A Mild and Efficient Flow Procedure for the Transfer Hydrogenation of Ketones and Aldehydes using Hydrous Zirconia

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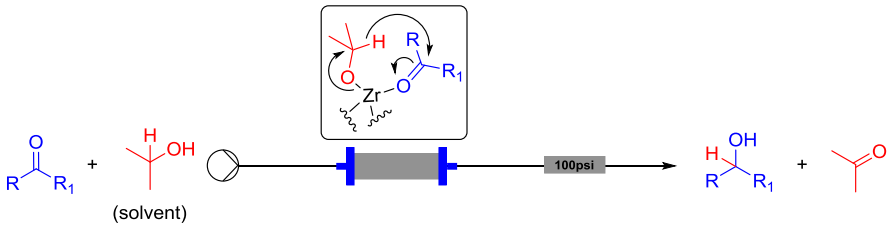
General experimental section. ^{1}H-NMR spectra were recorded on a Bruker Avance DPX-400 spectrometer with the residual solvent peak as the internal reference ($CDCl_3$ = 7.26 ppm, d_6-DMSO = 2.50 ppm). ^{1}H resonances are reported to the nearest 0.01 ppm. ^{13}C-NMR spectra were recorded on the same spectrometer with the central resonance of the solvent peak as the internal reference ($CDCl_3$ = 77.16 ppm, d_6-DMSO = 39.52 ppm). All ^{13}C resonances are reported to the nearest 0.01 ppm. DEPT 135, COSY, HMQC, and HMBC experiments were used to aid structural determination and spectral assignment. The multiplicity of ^{1}H signals are indicated as: s = singlet, d = doublet, dd = doublet of doublet, ddd = doublet of doublet of doublet, t = triplet, q = quadruplet, sext = sextet, m = multiplet, br. = broad, or combinations of thereof. Coupling constants (*J*) are quoted in Hz and reported to the nearest 0.1 Hz. Where appropriate, averages of the signals from peaks displaying multiplicity were used to calculate the value of the coupling constant. Infrared spectra were recorded neat on a PerkinElmer Spectrum One FT-IR spectrometer using Universal ATR sampling accessories. Unless stated otherwise, reagents were obtained from commercial sources and used without purification. The removal of solvent under reduced pressure was carried out on a standard rotary evaporator. Melting points were performed on either a Stanford Research Systems MPA100 (OptiMelt) automated melting point system and are uncorrected. High resolution mass spectrometry (HRMS) was performed using a Waters Micromass LCT Premier™ spectrometer using time of flight with positive ESI, or conducted by Mr Paul Skelton (Department of Chemistry, University of Cambridge) on a Bruker BioApex 47e FTICR spectrometer using (positive or negative) ESI or EI at 70 ev to within a tolerance of 5 ppm of the theoretically calculated value. LC-MS analysis was performed on an Agilent HP 1100 series chromatography (Mercury Luna 3u C18 (2) column) attached to a Waters ZQ2000 mass spectrometer with ESCi ionization source in ESI mode. Elution was carried out at a flow rate of 0.6 mL min^{-1} using a reverse phase gradient of acetonitrile and water containing 0.1% formic acid. Retention time (Rt) is given in min to the nearest 0.1 min and the *m/z* value is reported to the nearest mass unit (m.u.). Unless otherwise specified all the flow reactions were performed using a Vapourtec R2/R4+ flow platform.[1]

General procedure for the reduction of aldehydes and ketones under flow conditions.



Scheme 1. Flow protocol for the MPV reduction of compounds **1-35**.

A solution of the carbonyl compound (2 mmol)* in isopropanol (IPA)[2] (8 mL) was pumped through a glass column (Omnifit®, 6.6 mm i.d. × 100.0 mm length) packed with hydrous zirconium oxide (1.6 g, void volume 1.5 mL) (**Scheme 1**). The temperature and residence time (flow rate) were dependent on the nature of the substrate. A 100 psi back pressure regulator was placed after the reactor. The solution obtained was concentrated *in vacuo* to obtain the product in good to excellent yield. Unless otherwise stated, purification of compounds has been carried out using a polymer supported hydrazine (PS-TsNHNH$_2$).[3]

*For compounds **29**, **30** and **32** the solution in IPA was 0.025M due to the insoluble nature of the starting materials under the optimized conditions.

Calcination of zirconium hydroxide.

The calcination of zirconium hydroxide[4] was carried out using a commercially available Buchi TO-51 (**Figure 1**).[5] The catalyst was obtained via calcination at 270 °C for 10 h, under atmospheric pressure.

Figure 1. Commercially available Buchi TO-51 used for calcinating zirconium hydroxide.

Characterization of compounds **1-35**.

Benzyl alcohol (1). Flow rate = 125 µLmin^{-1}, residence time (τ) = 12 min, temperature = 60 °C; colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.38 (m, 4H), 7.32 (m, 1H), 4.68 (s, 2H), 2.21 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 140.90 (C), 128.55 (CH), 127.59 (CH), 127.00 (CH), 65.26 (CH_2); FT-IR (neat): 3327, 3029, 2872, 1606, 1496, 1453, 1368, 1206, 1035, 1009, 732, 689 cm^{-1}; LC-MS: retention time 0.29 min, *m/z* [M+H] = 109.01; HRMS (ESI): *m/z* calcd. for $C_7H_8ONa^+$: 131.0467; found 131.0466.

4-Chlorobenzyl alcohol (2). Flow rate = 165 µLmin^{-1}, residence time (τ) = 8 min, temperature = 60 °C; yellowish solid, m.p. 70-73 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.31 (d, 2H, *J* = 8.4 Hz), 7.26 (d, 2H, *J* = 8.4 Hz), 4.62 (s, 2H), 2.20 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 139.24 (C), 133.31 (C), 128.64 (CH), 128.25 (CH), 64.43 (CH_2); FT-IR (neat): 3335, 2924, 1597, 1578, 1490, 1404, 1354, 1207, 1085, 1009, 830, 797 cm^{-1}; LC-MS: retention time 3.85 min, *m/z* [M+H]^{dehydrated} = 125.01; HRMS (ESI): *m/z* calcd. for $C_7H_6ClO^-$: 141.0113; found 141.0115.

Methyl 4-(hydroxymethyl)benzoate (3). Flow rate = 165 µLmin^{-1}, residence time (τ) = 8 min, temperature = 60 °C; white solid, m.p. 49-51 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 8.01 (d, 2H, *J* = 8.0 Hz), 7.42 (d, 2H, *J* = 8.0 Hz), 4.76 (s, 2H), 3.91 (s, 3H), 2.08 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 166.97 (C), 146.00 (C), 129.82 (CH), 129.29 (C), 126.44 (CH), 64.65 (CH_2), 64.43 (CH_3); FT-IR (neat): 3301, 3212, 2959, 2861, 1719, 1612, 1572, 1502, 1432, 1358, 1311, 1278, 1176, 1191, 1107, 1045, 1011, 953, 838, 750 cm^{-1}; LC-MS: retention time 4.55 min, *m/z* [M+H] ^{dehydrated} = 149.15; HRMS (ESI): *m/z* calcd. for $C_9H_9O_3^-$: 165.0557; found 165.0558.

4-Benzyloxy-benzyl alcohol (4). Flow rate = 100 µLmin^{-1}, residence time (τ) = 15 min, temperature = 60 °C; white solid, m.p. 80-82 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.46-7.32 (m, 5H), 7.28 (d, 2H, *J* = 8.7 Hz), 6.98 (d, 2H, *J* = 8.7 Hz), 5.07 (s, 2H), 4.58 (s, 2H), 2.21 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 158.37 (C), 137.00 (C), 133.49 (C), 128.63 (CH), 128.61 (CH), 127.47 (CH), 114.93 (CH), 70.06 (CH_2), 64.85 (CH_2); FT-IR (neat): 3334, 2979, 2867, 1608, 1584, 1509, 1454, 1381, 1297, 1237, 1171, 1110, 996, 811, 738, 694 cm^{-1}; LC-MS: retention time 4.50 min, *m/z* [M+H]^{dehydrated} = 197.15; HRMS (ESI): *m/z* calcd. for $C_{14}H_{13}O_2^-$: 213.0921; found 213.0920.

4-(Imidazol-1yl)benzyl alcohol (5). Flow rate = 125 µLmin^{-1}, residence time (τ) = 12 min, temperature = 60 °C; white solid, m.p. 128-131 °C; ^{1}H-NMR (400 MHz, DMSO-*d6*, 25 °C): δ= 8.29 (br. s, 1H), 7.77 (br. s, 1H), 7.60 (d, 2H, *J* = 8.5 Hz), 7.47 (d, 2H, *J* = 8.5 Hz), 7.16 (br. s, 1H), 5.31 (t, 1H, *J* = 5.7 Hz), 4.54 (d, 2H, *J* = 5.7 Hz); ^{1}H-NMR (400 MHz, $CDCl_3$, 120 °C): δ= 8.06 (s, 1H), 7.56 (s, 1H), 7.52 (d, 2H, *J* = 8.5 Hz), 7.45 (d, 2H, *J* = 8.5 Hz), 7.09 (s, 1H), 4.78 (s, 1H), 4.57 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$, 120 °C): δ= 141.01 (C), 135.26 (CH), 135.01

(C), 129.22 (CH), 127.23 (CH), 119.89 (CH), 117.55 (CH), 62.00 (CH_2); FT-IR (neat): 3135, 2888, 2737, 1609, 1585, 1524, 1491, 1454, 1304, 1248, 1059, 1031, 1016, 917, 859, 806, 734 cm^{-1}; LC-MS: retention time 4.20 min, m/z [M+H] = 175.14; HRMS (ESI): *m/z* calcd. for $C_{10}H_{11}N_2O^+$: 175.0871; found 175.0870.

1,2-Benzenedimethanol (6). Flow rate = 125 µLmin^{-1}, residence time (τ) = 12 min, temperature = 60 °C; white solid, m.p. 62-65 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.26 (s, 4H), 4.55 (s, 4H), 4,32 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 139.58 (C), 128.09 (CH), 127.20 (CH), 63.35 (CH_2); FT-IR (neat): 3325, 2878, 1619, 1453, 1366, 1290, 1219, 1184, 1106, 998, 911, 733 cm^{-1}; LC-MS: retention time 4.44 min, *m/z* $[M+H]^{dehydrated}$ = 119.03; HRMS (ESI): *m/z* calcd. for $C_8H_9O_2^-$: 137.0608; found 137.0609.

2-Nitrobenzyl alcohol (7). Flow rate = 250 µLmin^{-1}, residence time (τ) = 6 min, temperature = 60 °C; yellowish solid, m.p. 68-71 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 8.11 (d, 1H, *J* = 8.4 Hz), 7.72 (d, 1H, *J* = 8.4 Hz), 7.68 (t, 1H, *J* = 8.4 Hz), 7.48 (t, 1H, *J* = 8.4 Hz), 4.98 (s, 2H), 2.48 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 148.84 (C), 136.74 (C), 134.12 (CH), 130.02 (CH), 128.51 (CH), 125.02 (CH), 62.58 (CH_2); FT-IR (neat): 3311, 3112, 2936, 2849, 1612, 1574, 1516, 1447, 1434, 1366, 1336, 1304, 1251, 1188, 1084, 1050, 1036, 859, 791, 724 cm^{-1}; LC-MS: retention time 2.11 min, *m/z* $[M+H]^{dehydrated}$ = 136.08; HRMS (ESI): *m/z* calcd. for $C_7H_6O_3N^-$: 152.0353; found 152.0352.

2-Methyl benzyl alcohol (8). Flow rate = 100 µLmin^{-1}, residence time (τ) = 15 min, temperature = 60 °C; colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.34 (m, 1H), 7.21 (m, 3H), 4.67 (s, 2H), 2.36 (s, 3H), 1.98 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 138.72 (C), 136.07 (C), 130.29 (CH), 127.74 (CH), 126.03 (CH), 63.39 (CH_2), 18.61 (CH_3); FT-IR (neat): 3310, 3022, 2871, 1606, 1492, 1460, 1379, 1286, 1214, 1183, 1113, 1035, 1001, 738 cm^{-1}; LC-MS: retention time 0.39 min, *m/z* $[M+H]^{dehydrated}$ = 105.07; HRMS (ESI): *m/z* calcd. for $C_8H_9O^-$: 121.0659; found 121.0660.

2-Hydroxymethyl benzo[b]thiopene (9). Flow rate = 125 µLmin^{-1}, residence time (τ) = 12 min, temperature = 60 °C; white solid, m.p. 94-96 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.82 (d, 1H, *J* = 7.3 Hz), 7.73 (d, 1H, *J* = 7.3 Hz), 7.33 (m, 2H), 7.26 (s, 1H), 4.93 (d, 2H, *J* = 6.2 Hz), 2.09 (s, 1H, *J* = 6.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 144.77 (C), 139.95 (C), 139.54 (C), 124.33 (CH), 124.31 (CH), 123.53 (CH), 122.47 (CH), 121.48 (CH), 60.90 (CH_2); FT-IR (neat): 3298, 3057, 2919, 2861, 1540, 1457, 1435, 1364, 1329, 1235, 1135, 1126, 1008, 938, 838, 739, 720 cm^{-1}; LC-MS: retention time 4.16 min, *m/z* $[M+H]^{dehydrated}$ = 147.11; HRMS (ESI): *m/z* calcd. for $C_9H_7OS^-$: 163.0223; found 163.0226.

2-Hydroxymethylphenyl-diphenylphosphine (10). Flow rate = 100 µLmin^{-1}, residence time (τ) = 15 min, temperature = 60 °C; colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.81-6.92 (m, 14H), 4.88 (s, 2H), 2.92 (br. s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 144.77 (C),

139.95 (C), 139.54 (C), 124.33 (CH), 124.31 (CH), 123.53 (CH), 122.47 (CH), 121.48 (CH), 60.90 (CH_2); FT-IR (neat): 3336, 3056, 2863, 2841, 1602, 1580, 1495, 1452, 1437, 1359, 1291, 1231, 1111, 1038, 962, 841, 741 cm^{-1}; LC-MS: retention time 5.08 min, m/z $[M+H]^{dehydrated}$ = 275.20; HRMS (ESI): *m/z* calcd. for $C_{19}H_{18}OP^+$: 293.1090; found 293.1085.

(±)-1-(Phenyl)ethanol (11). Flow rate = 50 µLmin^{-1}, residence time (τ) = 30 min, temperature = 130 °C; colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.36 (m, 4H), 7.30 (m, 1H), 4.85 (q, 1H, J = 6.2 Hz), 3.25 (s, 1H), 3.13 (d, 3H, J = 6.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 146.06 (C), 128.44 (CH), 127.33 (CH), 125.51 (CH), 70.16 (CH), 25.21 (CH_3); FT-IR (neat): 3338,3061, 2973, 2928, 2880, 1601, 1493, 1450, 1368, 1283, 1203, 1075, 1009, 897, 758, 687 cm^1; LC-MS: retention time 2.16 min, *m/z* $[M+H]^{dehydrated}$ = 106.32; HRMS (ESI): *m/z* calcd. for $C_8H_9O^-$: 121.0659; found 121.0659.

(±)-1-(Phenyl)propanol (12). Flow rate = 50 µLmin^{-1}, residence time (τ) = 30 min, temperature = 130 °C; colourless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.36 (m, 4H), 7.30 (m, 1H),4.61 (t, 2H, J_1 = 6.6 Hz), 2.05 (s, 1H), 1.85 (sext, 1H, J_1 = J_2 = J_2 = 6.2 Hz), 1.78 (sext, 1H, J_1 = J_2 = J_3 = 6.2 Hz), 0.94 (t, 3H, J_3 = 6.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 144.62 (C), 128.39 (CH), 127.48 (CH), 125.99 (CH), 76.01 (CH), 31.89 (CH_2), 10.15 (CH_3); FT-IR (neat): 3376, 2964, 2933, 2876, 1603, 1492, 1452, 1328, 1269, 1200, 1095, 1044, 1011, 972, 916, 896, 834, 744, 697 cm^{-1}; LC-MS: retention time 3.98 min, *m/z* $[M+H]^{dehydrated}$ = 119.12; HRMS (ESI): *m/z* calcd. for $C_9H_{11}O^-$: 135.0815; found 135.0818.

(±)-1-(Furan-2yl)ethanol (13). Flow rate = 50 µLmin^{-1}, residence time (τ) = 30 min, temperature = 130 °C; yellowish oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.40 (d, 1H, J_1 = 1.8 Hz), 6.35 (dd, 1H, J_2 = 3.3 Hz, J_1 = 1.8 Hz), 6.25 (d, 1H, J_2 = 3.3 Hz), 4.90 (q, 1H, J_3 = 6.6 Hz), 1.90 (s, 1H), 1.57 (d, 3H, J_3 = 6.6 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 141.94 (CH), 138.70 (C), 110.14 (CH), 105.13 (CH), 63.65 (CH), 21.28 (CH_3); FT-IR (neat): 3403, 2979, 2935, 1505, 1449, 1373, 1328, 1287, 1230, 1149, 1077, 1009, 992, 927, 878, 810, 738 cm^{-1}; LC-MS: retention time 4.19 min, *m/z* $[M+H]^{dehydrated}$ = 113.16; HRMS (ESI): *m/z* calcd. for $C_6H_7O_2^-$: 111.0452; found 111.0452.

(±)-1-Tetralol (14). Flow rate = 25 µLmin^{-1}, residence time (τ) = 60 min, temperature = 130 °C; white solid, m.p. 38-40 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.44 (m, 1H), 7.20 (m, 2H), 7.11 (m, 1H), 4.79 (m, 1H), 2.83 (m, 1H), 2.75 (m, 1H), 2.10-1.93 (m, 3H), 1.80 (m, 1H), 1.69 (d, 1H, J = 6.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 138.80 (C), 129.00 (CH), 128.62 (CH), 127.57 (CH), 126.45 (C), 126.17 (CH), 68.16 (CH), 32.28 (CH_2), 29.23 (CH_2), 18.07 (CH_2); FT-IR (neat): 3328, 2924, 2842, 1581, 1495, 1452, 1437, 1361, 1290, 1230, 1111, 1038, 962, 741 cm^{-1}; LC-MS: retention time 4.04 min, *m/z* $[M+H]^{dehydrated}$ = 131.12; HRMS (ESI): *m/z* calcd. for $C_{10}H_{11}O^-$: 147.0815; found 147.0815.

(±)-1-(Naphthyl)ethanol (15). Flow rate = 50 μLmin^{-1}, residence time (τ) = 30 min, temperature = 130 °C; white solid, m.p. 75-79 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.88-781 (m, 4H), 7.53-7.50 (m, 3H), 5.04 (q, 1H, *J* = 6.6 Hz), 2.58 (br. s, 1H), 1.60 (d, 3H, *J* = 6.6 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 143.29 (C), 133.37 (CH), 132.94 (C), 128.30 (C), 127.99 (CH), 127.72 (CH), 126.15 (CH), 125.80 (CH), 123.85 (CH), 70.45 (CH), 25.01 (CH_3); FT-IR (neat): 3302, 2923, 1600, 1506, 1453, 1409, 1362, 1322, 1275, 1166, 1123, 1072, 1024, 901, 861, 823, 741 cm^{-1}; LC-MS: retention time 4.37 min, *m/z* $[M+H]^{dehydrated}$ = 155.03; HRMS (ESI): *m/z* calcd. for $C_{12}H_{11}O^-$: 171.0815; found 171.0816.

(±)-α-(4-Chlorophenyl)-benzyl alcohol (16). Flow rate = 60 μLmin^{-1}, residence time (τ) = 25 min, temperature = 130 °C; white solid, m.p. 57-59 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.38-7.28 (m, 9H), 5.71 (d, 1H, *J* = 2.6 Hz), 2.90 (d, 1H, *J* = 2.6 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 143.44 (C), 142.26 (C), 133.24 (C), 128.65 (CH), 128.44 (CH), 127.03 (CH), 126.82 (CH), 126.58 (CH), 75.54 (CH); FT-IR (neat): 3356, 3062, 3029, 2886, 1597, 1488, 1452, 1402, 1375, 1317, 1285, 1229, 1217, 1184, 1088, 1034, 1012, 918, 845, 792, 753, 677 cm^{1}; LC-MS: retention time 4.78 min, *m/z* $[M+H]^{dehydrated}$ = 201.11; HRMS (ESI): *m/z* calcd. for $C_{13}H_{10}OCl^-$: 217.0426; found 217.0426.

(±)-1-(Thiazol-yl)ethanol (17). Flow rate = 43 μLmin^{-1}, residence time (τ) = 35 min, temperature = 130 °C; yellowish oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.71 (d, 1H, *J* = 8.0 Hz), 7.20 (d, 1H, *J* = 8.0 Hz), 5.18 (q, 1H, *J* = 6.6 Hz), 3.42 (br. s, 1H), 1.65 (d, 3H, *J* = 6.6 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 176.10 (C), 142.02 (CH), 118.95 (CH), 67.97 (CH), 24.09 (CH_3); FT-IR (neat): 3220, 2971, 2869, 1504, 1480, 1432, 1366, 1229, 1217, 1145, 1106, 1056, 1003, 936, 896, 766, 728 cm^{1}; LC-MS: retention time 0.31 min, *m/z* $[M+H]^{dehydrated}$ = 111.96; HRMS (ESI): *m/z* calcd. for $C_5H_6ONS^-$: 128.0176; found 128.0178.

(±)-1-(4-Iodophenyl)ethanol (18). Flow rate = 67 μLmin^{-1}, residence time (τ) = 25 min, temperature = 130 °C; yellow oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.69 (d, 2H, *J* = 8.4 Hz), 7.13 (d, 2H, *J* = 8.4 Hz), 4.86 (m, 1H), 1.75 (s, 1H), 1.47 (d, 3H, *J* = 6.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 145.45 (C), 137.53 (CH), 127.40 (CH), 92.69 (C), 69.84 (CH), 25.22 (CH_3); FT-IR (neat): 3337, 2971, 2873, 1588, 1484, 1449, 1396, 1368, 1295, 1201, 1109, 1083, 1004, 895, 817, 767 cm^{-1}; LC-MS: retention time 4.39 min, *m/z* $[M+H]^{dehydrated}$ = 230.97; HRMS (ESI): *m/z* calcd. for $C_8H_8OI^+$: 246.9614; found 246.9606.

(±)-1-(4-Bromophenyl)ethanol (19). Flow rate = 67 μLmin^{-1}, residence time (τ) = 25 min, temperature = 130 °C; yellowish solid, m.p. 39-42 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.50 (d, 2H, *J* = 8.4 Hz), 7.28 (d, 2H, *J* = 8.4 Hz), 4.86 (m, 1H), 1.75 (s, 1H), 1.50 (d, 1H, *J* = 6.6 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 144.77 (C), 131.00 (C), 127.14 (CH), 121.17 (CH), 69.81 (CH), 25.27 (CH_3); FT-IR (neat): 3333, 2971, 2927, 1592, 1488, 1447, 1401, 1366, 1229, 1217, 1204, 1111, 1069, 1008, 896, 821, 770, 715 cm^{1}; LC-MS: retention time 4.26 min, *m/z* $[M+H]^{dehydrated}$ = 183.05; HRMS (ESI): *m/z* calcd. for $C_8H_8OBr^-$: 198.9764; found 198.9759.

(±)-1-(4-Fluorophenyl)ethanol (20). Flow rate = 60 µLmin^{-1}, residence time (τ) = 25 min, temperature = 130 °C; yellowish oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.34 (m, 2H), 7.00 (m, 2H), 4.87 (m, 1H), 2.02 (s, 1H), 1.47 (d, 3H, J = 6.6 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 162.10 (C_F), 141.55 (C), 127.06 (CH), 115.11 (CH), 69.73 (CH), 25.27 (CH_3); FT-IR (neat): 3177, 2894, 2849, 1464, 1447, 1374, 1360, 1101, 1088, 1056, 1022, 978, 959, 938 cm^{1}; LC-MS: retention time 3.65 min, *m/z* $[M+H]^{dehydrated}$ = 123.04; HRMS (ESI): *m/z* calcd. for $C_8H_8OF^-$: 139.0565; found 139.0567.

