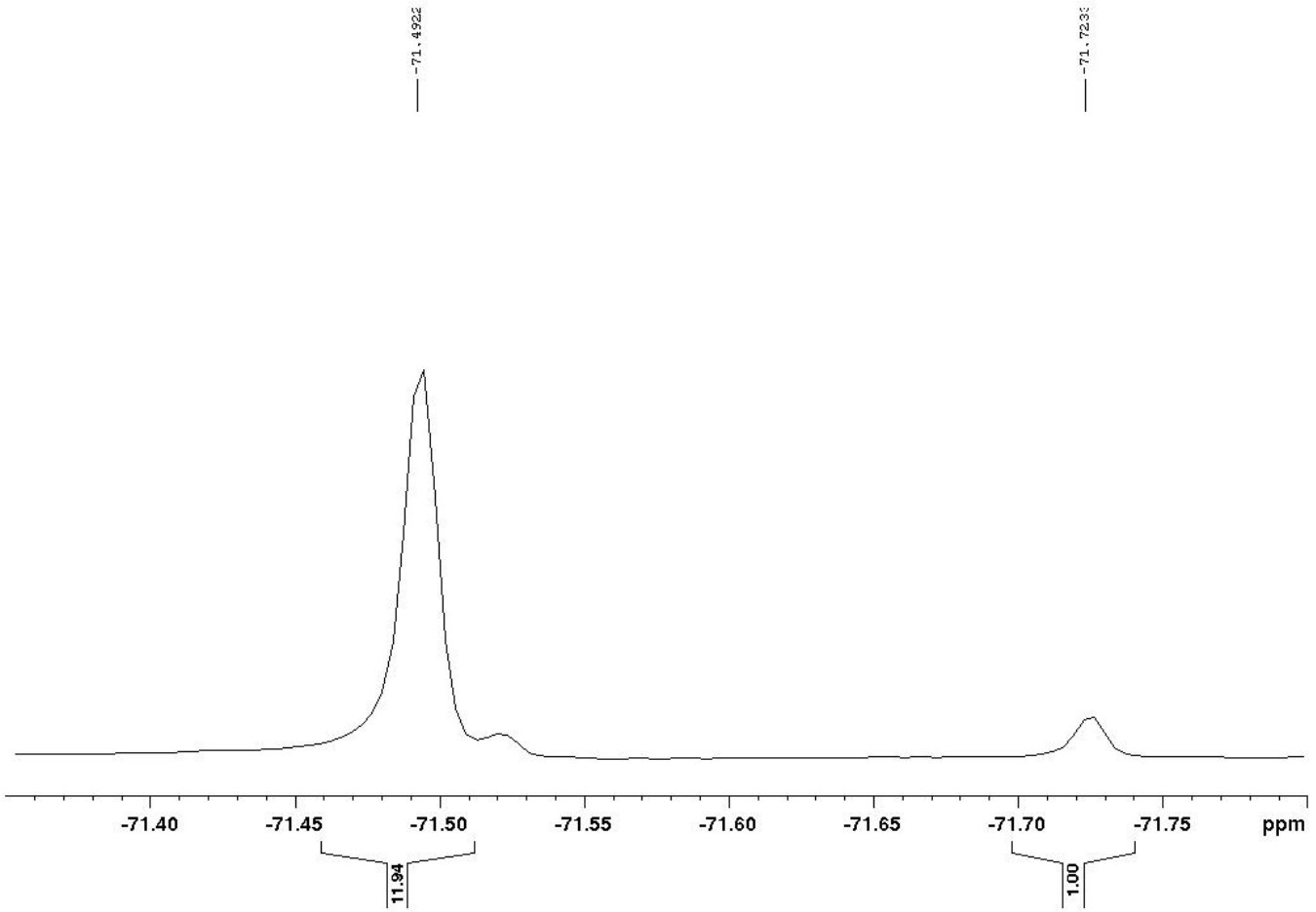
^{13}C NMR


172.95
143.42
139.09
138.36
129.17
128.84
127.95
127.64
108.55
66.54
43.75
37.26
36.13
21.65
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

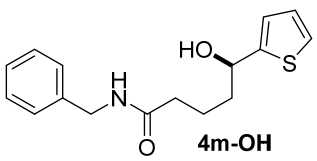
^{19}F NMR of Mosher's ester 4l-Mosher



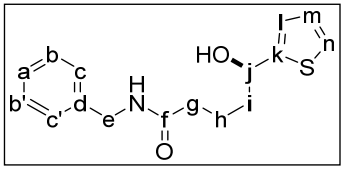
Mixture of diastereomeric 4l-Mosher


-71.4922
-71.7233
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
ppm
11.94
1.00

(*R*,*S*)-**4l-Mosher**

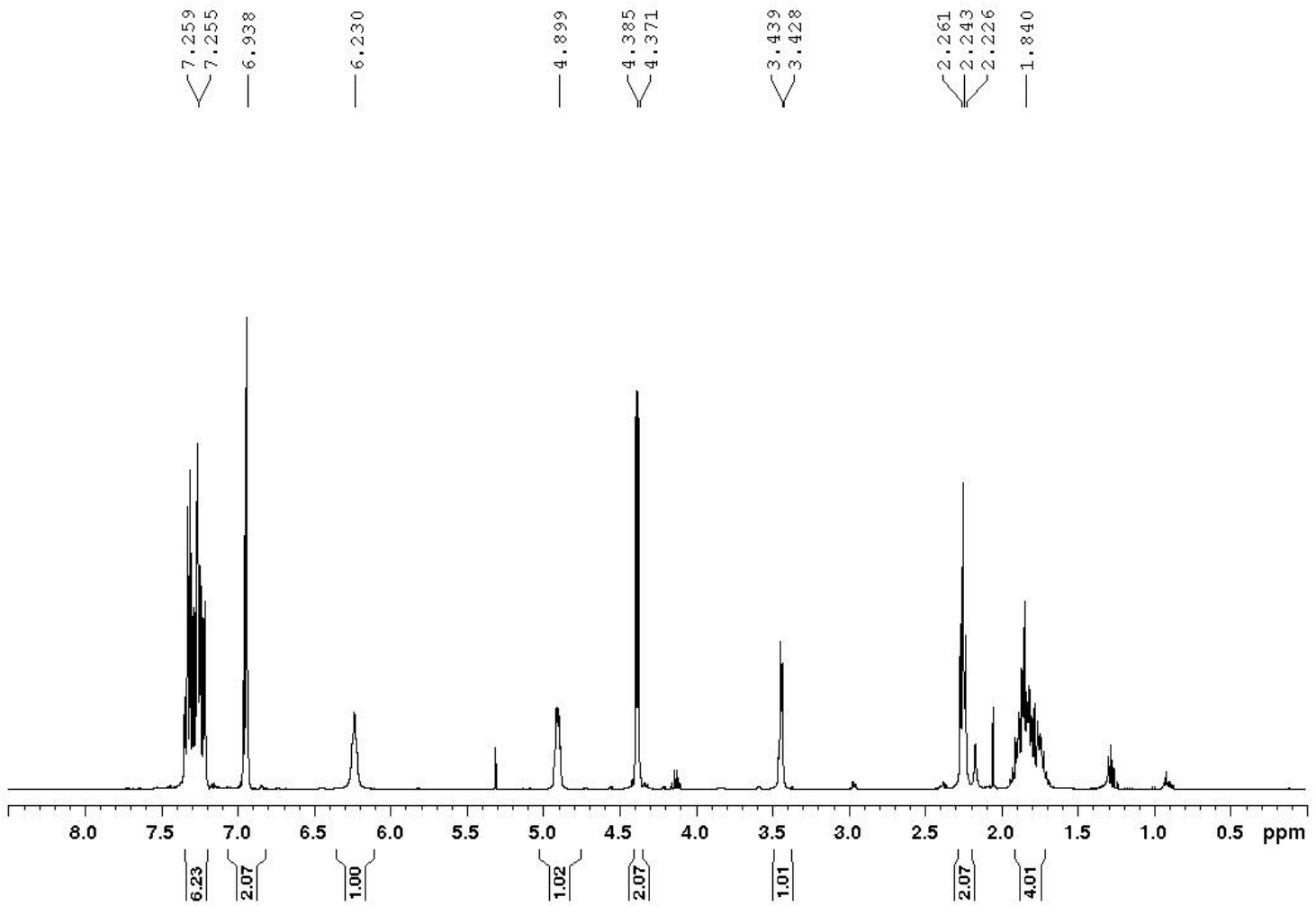


4m-OH

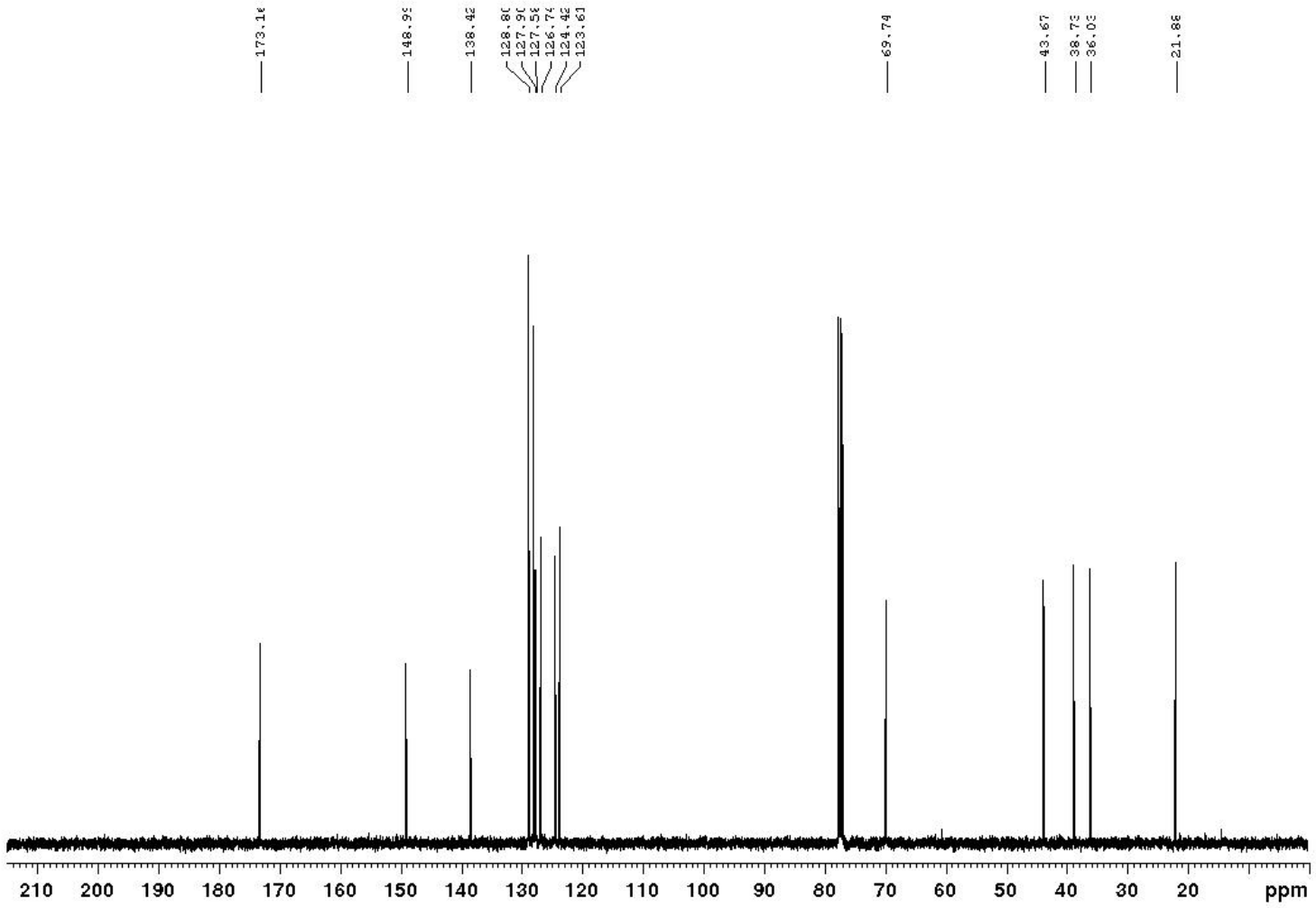


$[\alpha]_D^{20}$	–9.3° (*c* 1.0, $CHCl_3$)
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4m-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.32 (major 92.5%) and –71.74 (minor 7.5%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (6H, m, a,b,b',c,c',n), 6.90–7.00 (2H, m, l,m), 6.23 (1H, br s), 4.85–4.95 (1H, m, j), 4.38 (2H, d, *J* = 5.7 Hz, e), 3.43 (1H, d, *J* = 4.3 Hz, OH), 2.24 (2H, t, *J* = 7.2 Hz, g), 1.70–1.90 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.16 (f), 148.99 (k), 138.42 (d), 128.80 (b,b'), 127.90 (c,c'), 127.58 (a), 126.74 (m), 124.42 (n), 123.61 (l), 69.74 (j), 43.67 (e), 38.73 (i), 36.03 (g), 21.88 (h)
IR (neat)	3292 (O–H stretch), 2916, 1640 (C=O stretch), 1539, 1495, 1454, 1357, 1250, 1077, 1028, 694 cm^{-1}
HRMS (ESI)	$C_{16}H_{19}NNaO_2S$ (M+Na): 312.1034, found 312.1039 *m/z*

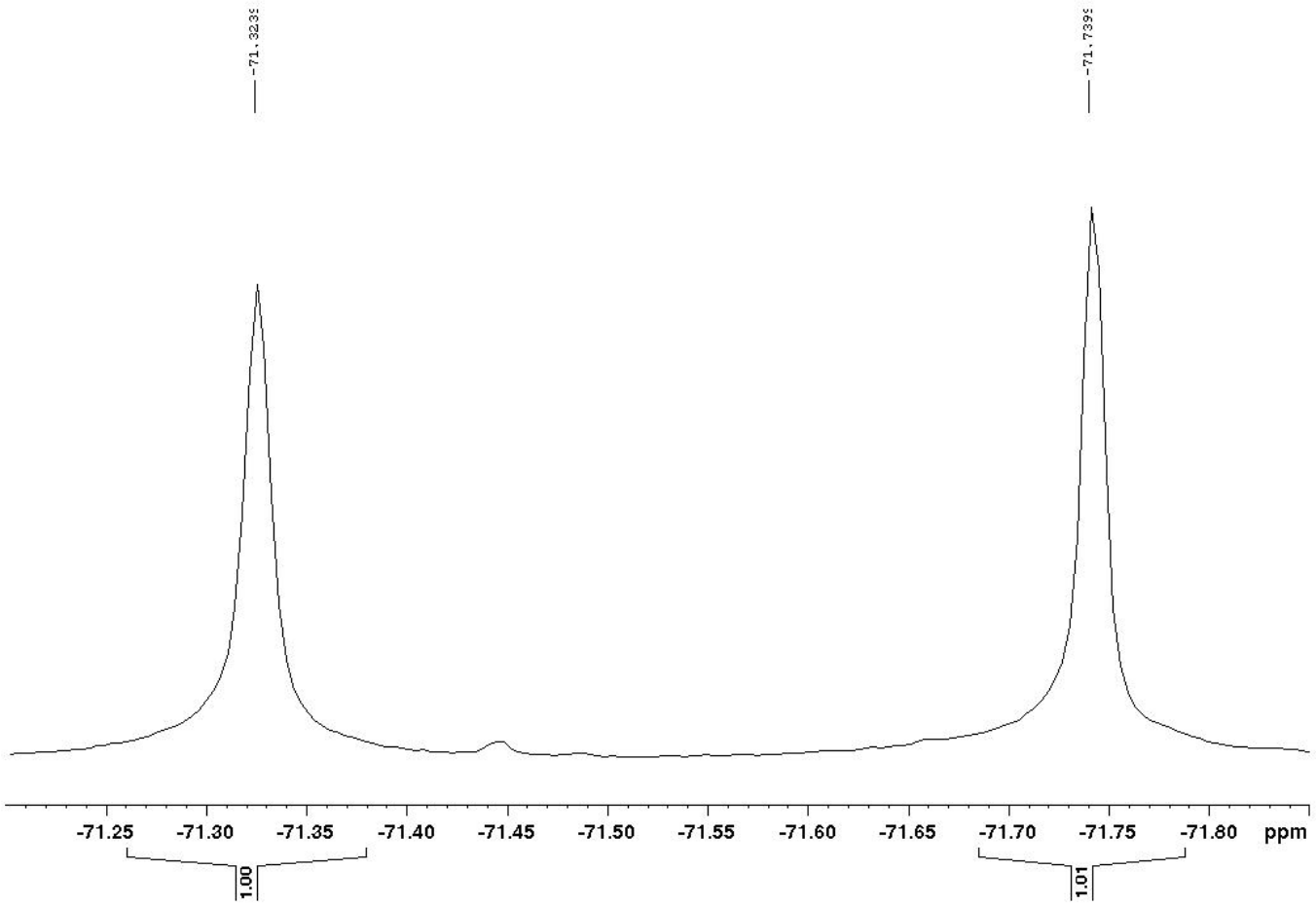
^{1}H NMR


7.259
7.255
6.938
6.230
4.899
4.385
4.371
3.439
3.428
2.261
2.243
2.226
1.840
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
6.23
2.07
1.00
1.02
2.07
1.01
2.07
4.01

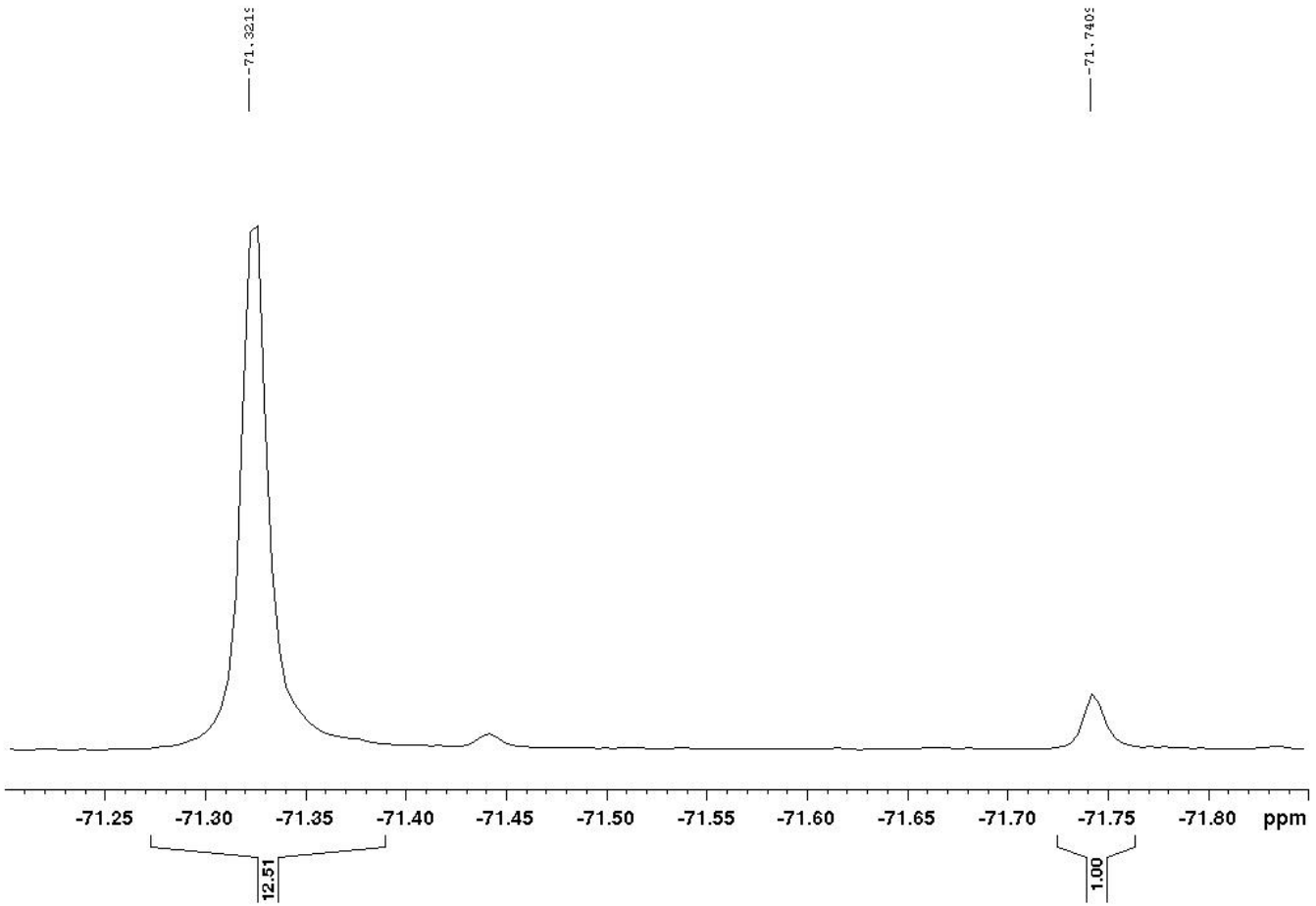
^{13}C NMR


173.1
148.9
138.4
128.8
127.9
127.5
126.7
124.4
123.6
69.74
43.67
38.7
36.0
21.8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

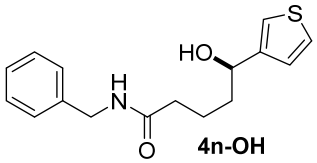
^{19}F NMR of Mosher's ester 4m-Mosher

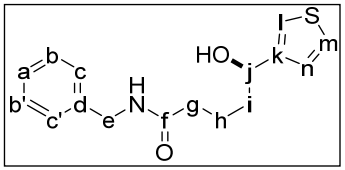


Mixture of diastereomeric 4m-Mosher


-71.3215
-71.7405
-71.25
-71.30
-71.35
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
-71.80
ppm
12.51
1.00

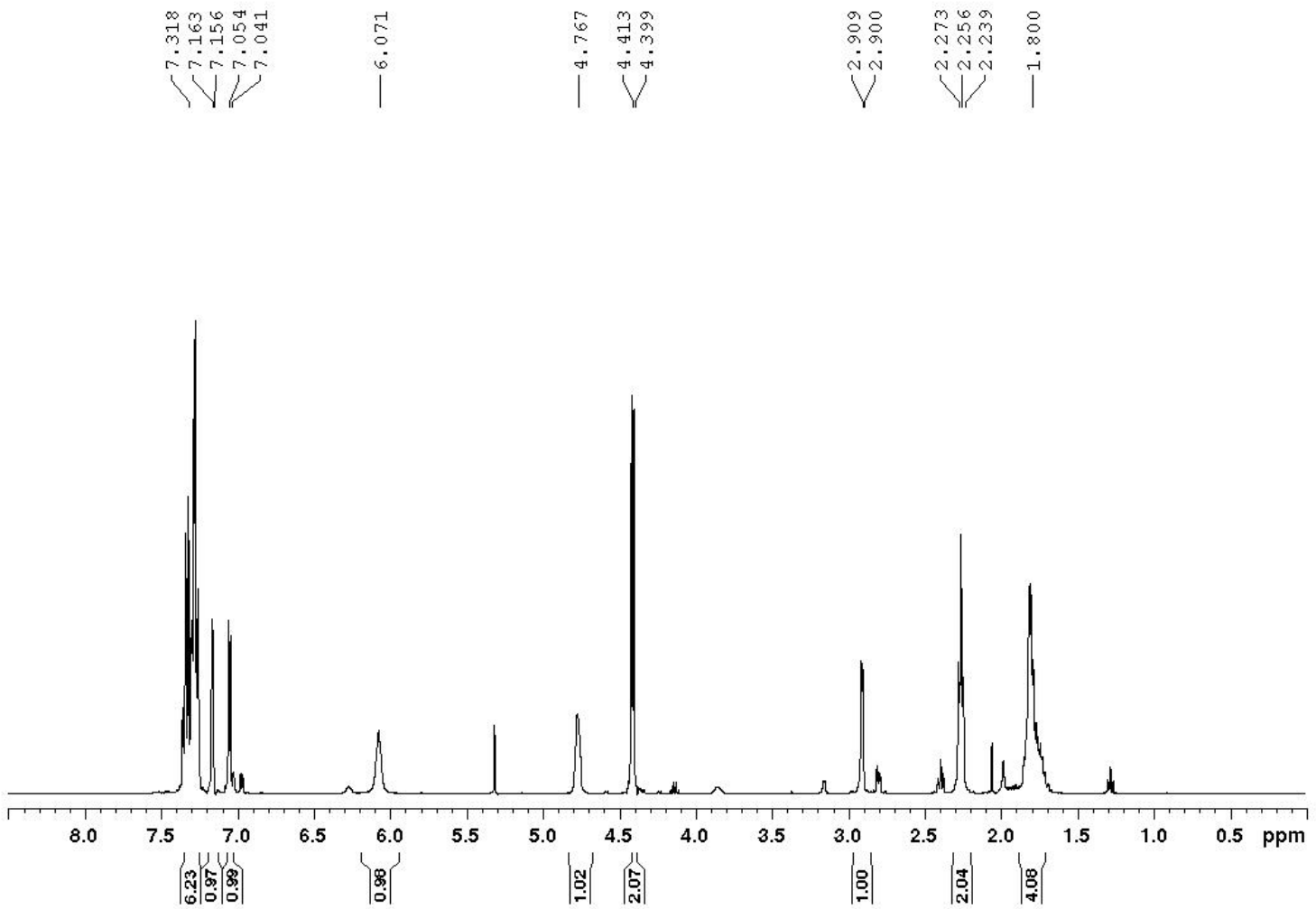
(*R*,*S*)-**4m-Mosher**



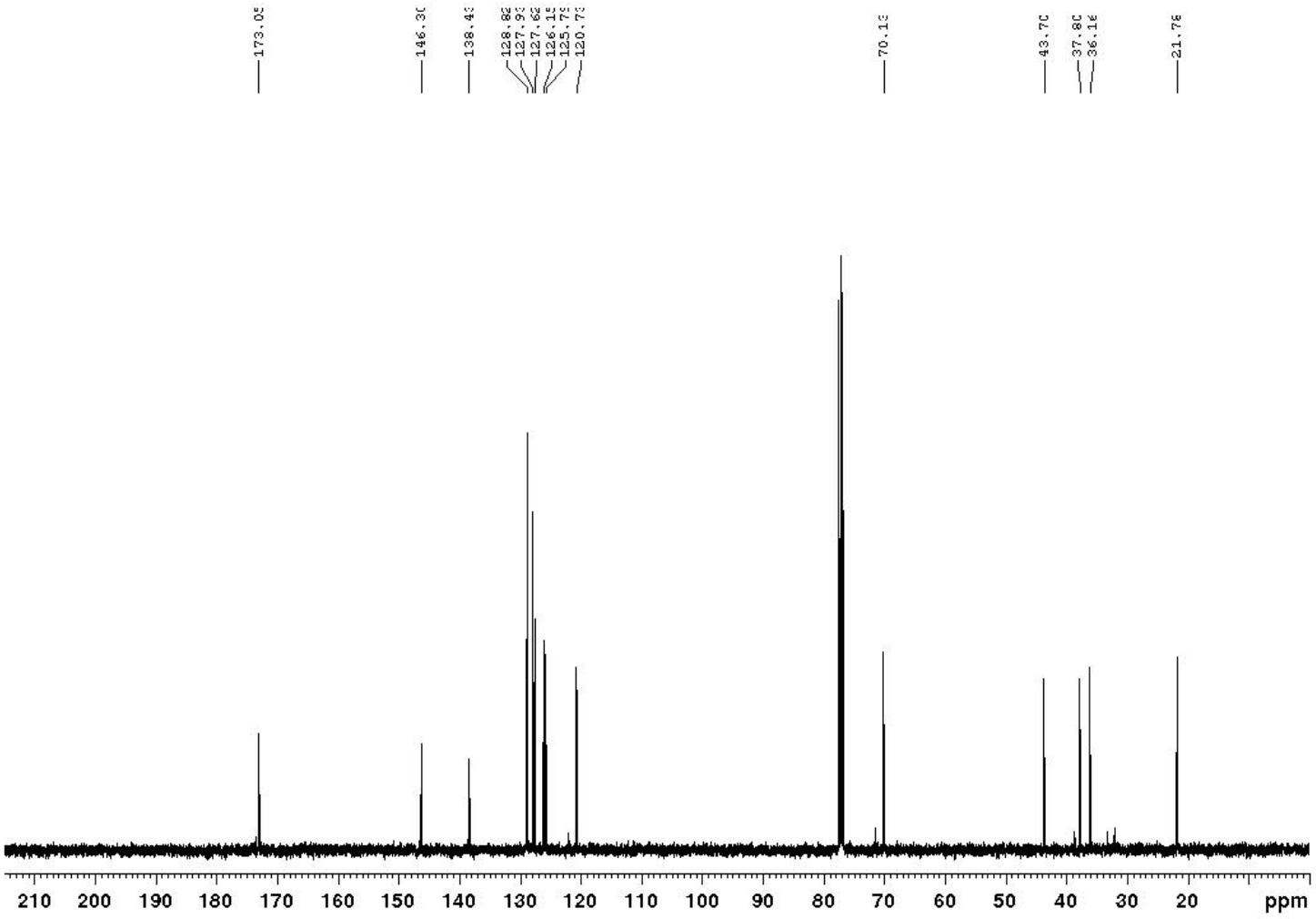


$[\alpha]_D^{20}$	–18.2º (*c* 1.0, $CHCl_3$)
m.p.	81.0–82.5 ºC
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **4n-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.34 (major 94%) and –71.60 (minor 6%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.35 (7H, m, a,b,b',c,c',m), 7.16 (1H, d, *J* = 2.6 Hz, n), 7.05 (1H, d, *J* = 5.0 Hz, l), 6.07 (1H, br s), 4.70–4.80 (1H, m, j), 4.41 (2H, d, *J* = 5.7 Hz, e), 2.90 (1H, d, *J* = 3.6 Hz, OH), 2.26 (2H, t, *J* = 6.7 Hz, g), 1.70–1.90 (4H, m, h,i)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 173.04 (f), 146.30 (k), 138.43 (d), 128.82 (b,b'), 127.93 (c,c'), 127.62 (a), 126.15 (m), 125.79 (n), 120.73 (l), 70.13 (j), 43.70 (e), 37.80 (i), 36.16 (g), 21.78 (h)
IR (neat)	3284 (O–H stretch), 2360, 2341, 1630 (C=O stretch), 1537, 1454, 1068, 844, 789, 747, 696 cm^{-1}
HRMS (ESI)	$C_{16}H_{19}NNaO_2S$ (M+Na): 312.1034, found 312.1036 *m/z*

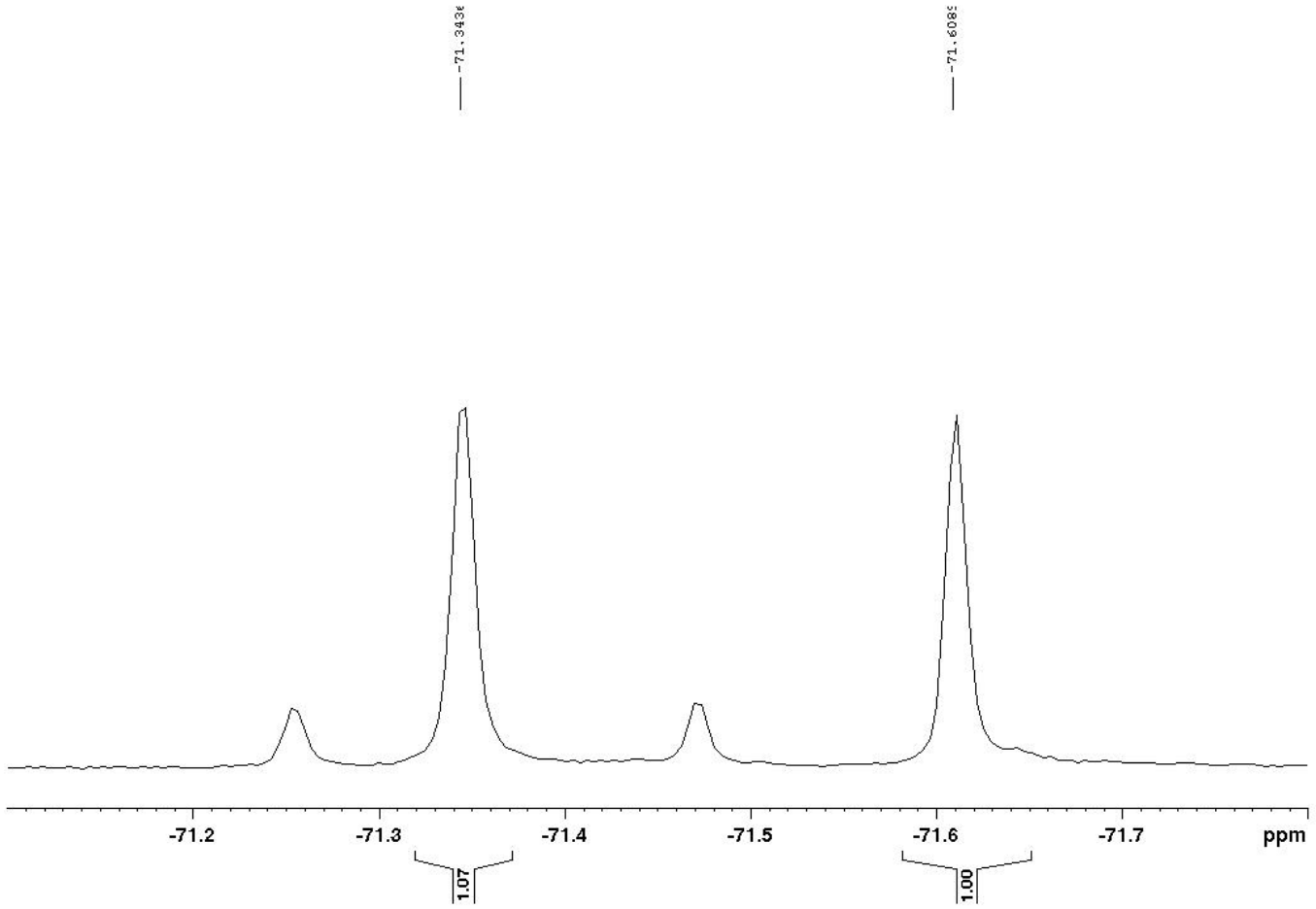
^{1}H NMR


7.318
7.163
7.156
7.054
7.041
6.071
4.767
4.413
4.399
2.909
2.900
2.273
2.256
2.239
1.800
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
6.23
0.97
0.99
0.96
1.02
2.07
1.00
2.04
4.08

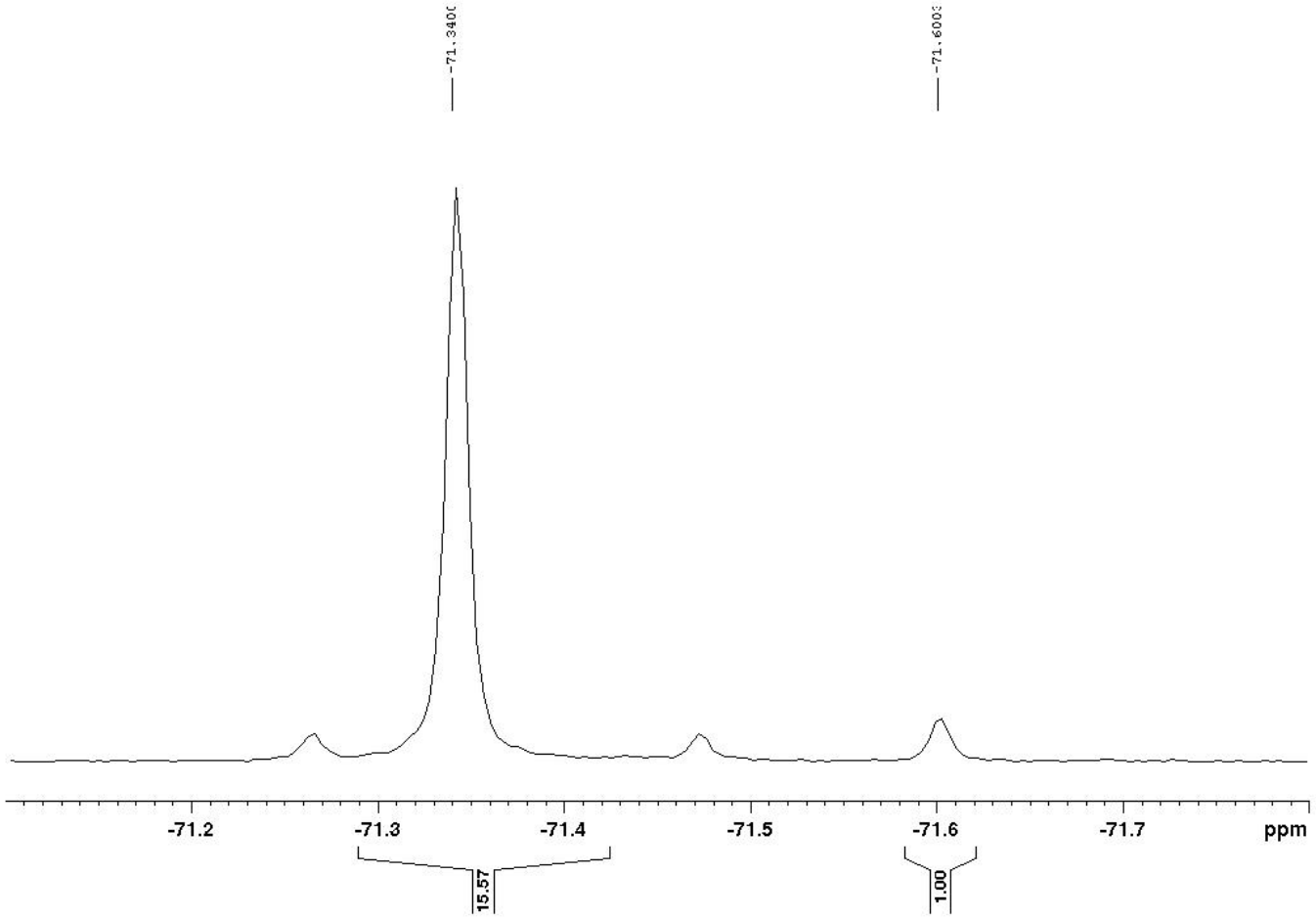
^{13}C NMR


173.05
146.30
138.43
128.86
127.93
127.65
126.15
125.75
120.73
70.13
43.70
37.80
36.18
21.76
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

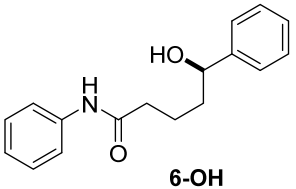
^{19}F NMR of Mosher's ester 4n-Mosher



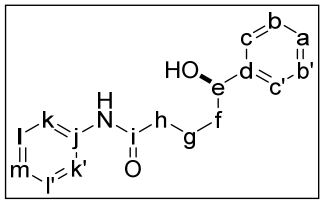
Mixture of diastereomeric 4n-Mosher



(*R*,*S*)-**4n-Mosher**

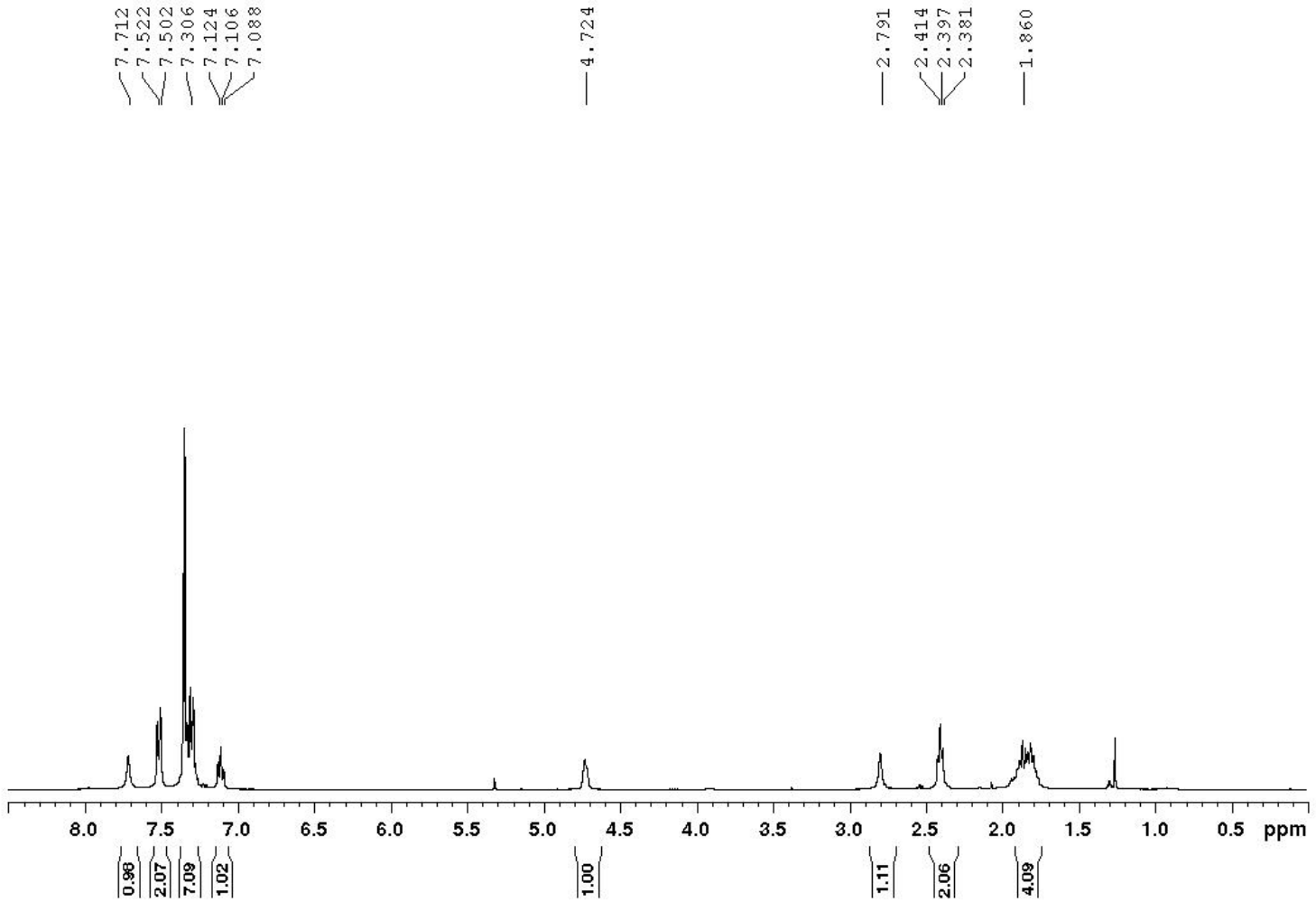


6-OH

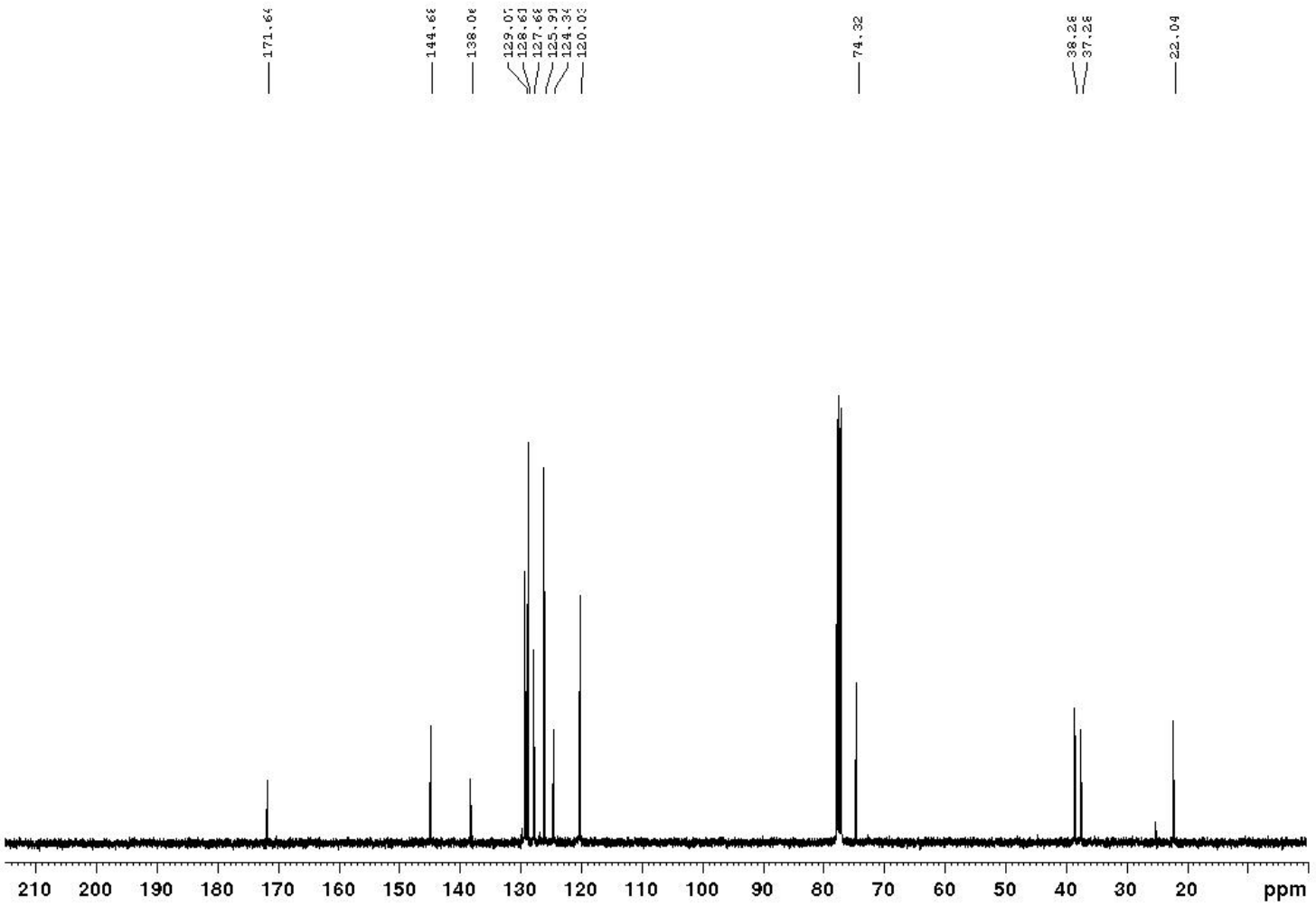


$[\alpha]_D^{20}$	–21.6° (*c* 1.0, $CHCl_3$)
m.p.	84.5–86.0 °C
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **6-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.41 (major 94%) and –71.61 (minor 6%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.71 (1H, br s, NH), 7.51 (2H, d, *J* = 7.9 Hz, k,k'), 7.25–7.40 (7H, m, a,b,b',c,c',l,l'), 7.11 (1H, t, *J* = 7.4 Hz, m), 4.65–4.80 (1H, m, e), 2.79 (1H, br s, OH), 2.40 (2H, t, *J* = 6.4 Hz, h), 1.75–1.95 (4H, m, f,g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.64 (i), 144.68 (d), 138.06 (j), 129.07 (l,l'), 128.61 (b,b'), 127.68 (m), 125.91 (c,c'), 124.34 (a), 120.03 (k,k'), 74.32 (e), 38.28 (g), 37.28 (h), 22.04 (f)
IR (neat)	3294 (O–H stretch), 2360, 2342, 1668 (C=O stretch), 1655, 1600, 1557, 1499, 1489, 1444, 1088, 951, 699, 691 cm^{-1}
HRMS (EI)	$C_{17}H_{19}NO_2$ (M): 269.1416, found 269.1422 *m/z*