(±)-1-(Pyrid-3yl)ethanol (21). Flow rate = 60 µLmin^{-1}, residence time (τ) = 25 min, temperature = 130 °C; colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 8.58 (br. s, 1H), 8.50 (br. s, 1H), 7.77 (m, 1H), 7.32 (m, 1H), 4.97 (q, 1H, J = 6.3 Hz), 2.59 (br. s, 1H), 1.54 (d, 3H, J = 6.3 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 147.76 (CH), 146.89 (CH), 142.00 (CH), 133.59 (CH), 123.55 (C), 67.28 (CH), 25.16 (CH_3); FT-IR (neat): 3321, 2973, 2868, 1594, 1580, 1479, 1424, 1369, 1314, 1190, 1088, 1009, 900, 809 cm^{-1}; LC-MS: retention time 0.29 min, m/z [M+H] = 124.04; HRMS (ESI): *m/z* calcd. for $C_7H_{10}ON^+$: 124.0757; found 124.0755.

(±)-1-(2,6-Dimethoxyphenyl)ethanol (22). Flow rate = 33 µLmin^{-1}, residence time (τ) = 45 min, temperature = 130 °C; colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.16 (t, 1H, J = 8.2 Hz), 6.58 (d, 2H, J = 8.2 Hz), 5.33 (m, 1H), 3.88 (br. s, 1H), 3.85 (s, 6H), 1.50 (d, 1H, J = 6.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 157.44 (C), 128.05 (C), 121.01 (CH), 104.32 (CH), 64.01 (CH), 55.69 (CH_3), 23.58 (CH_3); FT-IR (neat): 3552, 2965, 2840, 1595, 1475, 1443, 1409, 1234, 1106, 1062, 1007, 891, 783, 730 cm^{1}; LC-MS: retention time 4.11 min, *m/z* $[M+H]^{dehydrated}$ = 165.13; HRMS (ESI): *m/z* calcd. for $C_{10}H_{15}O_3^+$: 183.1023; found 183.1024.

***N*-Boc-piperidin-4-ol (23).** Flow rate = 38 µLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; yellowish oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 3.85-3.72 (m, 3H), 2.98 (ddd, 2H, J_1 =J_2 = 6.3 Hz, J_3 = 2.1 Hz), 2.43 (br. s, 1H), 1.80 (m, 2H), 1.45 (m, 2H), 1.42 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 154.84 (C), 79.55 (CH), 70.19 (C), 67.50 (CH_2), 34.08 (CH_2), 28.39 (CH_3); FT-IR (neat): 3385, 2971, 2936, 2857, 1664, 1473, 1422, 1365, 1271, 1231, 1189, 1130, 1067, 1026, 975, 863, 810, 768, 731, 700 cm^{1}; LC-MS: retention time 3.88 min, *m/z* $[M+H]^{dehydrated}$ = 202.06; HRMS (ESI): *m/z* calcd. for $C_{12}H_{18}NO^+$: 202.1443; found 202.1445.

***N*-Benzyl-piperidin-4-ol (24).** Flow rate = 38 µLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; yellowish oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.41-7.34 (m, 5H), 3.81 (br. s, 1H), 3.66 (s, 2H), 2.86 (br. s, 2H), 2.39 (br. s, 2H), br. s, 2H), 1.82 (br. s, 1H), 1.69 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 136.47 (C), 129.57 (CH), 128.37 (CH), 127.58 (CH), 62.48 (CH), 60.40 (CH_2),50.47 (CH_2), 33.57 (CH_2); FT-IR (neat): 3341, 3030, 2938, 2800, 1495, 1453, 1362, 1336, 1282, 1138, 1061, 978, 953, 910, 737, 698 cm^{-1}; LC-MS: retention time 0.28 min, m/z [M+H] = 192.22; HRMS (ESI): *m/z* calcd. for $C_{12}H_{18}NO^+$: 192.1388; found 192.1388.

(±)-2-Tetralol (25). Flow rate = 38 µLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; yellowish oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 7.19-7.11 (m, 4H), 4.17 (br. s, 1H), 3.12 (dd, 1H, J = 16.5 Hz, J = 5.9 Hz), 3.05 (t, 1H, J = 5.9 Hz), 2.98 (t, 1H, J = 5.9 Hz), 2.90 (m, 1H), 2.80 (m, 1H), 2.65 (s, 1H), 2.08 (m, 1H), 1.85 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 135.76 (C), 134.43 (C), 129.55 (CH), 128.64 (CH), 125.93 (CH), 125.89 (CH), 67.18 (CH), 38.39 (CH_2), 31.52 (CH_2), 27.13 (CH_2); FT-IR (neat): 3328, 3062, 2926, 2841, 1602, 1580, 1495, 1452, 1437, 1359, 1291, 1231, 1111, 1038, 962, 841, 741 cm^{-1}; LC-MS: retention time 4.02 min, *m/z* $[M+H]^{dehydrated}$ = 131.14; HRMS (ESI): *m/z* calcd. for $C_{10}H_{11}O^-$: 147.0815; found 147.0818.

(±)-3-(3,4-Dimethoxyphenyl)-propan-2ol (26). Flow rate = 38 µLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; yellowish oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 6.84 (d, 1H, J = 7.7 Hz), 6.76 (m, 2H), 4.01 (m, 1H), 3.89 (s, 3H), 3.87 (s, 3H), 2.75 (dd, 1H, J = 13.5 Hz, J = 4.4 Hz), 2.64 (dd, 1H, J = 13.5 Hz, J = 8.0 Hz), 1.72 (br. s, 1H), 1.26 (d, 1H, J = 6.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 148.97 (C), 147.23 (CH), 131.04 (CH), 121.31 (CH), 112.57 (C), 111.39 (CH), 68.88 (CH), 55.93 (CH_2), 45.35 (CH_2), 22.73 (CH_2); FT-IR (neat): 3470, 2967, 2933, 2835, 1607, 1590, 1514, 1463, 1417, 1317, 1330, 1259, 1234, 1155, 1139, 1078, 1026, 946, 802 cm^{1}; LC-MS: retention time 3.77 min, *m/z* $[M+H]^{dehydrated}$ = 179.17; HRMS (ESI): *m/z* calcd. for $C_{11}H_{17}O_3^+$: 197.1178; found 197.1179.

Cyclopentanol (27). Flow rate = 38 µLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; colourless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 4.34 (br. s, 1H), 2.17 (s, 1H), 1.77 (m, 4H), 1.57 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 138.80 (CH), 35.61 (CH_2), 23.67 (CH_2); FT-IR (neat): 3346, 2961, 2871, 1454, 1437, 1340, 1307, 1171, 1033, 995, 894, 835 cm^{1}; LC-MS: retention time 0.20 min, *m/z* $[M+H]^{dehydrated/combined\ with\ acetonitrile}$ = 110.85.

(±)Pentan-1,4-diol (28). Flow rate = 38 µLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; colourless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 4.14 (br. s, 1H), 3.98 (br. s, 1H), 3.75 (br. s, 1H), 3.55 (m, 2H), 1.65-1.40 (m, 4H), 1.11 (d, 3H, J = 6.3 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 67.61 (CH), 62.42 (CH_2), 36.09 (CH_2), 28.97 (CH_2), 23.31 (CH_2); FT-IR (neat): 3177, 2894, 2849, 1464, 1447, 1374, 1360, 1101, 1088, 1056, 1022, 978, 959, 938 cm^{1}; LC-MS: retention time 0.35 min, *m/z* $[M+H]^{dehydrated/combined\ with\ acetonitrile}$ = 129.10; HRMS (ESI): *m/z* calcd. for $C_5H_{13}O_2^+$: 105.0915; found 105.0912.

Adamantan-2-ol (29). Flow rate = 33 µLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; white solid, mp 257-260°C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): δ= 3.83 (s, 1H), 2.08 (d, 2H), 7.11 (m, 1H), 4.79 (m, 1H), 2.83 (m, 1H), 2.75 (m, 1H), 2.10-1.93 (m, 3H), 1.80 (m, 1H), 1.69 (d, 1H, J = 6.2 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): δ= 74.52 (CH), 37.59 (CH_2), 36.52 (CH_2), 34.55 (CH), 31.02 (CH_2), 27.53 (CH), 27.07 (CH); FT-IR (neat): 3177, 2894, 2849, 1464, 1447, 1374, 1360, 1101, 1088, 1056, 1022, 978, 959, 938 cm^{1}; LC-MS: retention

time 4.39 min, *m/z* $[M+H]^{dehydrated}$ = 135.21; HRMS (ESI): *m/z* calcd. for $C_{10}H_{15}O^-$: 151.1128; found 151.1130.

(±)Dihydrocholesterol (30). Flow rate = 20 μLmin^{-1}, residence time (τ) = 75 min, temperature = 130 °C; white solid**,** mp 257-260°C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): *δ*= 3.60 (br. s, 1H), 2.00 (m, 1H), 1.80 (m, 2H), 1.71 (m, 2H), 1.59-1.40 (m, 6H), 1.38-1.25 (m, 12H), 1.18-0.89 (m, 22H), 0.82 (s, 3H), -.67 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): *δ*= 56.49 (C), 56.31 (CH), 54.37 (CH), 44.86 (CH_2), 42.59 (CH_2), 40.05 (CH_2), 39.51 (CH_2), 38.18 (CH_2), 37.02 (CH_2), 36.18 (CH_2), 35.79 (CH_2), 35.45 (CH_2), 32.08 (CH_2), 31.49 (CH_2), 28.74 (CH_2), 28.24 (CH_2), 27.99 (CH_2), 24.21 (CH_2), 23.85 (CH), 22.81 (CH_2), 22.56 (CH_2), 21.26 (CH_2), 18.67 (CH_3), 12.31 (CH_3), 12.06 (CH_3); FT-IR (neat): 3396, 2929, 2866, 2848, 1467, 1444, 1381, 1170, 1133, 1077, 1043, 956 cm^1; LC-MS: retention time 5.79 min, *m/z* $[M+H]^{dehydrated}$ = 371.12; HRMS (ESI): *m/z* calcd. for $C_{27}H_{49}O^+$: 389.3702; found 389.3705.

Isoborneol/borneol (31).[6] Flow rate = 20 μLmin^{-1}, residence time = 75 min, temperature = 130 °C.

(±)6-Methyl -2,3,4,9-tetrahydro-1*H*-carbazol-1-ol (32). Flow rate = 33 μLmin^{-1}, residence time (τ) = 45 min, temperature = 130 °C; white solid**,** mp 123-125 °C; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): *δ*= 8.01 (br. s, 1H), 7.29 (s, 1H), 7.20 (d, 1H, *J* = 8.0 Hz), 7.01 (d, 1H, *J* = 8.0 Hz), 4.88 (m, 1H), 2.72 (m, 1H), 2.65 (m, 1H), 2.45 (s, 3H), 2.15 (m, 1H), 2.01 (m, 1H), 1.88 (m, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): *δ*= 135.10 (C), 134.51 (C), 128.33 (CH), 127.18 (C), 123.84 (C), 118.54 (CH), 111.89 (C), 110.69 (CH), 64.52 (CH), 33.63 (CH_2), 21.44 (CH_2), 20.95 (CH_2), 20.32 (CH_3); FT-IR (neat): 3389, 2930, 1853, 1454, 1436, 1409, 1313, 1295, 1251, 1238, 1121, 1061, 986, 932, 907, 797 cm^1; LC-MS: retention time 4.55 min, *m/z* $[M+H]^{dehydrated}$ = 184.24; HRMS (ESI): *m/z* calcd. for $C_{13}H_{16}NO^+$: 202.1232.; found 202.1225.

3-Phenyl propanol (33). Flow rate = 80 μLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): *δ*= 7.31-7.27 (m, 3H), 7.25-7.18 (m, 2H), 3.68 (t, 2H, *J* = 6.6 Hz), 2.72 (t, 1H, *J* = 6.6 Hz), 1.91 (m, 2H), 1.49 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): *δ*= 141.92 (C), 128.42 (CH), 128.39 (CH), 125.86 (CH), 62.25 (CH), 34.21 (CH_2), 32.07 (CH_2); FT-IR (neat): 3318, 3027, 2938, 2862, 1603, 1492, 1453, 1365, 1228, 1217, 1030, 915, 815, 743, 697 cm^1; LC-MS: retention time 3.96 min, *m/z* $[M+H]^{dehydrated}$ = 119.21; HRMS (ESI): *m/z* calcd. for $C_9H_{13}O_3^+$: 137.0917; found 137.0916.

(±)Citronellol (34). Flow rate = 80 μLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; colorless oil; ^{1}H-NMR (400 MHz, $CDCl_3$, 25 °C): *δ*= 5.10 (app. t, 1H, *J* = 7.3 Hz), 3.68 (m, 2H), 2.00 (m, 2H), 1.68 (s, 3H), 1.56 (m, 5H), 1.35-1.30 (m, 3H), 1.20 (m, 2H), 0.92 (d, 3H, *J* = 7.6 Hz); ^{13}C-NMR (100 MHz, $CDCl_3$, 25 °C): *δ*= 131.21 (C), 124.69 (CH), 61.13 (CH_2), 39.88 (CH), 37.19 (CH_2), 29.15 (CH), 25.67 (CH_2), 25.62 (CH_3), 19.29 (CH_3), 17.60 (CH_3); FT-IR (neat): 3323, 2960, 2916, 1452, 1376, 1216, 1056, 1009, 828, 741, 673 cm^1; LC-MS: retention

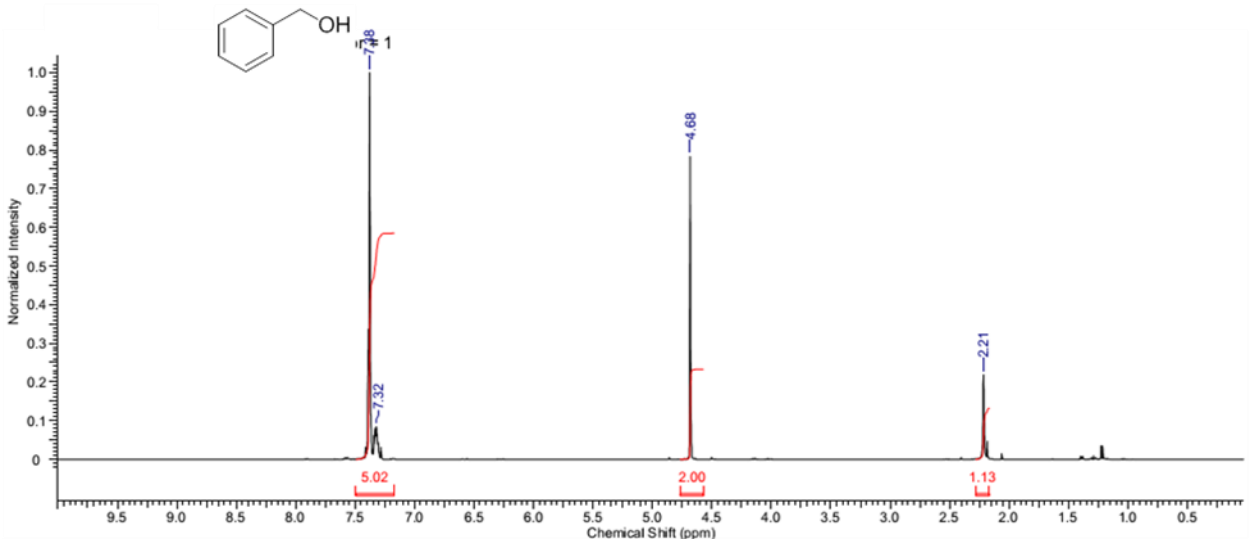
time 3.95 min, *m/z* $[M+H]^{dehydrated}$ = 140.30; HRMS (ESI): *m/z* calcd. for $C_{10}H_{19}O^-$: 155.1441; found 155.1445.

(*E*)-Cinnamyl alcohol (35). Flow rate = 68 µLmin^{-1}, residence time (τ) = 40 min, temperature = 130 °C; colorless oil; ^{1}H-NMR (400 MHz, DMSO-*d6*, 25 °C): *δ*= 7.44 (m, 1H), 7.20 (m, 2H), 7.11 (m, 1H), 4.79 (m, 1H), 2.83 (m, 1H), 2.75 (m, 1H), 2.10-1.93 (m, 3H), 1.80 (m, 1H), 1.69 (d, 1H, *J* = 6.2 Hz); ^{13}C-NMR (100 MHz, DMSO-*d6*, 25 °C): *δ*= 137.36 (C), 131.26 (CH), 129.04 (CH), 129.00 (CH), 127.63 (CH), 126.57 (CH), 61.99 (CH_2); FT-IR (neat): 3327, 3081, 3025, 2922, 2861, 1656, 1598, 1577, 1493, 1448, 1367, 1216, 1091, 1069, 1007, 964, 918, 732, 690 cm^1; LC-MS: retention time 3.95 min, *m/z* $[M+H]^{dehydrated}$ = 118.30; HRMS (ESI): *m/z* calcd. for $C_9H_9O^-$: 133.0659; found 133.0661.

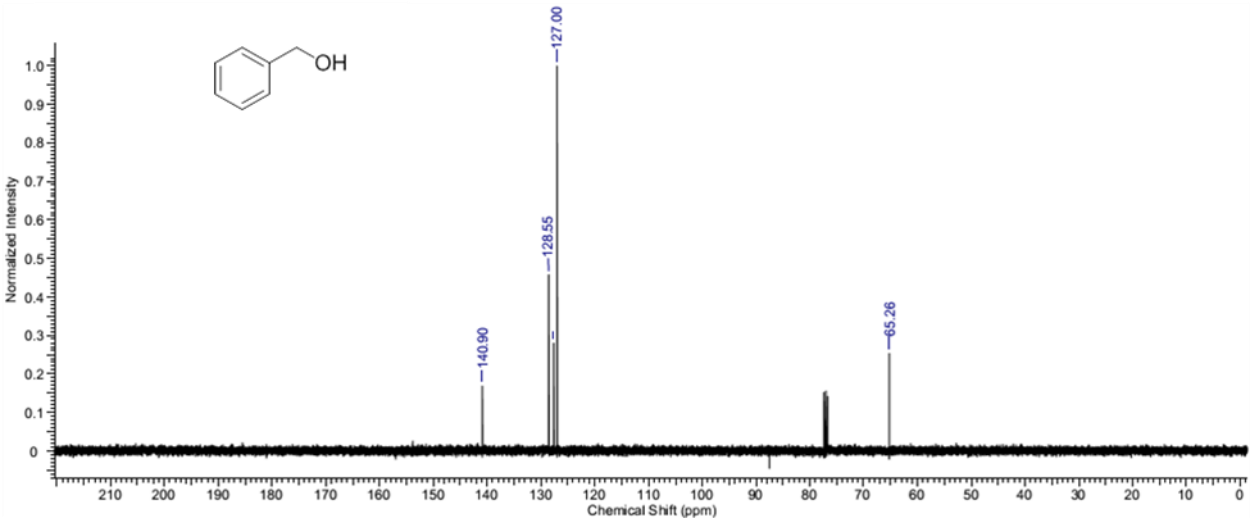
[1]H-NMR and [13]C-NMR spectra.

Benzyl alcohol (1).

[1]H-NMR (25 °C)

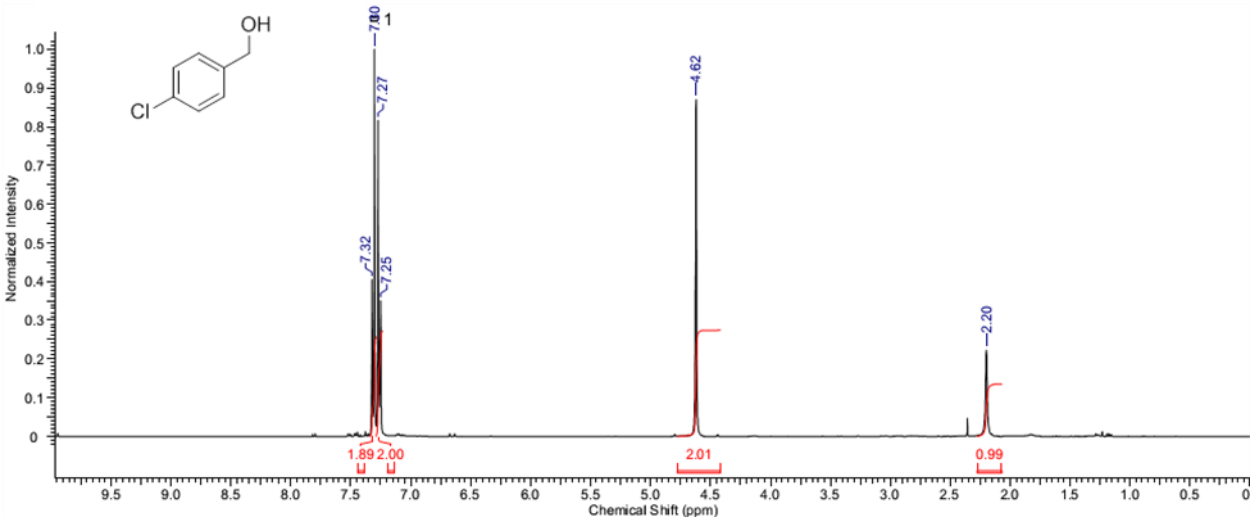


[13]C-NMR (25 °C)

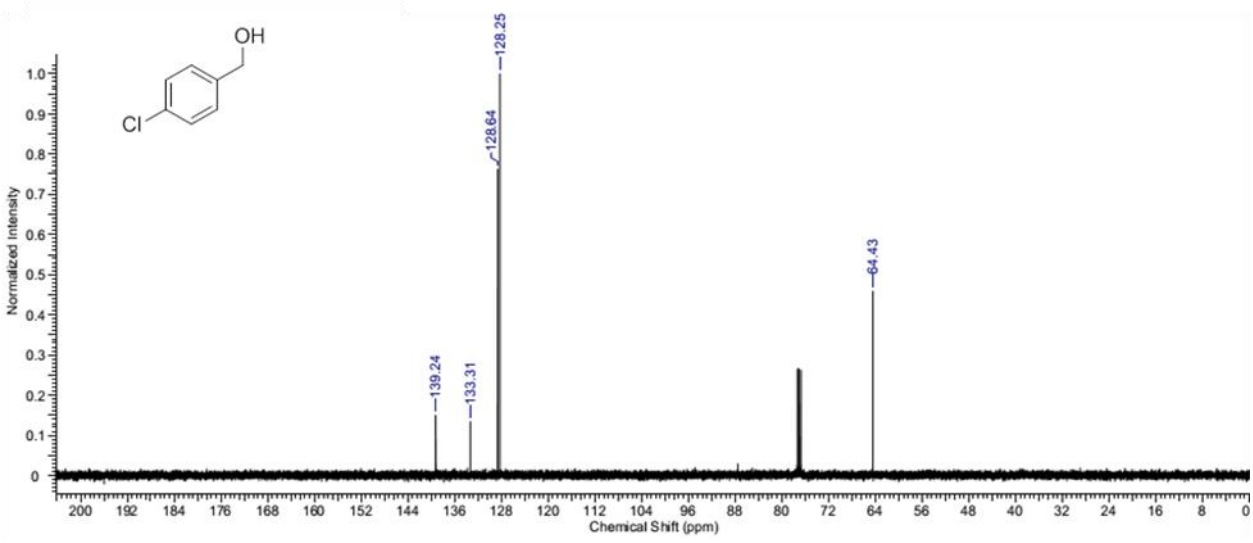


4-Chlorobenzyl alcohol (2).

^{1}H-NMR (25 °C)

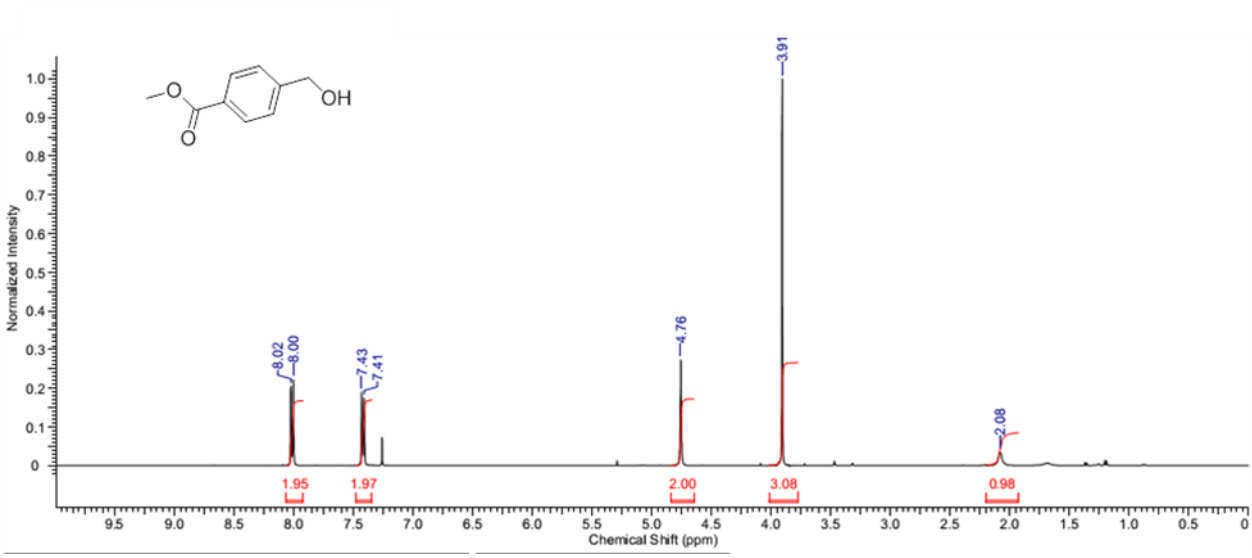


^{13}C-NMR (25 °C)

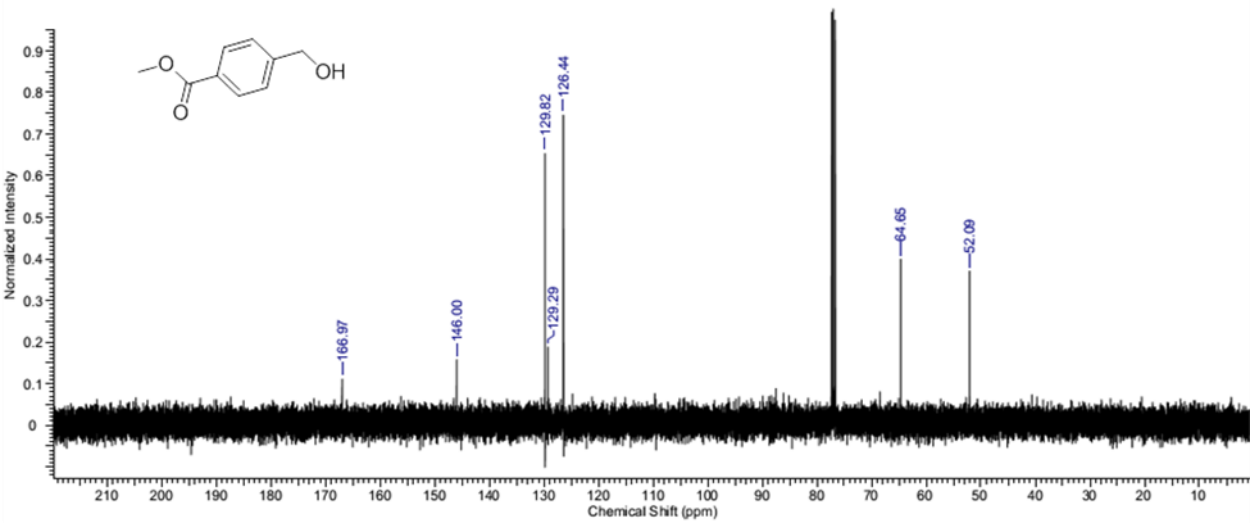


Methyl 4-(hydroxymethyl)benzoate (3).

^{1}H-NMR (25 °C)

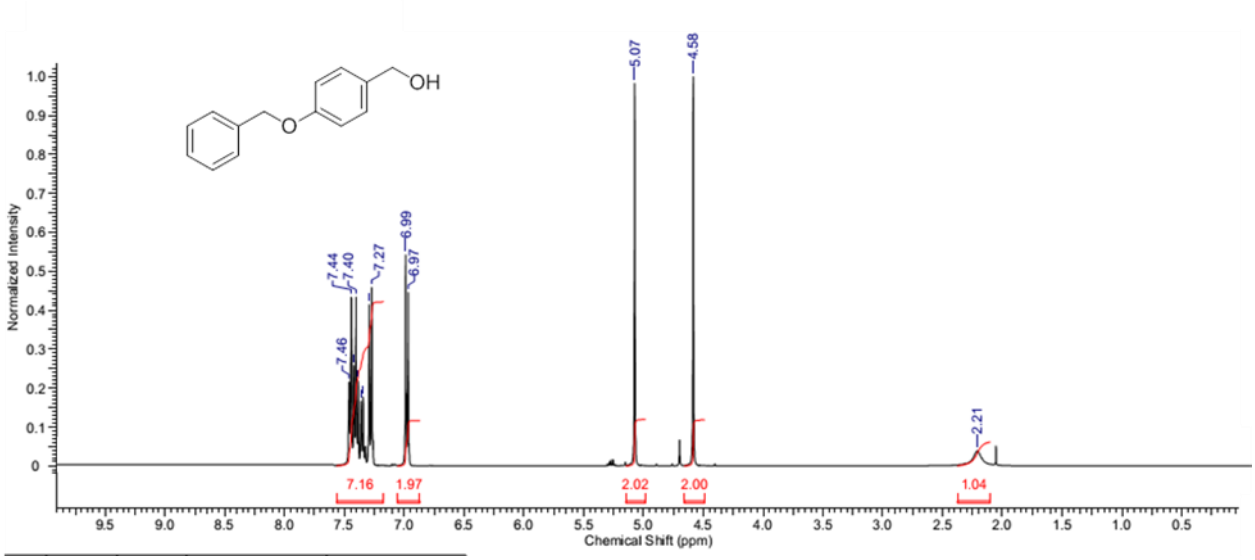


^{13}C-NMR (25 °C)

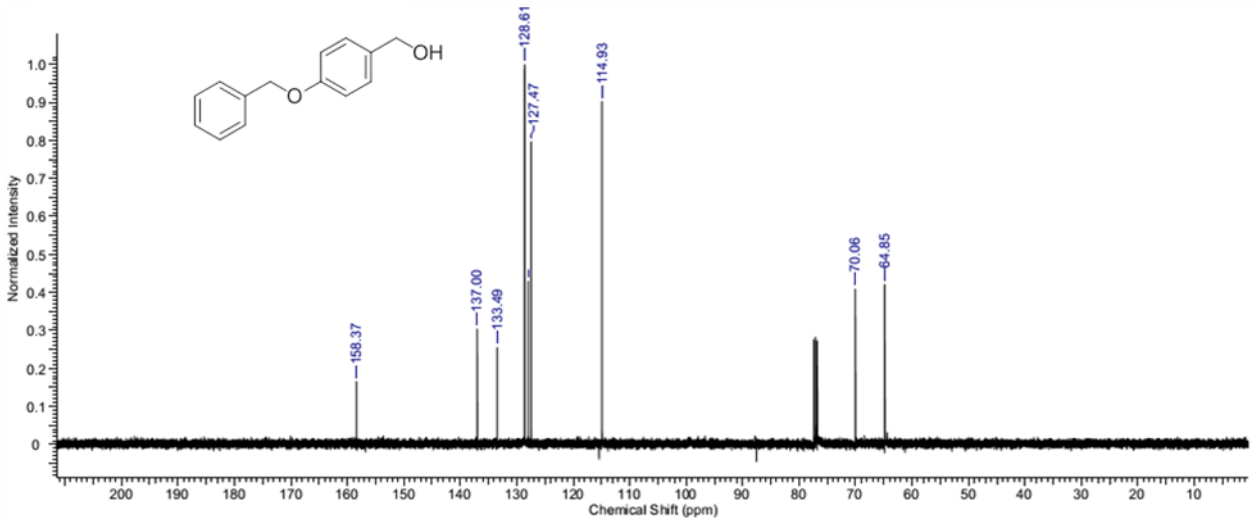


4-Benzyloxy-benzyl alcohol (4).

^{1}H-NMR (25 °C)

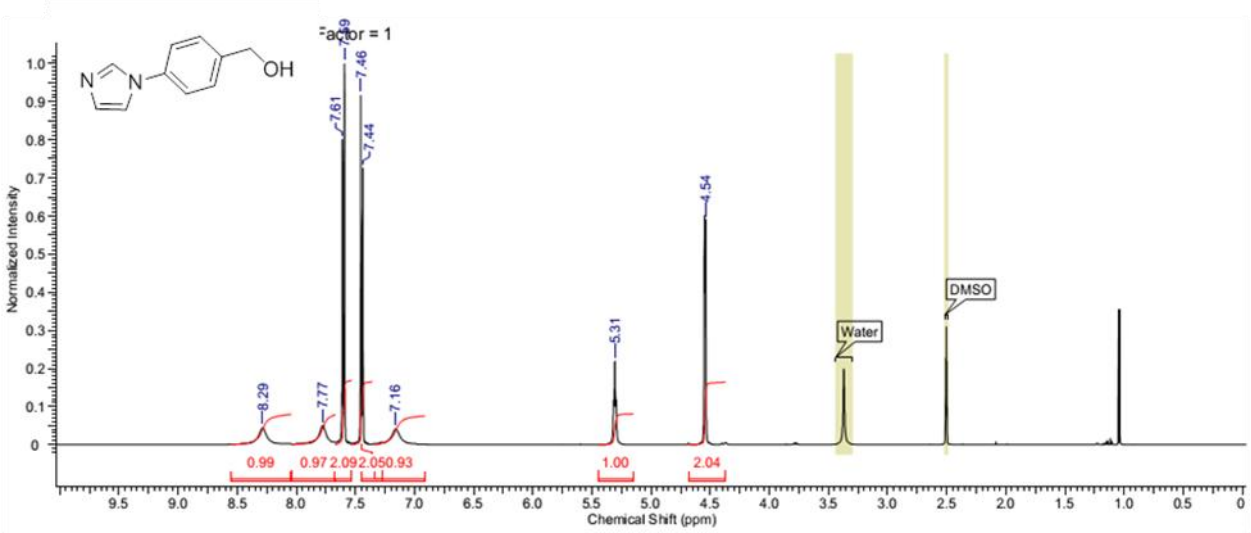


^{13}C-NMR (25 °C)

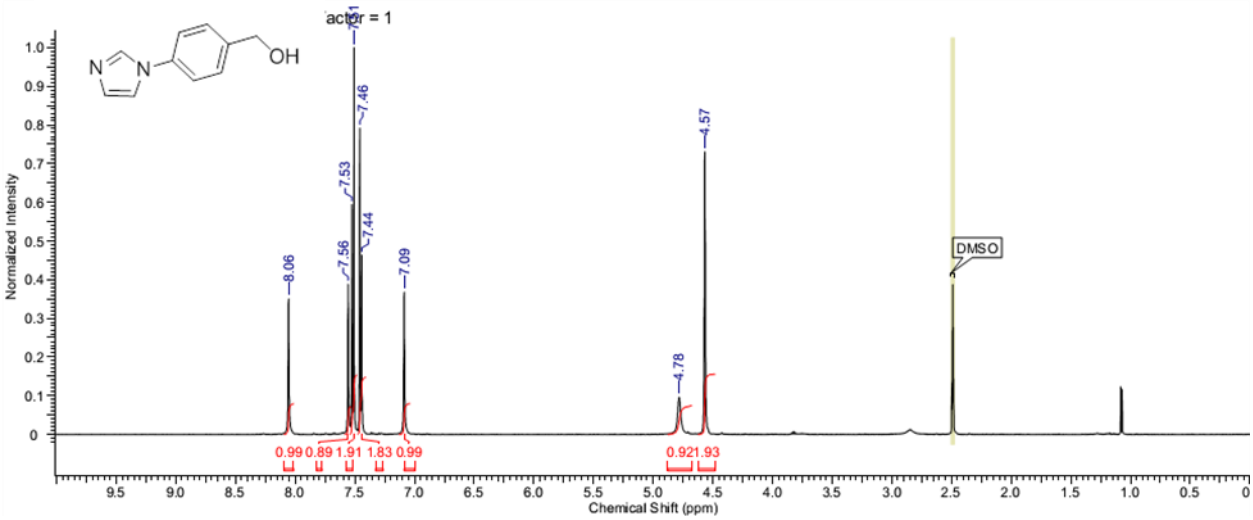


4-(Imidazol-1yl)benzyl alcohol (5).

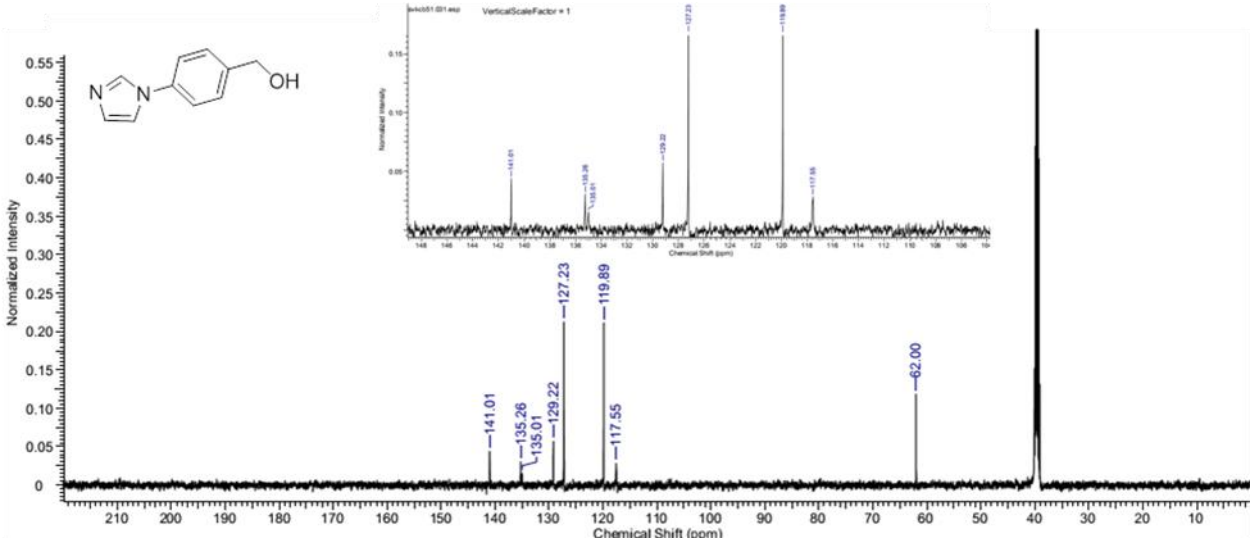
^{1}H-NMR (25 °C)



^{1}H-NMR (120 °C)



^{13}C-NMR (120 °C)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N
N
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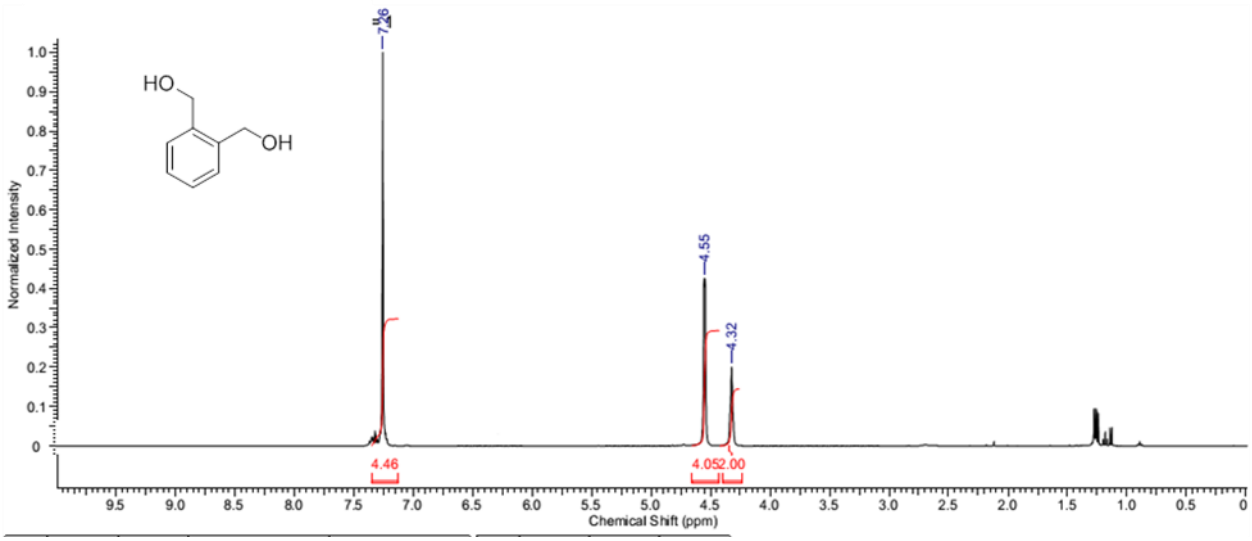
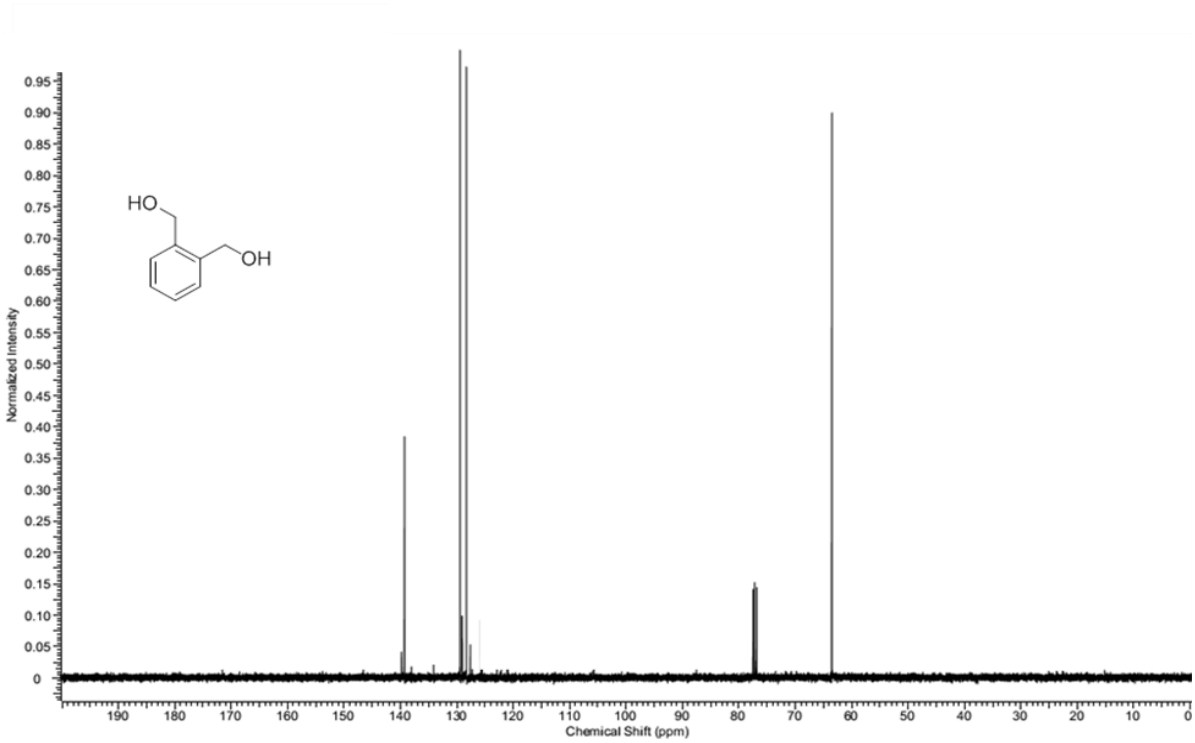
Normalized Intensity
Chemical Shift (ppm)
141.01
135.26
135.01
129.22
127.23
119.89
117.55
62.00

1,2-Benzenedimethanol (6).

^{1}H-NMR (25 ℃)

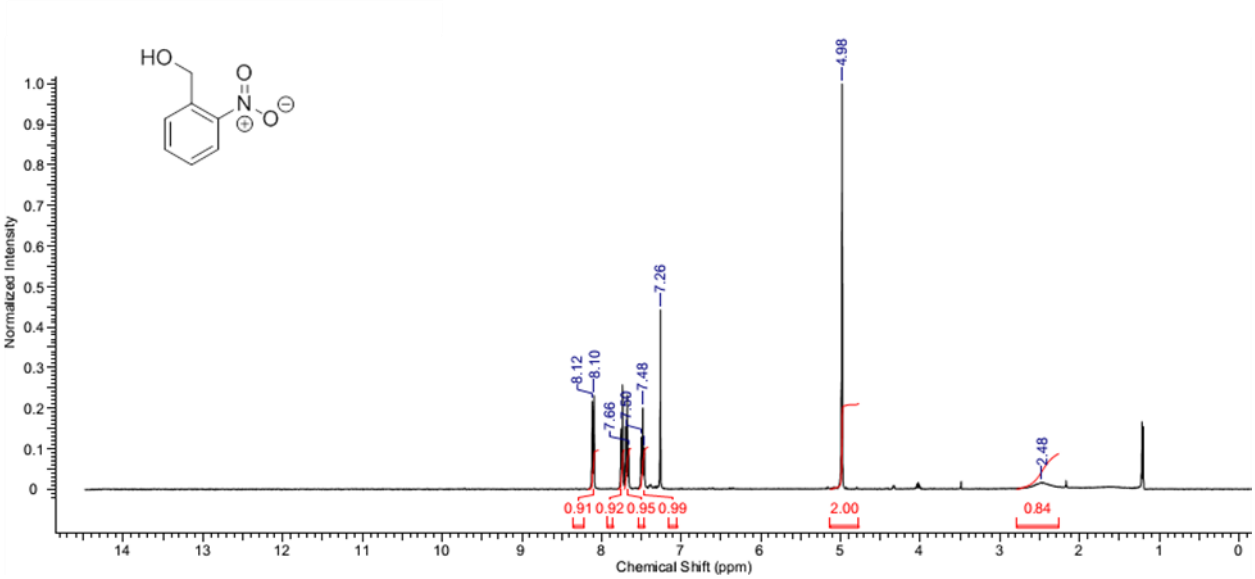


^{13}C-NMR (25 ℃)

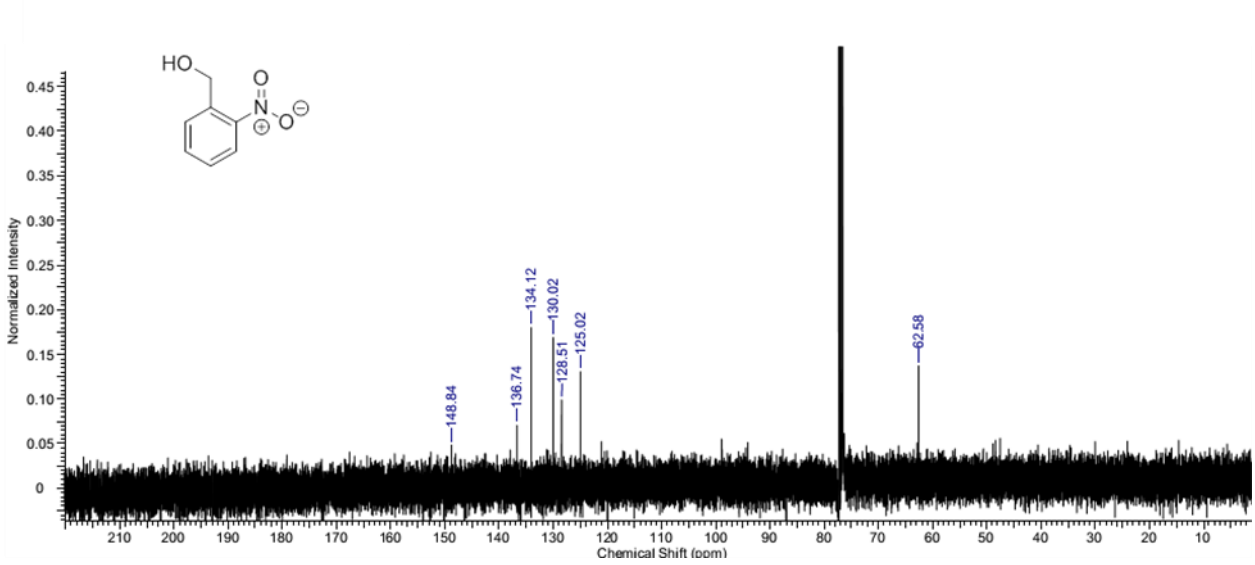


2-Nitrobenzyl alcohol (7).