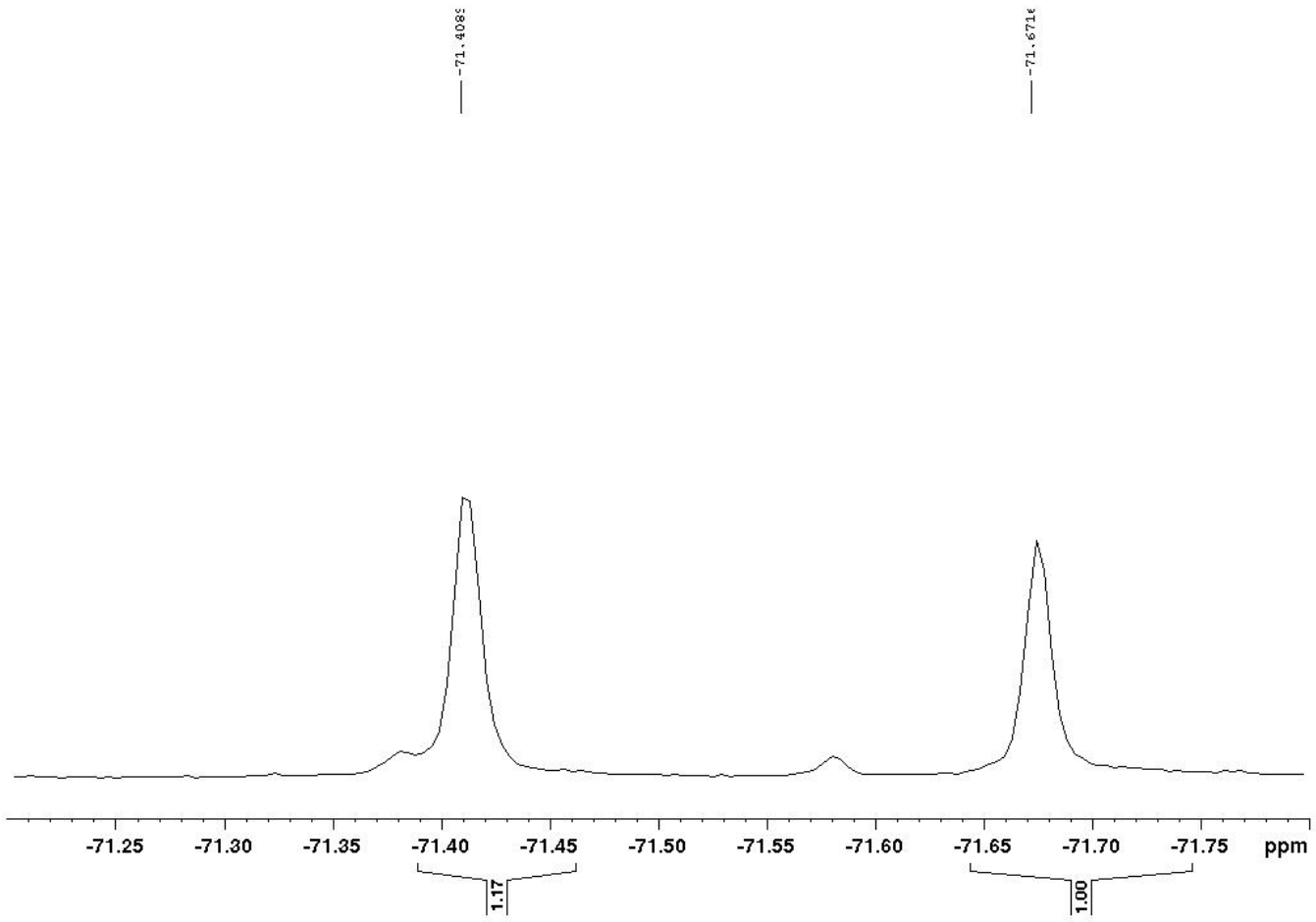
^{1}H NMR


7.712
7.522
7.502
7.306
7.124
7.106
7.088
4.724
2.791
2.414
2.397
2.381
1.860
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.98
2.07
7.09
1.02
1.00
1.11
2.06
4.09

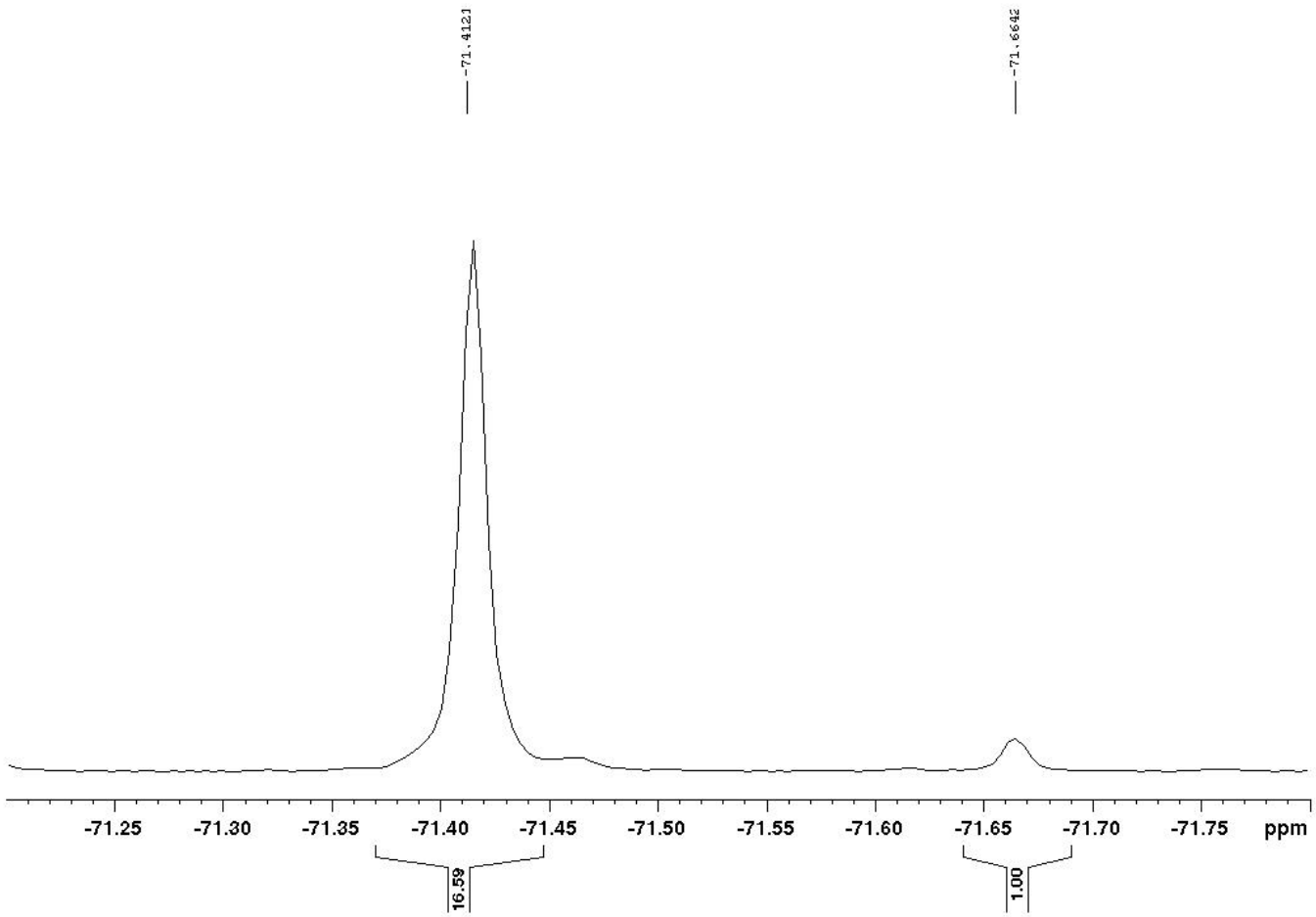
^{13}C NMR


171.64
144.68
138.0
129.07
128.61
127.68
125.93
124.34
120.03
74.32
38.26
37.28
22.04
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

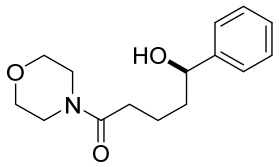
^{19}F NMR of Mosher's ester 6-Mosher



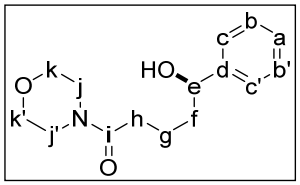
Mixture of diastereomeric 6-Mosher


-71.4121
-71.6642
-71.25
-71.30
-71.35
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
-71.75
ppm
16.59
1.00

(*R*,*S*)-**6-Mosher**

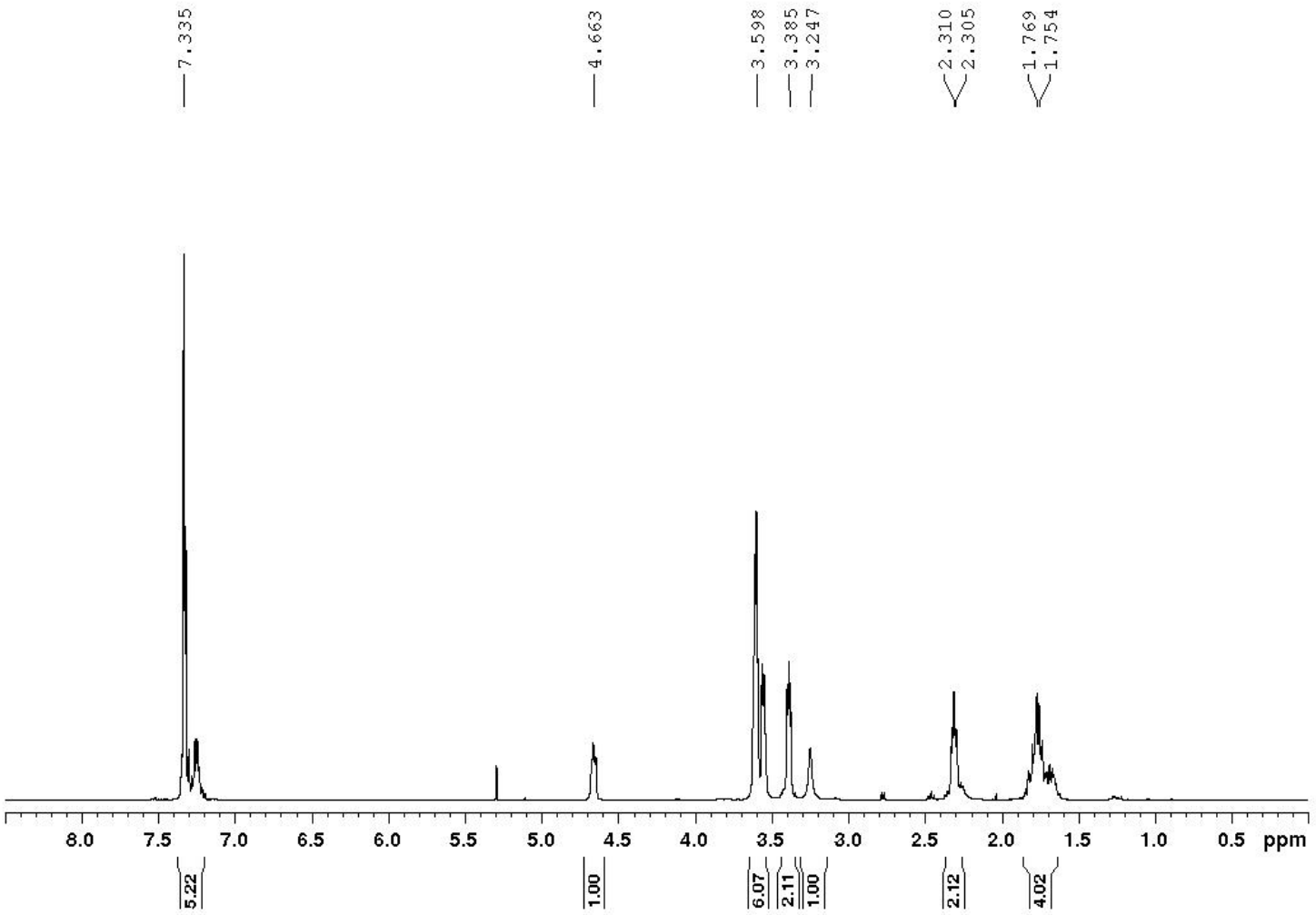


7-OH

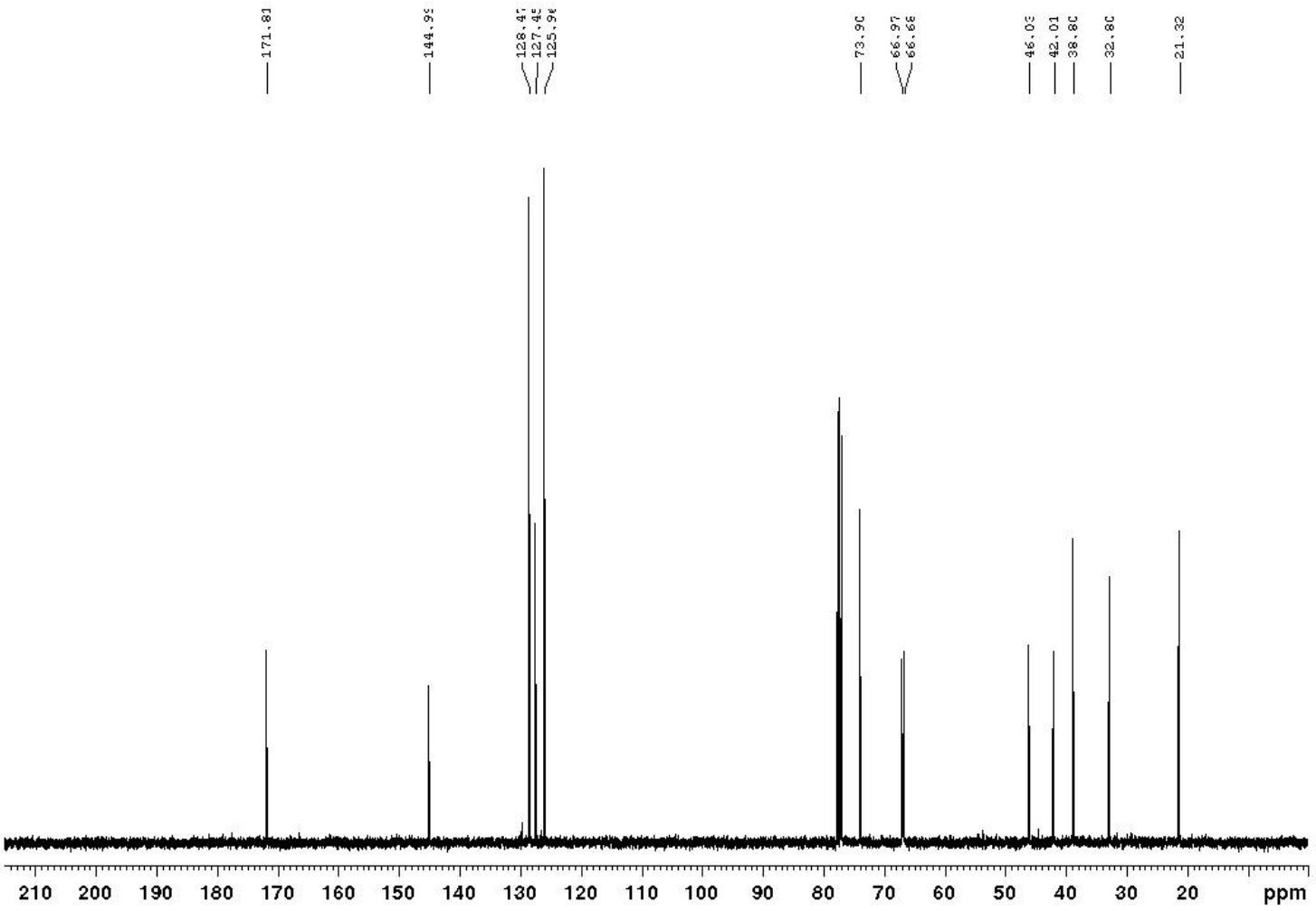


$[\alpha]_D^{20}$	–22.7° (*c* 2.0, $CHCl_3$)
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **7-Mosher**; ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.22 (major 95.5%) and –71.61 (minor 4.5%)
^1H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (5H, m, aryl), 4.60–4.70 (1H, m, e), 3.55–3.65 (6H, m, j,j',k'k'), 3.35–3.45 (2H, m, k,k'), 3.25 (1H, br s, OH), 2.25–2.35 (2H, m, h), 1.60–1.85 (4H, m, f,g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 171.81 (i), 144.99 (d), 128.47 (b,b'), 127.45 (a), 125.96 (c,c'), 73.90 (e), 66.97 and 66.68 (k,k'), 46.03 and 42.01 (j,j'), 38.80 (g), 32.80 (h), 21.32 (f)
IR (neat)	3410 (O–H stretch), 2915, 2856, 1621 (C=O stretch), 1433, 1271, 1237, 1113, 1066, 1026, 701 cm^{-1}
HRMS (EI)	$C_{15}H_{21}NO_3$ (M): 263.1521, found 263.1523 *m/z*

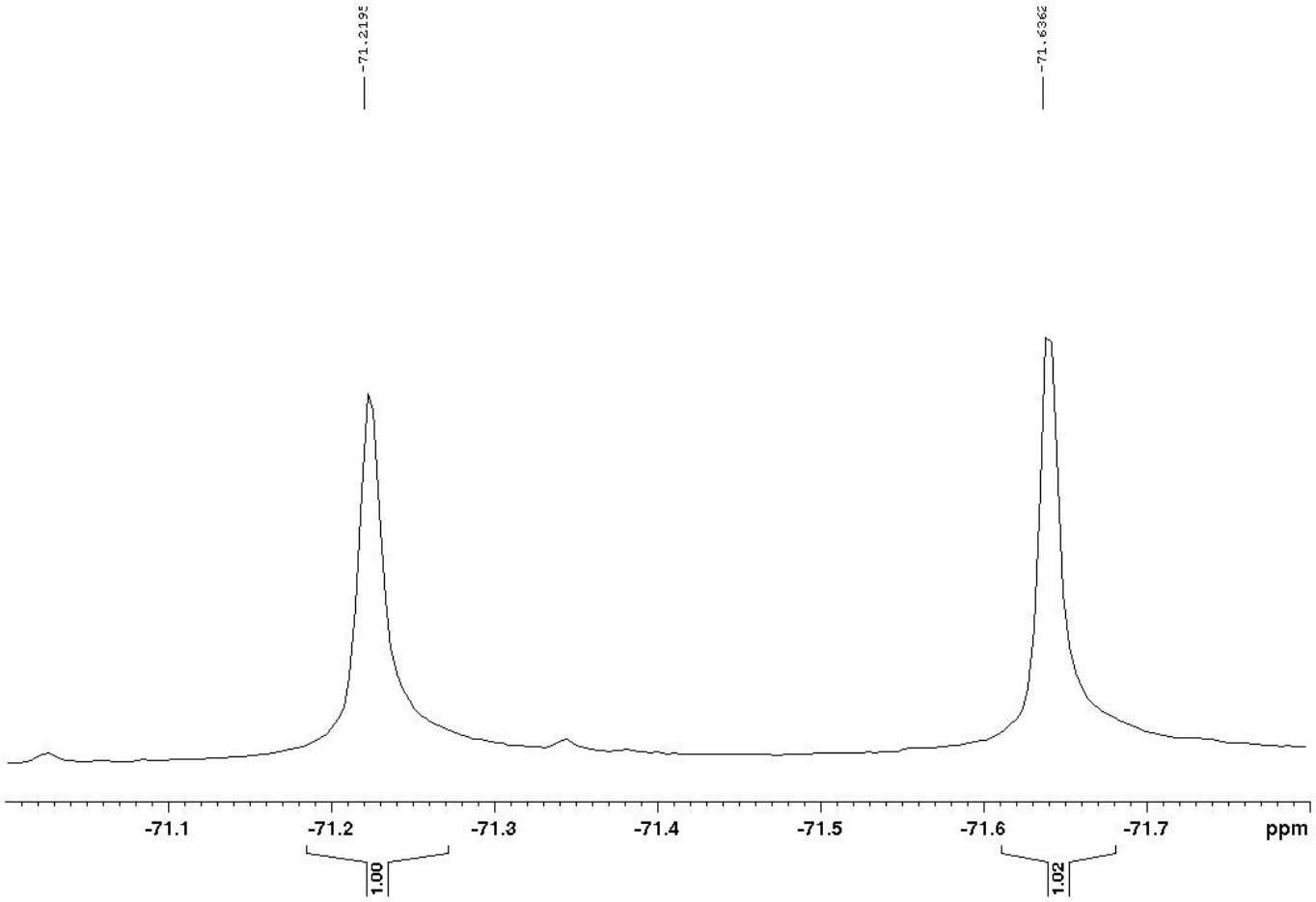
^{1}H NMR


7.335
4.663
3.598
3.385
3.247
2.310
2.305
1.769
1.754
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.22
1.00
6.07
2.11
1.00
2.12
4.02