^{1}H-NMR (25 °C)

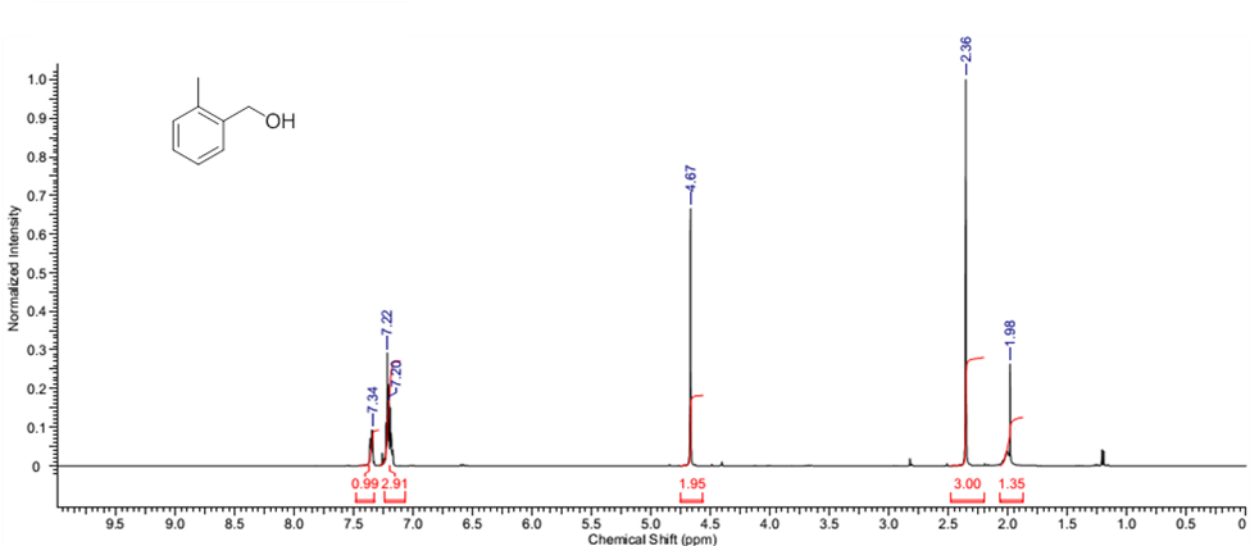


^{13}C-NMR (25 °C)

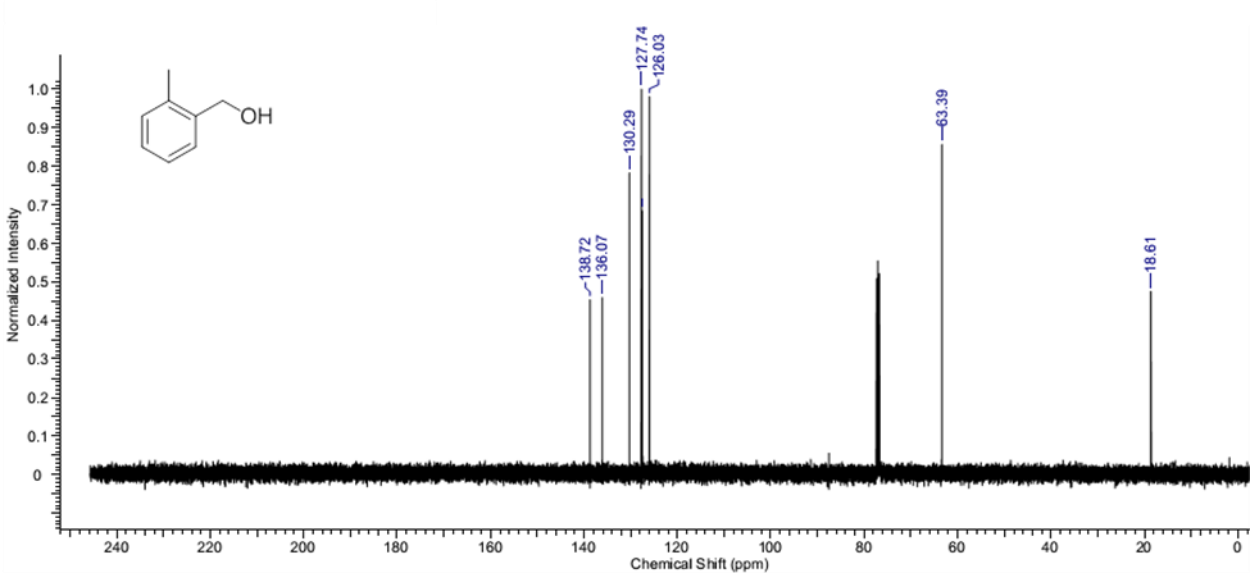


2-Methyl benzyl alcohol (8).

^{1}H-NMR (25 °C)

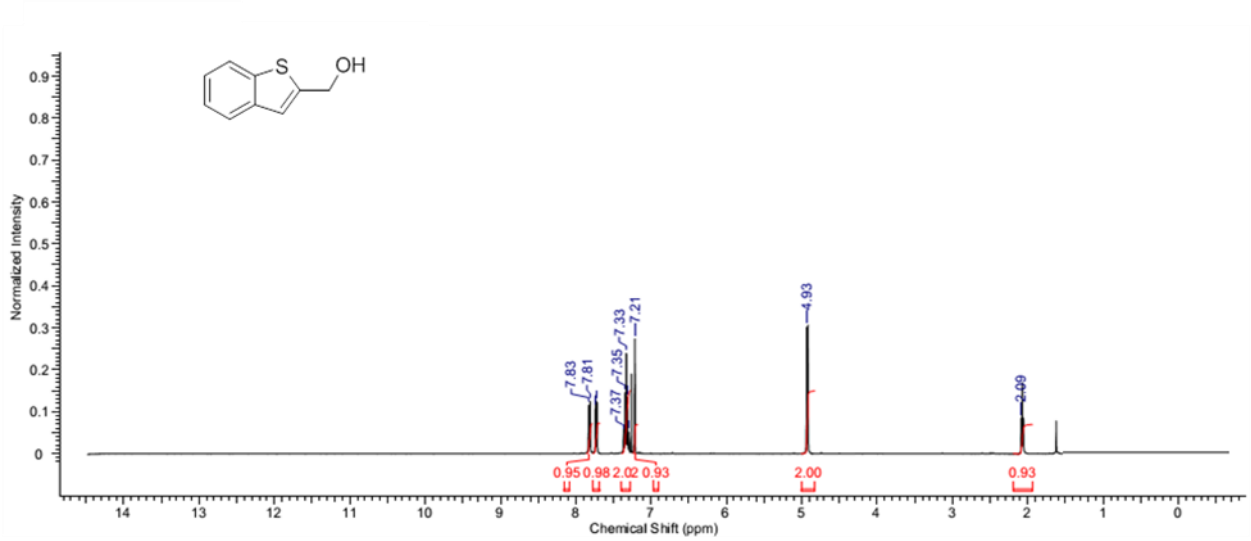


^{13}C-NMR (25 °C)

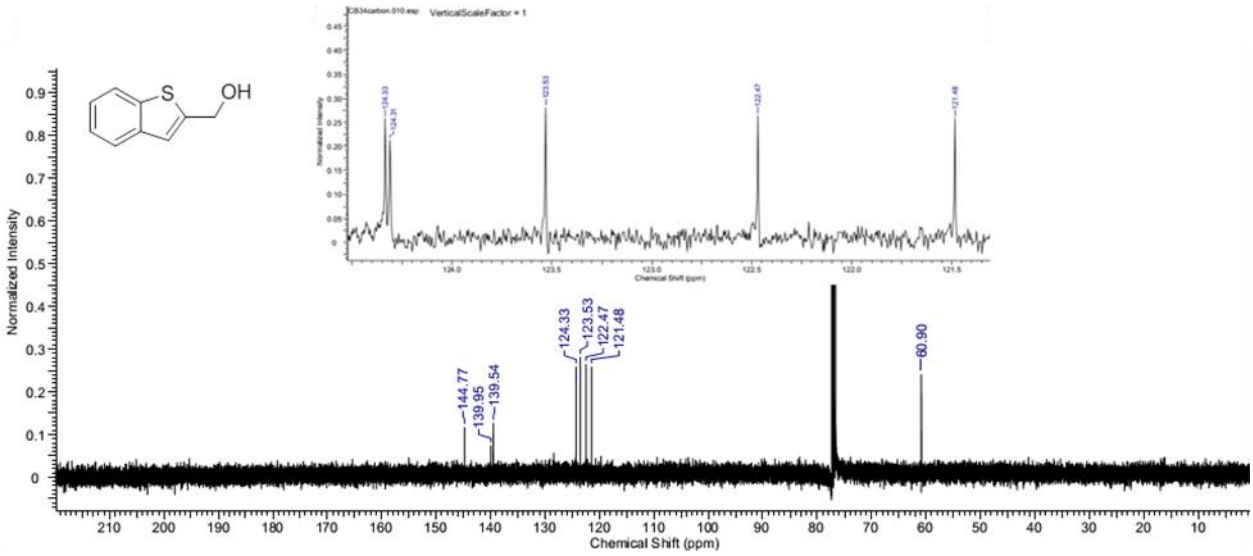


2-Hydroxymethyl benzo[b]thiopene (9).

^{1}H-NMR (25 °C)

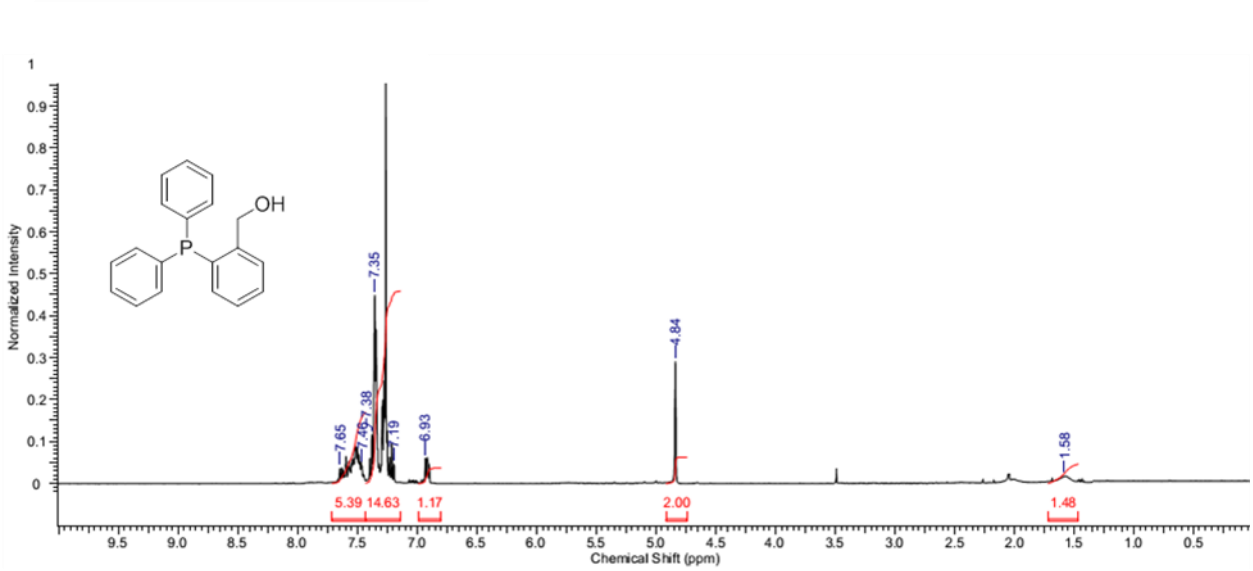


^{13}C-NMR (25 °C)

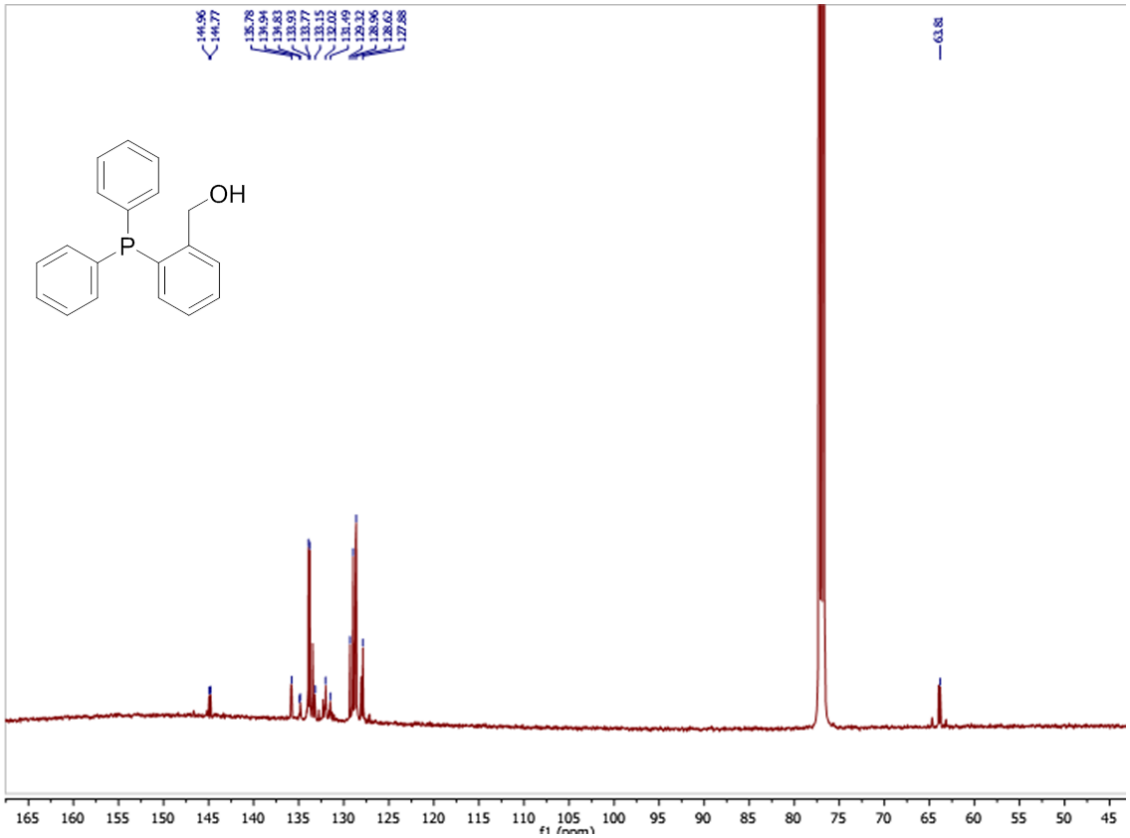


2-Hydroxymethylphenyl-diphenylphosphine (10).

^{1}H-NMR (25 °C)

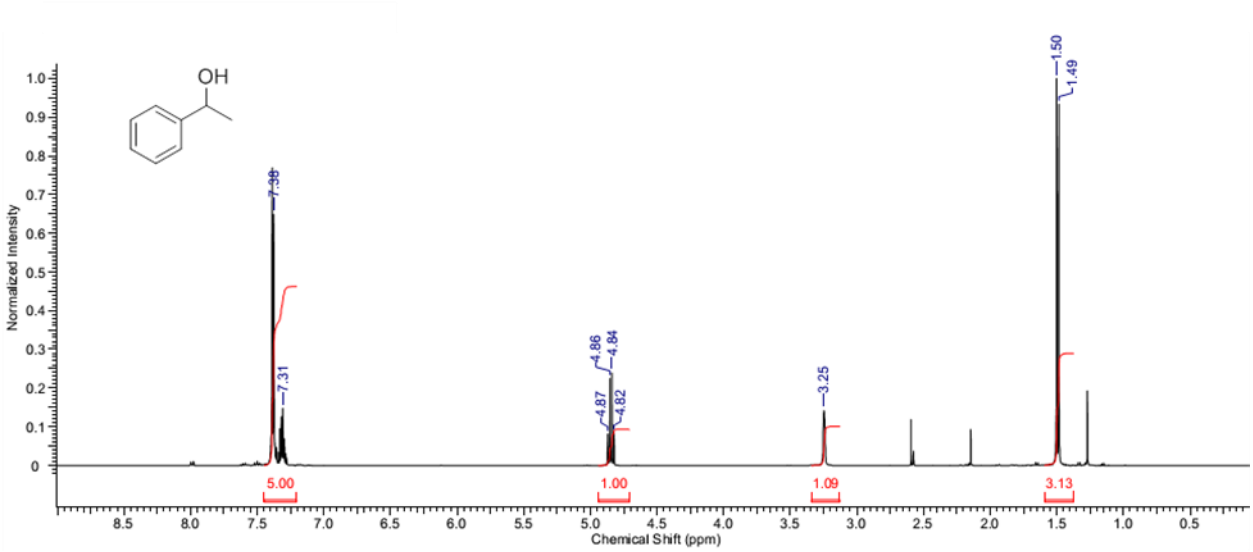


^{13}C-NMR (25 °C)

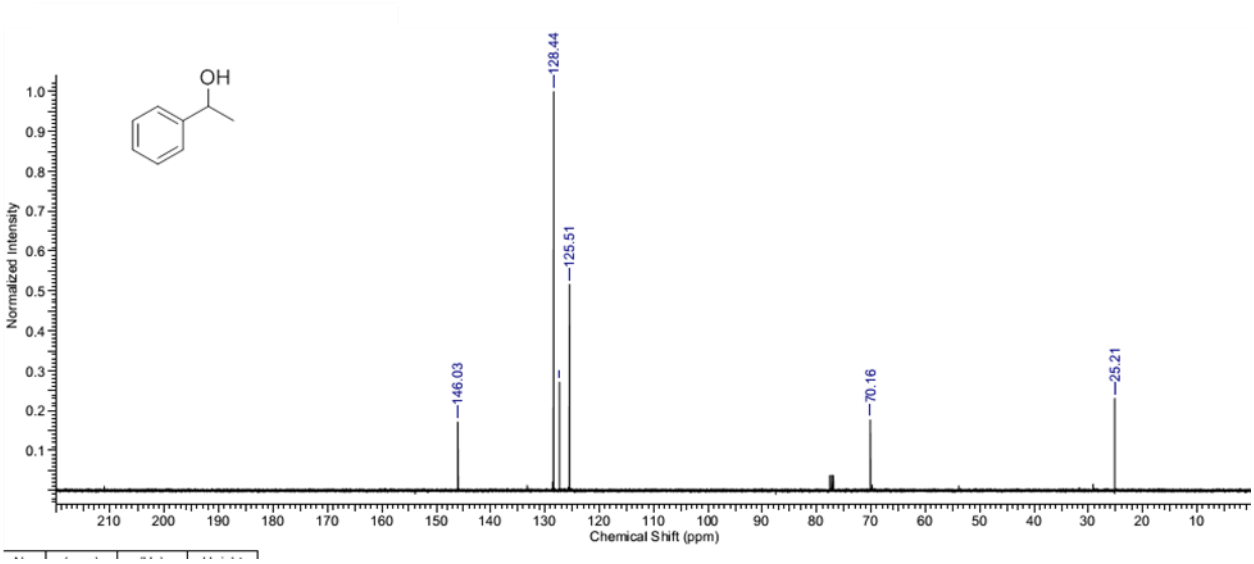


(±)-1-(Phenyl)ethanol (11).

^{1}H-NMR (25 °C)

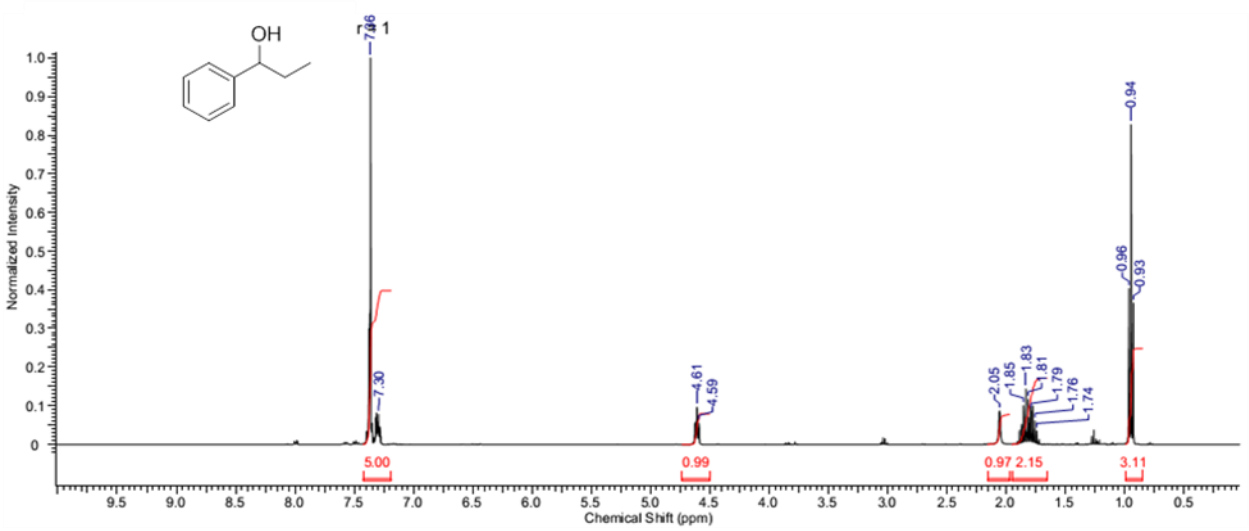


^{13}C-NMR (25 °C)

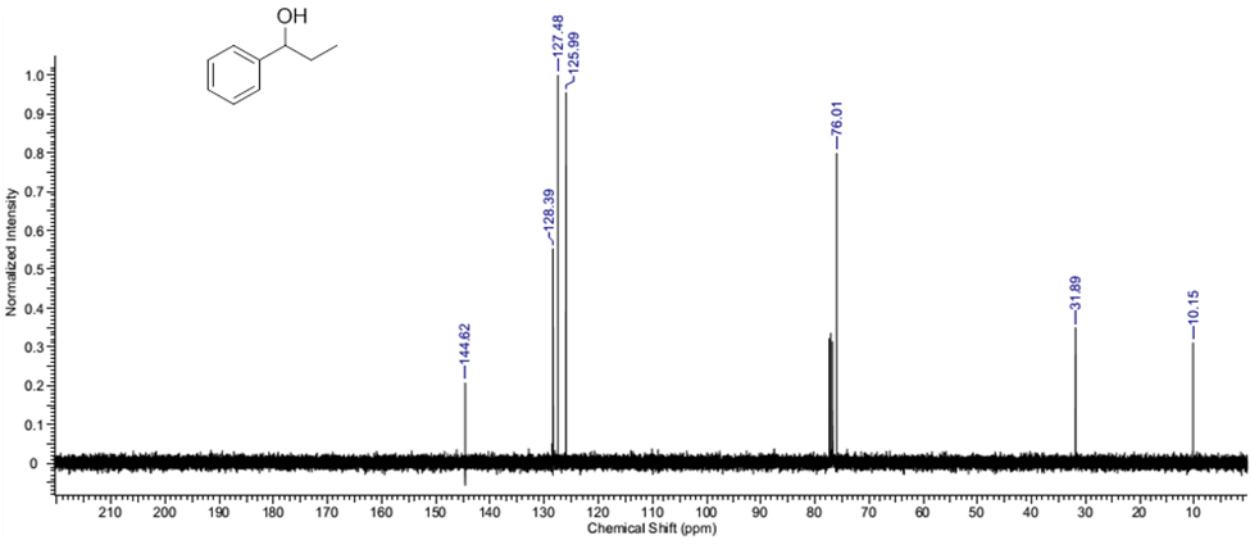


(±)-1-(Phenyl)propanol (12).