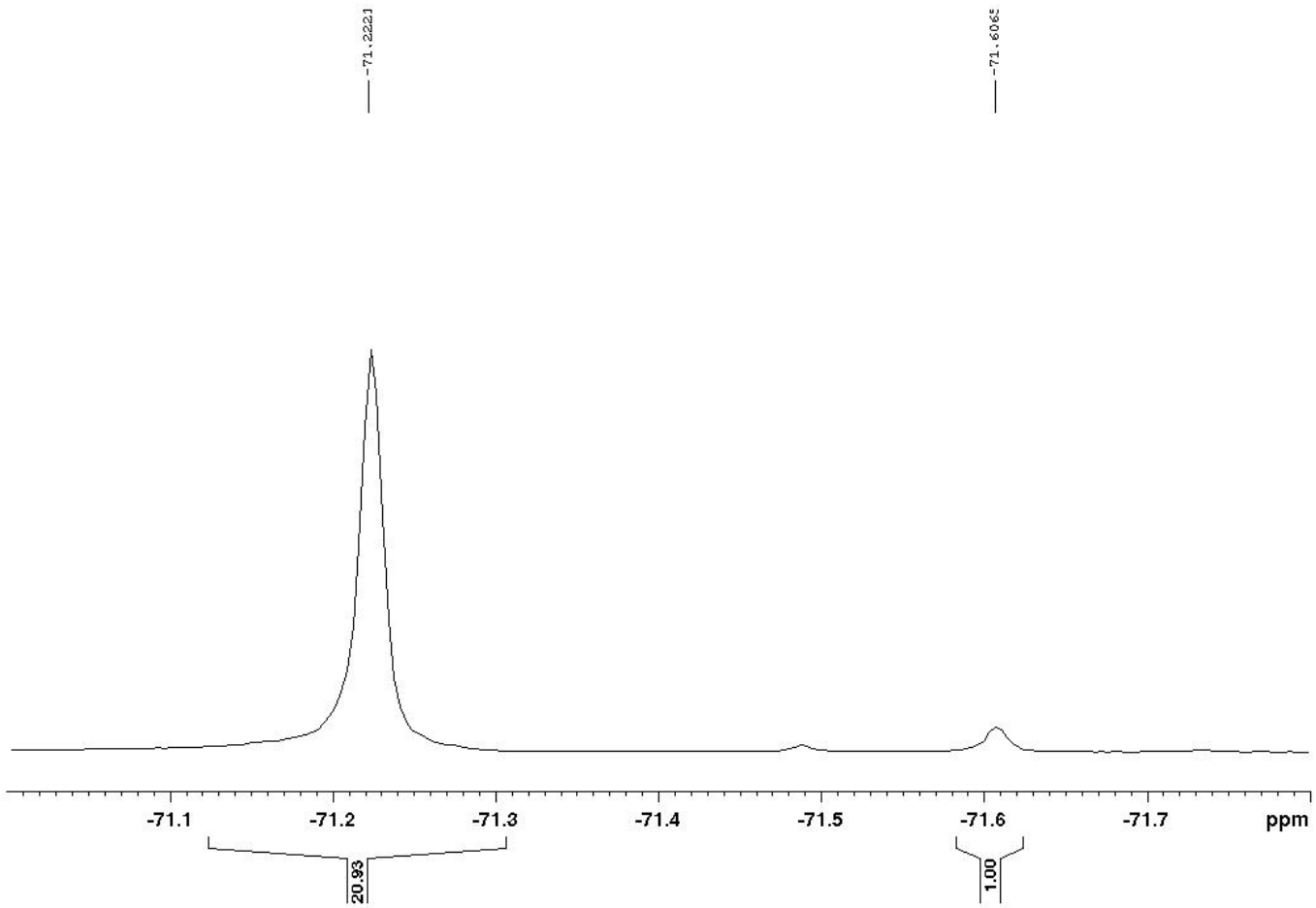
^{13}C NMR


171.81
144.95
128.47
127.45
125.96
73.90
66.97
66.68
46.03
42.01
38.80
32.80
21.32
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

^{19}F NMR of Mosher's ester 7-Mosher

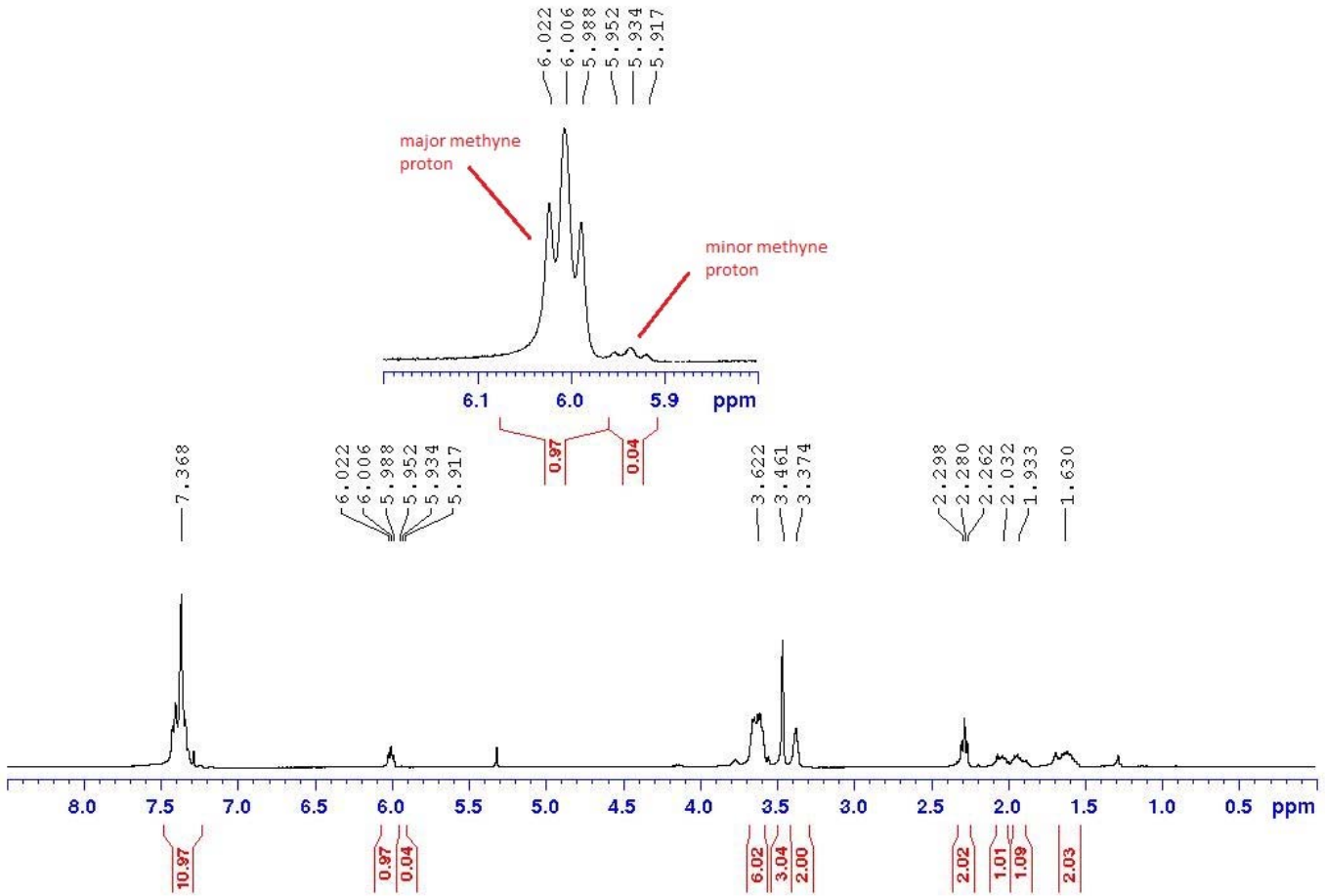


Mixture of diastereomeric 7-Mosher

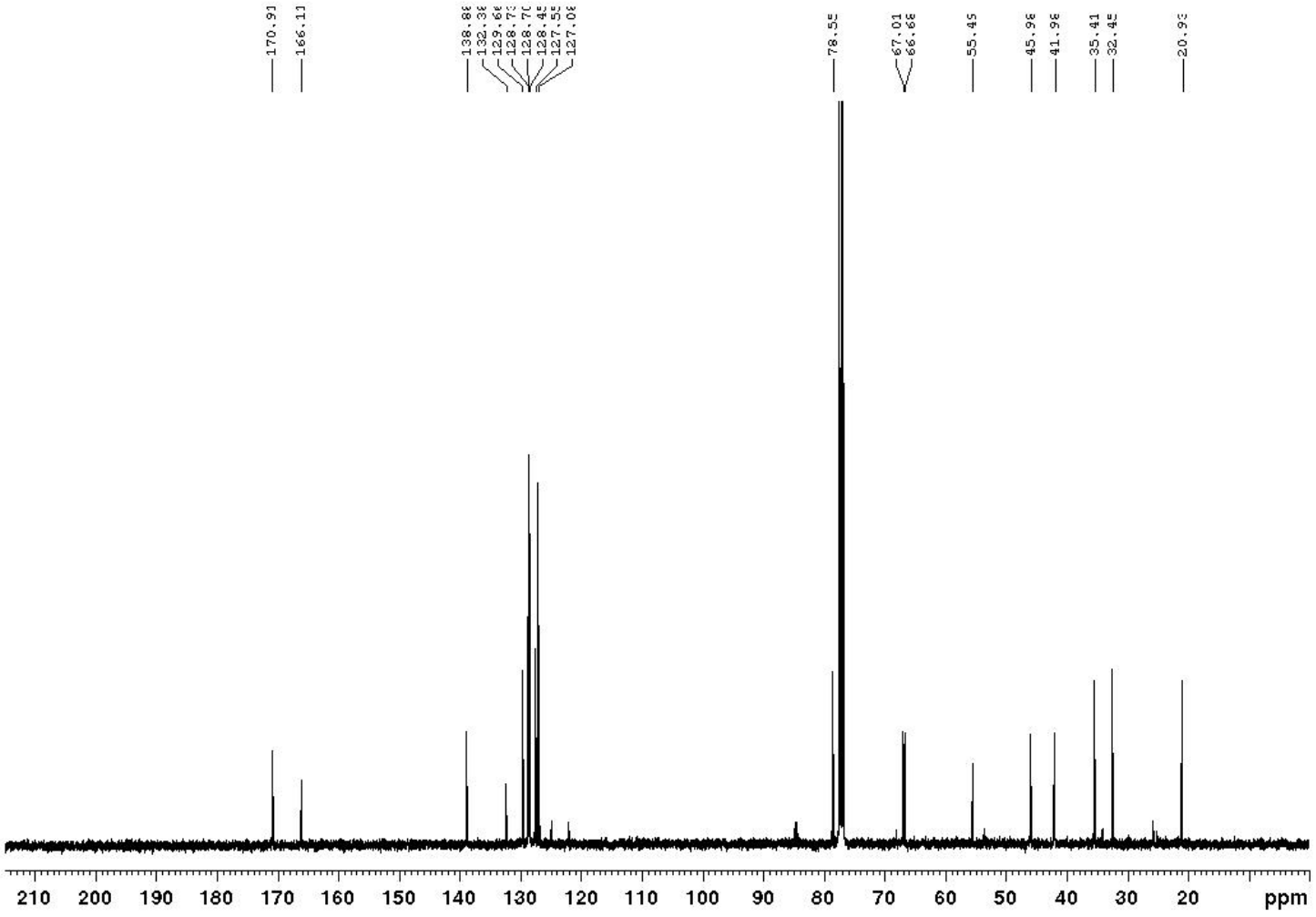


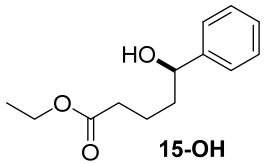
(*R*,*S*)-**7-Mosher**

[1]H NMR of Mosher's ester 7-Mosher

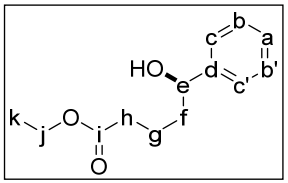


^{13}C NMR of Mosher's ester 7-Mosher



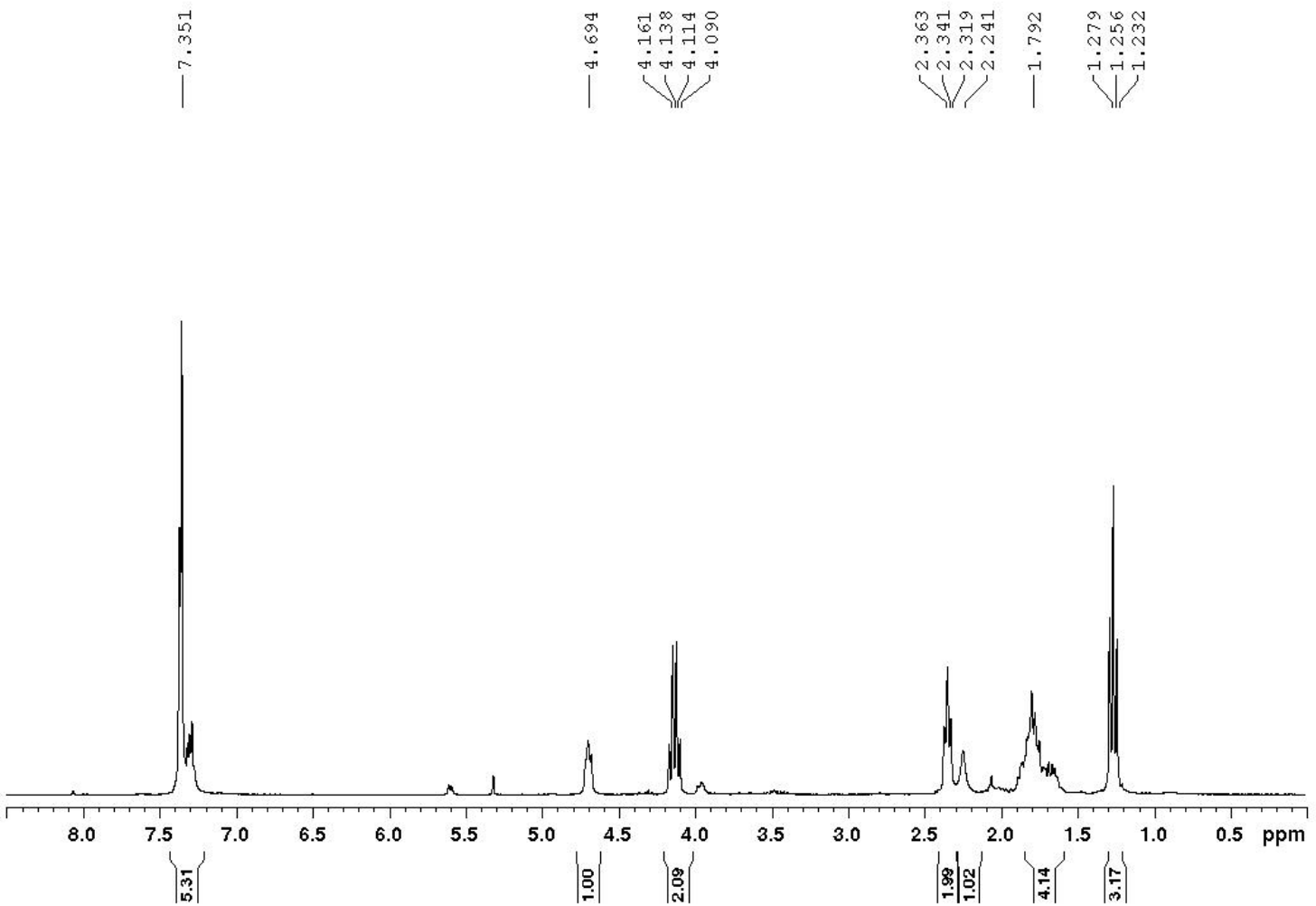


15-OH

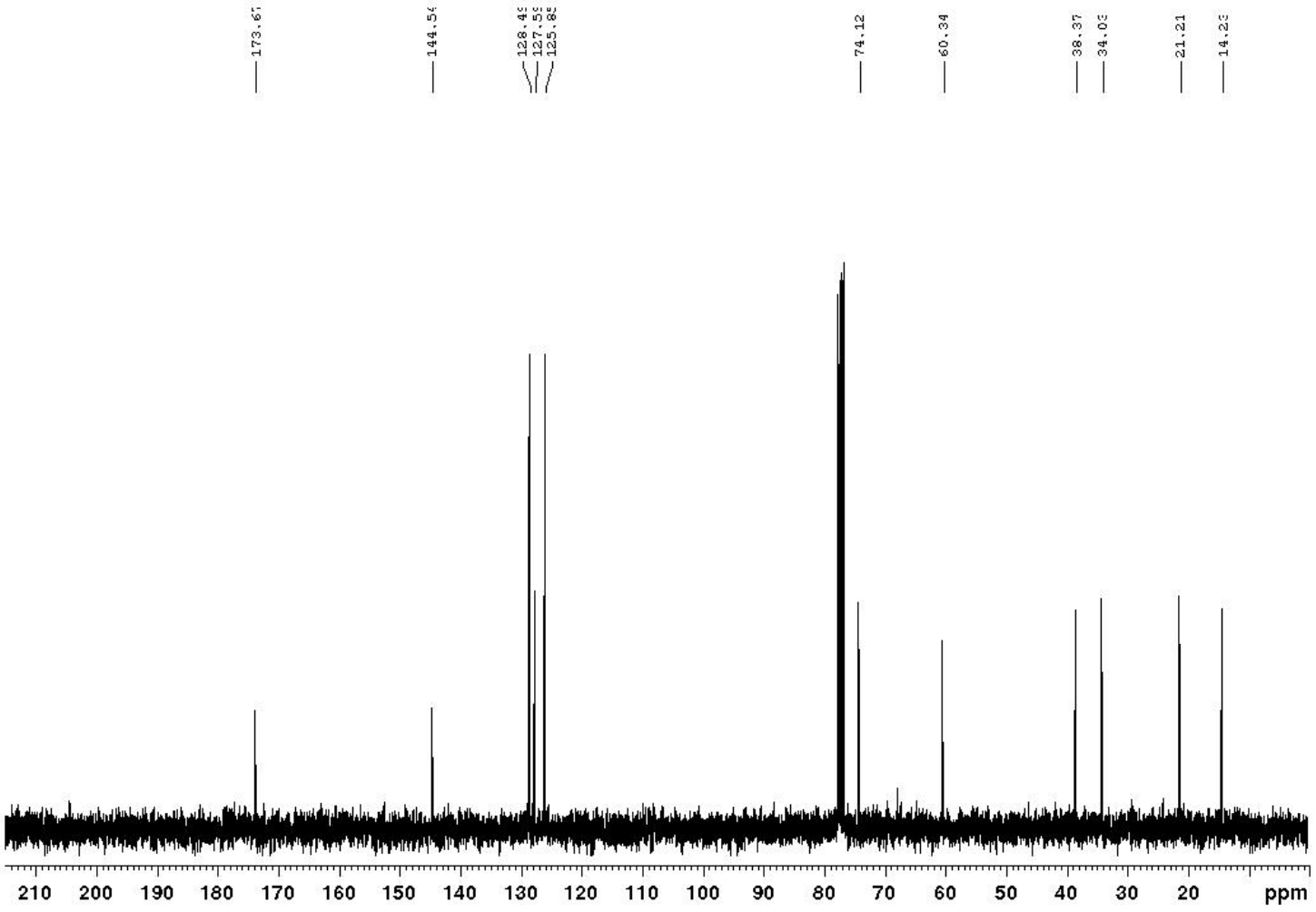


$[\alpha]_D^{20}$	–19.5° (*c* 2.0, $CHCl_3$)
Enantiomeric ratio analysis	Enantiomeric excess was checked by converting to the corresponding Mosher's ester **15-Mosher**; crude ^{19}F NMR (375 MHz, $CDCl_3$) shows δ –71.44 (major 85%) and –71.63 (minor 15%)
^1H NMR (300 MHz, $CDCl_3$)	δ 7.25–7.45 (5H, m, aryl), 4.65–4.75 (1H, m, e), 4.13 (2H, q, *J* = 9.5 Hz, j), 2.34 (2H, t, *J* = 8.7 Hz, h), 2.24 (1H, br s, OH), 1.60–1.85 (4H, m, f,g), 1.26 (3H, t, *J* = 9.5 Hz, k)
^{13}C NMR (75 MHz, $CDCl_3$)	δ 173.67 (i), 144.54 (d), 128.49 (b,b'), 127.59 (a), 125.85 (c,c'), 74.12 (e), 60.34 (j), 38.37 (f), 34.03 (h), 21.21 (g), 14.23 (k)
IR (neat)	3342 (O–H stretch), 2979, 2936, 1731 (C=O stretch), 1453, 1373, 1238, 1185, 1146, 1065, 1026, 736, 700 cm^{-1}
HRMS (EI)	$C_{13}H_{18}O_3$ (M): 222.1256, found 222.1262 *m/z*