^{1}H-NMR (25 °C)

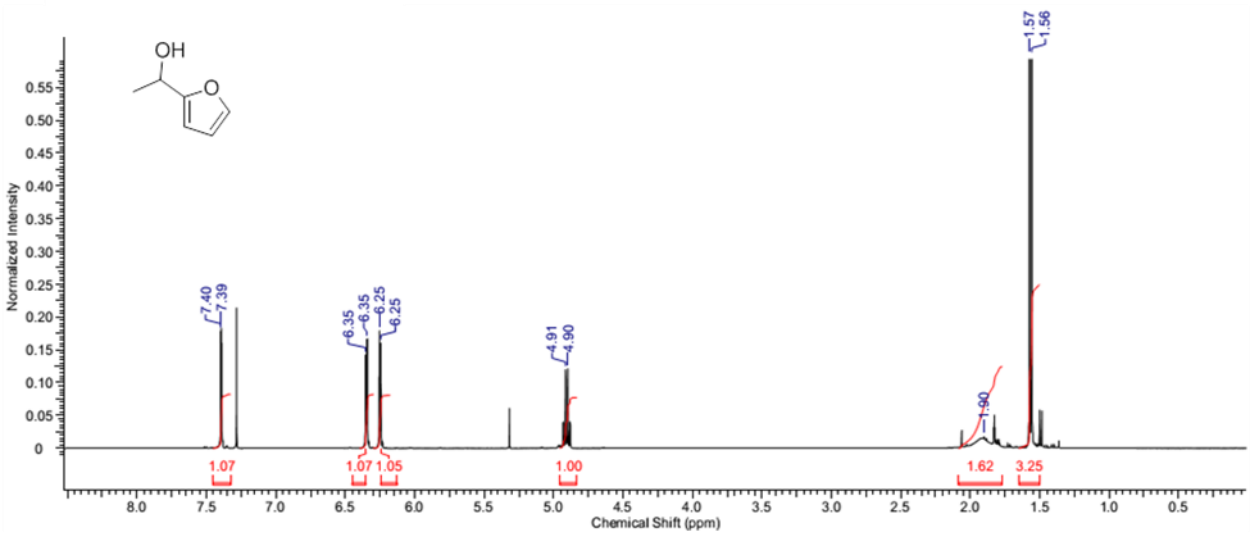


^{13}C-NMR (25 °C)

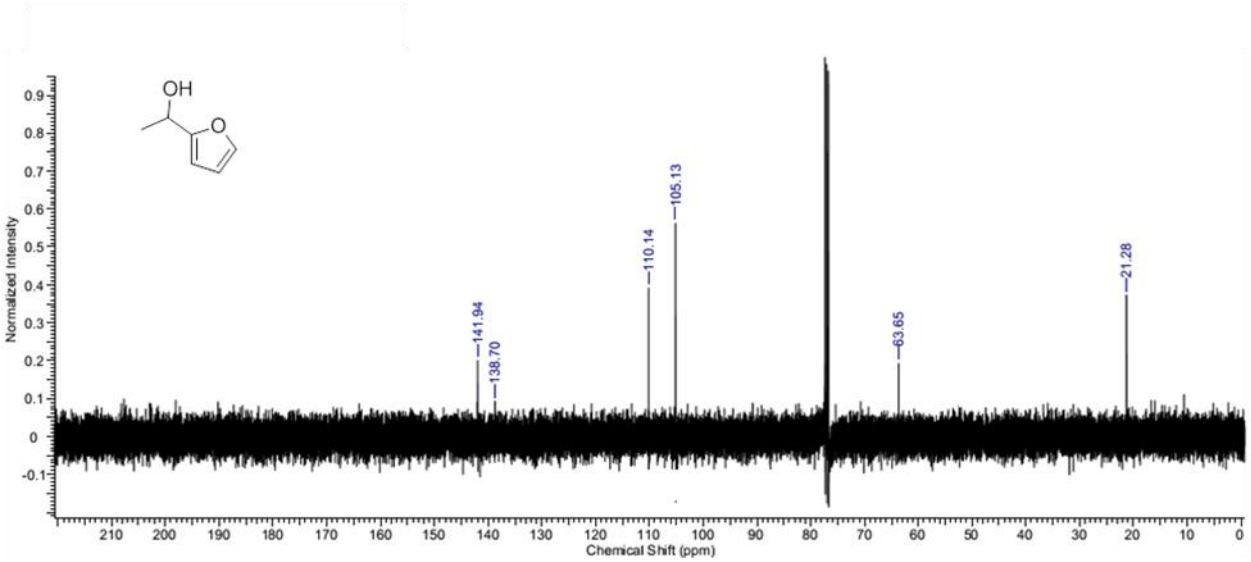


(±)-1-(Furan-2yl)ethanol (13).

^{1}H-NMR (25 °C)

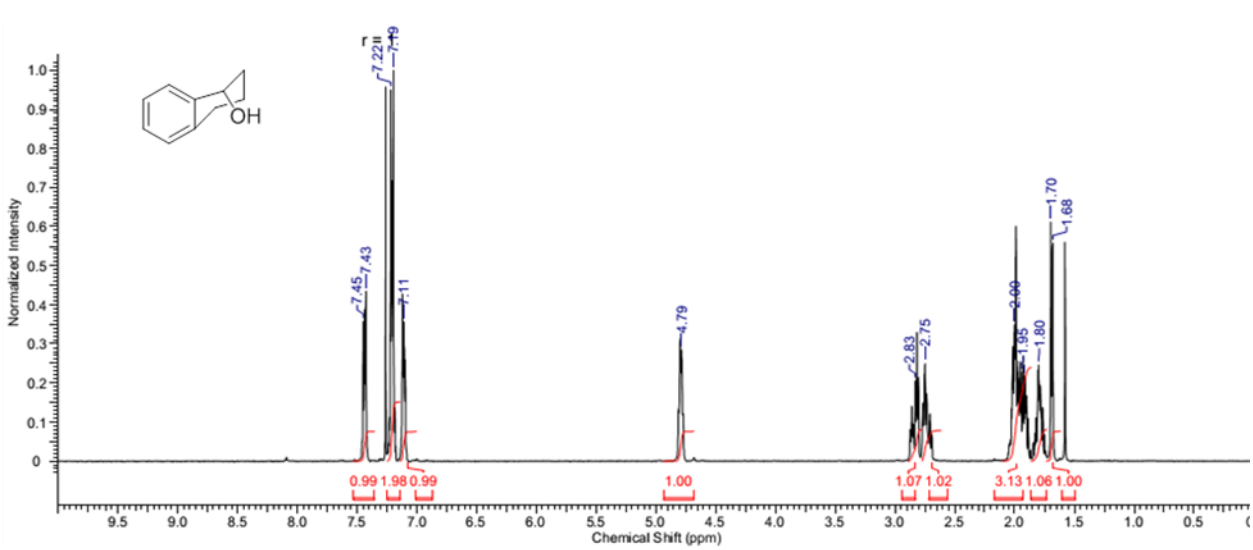


^{13}C-NMR (25 °C)

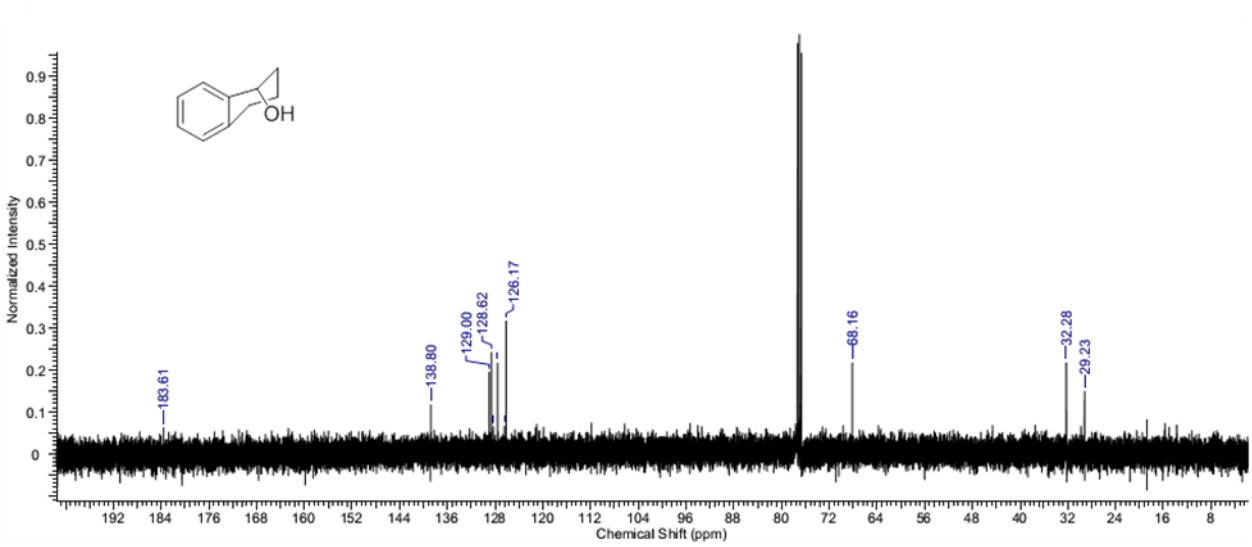


(±)-1-Tetralol (14).

^{1}H-NMR (25 °C)

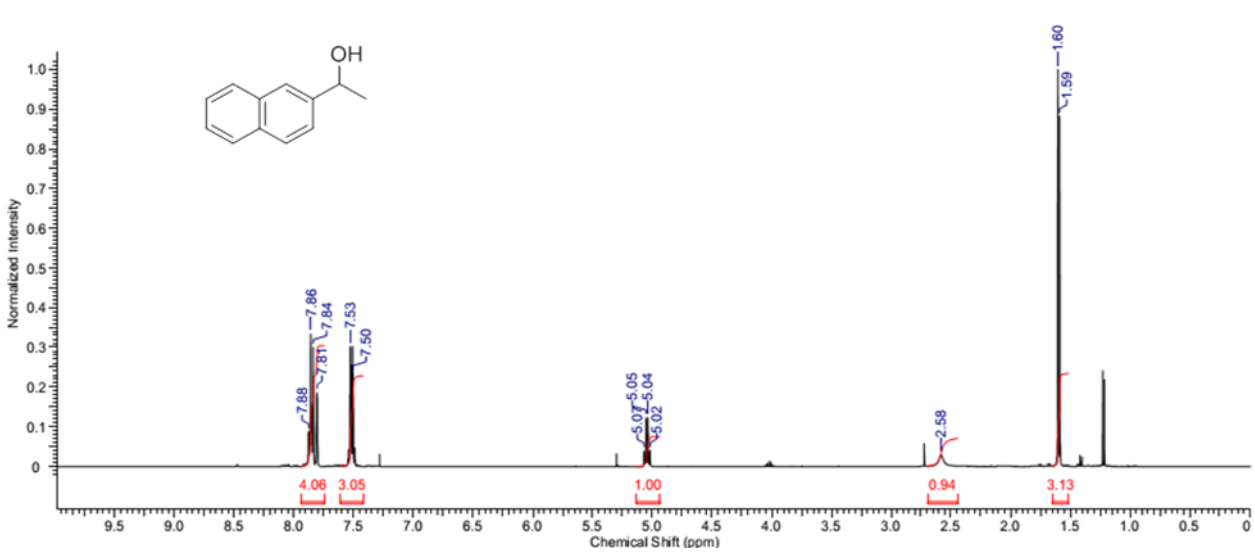


^{13}C-NMR (25 °C)

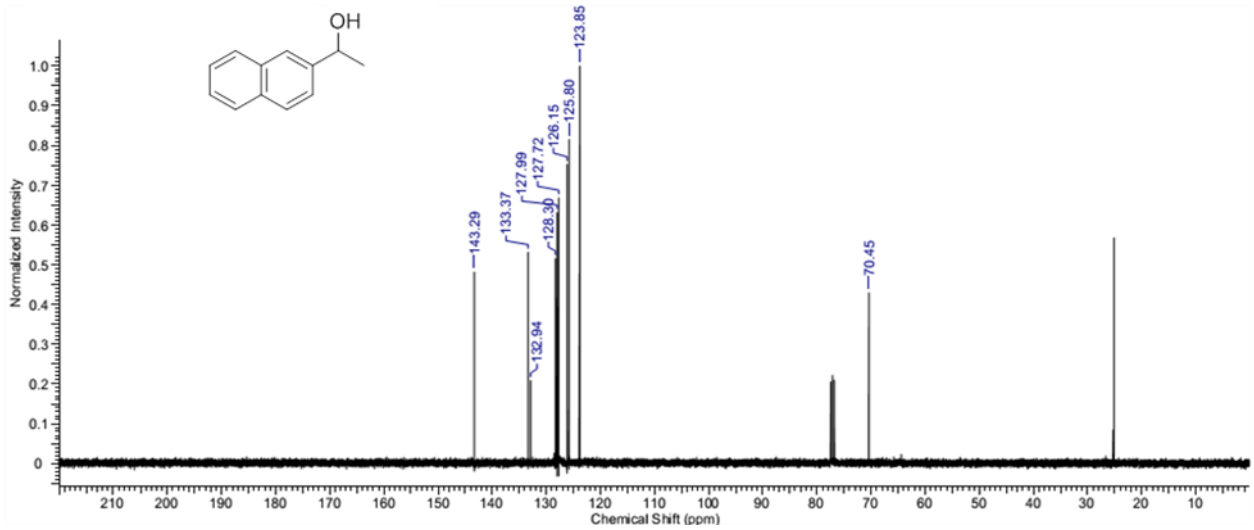


(±)-1-(Naphthyl)ethanol (15).

^{1}H-NMR (25 °C)

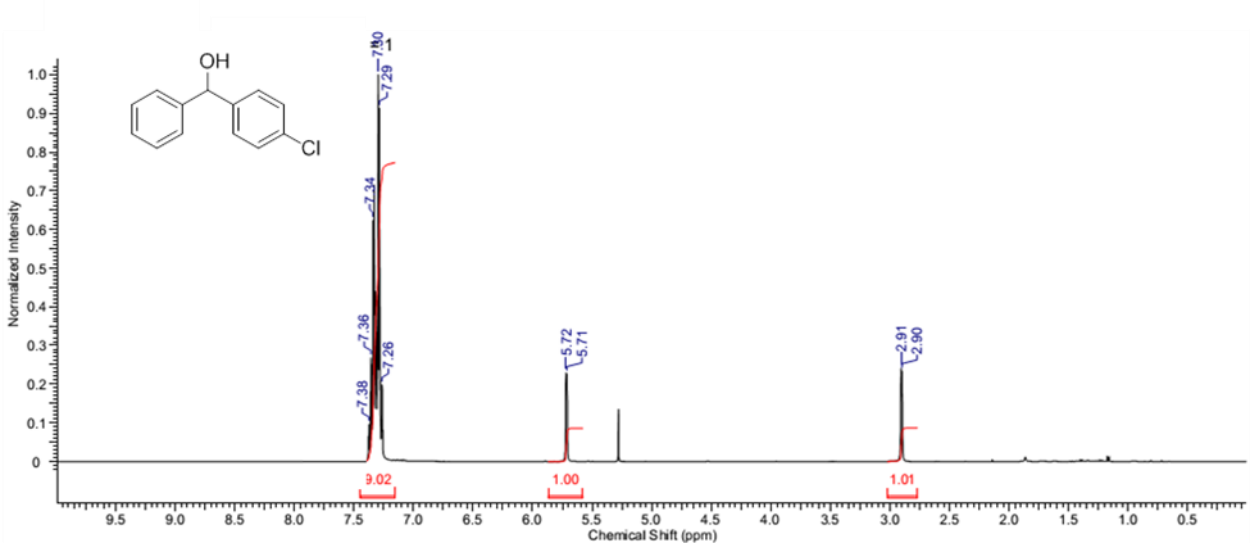


^{13}C-NMR (25 °C)

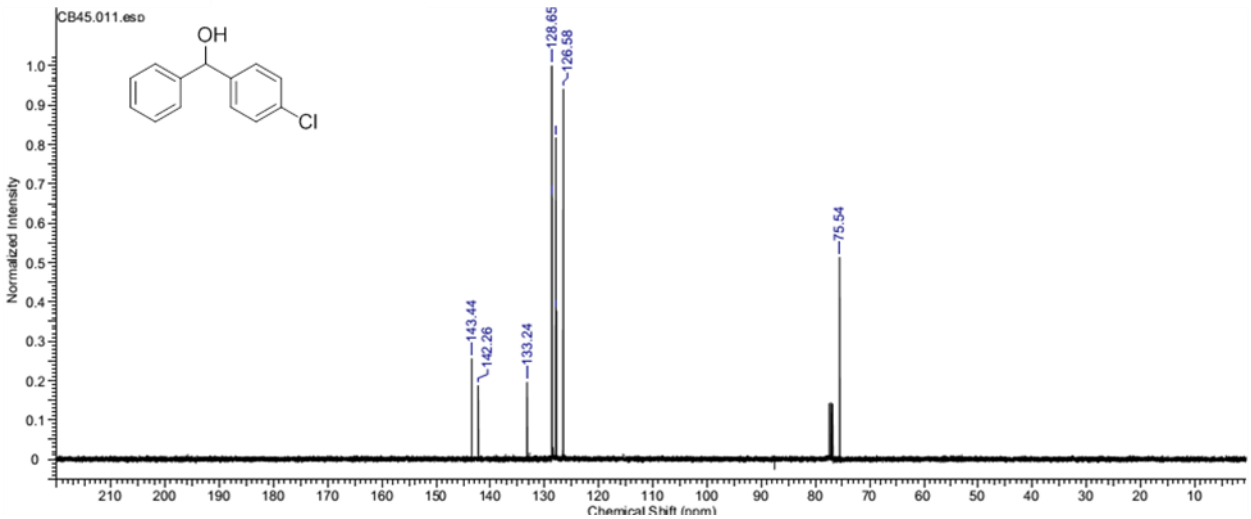


(±)-α-(4-Chlorophenyl)-benzyl alcohol (16).

^{1}H-NMR (25 °C)

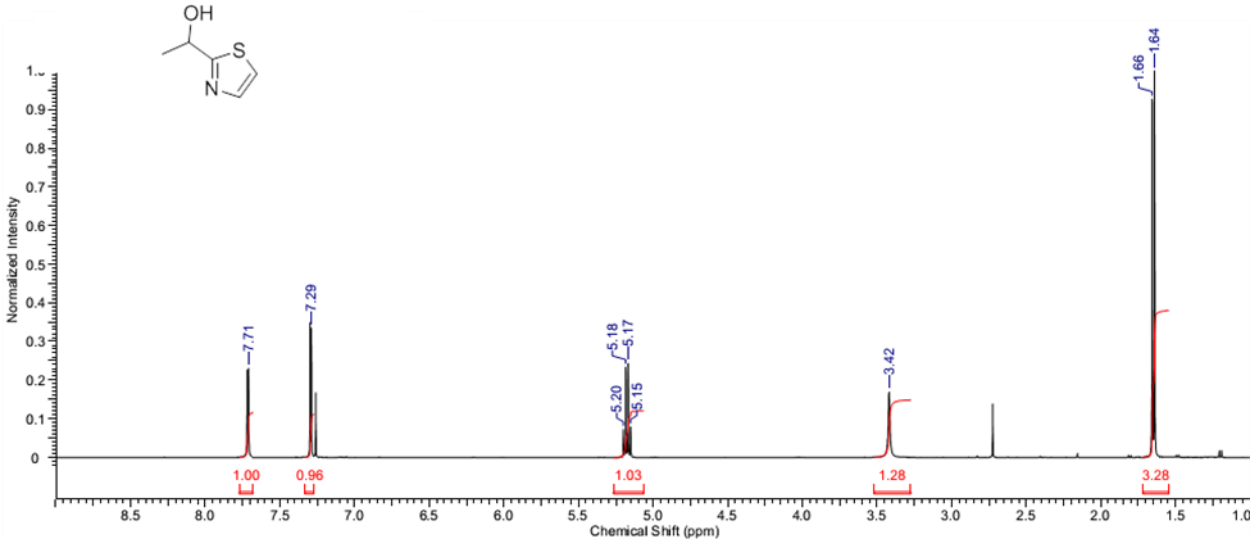


^{13}C-NMR (25 °C)

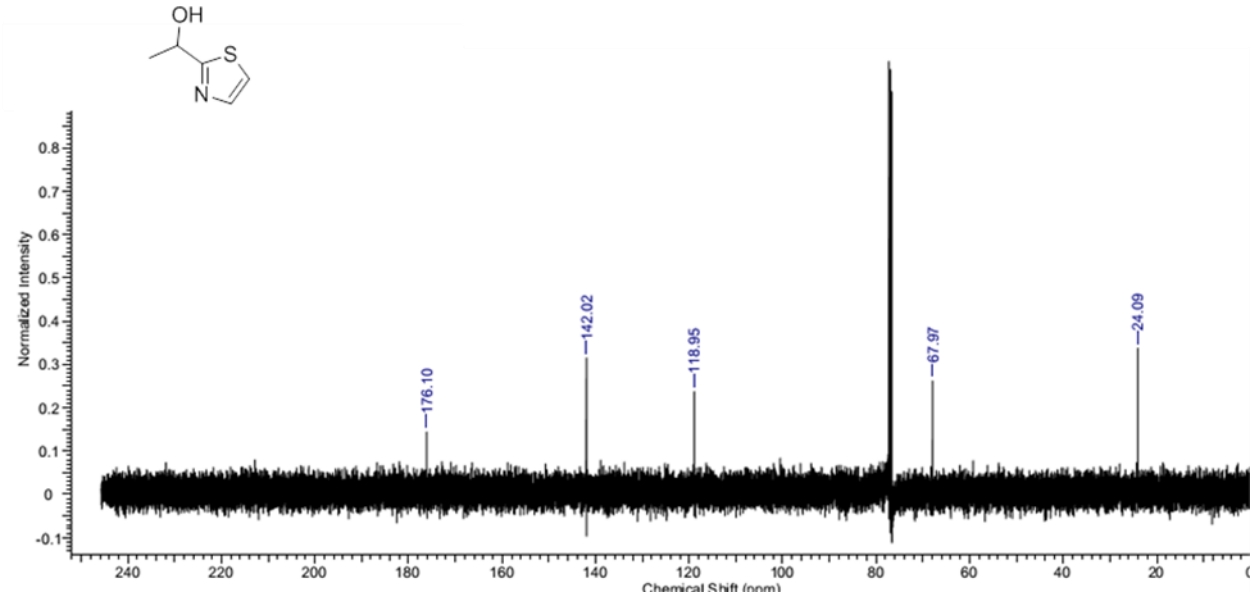


(±)-1-(Thiazol-yl)ethanol (17).

^{1}H-NMR (25 °C)

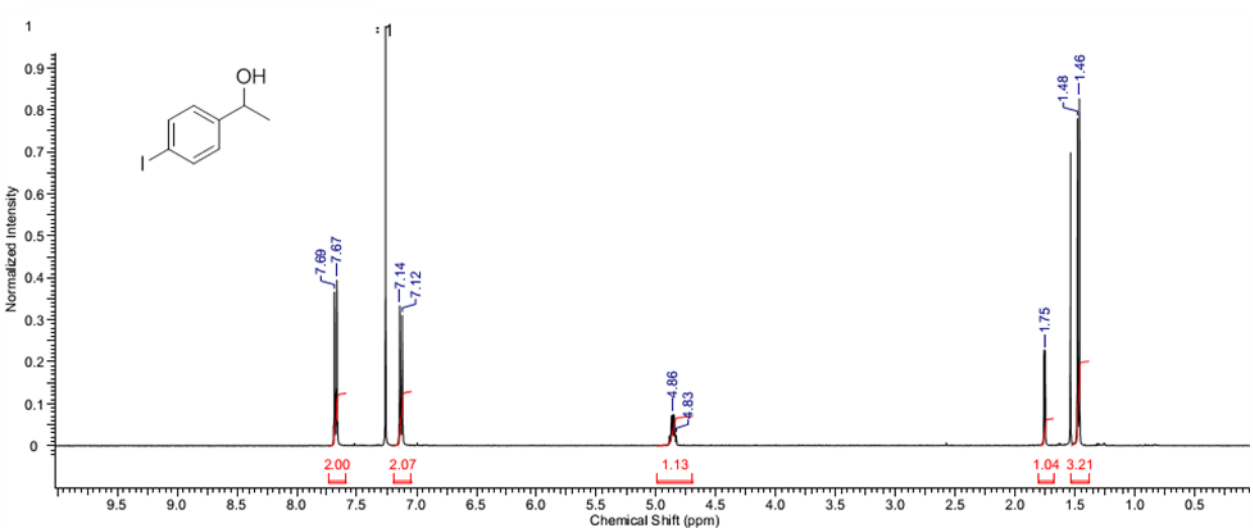


^{13}C-NMR (25 °C)

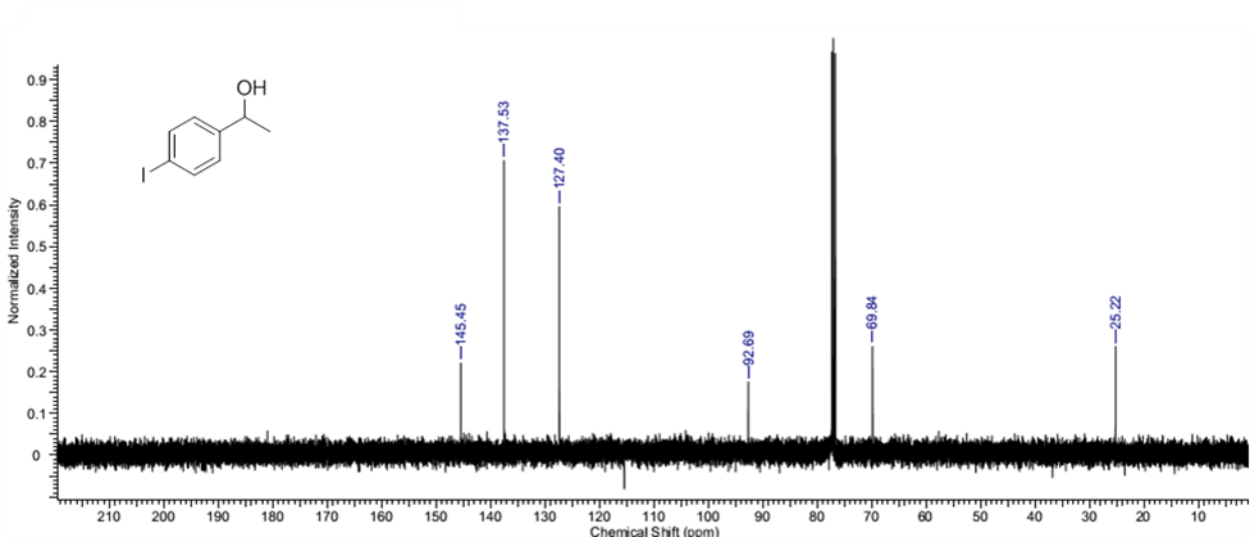


(±)-1-(4-Iodophenyl)ethanol (18).

^{1}H-NMR (25 °C)

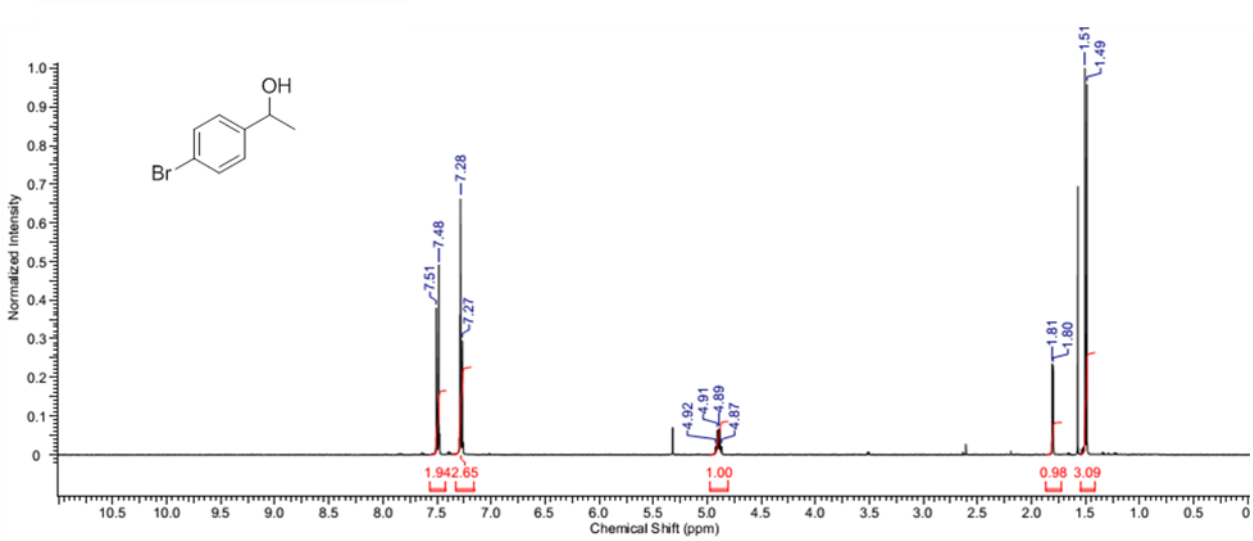


^{13}C-NMR (25 °C)

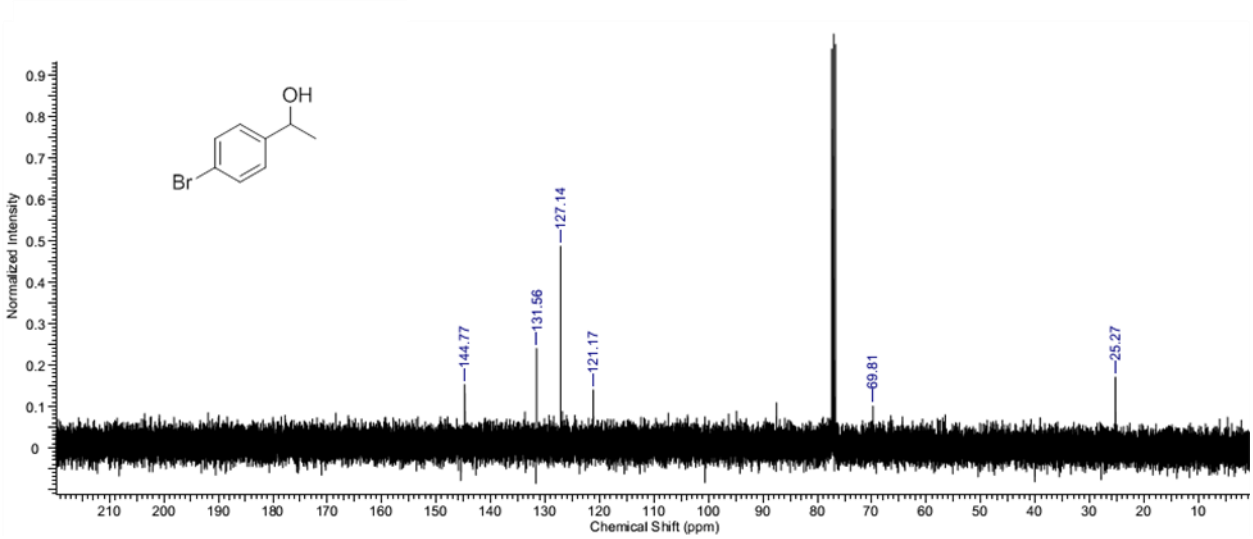


(±)-1-(4-Bromophenyl)ethanol (19).

^{1}H-NMR (25 °C)

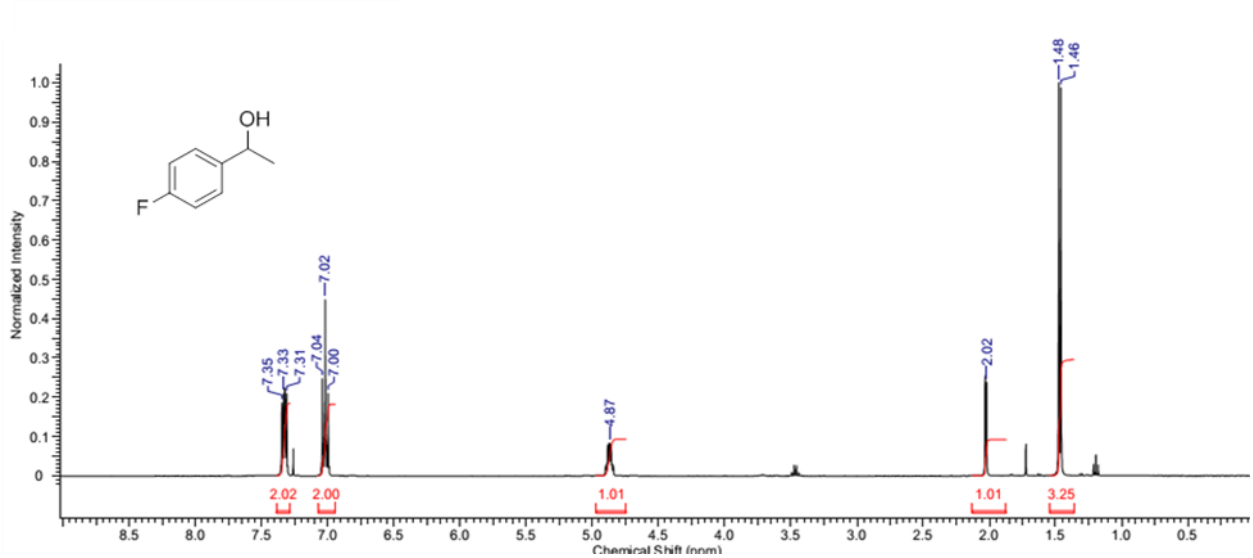


^{13}C-NMR (25 °C)

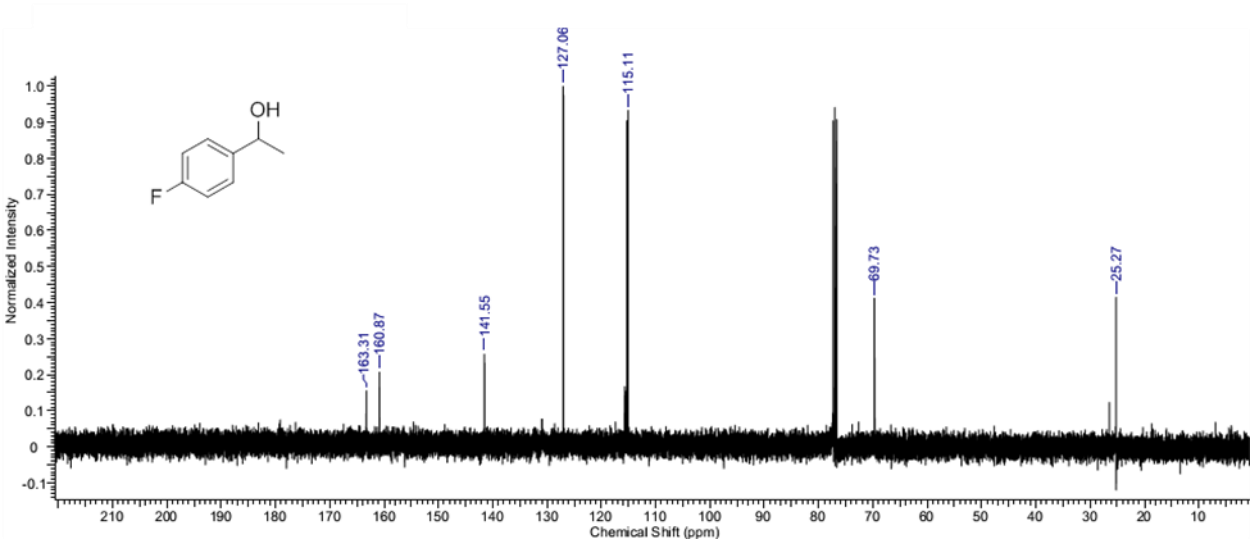


(±)-1-(4-Fluorophenyl)ethanol (20).

^{1}H-NMR (25 °C)

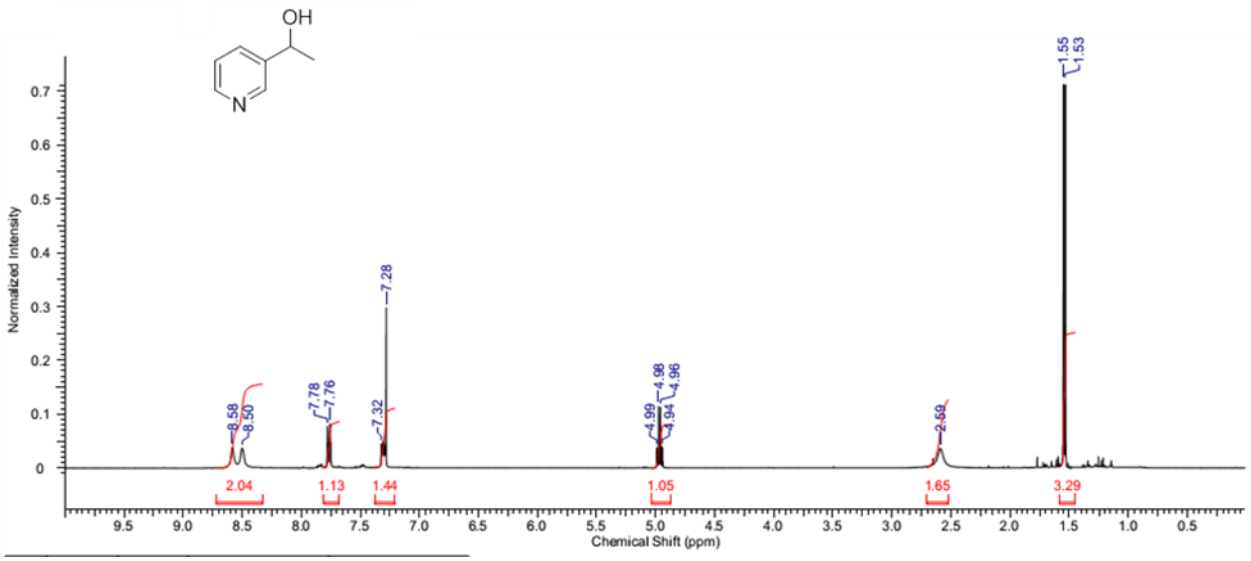


^{13}C-NMR (25 °C)

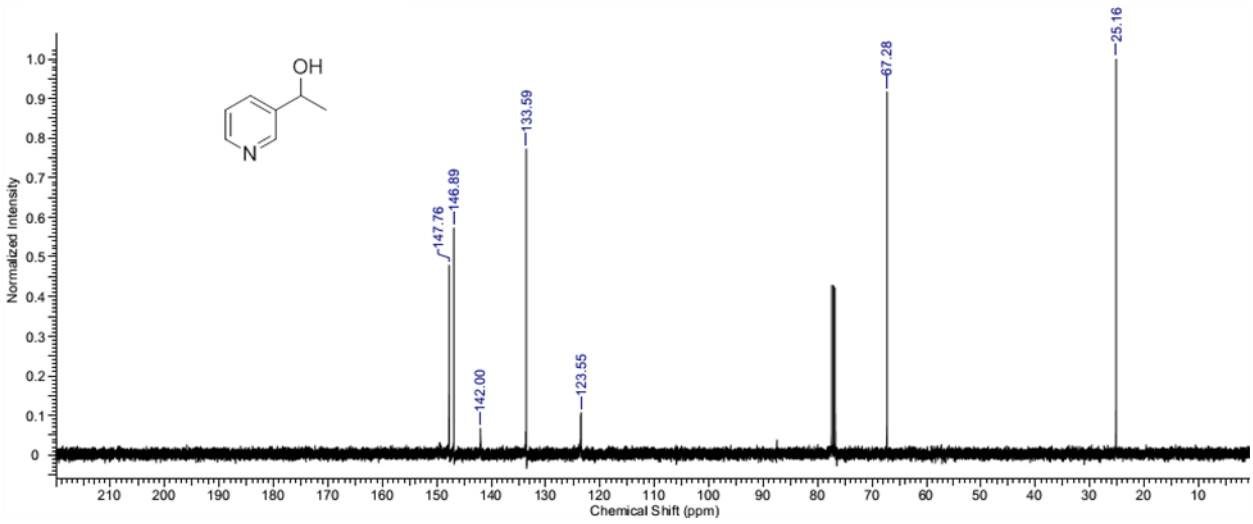


(±)-1-(Pyrid-3yl)ethanol (21).

^{1}H-NMR (25 °C)

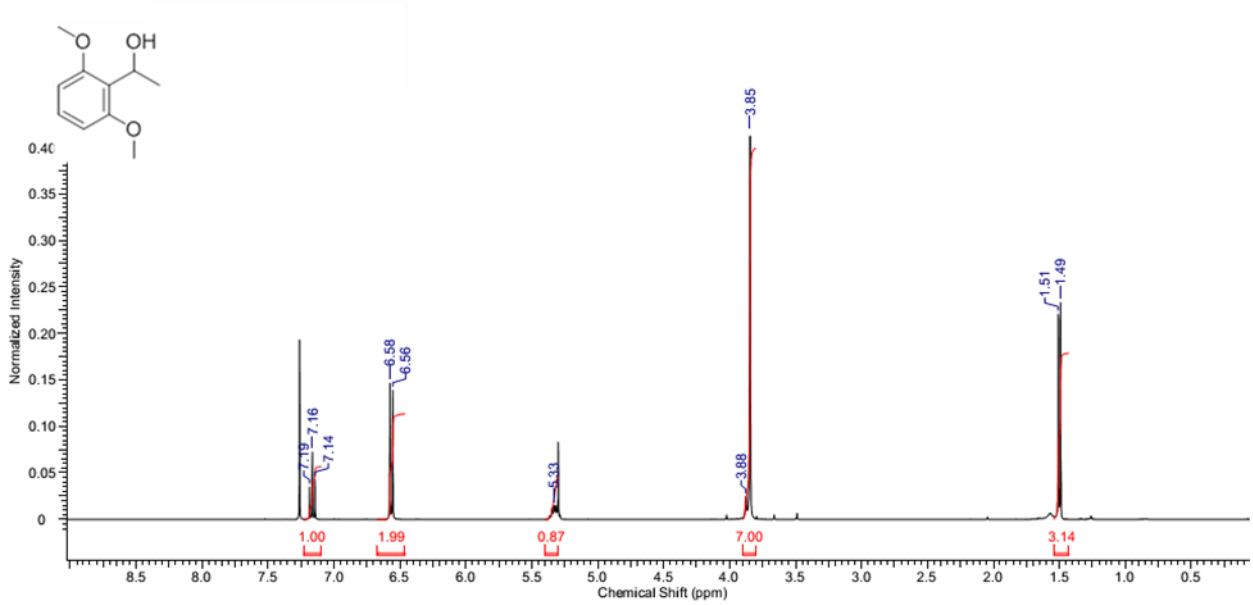


^{13}C-NMR (25 °C)

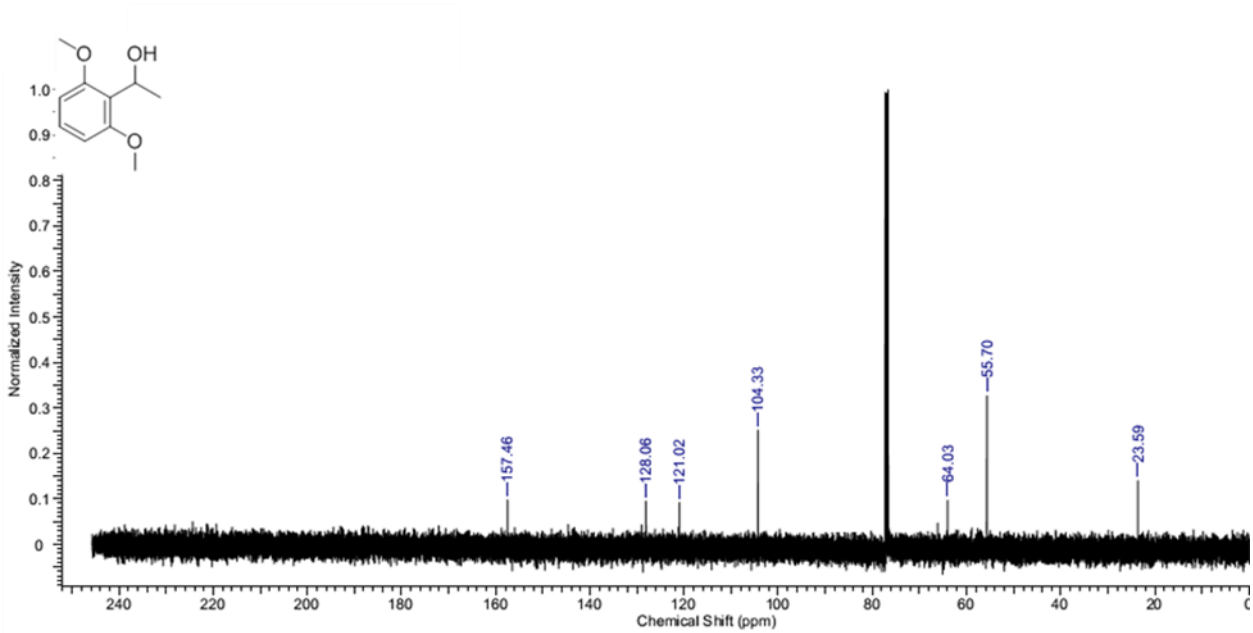


(±)-1-(2,6-Dimethoxyphenyl)ethanol (22).

^{1}H-NMR (25 °C)

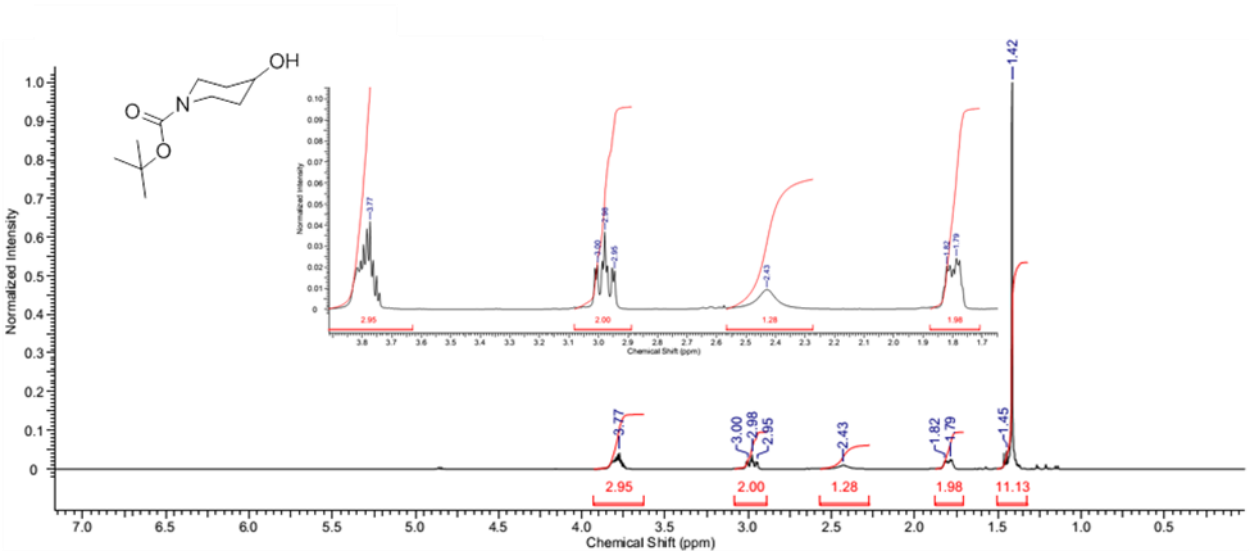


^{13}C-NMR (25 °C)

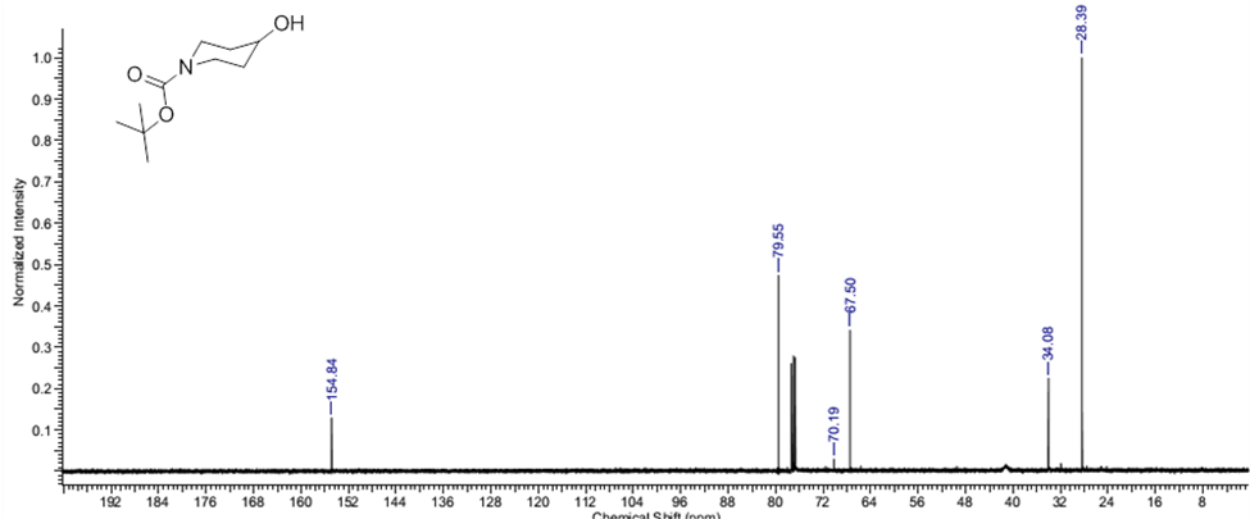


N-Boc-piperidin-4-ol (23).