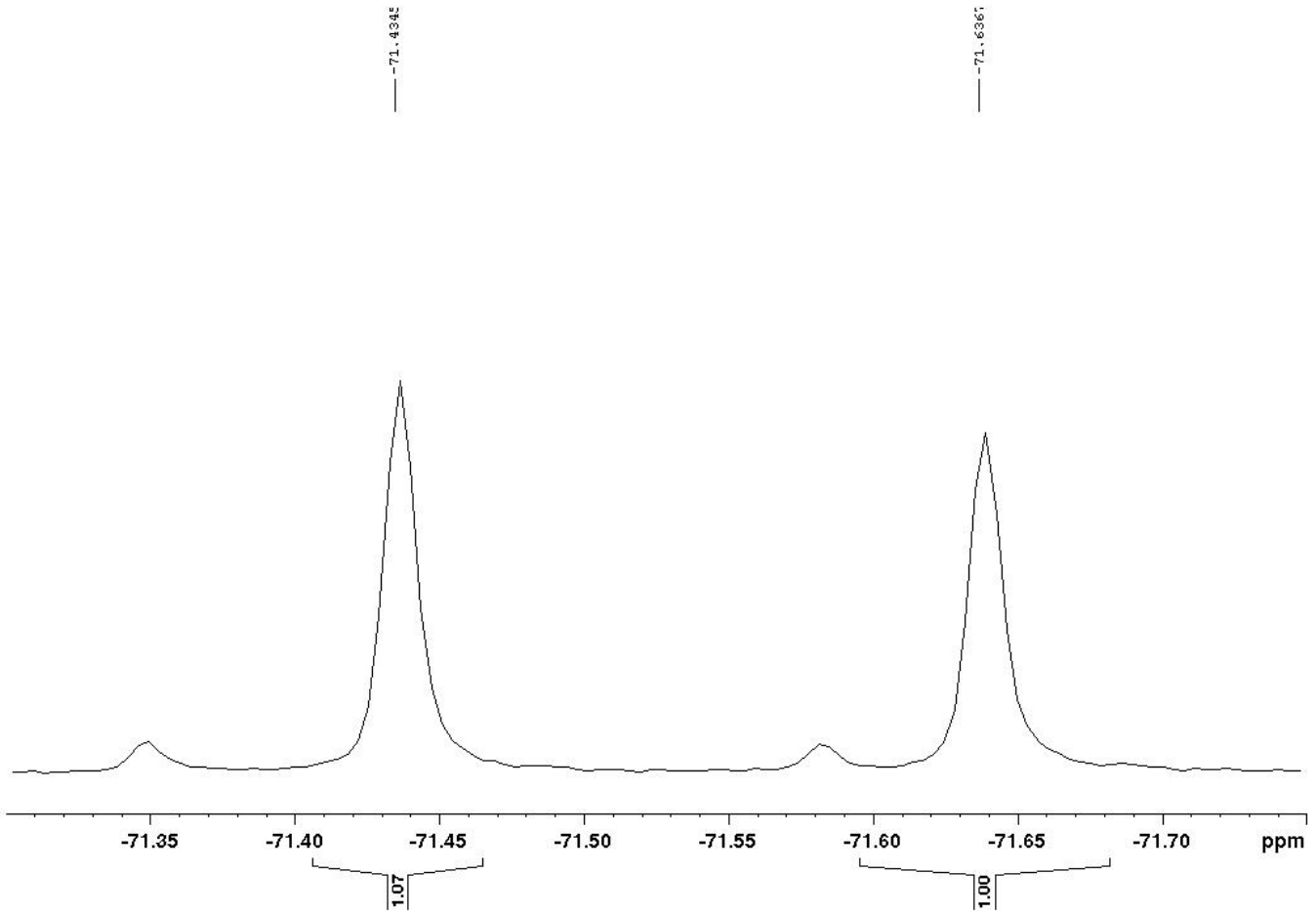
^{1}H NMR


7.351
4.694
4.161
4.138
4.114
4.090
2.363
2.341
2.319
2.241
1.792
1.279
1.256
1.232
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.31
1.00
2.09
1.99
1.02
4.14
3.17

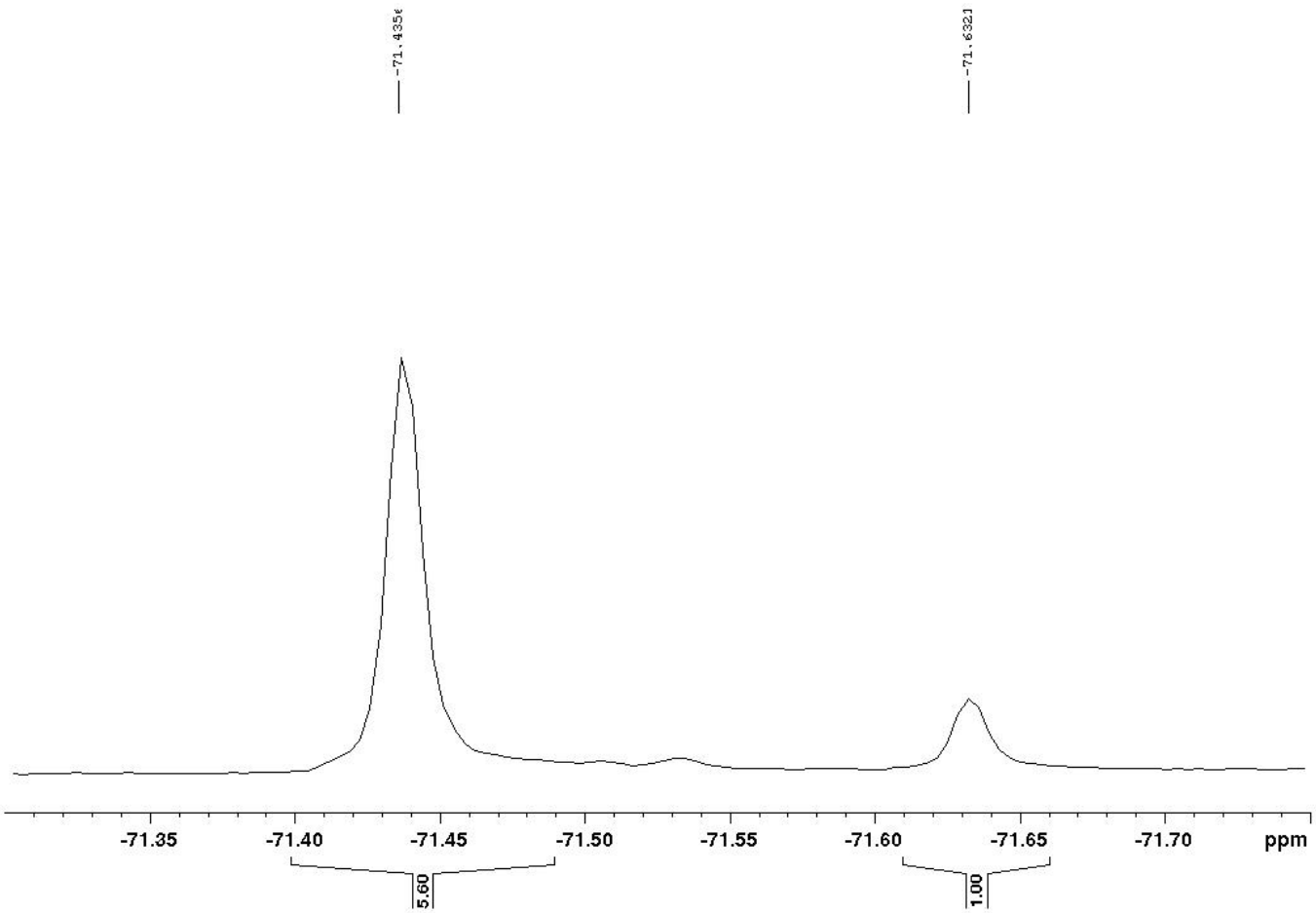
^{13}C NMR


74.12
60.34
38.37
34.03
21.21
14.23
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

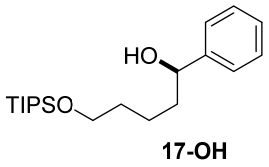
^{19}F NMR of Mosher's ester 15-Mosher



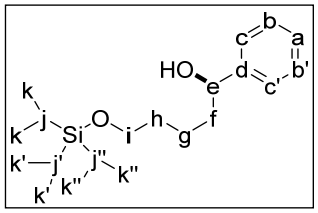
Mixture of diastereomeric 15-Mosher


-71.435
-71.6321
-71.35
-71.40
-71.45
-71.50
-71.55
-71.60
-71.65
-71.70
ppm
5.60
1.00

(*R*,*S*)-**15-Mosher**

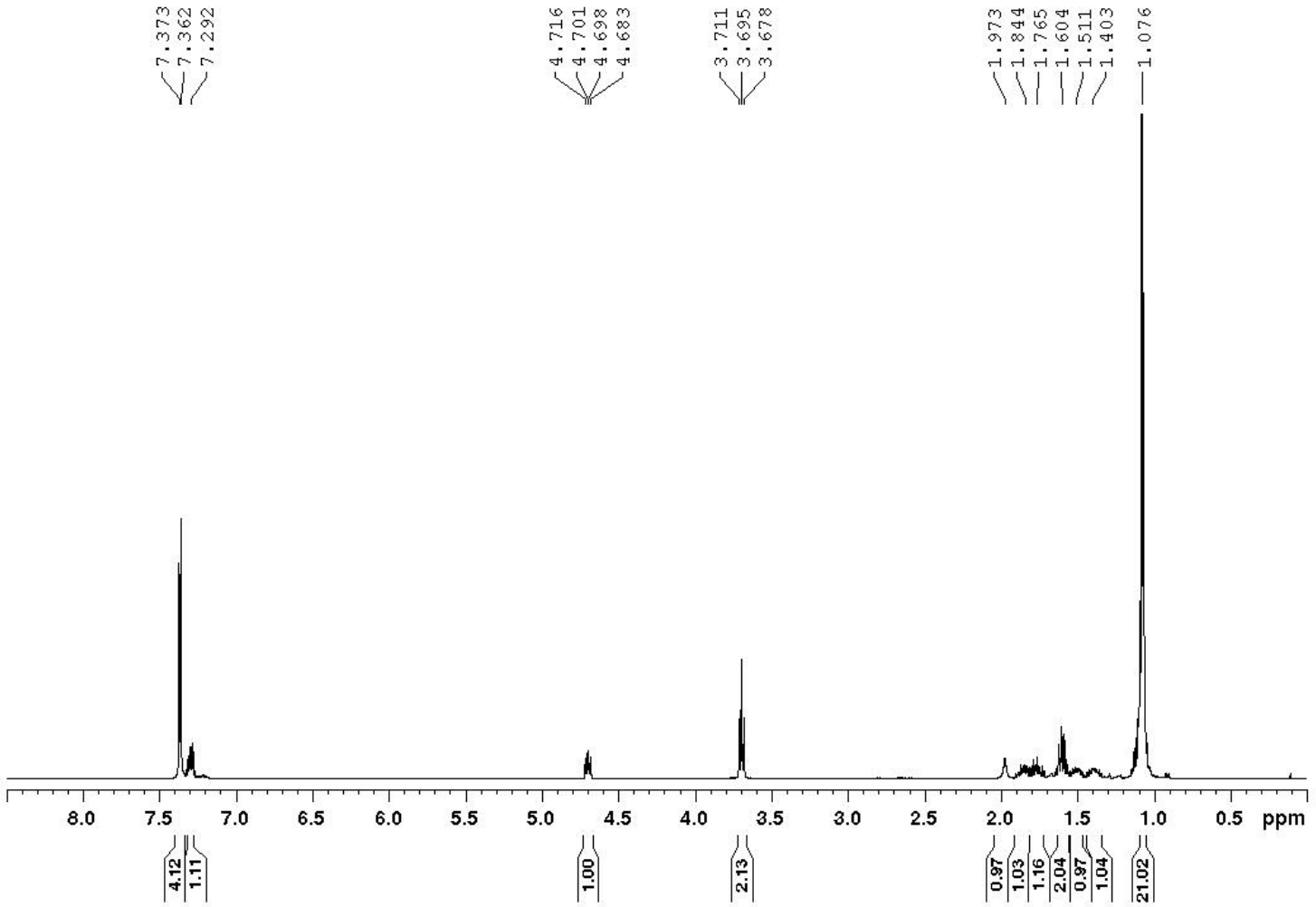


17-OH

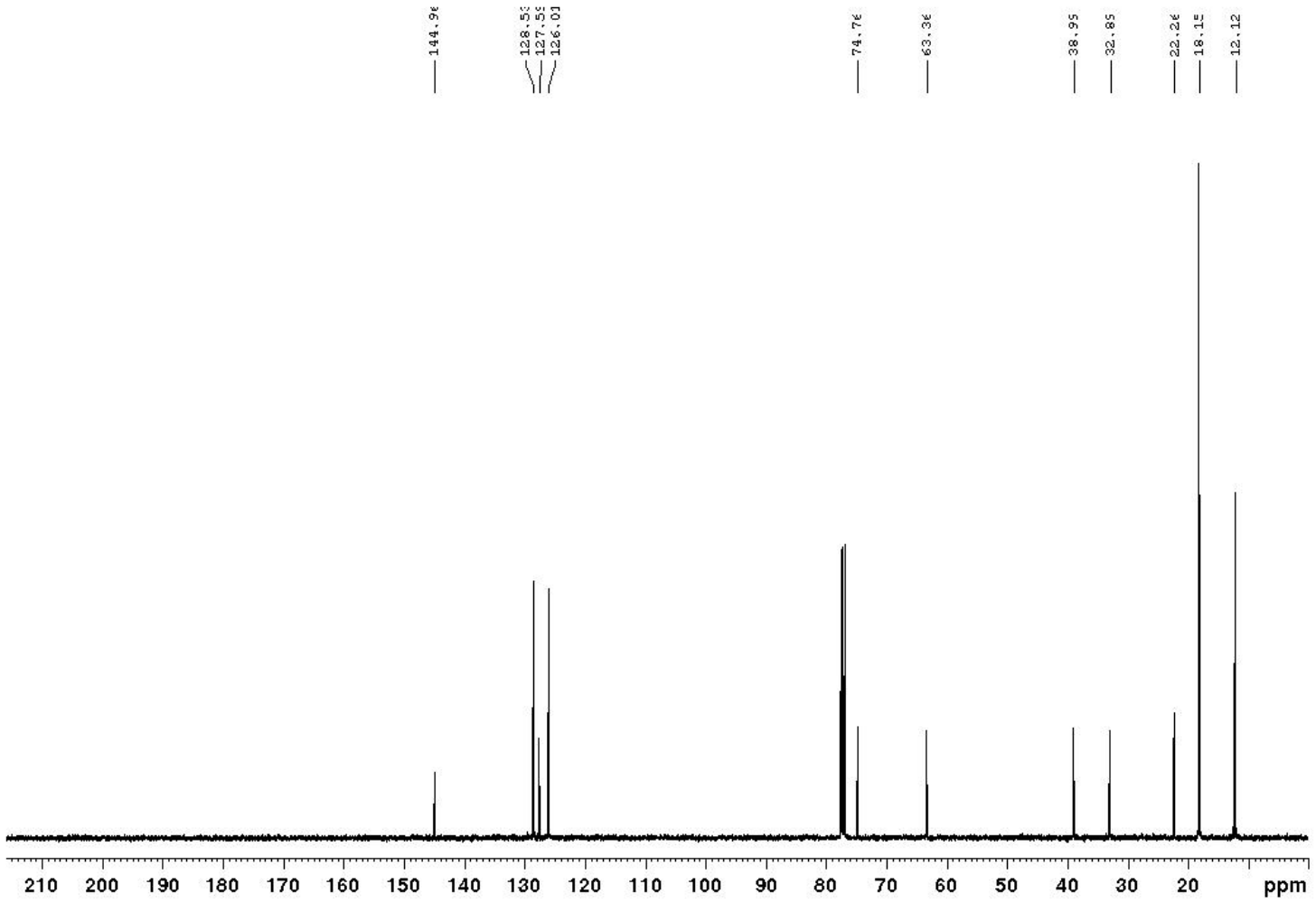


$[\alpha]_D^{20}$	–11.0° (*c* 2.0, $CHCl_3$)
Enantiomeric ratio analysis	Chiral HPLC analysis (Chiralcel-OD, 99:1 hexanes:isopropanol, flowrate = 1.0 mL/min) showed peaks at 35 minutes (10.0% (*S*)) and 42 minutes (90.0% (*R*))
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.35–7.40 (4H, m, b,b',c,c'), 7.25–7.35 (1H, m, a), 4.70 (1H, dd, *J* = 7.2 and 6.0 Hz, e), 3.69 (2H, d, *J* = 6.4 Hz, i), 1.97 (1H, br s, OH), 1.80–1.90 (1H, m, f), 1.70–1.80 (1H, m, f), 1.55–1.65 (2H, m, h), 1.45–1.55 (1H, m, g), 1.35–1.45 (1H, m, g), 1.05–1.10 (21H, m, j,j',j'',k,k',k'')
^{13}C NMR (100 MHz, $CDCl_3$)	δ 144.96 (d), 128.53 (b,b'), 127.59 (a), 126.01 (c,c'), 74.76 (e), 63.36 (i), 38.99 (f), 32.89 (h), 22.26 (g), 18.15 (k,k',k''), 12.12 (j,j',j'')
IR (neat)	3369 (O–H stretch), 2940, 2891, 2864, 1462, 1101, 1069, 881, 759, 698, 679, 657 cm^{-1}
HRMS (ESI)	$C_{20}H_{36}NaO_2Si$ (M+Na): 359.2382, found 359.2385 *m/z*

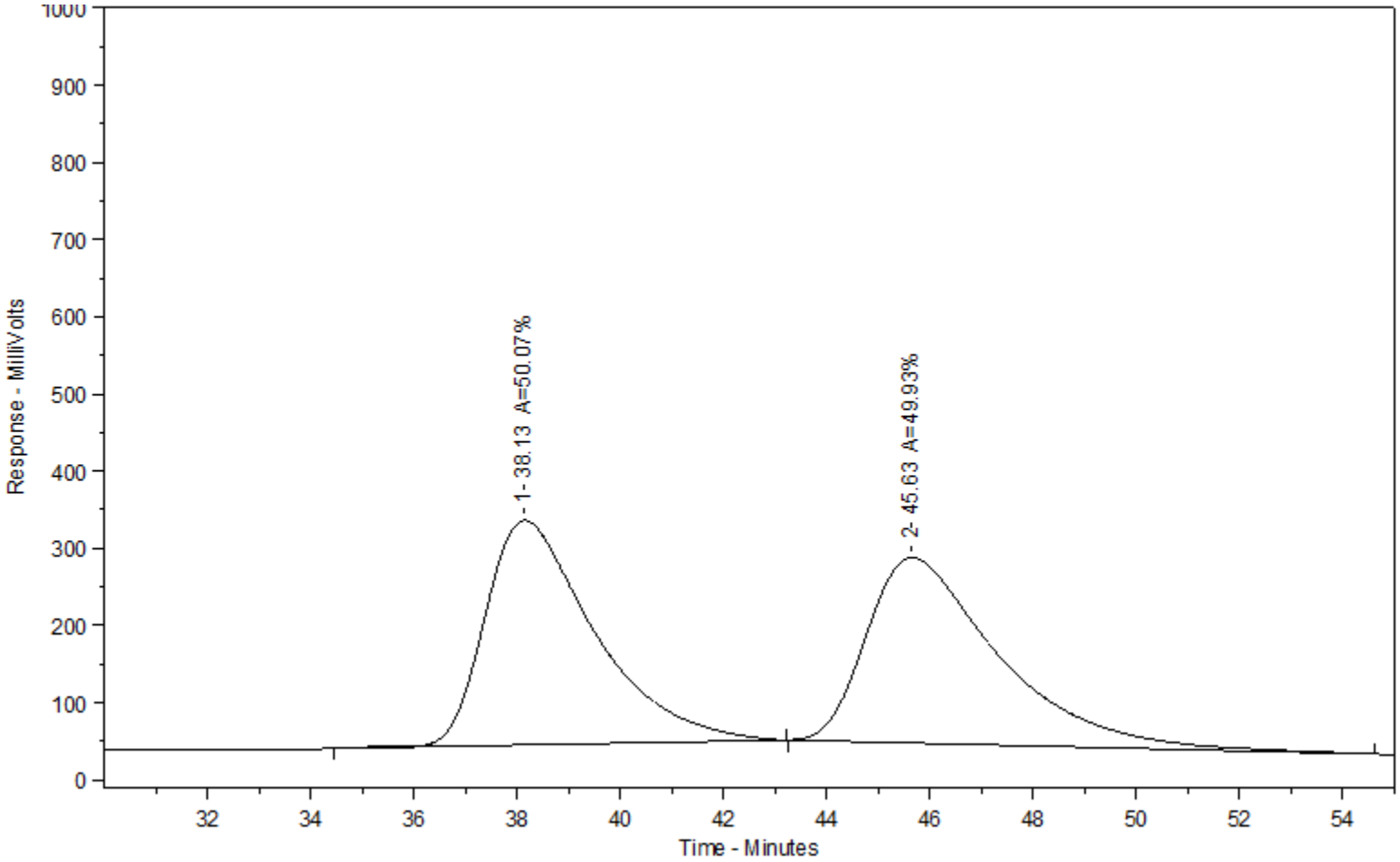
^{1}H NMR


7.373
7.362
7.292
4.716
4.701
4.698
4.683
3.711
3.695
3.678
1.973
1.844
1.765
1.604
1.511
1.403
1.076
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.12
1.11
1.00
2.13
0.97
1.03
1.16
2.04
0.97
1.04
21.02

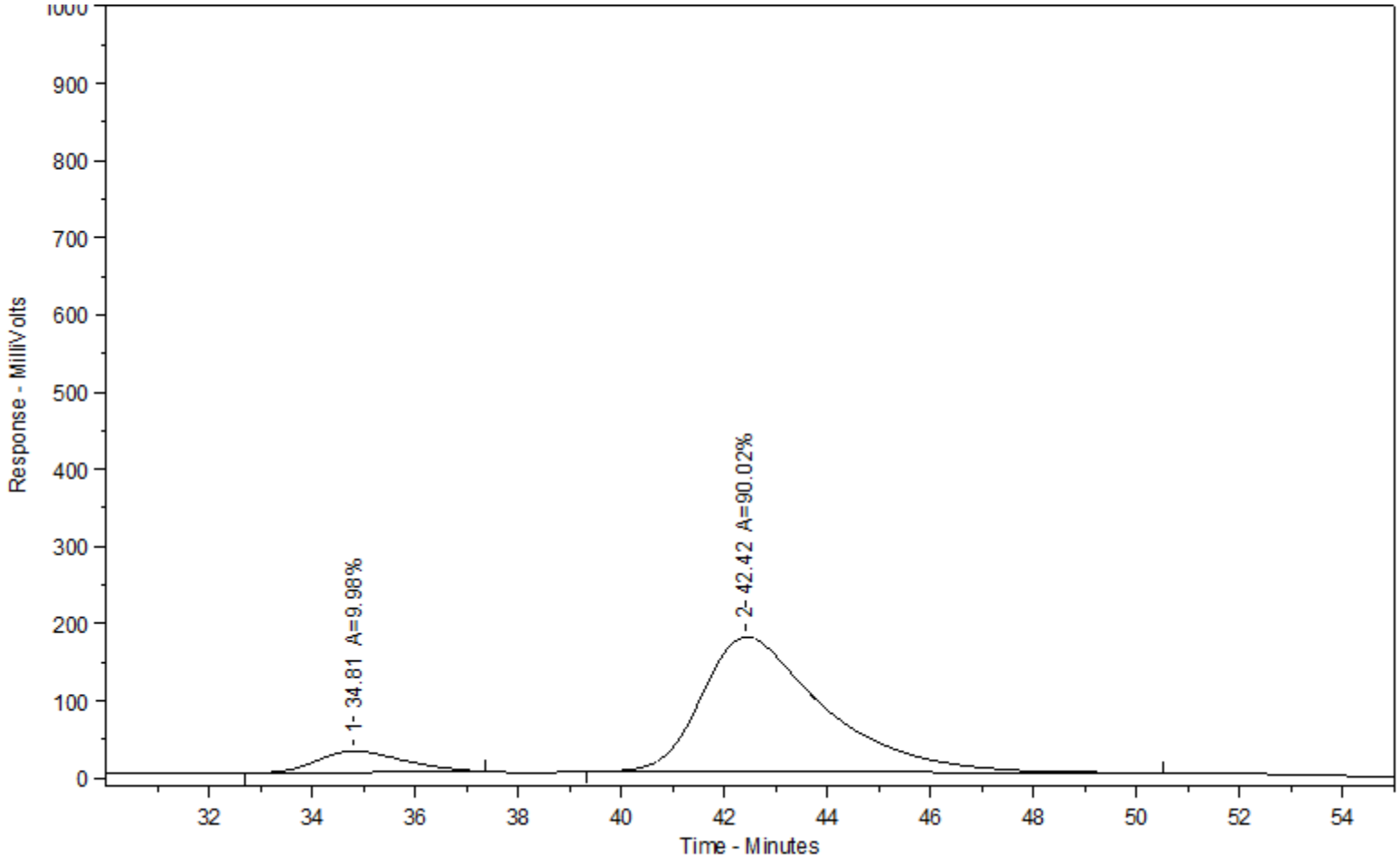
^{13}C NMR


144.9
128.5
127.5
126.0
74.7
63.3
38.9
32.8
22.2
18.1
12.1
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

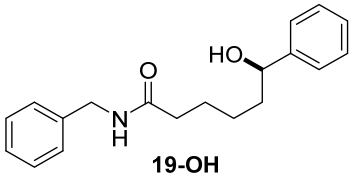
HPLC trace of 17-OH



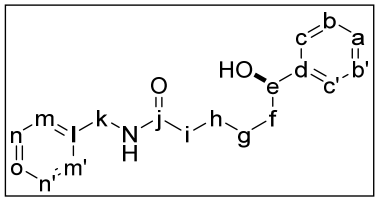
Racemic **17-OH**


Response - MilliVolts
1000
900
800
700
600
500
400
300
200
100
0
1- 34.81 A=9.98%
2- 42.42 A=90.02%
32
34
36
38
40
42
44
46
48
50
52
54
Time - Minutes

(*R*)-**17-OH**

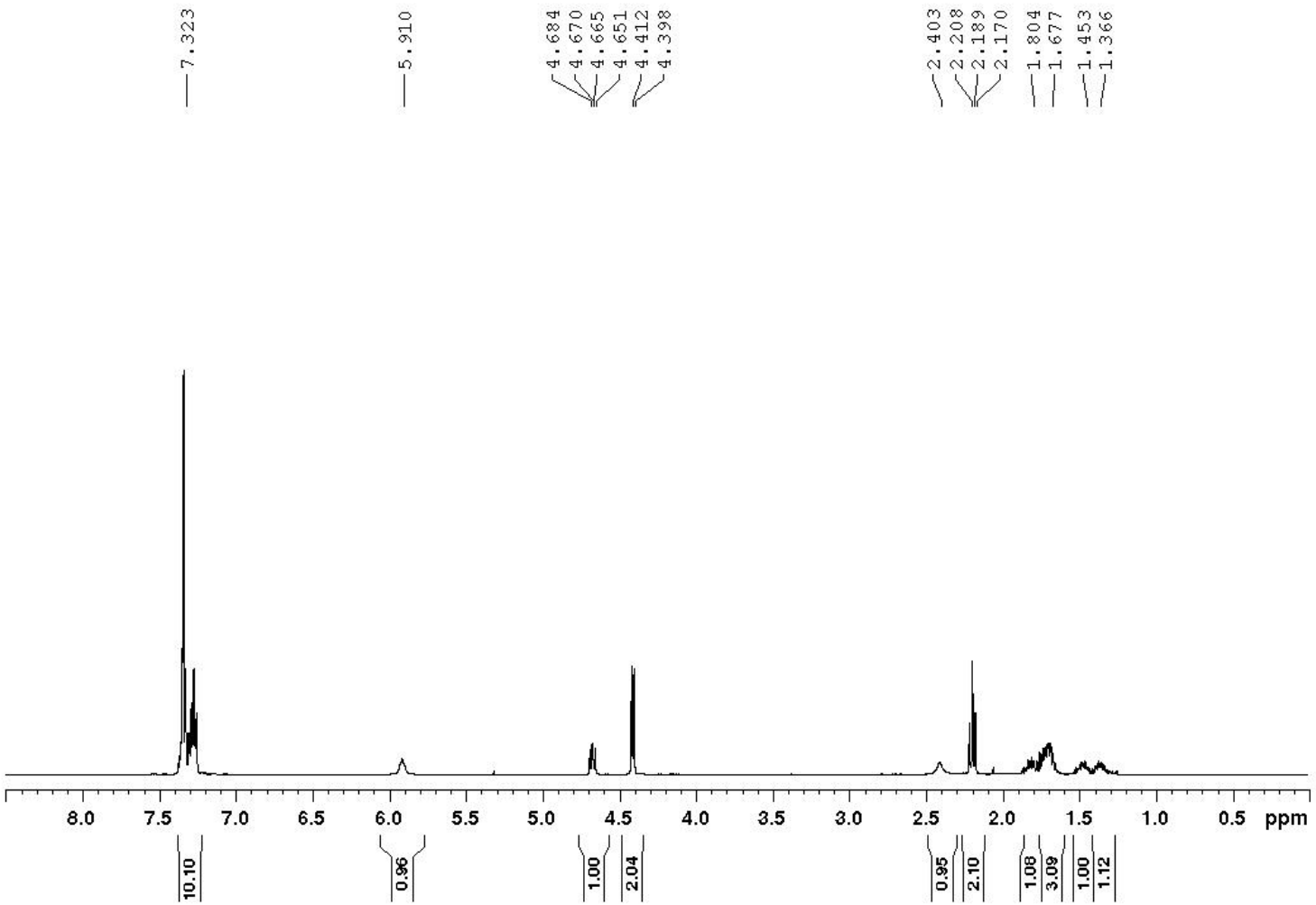


19-OH

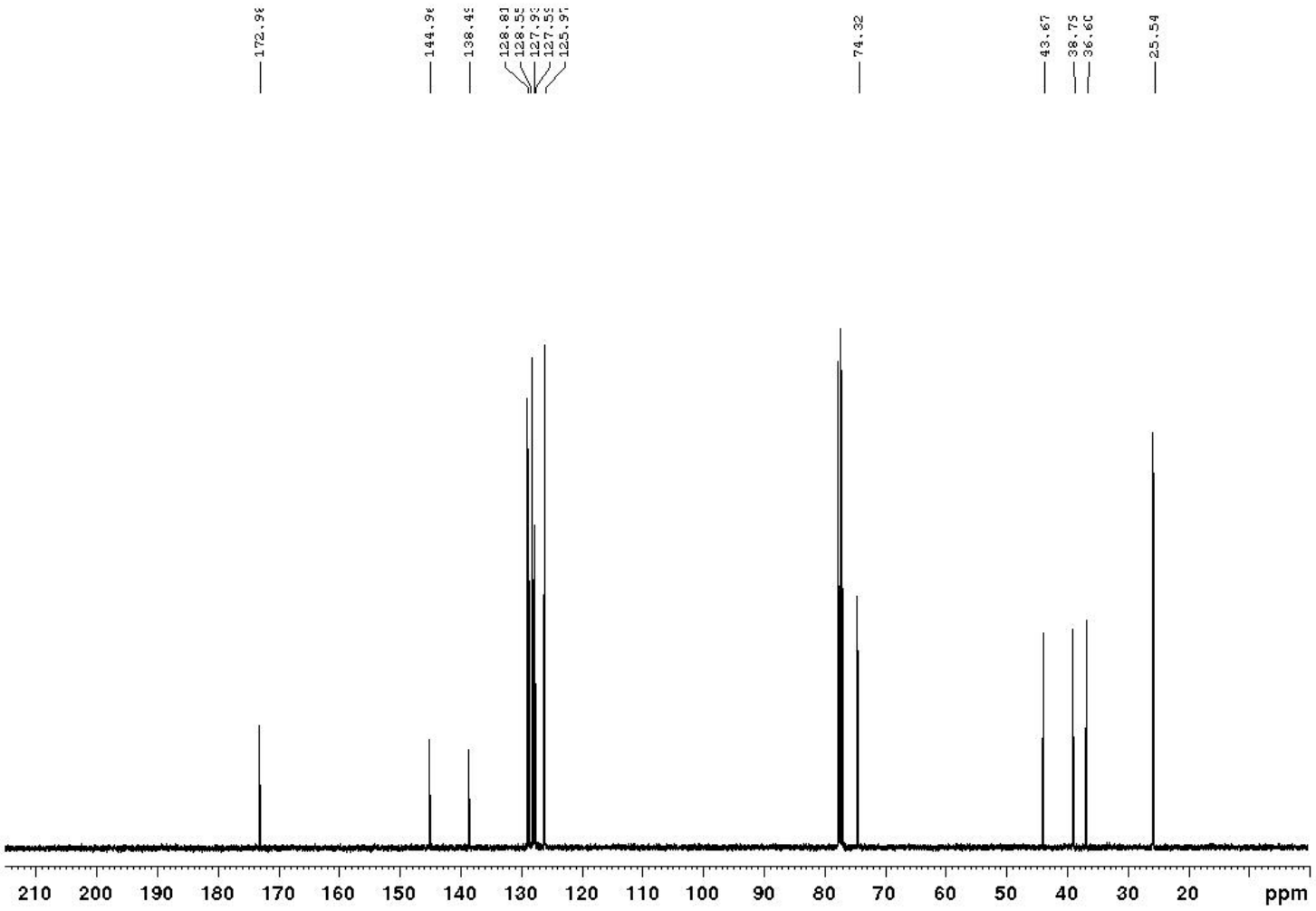


$[\alpha]_D^{20}$	–16.8° (*c* 2.0, $CHCl_3$)
HPLC analysis	Chiral HPLC analysis (Chiralpak-IC, 60:40 hexanes:isopropanol, flowrate = 1.0 mL/min) showed peaks at 47 minutes (90.5% (*R*)) and 56 minutes (9.5% (*S*))
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (10H, m, a,b,b',c,c',l,m,m',n,n',o), 5.91 (1H, br s, NH), 4.67 (1H, dd, *J* = 7.6 and 5.6 Hz, e), 4.40 (2H, d, *J* = 5.7 Hz, k), 2.40 (1H, br s, OH), 2.19 (2H, t, *J* = 7.5 Hz, i), 1.75–1.85 (1H, m, f), 1.65–1.75 (3H, m, f,h), 140–1.55 (1H, m, g), 1.30–1.40 (1H, m, g)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.98 (j), 144.96 (d), 138.49 (l), 128.81 (n,n'), 128.55 (b,b'), 127.93 (c,c'), 127.59 (a and o), 125.97 (m,m'), 74.32 (e), 43.67 (k), 38.79 (f), 36.60 (i), 25.54 (g and h)
IR (neat)	3281 (O–H stretch), 2929, 2858, 1642 (C=O stretch), 1544, 1494, 1453, 1028, 747, 696 cm^{-1}
HRMS (EI)	$C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1742 *m/z*

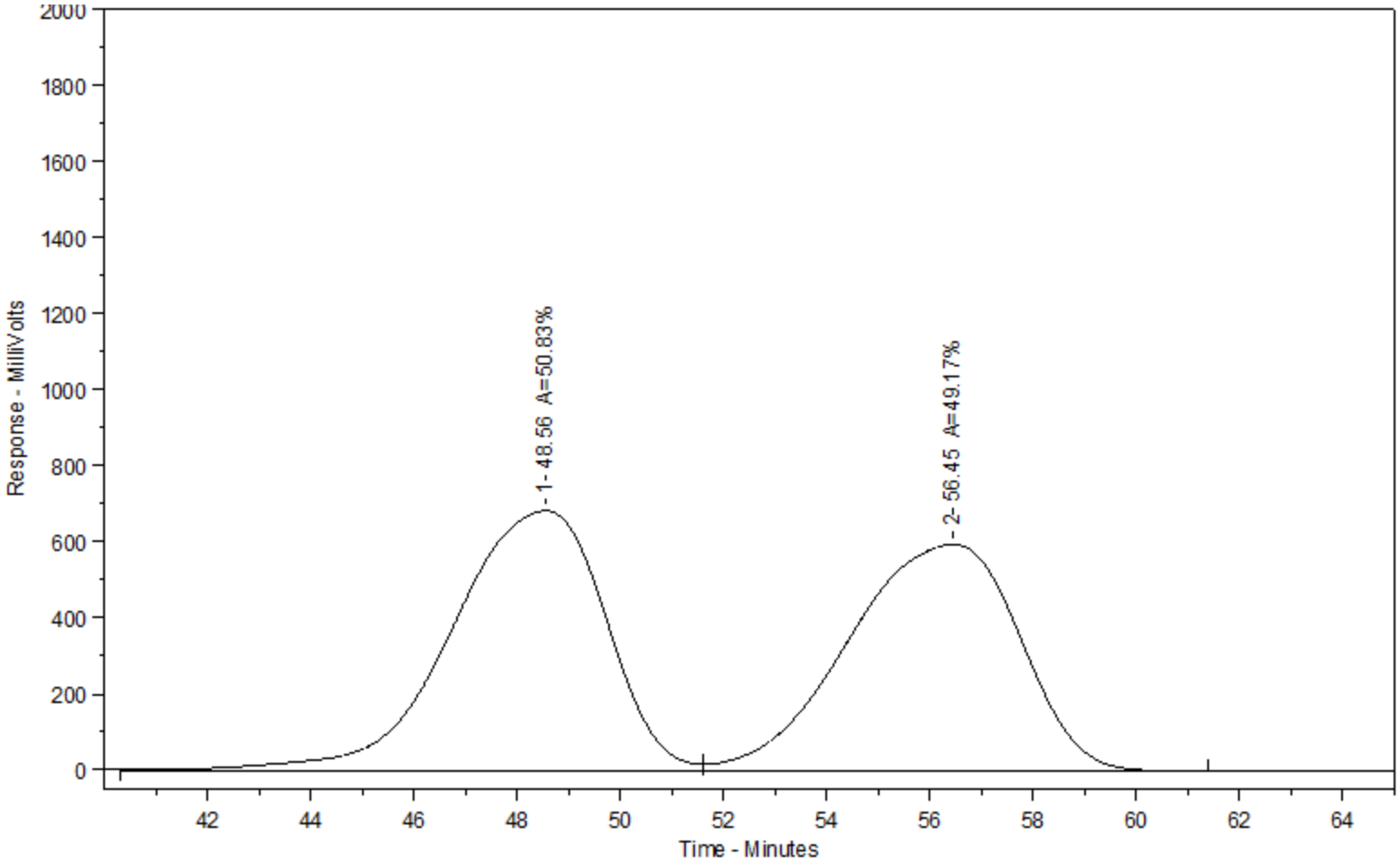
^{1}H NMR


7.323
5.910
4.684
4.670
4.665
4.651
4.412
4.398
2.403
2.208
2.189
2.170
1.804
1.677
1.453
1.366
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.10
0.96
1.00
2.04
0.95
2.10
1.08
3.09
1.00
1.12

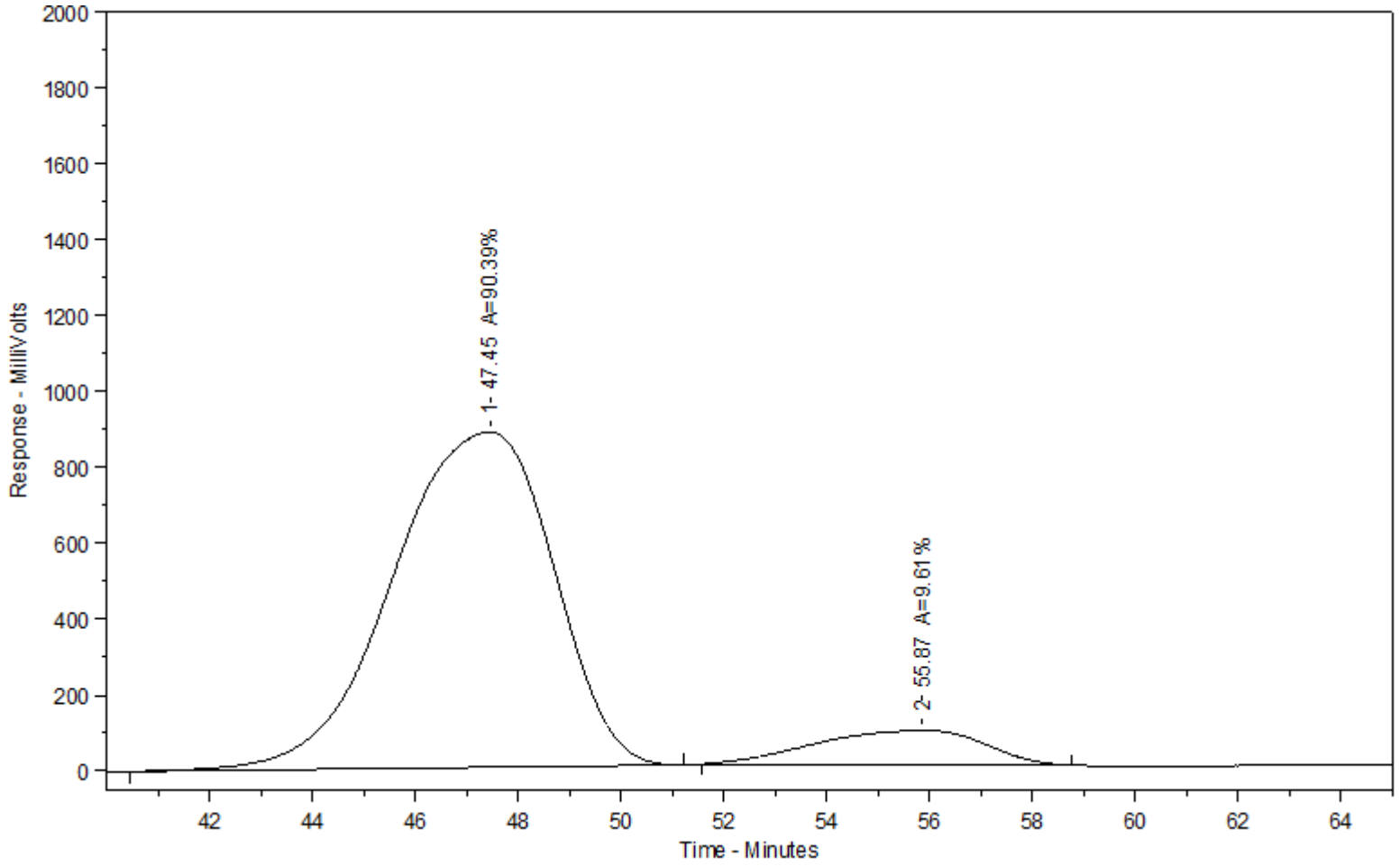
^{13}C NMR


172.98
144.96
138.49
128.81
128.58
127.90
127.59
125.97
74.32
43.67
38.75
36.60
25.54
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

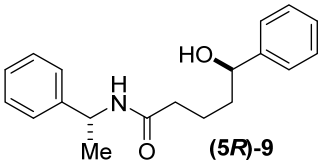
HPLC trace of 19-OH



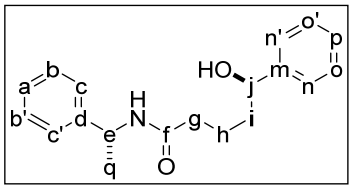
Racemic **19-OH**


Response - MilliVolts
Time - Minutes
1- 47.45 A=90.39%
2- 55.87 A=9.61%
2000
1800
1600
1400
1200
1000
800
600
400
200
0
42
44
46
48
50
52
54
56
58
60
62
64