^{1}H-NMR (25 °C)

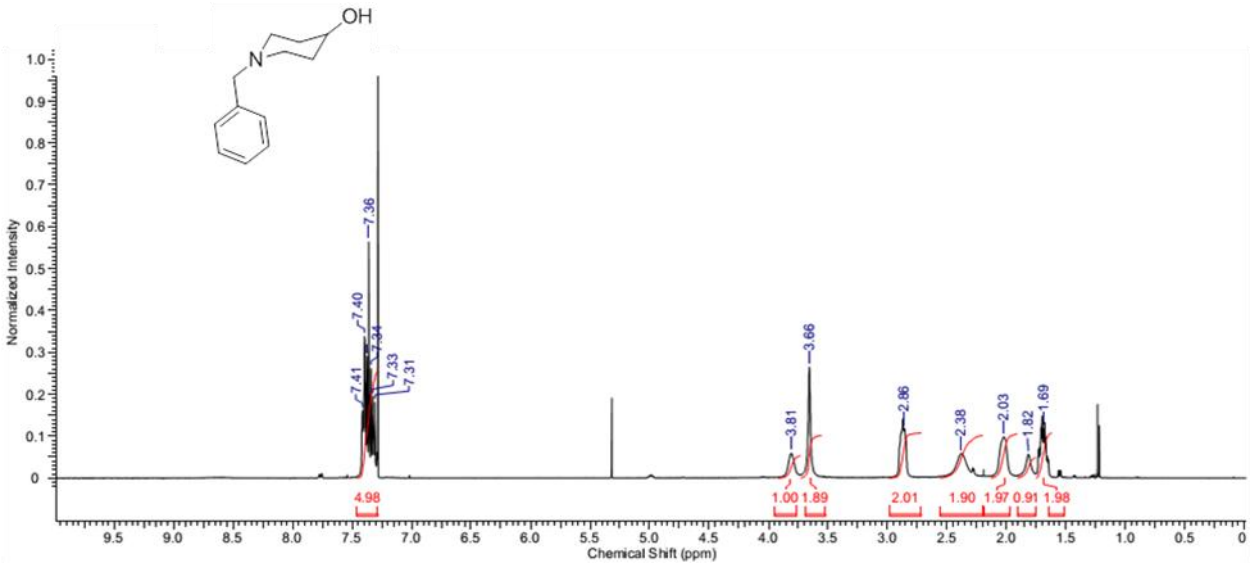


^{13}C-NMR (25 °C)

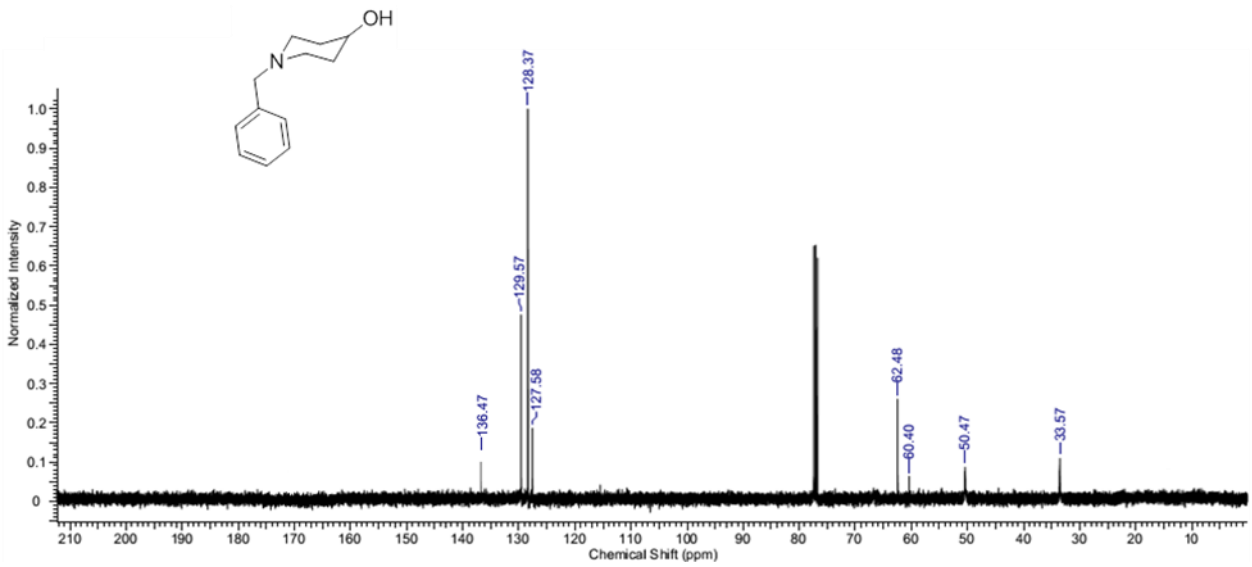


N-Benzyl-piperidin-4-ol (24).

^{1}H-NMR (25 °C)

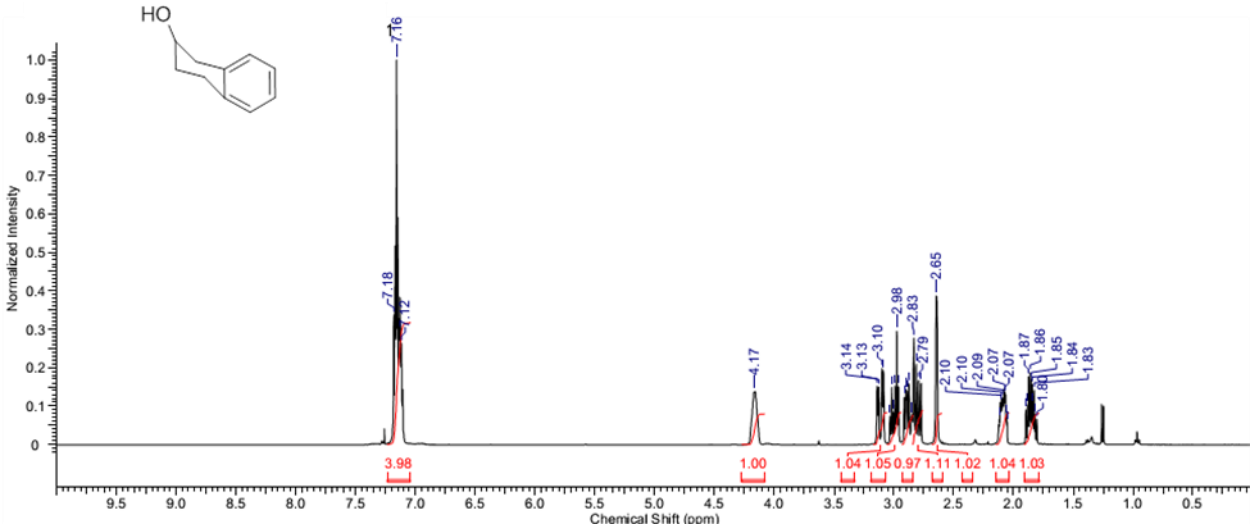


^{13}C-NMR (25 °C)

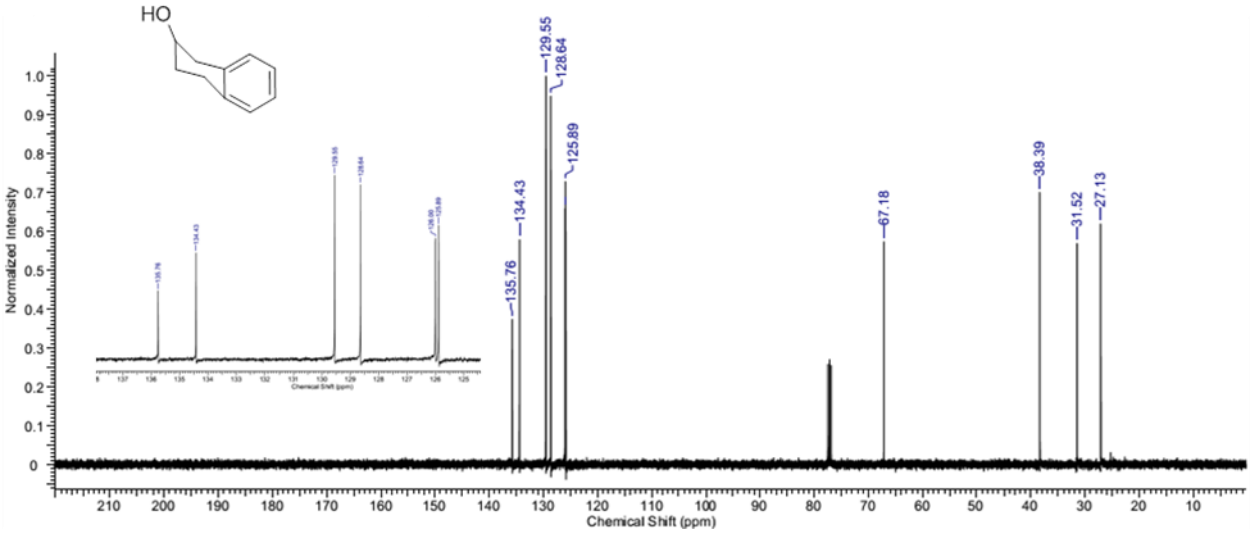


(±)-2-Tetralol (25).

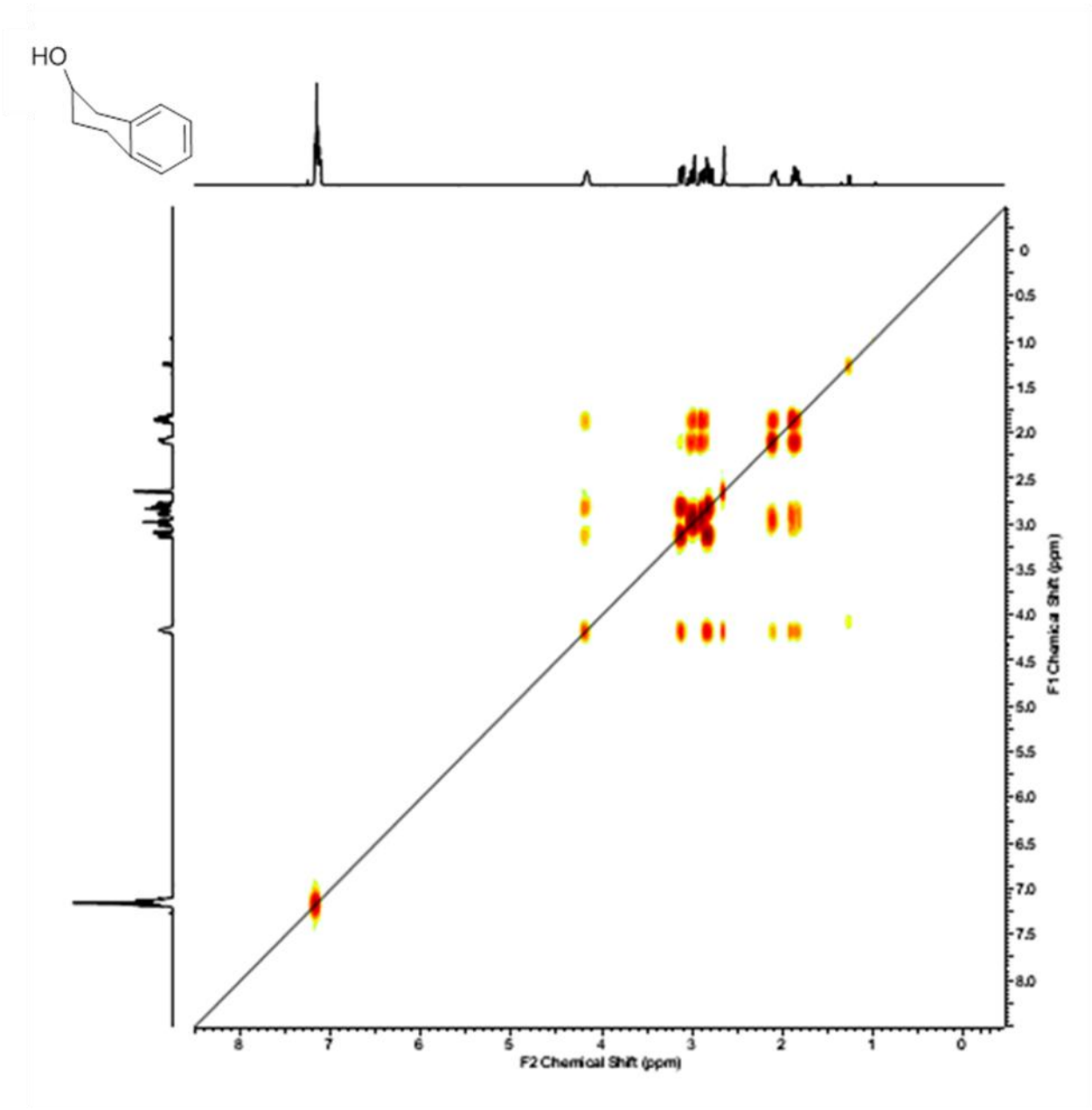
^{1}H-NMR (25 °C)



^{13}C-NMR (25 °C)

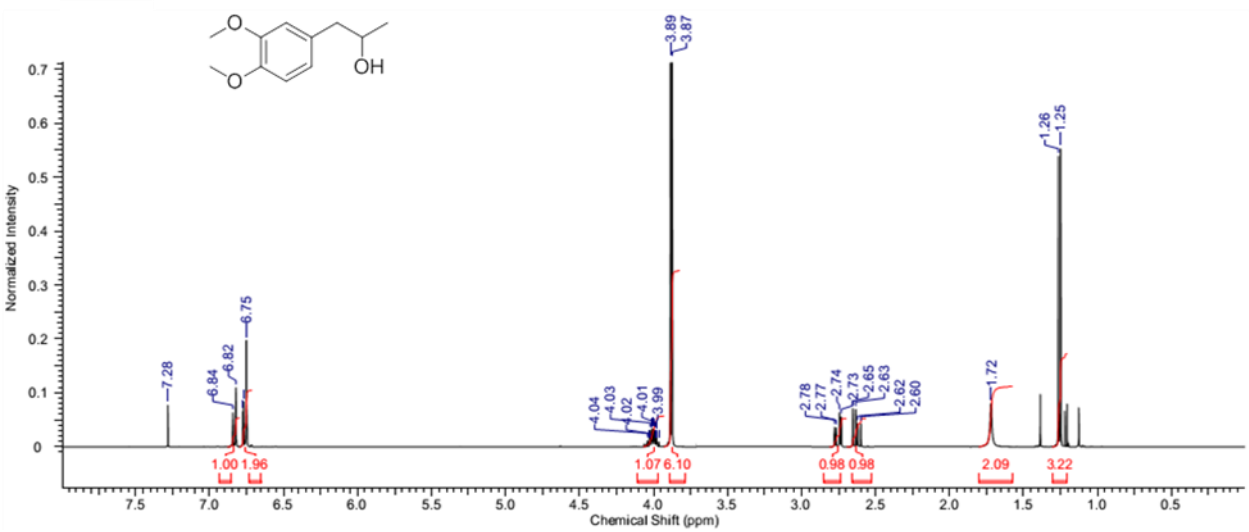


COSY-NMR (25 °C)

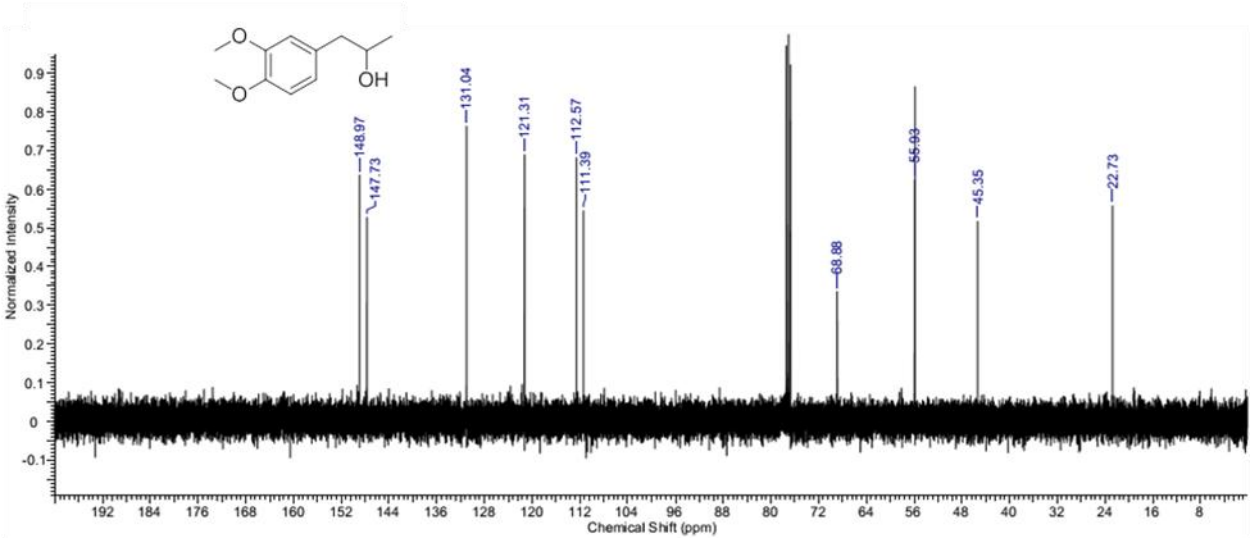


(±)-3-(3,4-Dimethoxyphenyl)-propan-2ol (26).

^{1}H-NMR (25 °C)

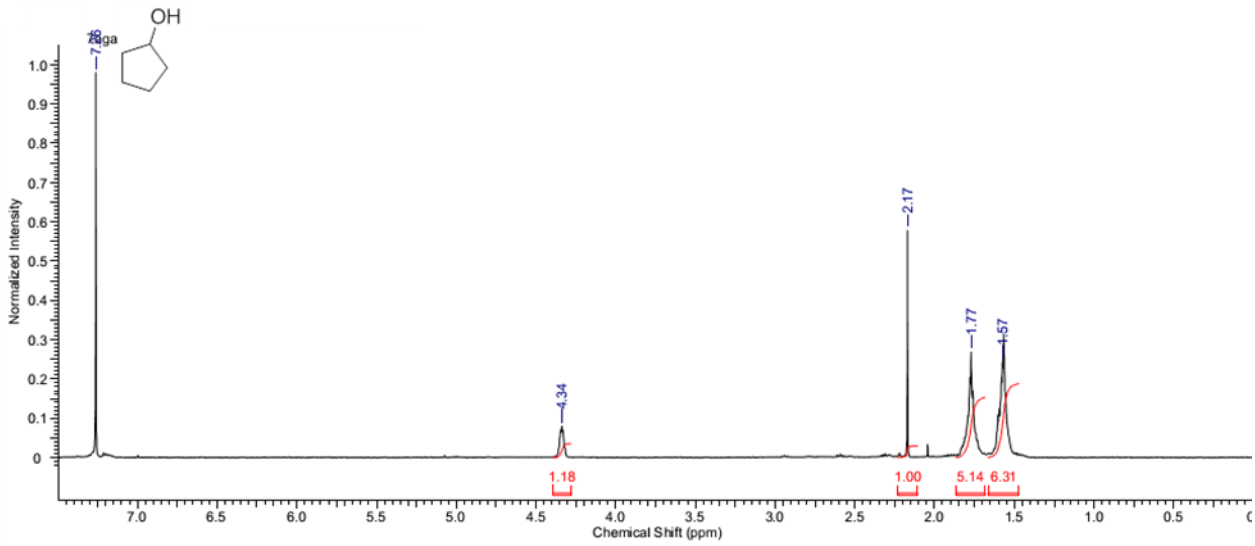


^{13}C-NMR (25 °C)

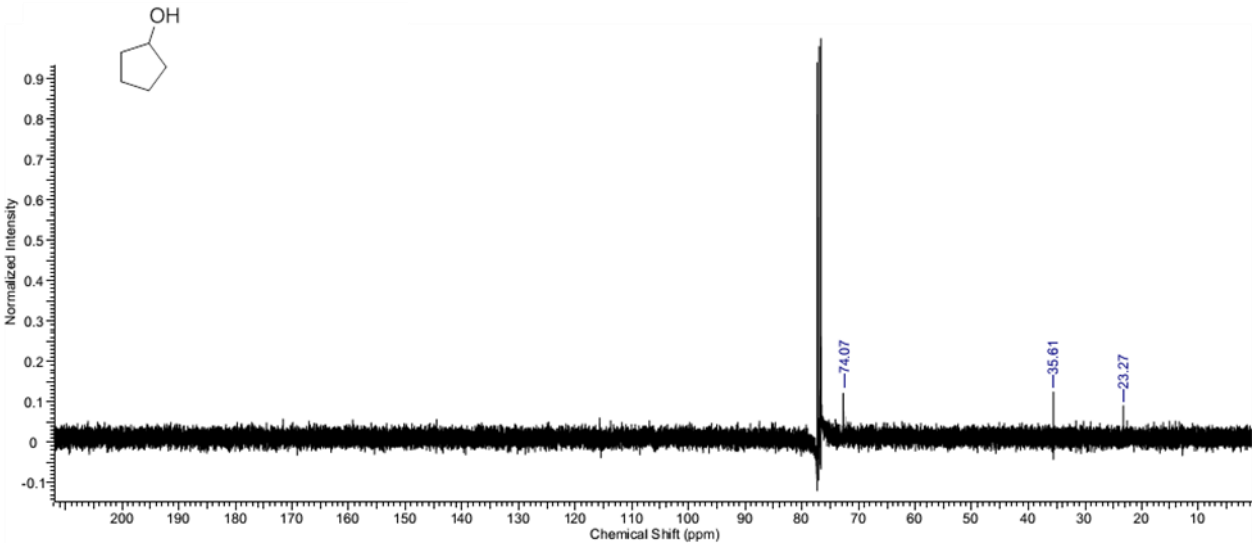


Cyclopentanol (27).

^{1}H-NMR (25 °C)

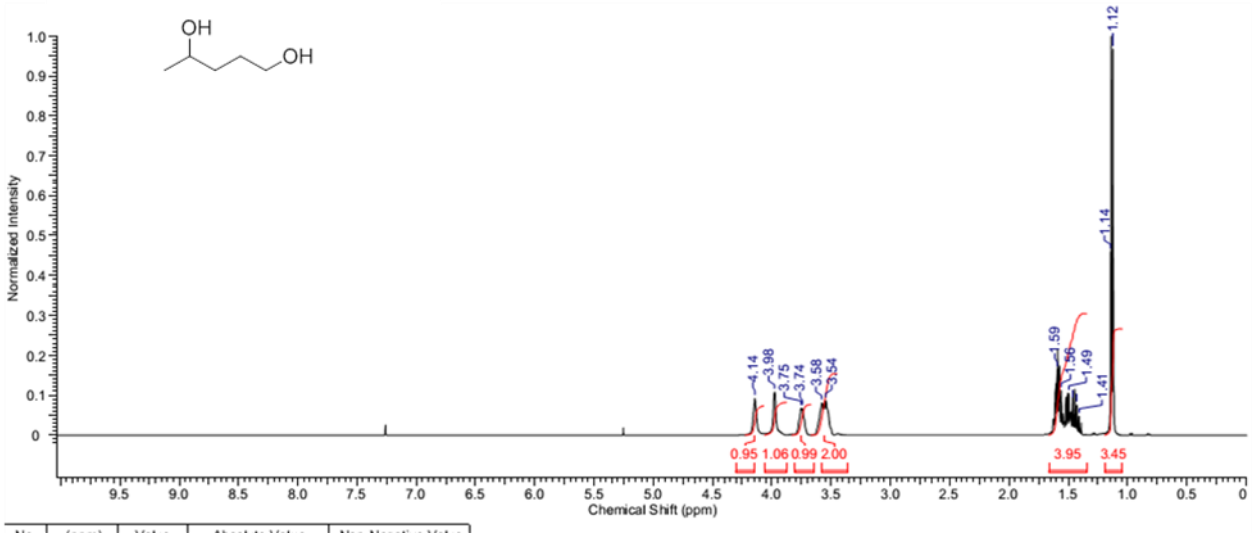


^{13}C-NMR (25 °C)

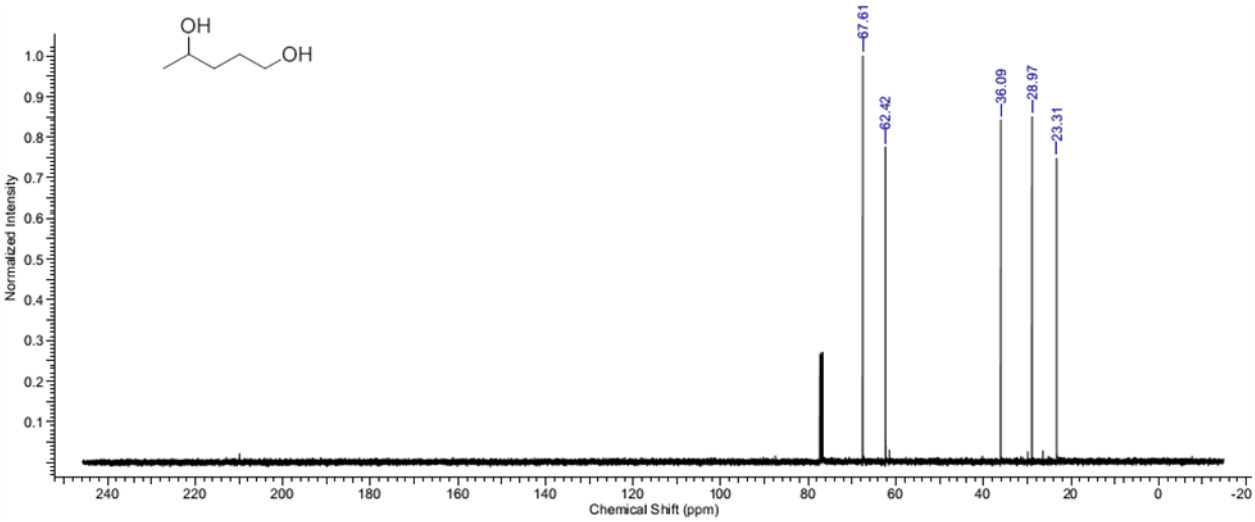


(±)Pentan-1,4-diol (28).