(*R*)-**19-OH**

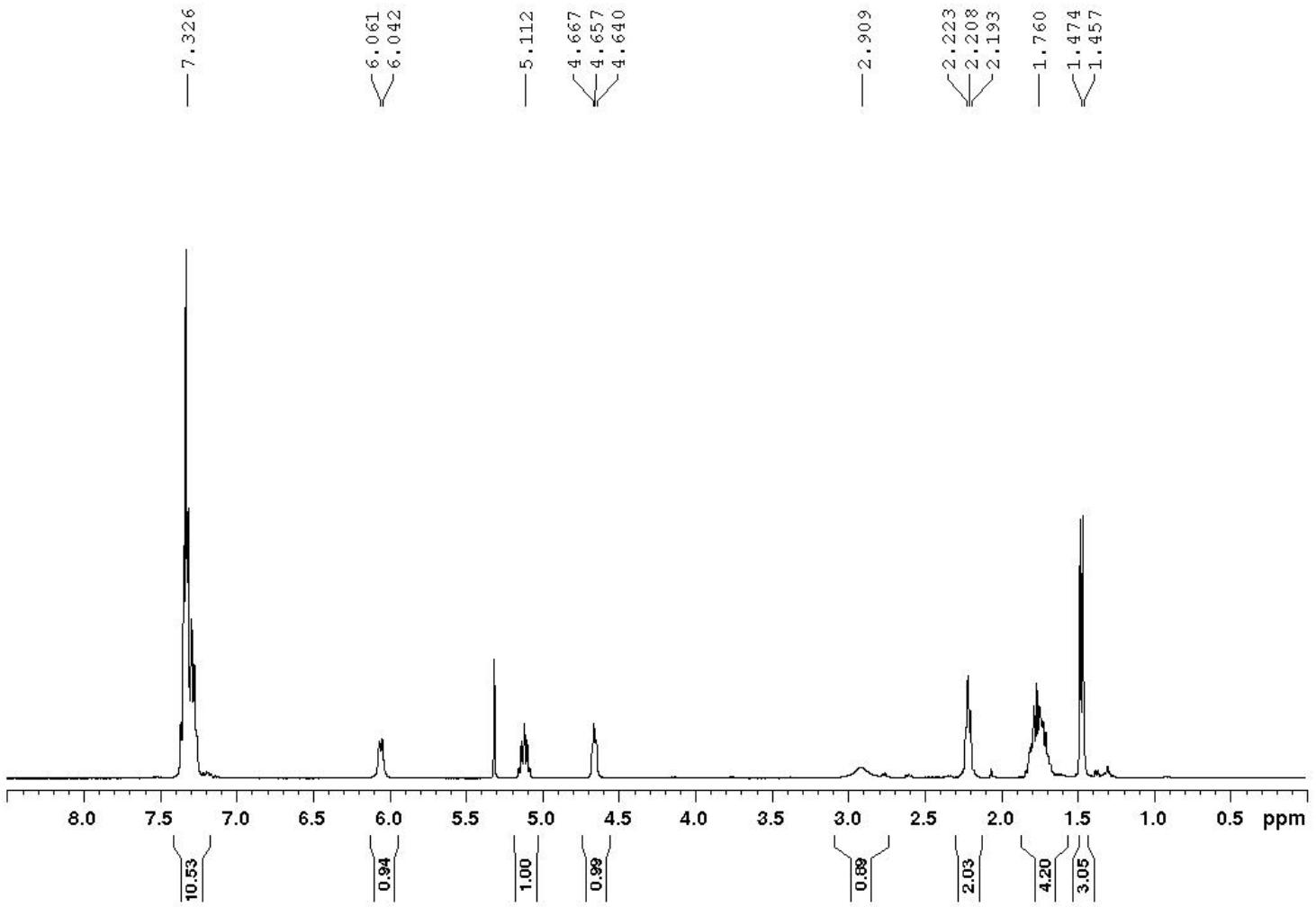


(5*R*)-9

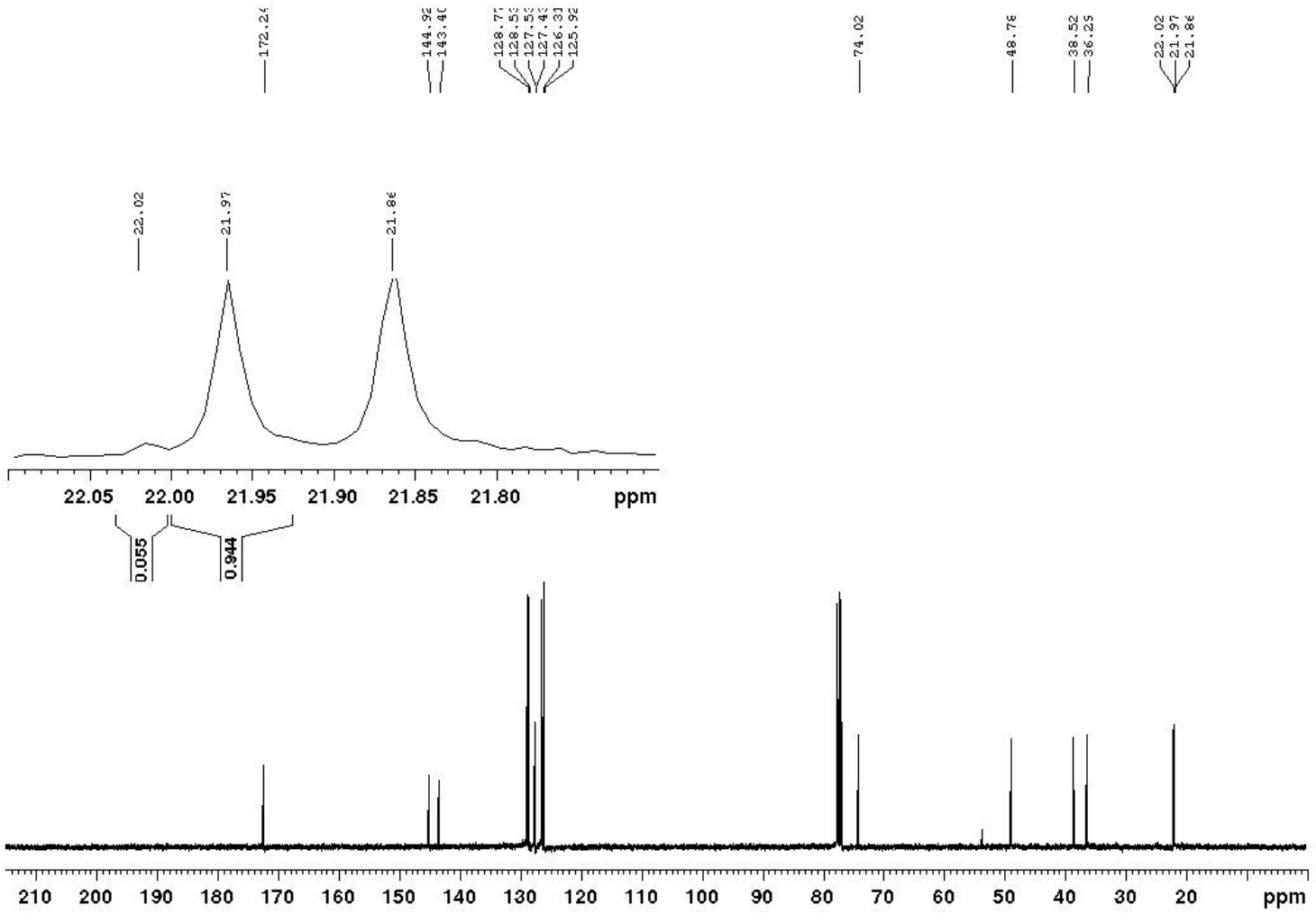


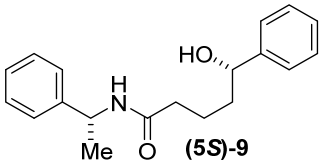
$[\alpha]_D^{20}$	+43.6º (*c* 2.0, $CHCl_3$)
m.p.	65.5–67.0 ºC
TLC analysis	R_f 0.4 (0:100 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (10H, m, aryl), 6.05 (1H, d, *J* = 7.6 Hz, NH), 5.05–5.15 (1H, m, e), 4.60–4.70 (1H, m, j), 2.91 (1H, br s, OH), 2.10–2.30 (2H, m, g), 1.60–1.80 (4H, m, h,i), 1.47 (3H, d, *J* = 6.9 Hz, q)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.24 (f), 144.92 (m), 143.40 (d), 128.77 (aryl), 128.53 (aryl), 127.53 (aryl), 127.43 (aryl), 126.31 (aryl), 125.92 (aryl), 74. 02 (j), 48.78 (e), 38.52 (i), 36. 29 (g), 22.02 (minor q, 5.5%), 21.97 (major q, 94.5%), 21.86 (h)
IR (neat)	3338 (O–H stretch), 2360, 2340, 1639 (C=O stretch), 1620, 1529, 1493, 1448, 1066, 1029, 758, 698 cm^{-1}
HRMS (EI)	$C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1737 *m/z*

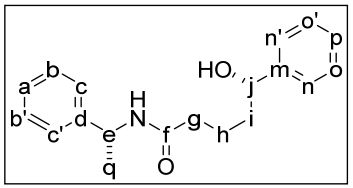
^{1}H NMR


7.326
6.061
6.042
5.112
4.667
4.657
4.640
2.909
2.223
2.208
2.193
1.760
1.474
1.457
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.53
0.94
1.00
0.99
0.89
2.03
4.20
3.05

^{13}C NMR

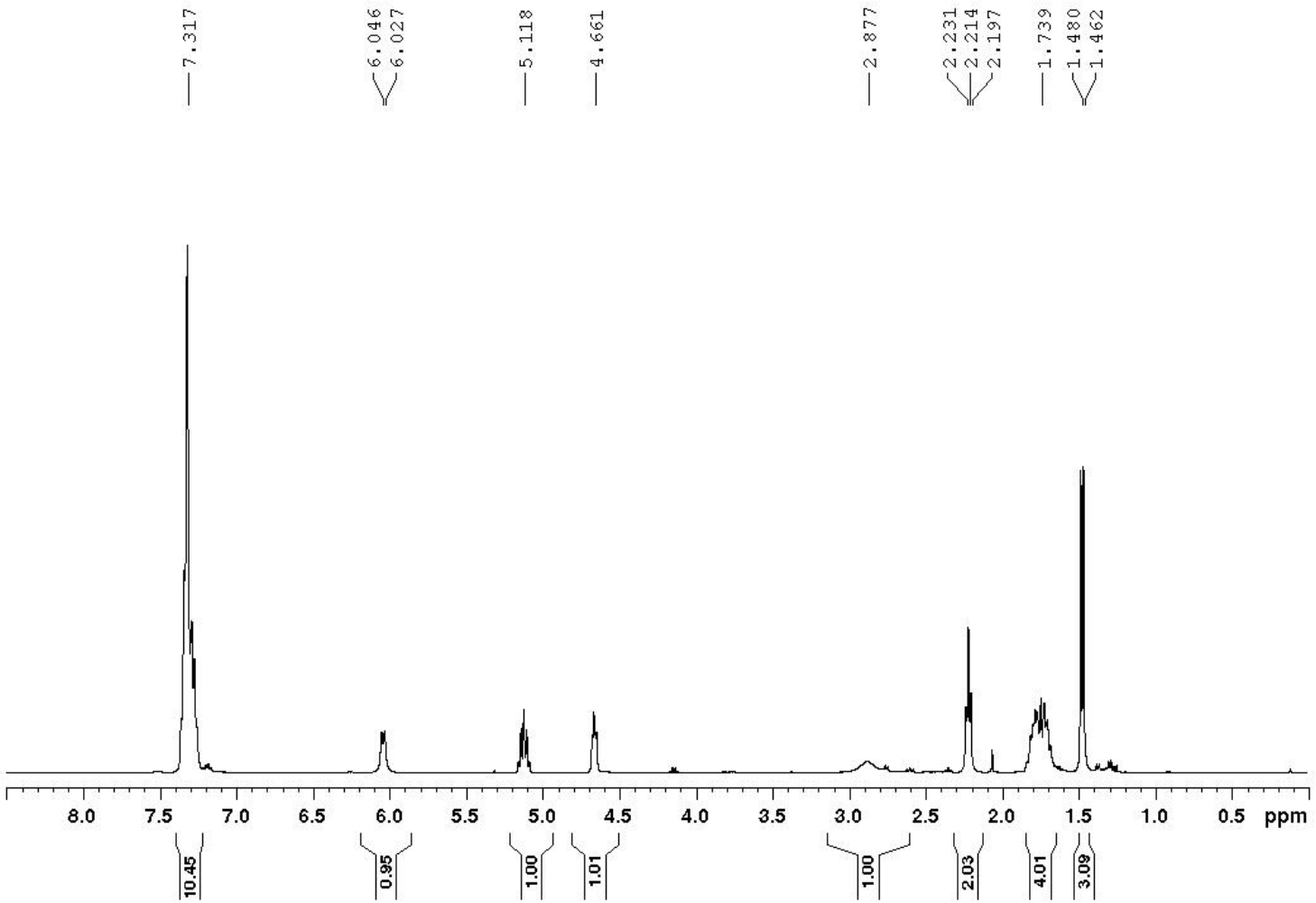

172.24
144.98
143.40
128.77
128.53
127.53
127.43
126.31
125.98
74.02
48.78
38.52
36.25
22.02
21.97
21.88
22.02
21.97
21.88
22.05
22.00
21.95
21.90
21.85
21.80
ppm
0.055
0.944
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm



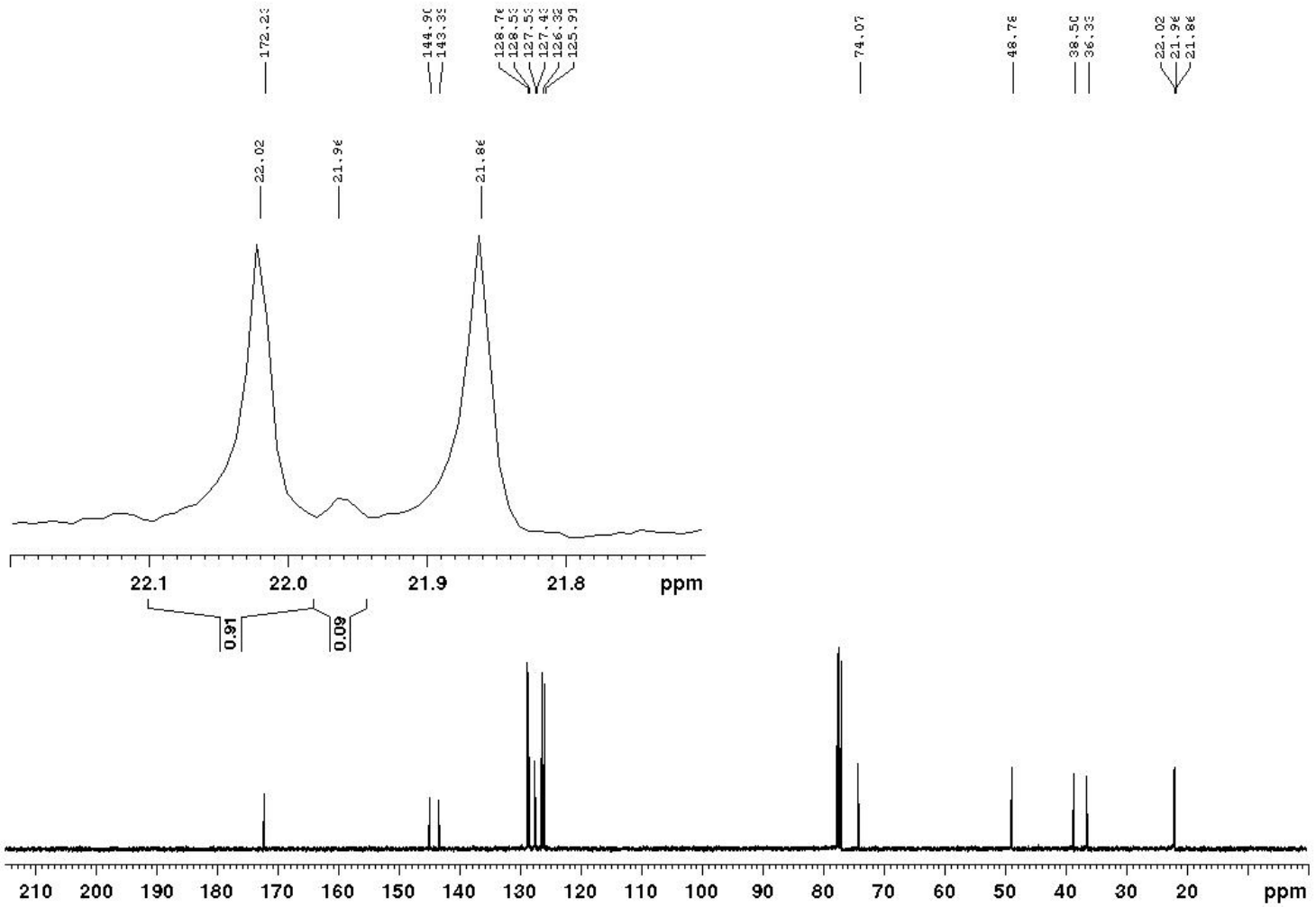


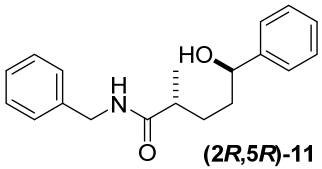
$[\alpha]_D^{20}$	+91.3° (*c* 2.0, $CHCl_3$)
m.p.	96.5–97.5 °C
TLC analysis	R_f 0.4 (0:100 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (10H, m, aryl), 6.04 (1H, d, *J* = 7.6 Hz, NH), 5.05–5.15 (1H, m, e), 4.60–4.70 (1H, m, j), 2.88 (1H, br s, OH), 2.21 (2H, t, *J* = 6.6 Hz, g), 1.60–1.85 (4H, m, h,i), 1.47 (3H, d, *J* = 6.9 Hz, q)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 172.23 (f), 144.90 (m), 143.39 (d), 128.76, 128.53, 127.53, 127.43, 126.32, 125.91, 74. 07 (j), 48.78 (e), 38.50 (i), 36. 33 (g), 22.02 (major q, 9.0%), 21.96 (major q, 91%), 21.86 (h)
IR (neat)	3318 (O–H stretch), 2360, 2341, 1640 (C=O stretch), 1532, 1493, 1449, 1074, 1028, 753, 697 cm^{-1}
HRMS (EI)	$C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1717 *m/z*

^{1}H NMR

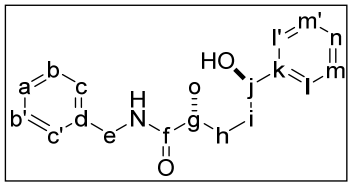

7.317
6.046
6.027
5.118
4.661
2.877
2.231
2.214
2.197
1.739
1.480
1.462
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.45
0.95
1.00
1.01
1.00
2.03
4.01
3.09

^{13}C NMR


172.23
144.90
143.38
128.78
128.53
127.53
127.43
126.38
125.91
74.07
48.78
38.50
36.38
22.02
21.96
21.88
22.1
22.0
21.9
21.8
ppm
0.91
0.09
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

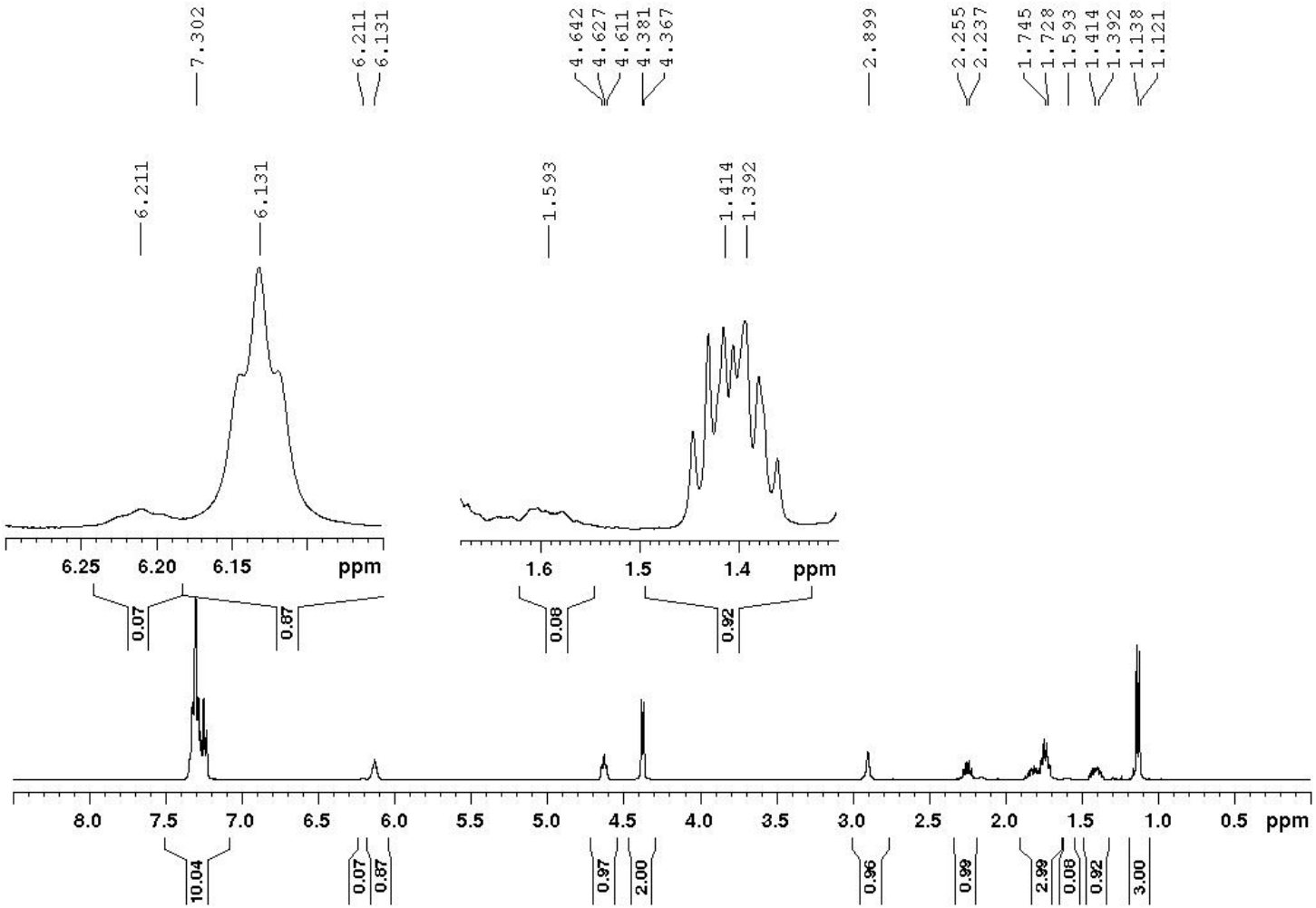


(2*R*,5*R*)-11

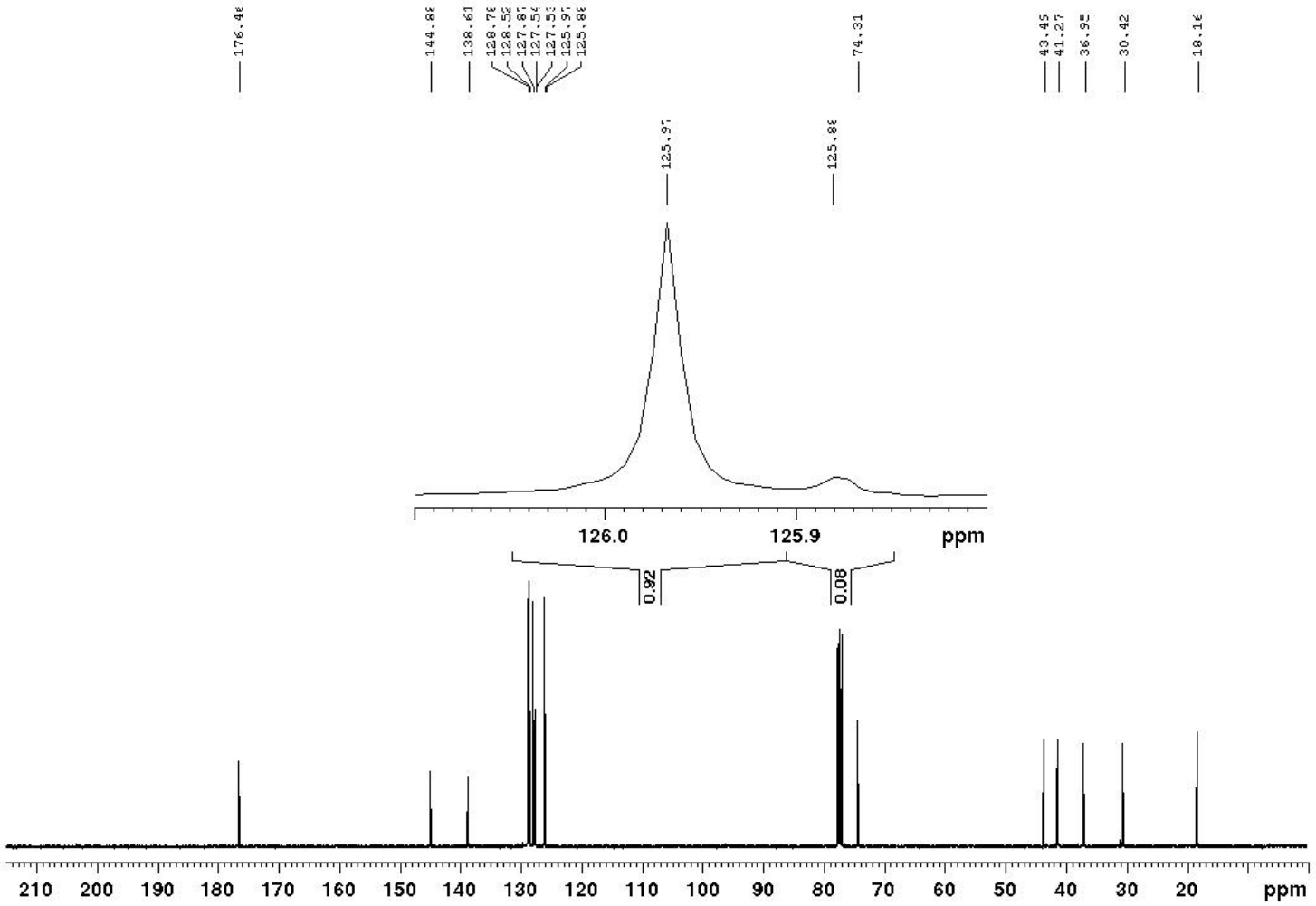


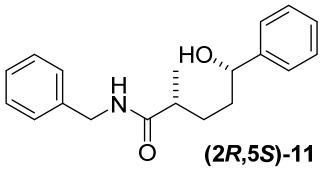
$[\alpha]_D^{20}$	–35.6º (*c* 2.0, $CHCl_3$)
m.p.	63.0–64.5 ºC
TLC analysis	R_f 0.4 (0:100 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.35 (10H, m, aryl), 6.21 (br s, minor 8.0%, NH), 6.13 (br s, major 92%, NH), 4.63 (1H, dd, *J* = 6.2 and 6.2 Hz, j), 4.38 (2H, d, *J* = 5.7 Hz, e), 2.90 (1H, br s, OH), 2.20–2.30 (1H, m, g), 1.70–1.90 (3H, m, h,i), 1.55–1.65 (0.08H, m, minor i), 1.35–1.50 (0.92H, m, major i), 1.13 (3H, d, *J* = 6.9 Hz, o)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 176.46 (f), 144.88 (k), 138.61 (d), 128.78 (aryl), 128.52 (aryl), 127.87 (aryl), 127.54 (aryl), 127.53 (aryl), 125.97 (aryl, 92.0%, major), 125.88 (aryl, 8.0%, minor), 74.31 (j), 43.49 (e), 41.27 (g), 36.95 (i), 30.42 (h), 18.16 (o)
IR (neat)	3571, 3542, 3446, 3307 (O–H stretch), 2916, 1640 (C=O stretch), 1545, 1494, 1243, 1056, 761, 728, 695 cm^{-1}
HRMS (EI)	$C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1724 *m/z*