^{1}H-NMR (25 °C)

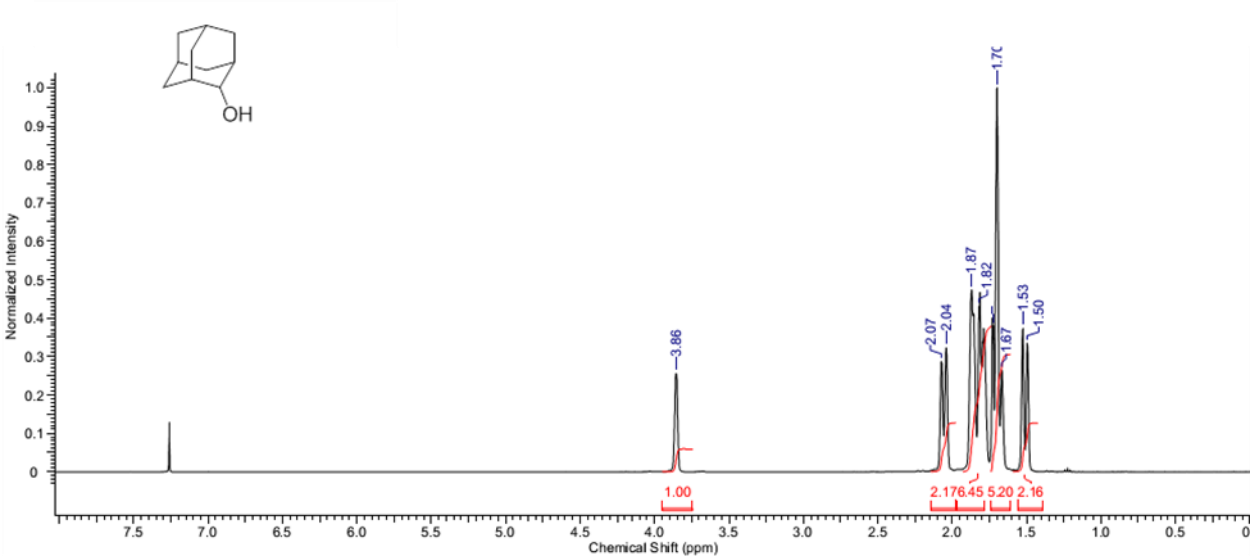


^{13}C-NMR (25 °C)

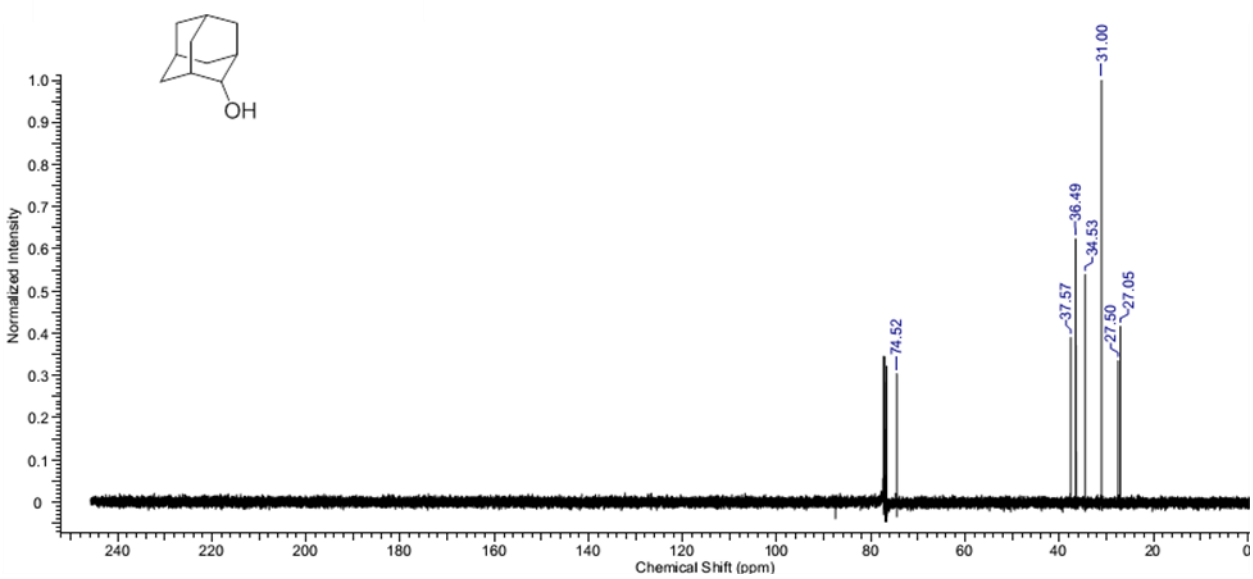


Adamantan-2-ol (29).

^{1}H-NMR (25 °C)

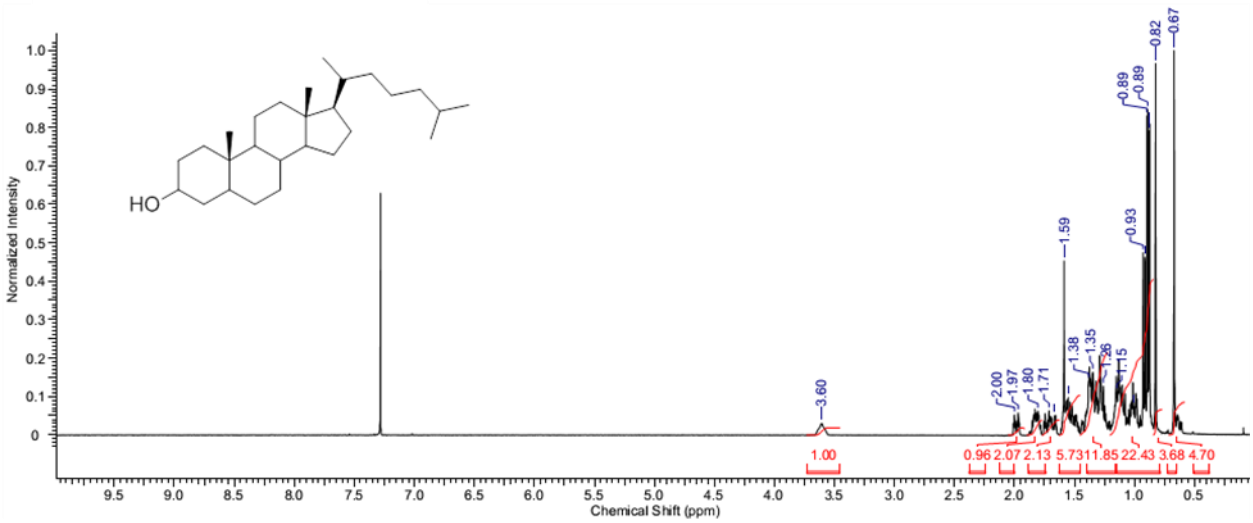


^{1}H-NMR (25 °C)

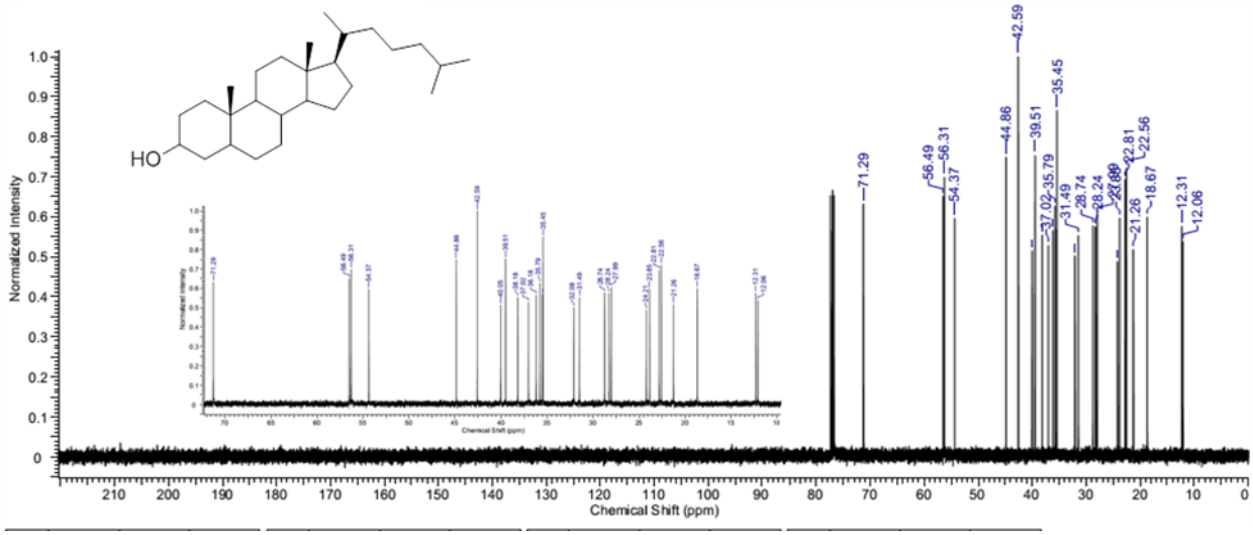


(±)Dihydrocholesterol (30).

^{1}H-NMR (25 °C)

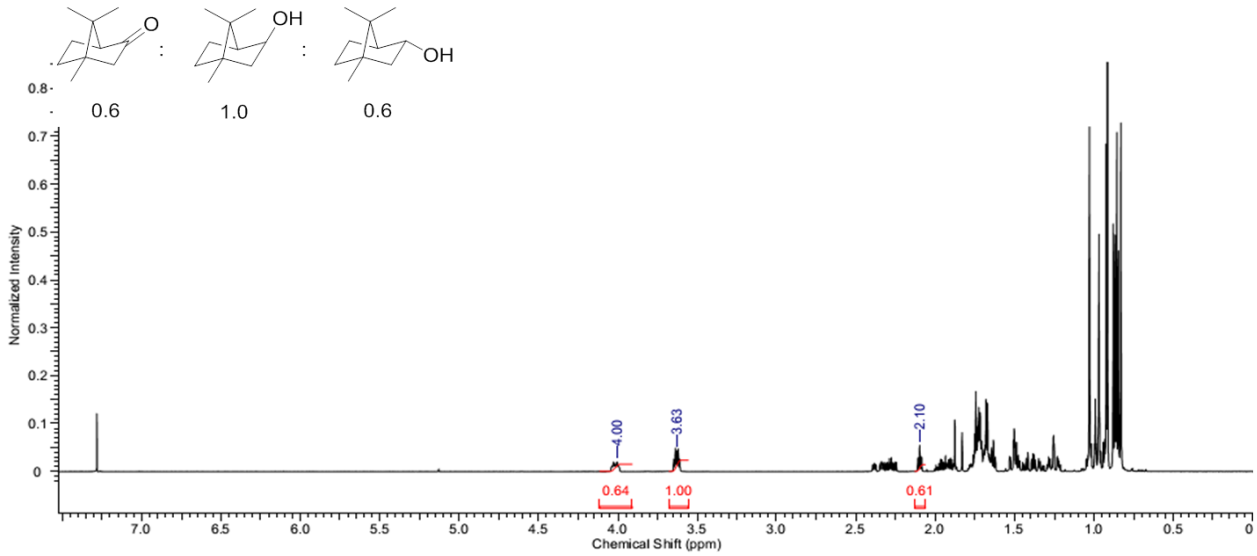


^{13}C-NMR (25 °C)

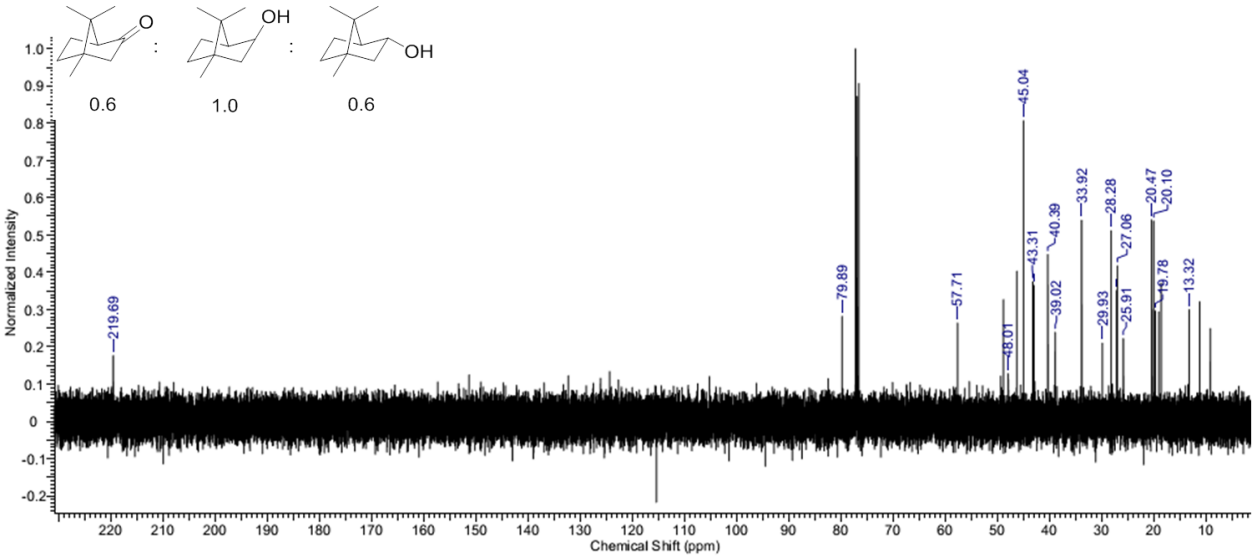


Isoborneol/borneol (31).[6]

^{1}H-NMR (25 °C)

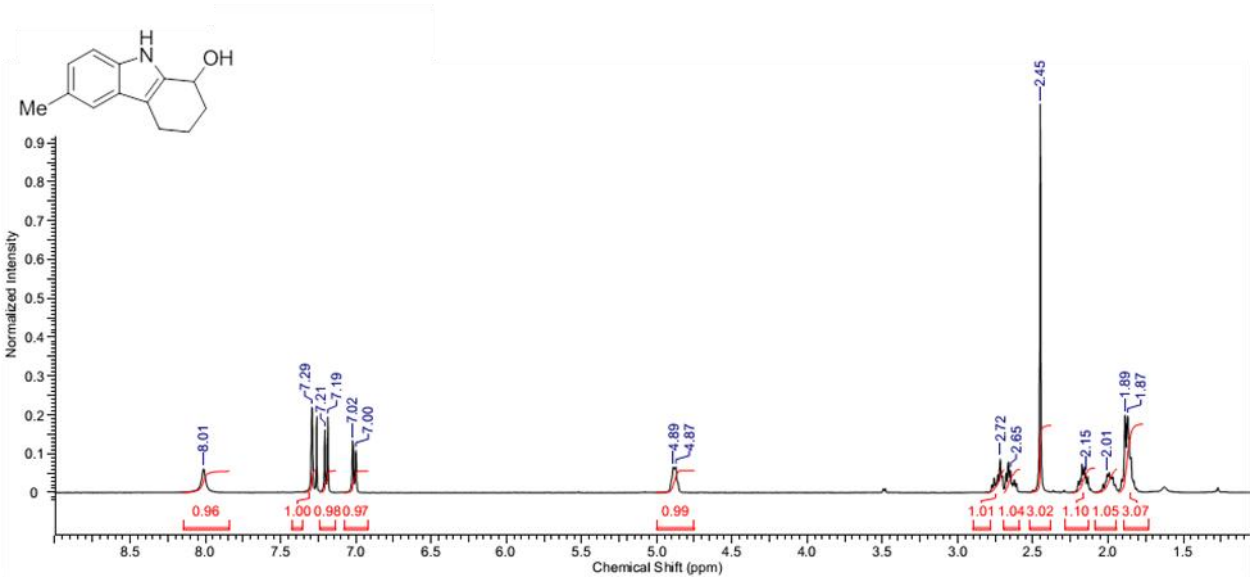


^{13}C-NMR (25 °C)

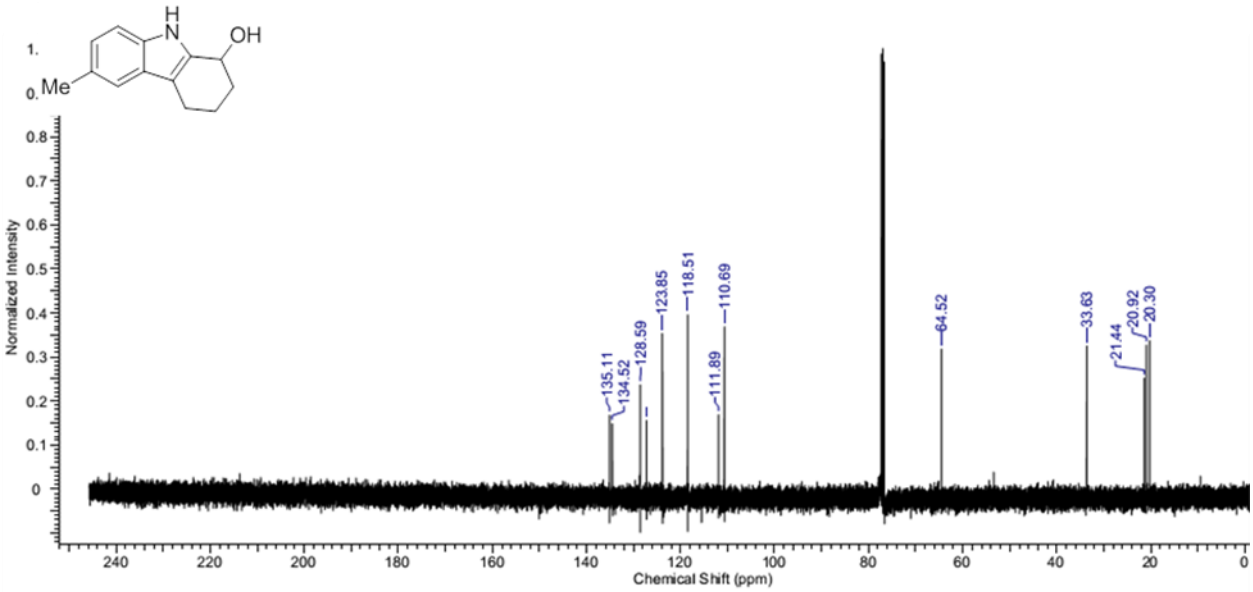


(±)6-Methyl -2,3,4,9-tetrahydro-1*H*-carbazol-1-ol (32).

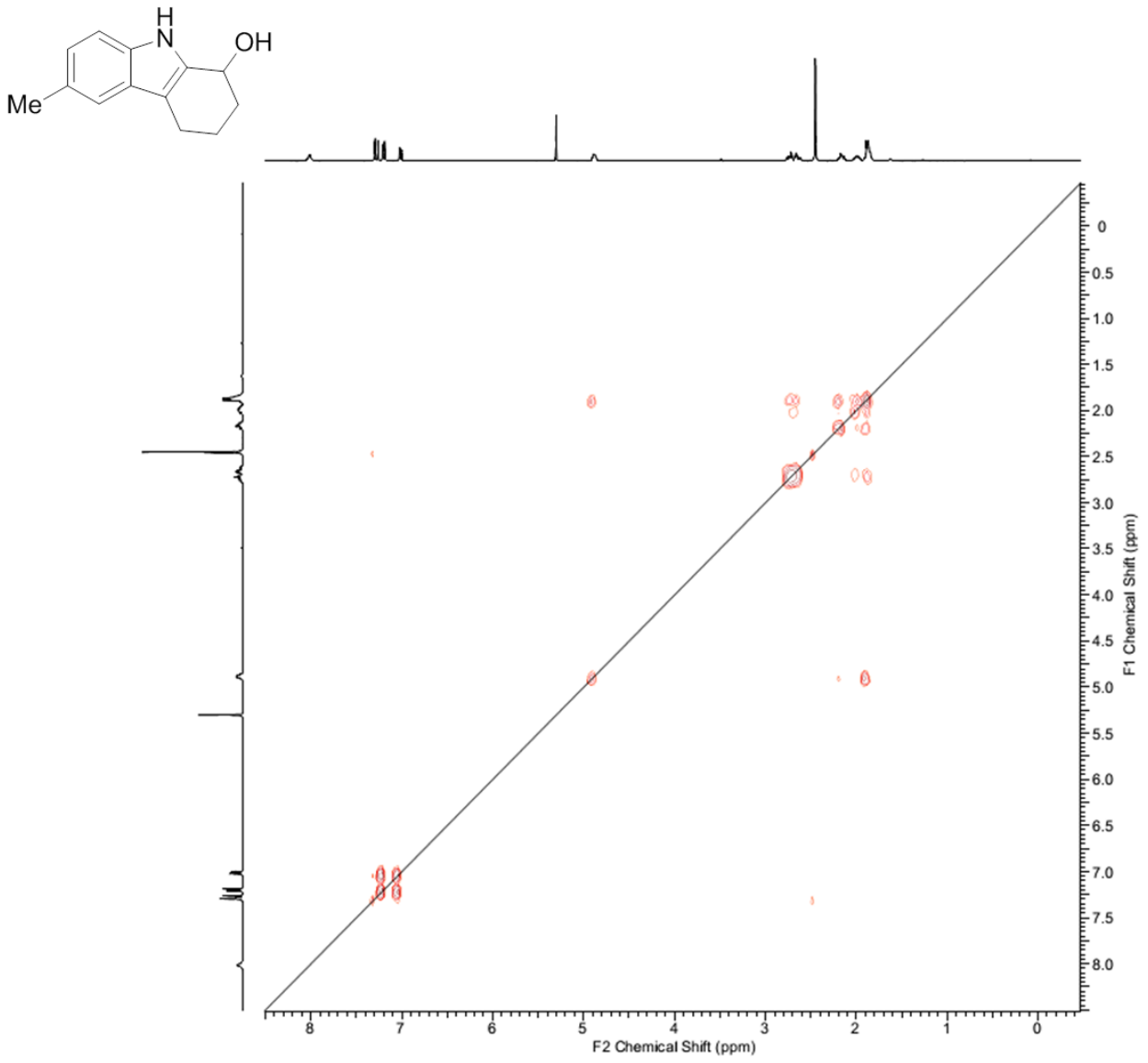
^{1}H-NMR (25 °C)



^{13}C-NMR (25 °C)