^{1}H NMR

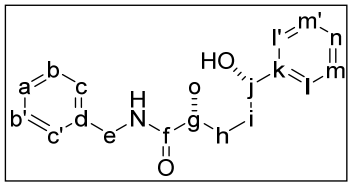

7.302
6.211
6.131
4.642
4.627
4.611
4.381
4.367
2.899
2.255
2.237
1.745
1.728
1.593
1.414
1.392
1.138
1.121
6.25
6.20
6.15
ppm
1.6
1.5
1.4
ppm
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.04
0.07
0.87
0.97
2.00
0.96
0.99
2.99
0.08
0.92
3.00

^{13}C NMR


176.4
144.8
138.6
128.7
128.5
127.8
127.5
125.9
125.8
74.31
43.45
41.27
36.95
30.42
18.16
125.9
125.8
126.0
125.9
ppm
0.92
0.08
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

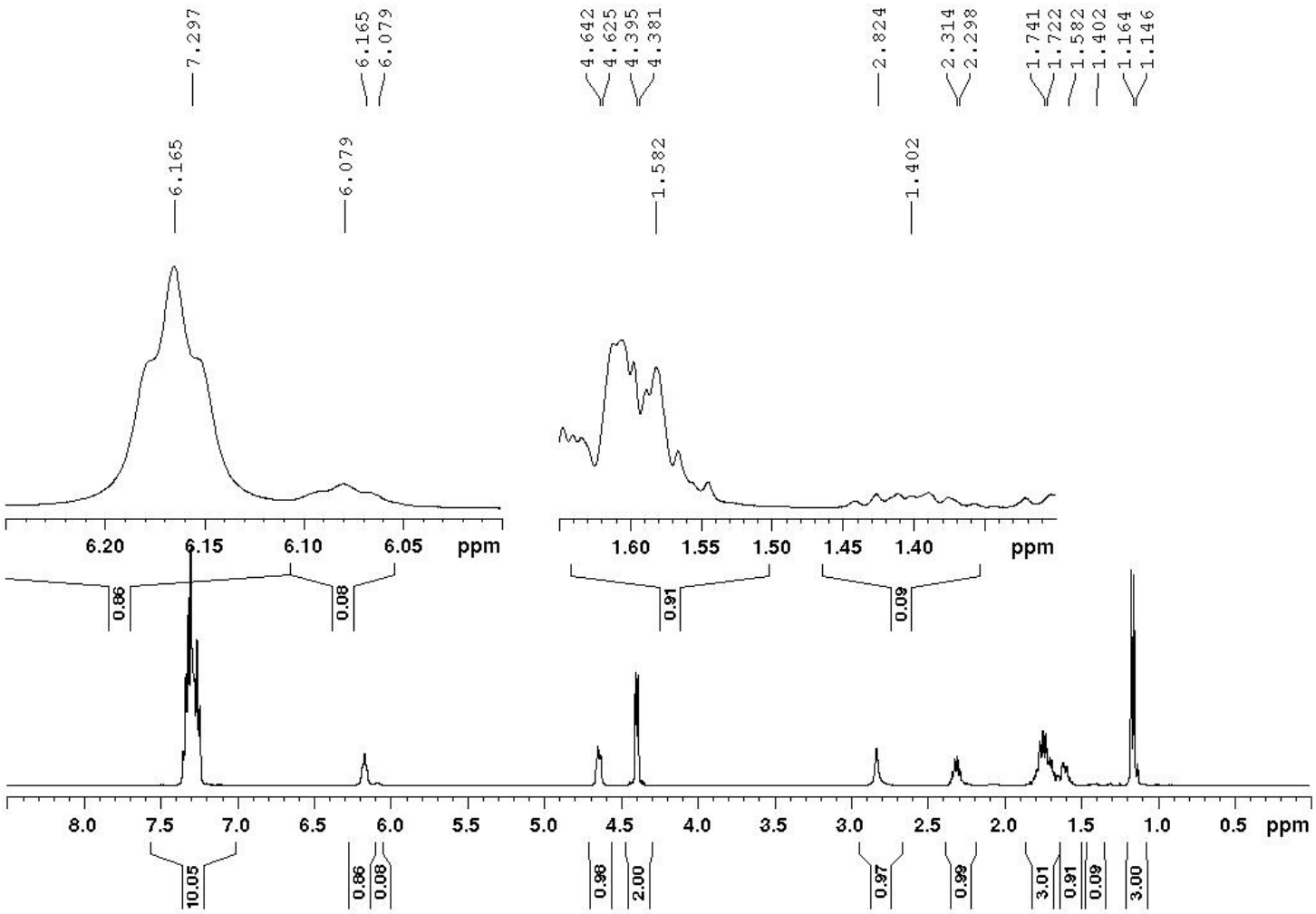


(2*R*,5*S*)-11

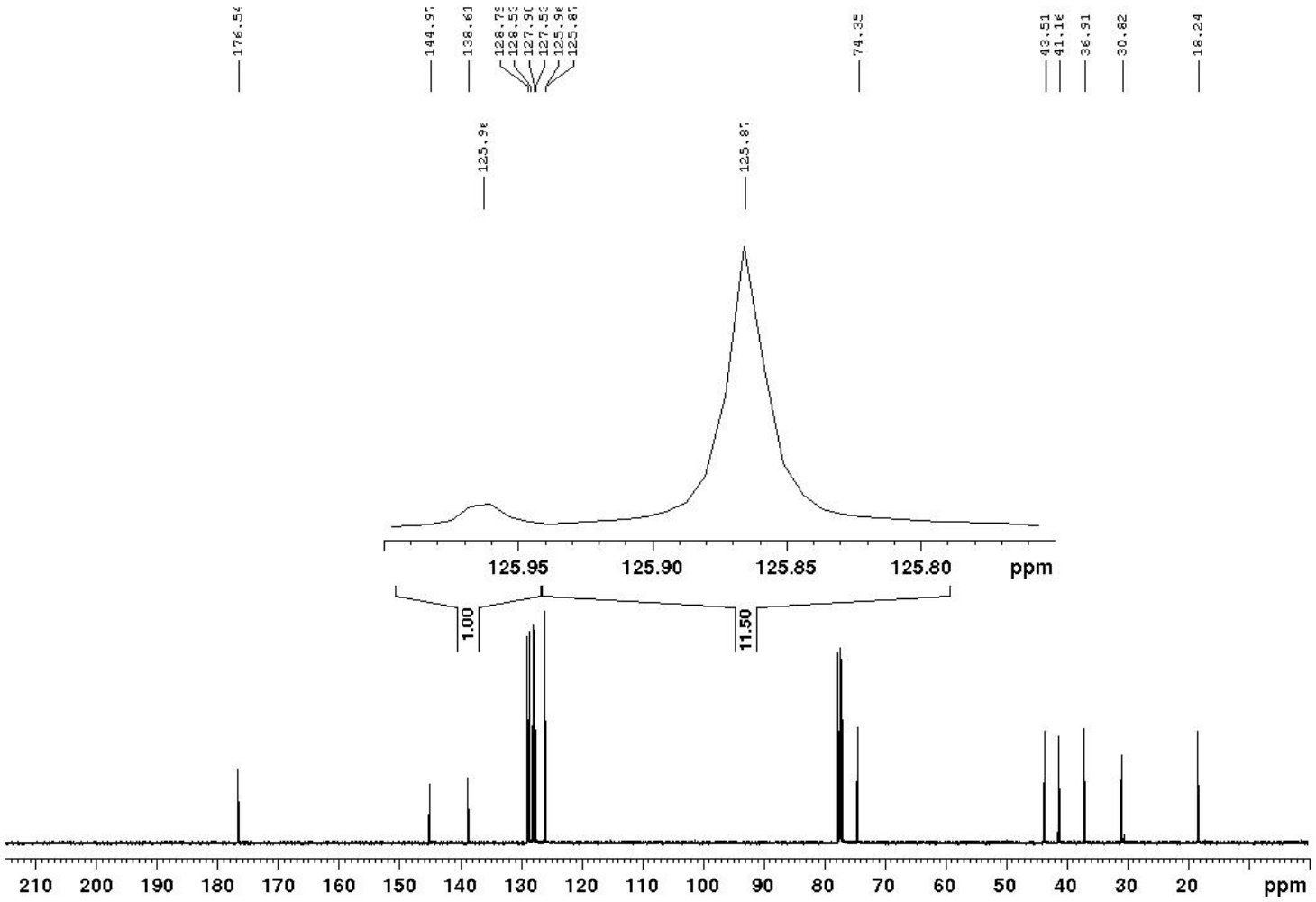


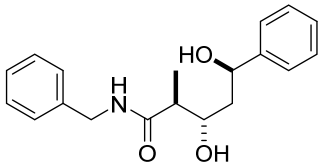
$[\alpha]_D^{20}$	+22.9º (*c* 2.0, $CHCl_3$)
m.p.	93.0–95.0 ºC
TLC analysis	R_f 0.4 (0:100 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.20–7.40 (10H, m, aryl), 6.17 (br s, major 91.0%, NH), 6.08 (br s, minor 9.0%, NH), 4.60–4.65 (1H, m, j), 4.39 (2H, d, *J* = 5.6 Hz, e), 2.82 (1H, br s, OH), 2.20–2.35 (1H, m, g), 1.65–1.80 (3H, m, h,i), 1.50–1.65 (0.91H, m, major i), 1.35–1.45 (0.09H, m, minor i), 1.15 (3H, d, *J* = 6.8 Hz, o)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 176.54 (f), 144.97 (k), 138.61 (d), 128.79 (aryl), 128.53 (aryl), 127.90 (aryl), 127.53 (aryl), 127.53 (aryl), 12.96 (aryl, 8.0%, minor), 125.87 (aryl, 92.0%, major), 74.35 (j), 43.51 (e), 41.16 (g), 36.91 (i), 30.82 (h), 18.24 (o)
IR (neat)	3285 (O–H stretch), 2919, 1636 (C=O stretch), 1548, 1495, 1451, 1350, 1225, 1089, 1056, 1027, 753, 735, 695 cm^{-1}
HRMS (EI)	$C_{19}H_{23}NO_2$ (M): 297.1729, found 297.1719 *m/z*

^{1}H NMR

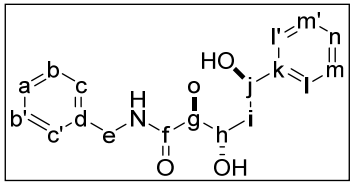

7.297
6.165
6.079
4.642
4.625
4.395
4.381
2.824
2.314
2.298
1.741
1.722
1.582
1.402
1.164
1.146
6.165
6.079
1.582
1.402
6.20
6.15
6.10
6.05
ppm
1.60
1.55
1.50
1.45
1.40
ppm
0.86
0.08
0.91
0.09
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
10.05
0.86
0.08
0.96
2.00
0.97
0.99
3.01
0.91
0.09
3.00

^{13}C NMR


176.54
144.97
138.63
128.75
128.53
127.90
127.53
125.96
125.87
74.35
43.51
41.16
36.91
30.82
18.24
125.95
125.87
125.95
125.90
125.85
125.80
ppm
1.00
11.50
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

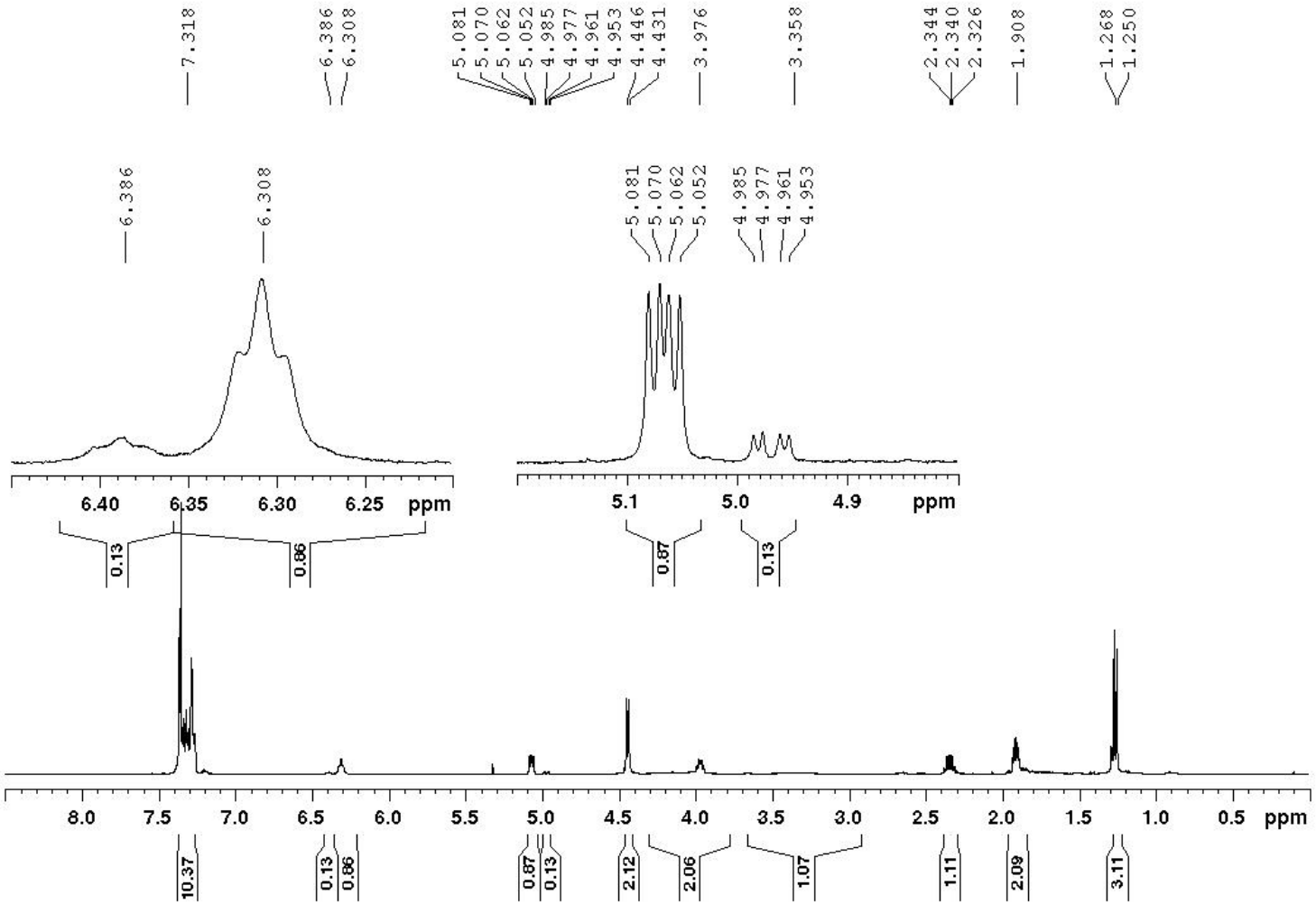


(2*S*,3*S*,5*R*)-13

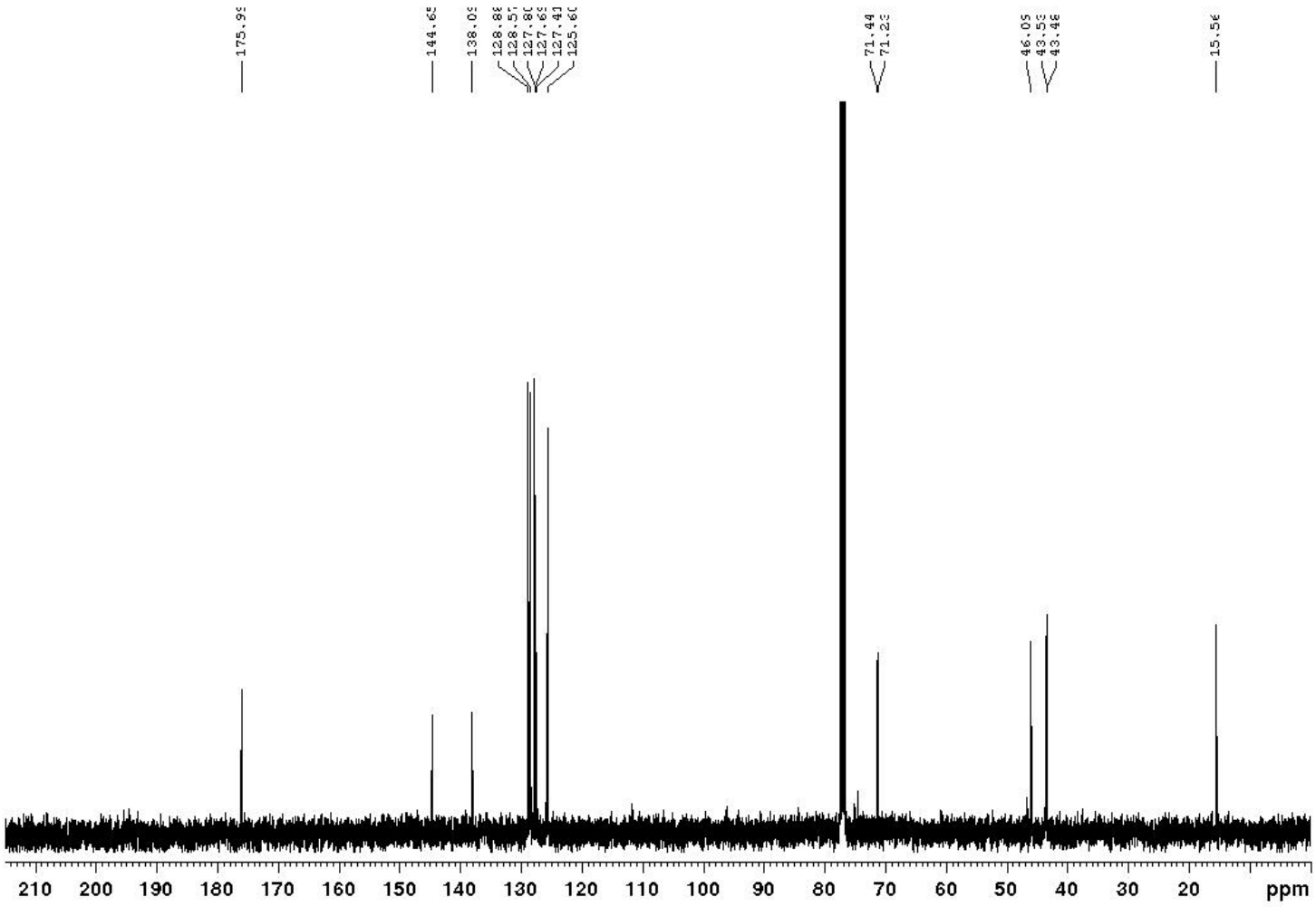


$[\alpha]_D^{20}$	–14.5° (*c* 0.775, $CHCl_3$)
m.p.	109.0–110.0 °C
TLC analysis	R_f 0.3 (0:100 hexanes:ethyl acetate)
^{1}H NMR (400 MHz, $CDCl_3$)	δ 7.25–7.40 (10H, m, aryl), 6.39 (br s, minor 13.0%, NH), 6.31 (br s, major 87.0%, NH), 5.07 (0.87H, dd, *J* = 7.3 and 4.1 Hz, j), 4.97 (0.13H, dd, *J* = 9.6 and 3.3 Hz, j), 4.44 (2H, d, *J* = 5.7 Hz, e), 3.80–4.20 (2H, m, h,OH), 3.36 (1H, br s, OH), 2.30–2.40 (1H, m, g), 1.85–1.95 (2H, m, i), 1.26 (3H, d, *J* = 7.2 Hz, o)
^{13}C NMR (100 MHz, $CDCl_3$)	δ 175.99 (f), 144.65 (k), 138.09 (d), 128.88 (aryl), 128.57 (aryl), 127.80 (aryl), 127.69 (aryl), 127.41 (aryl), 125.60 (aryl), 74.44 (j), 71.23 (h), 46.09 (g), 43.52 (i), 43.48 (e), 15.56 (o)
IR (neat)	3295 (O–H stretch), 2925, 2359, 2341, 1636 (C=O stretch), 1537, 1495, 1453, 1369, 1028, 751, 696 cm^{-1}
HRMS (ESI)	$C_{19}H_{23}NNaO_3$ (M+Na): 336.1576, found 336.1583 *m/z*

^{1}H NMR


7.318
6.386
6.308
5.081
5.070
5.062
5.052
4.985
4.977
4.961
4.953
4.446
4.431
3.976
3.358
2.344
2.340
2.326
1.908
1.268
1.250
6.40
6.35
6.30
6.25
ppm
5.1
5.0
4.9
ppm
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.13
0.86
0.87
0.13
10.37
0.13
0.86
0.87
0.13
2.12
2.06
1.07
1.11
2.09
3.11

^{13}C NMR


175.9
144.6
138.0
128.8
128.5
127.8
127.6
127.4
125.6
71.44
71.2
46.05
43.5
43.4
15.5
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm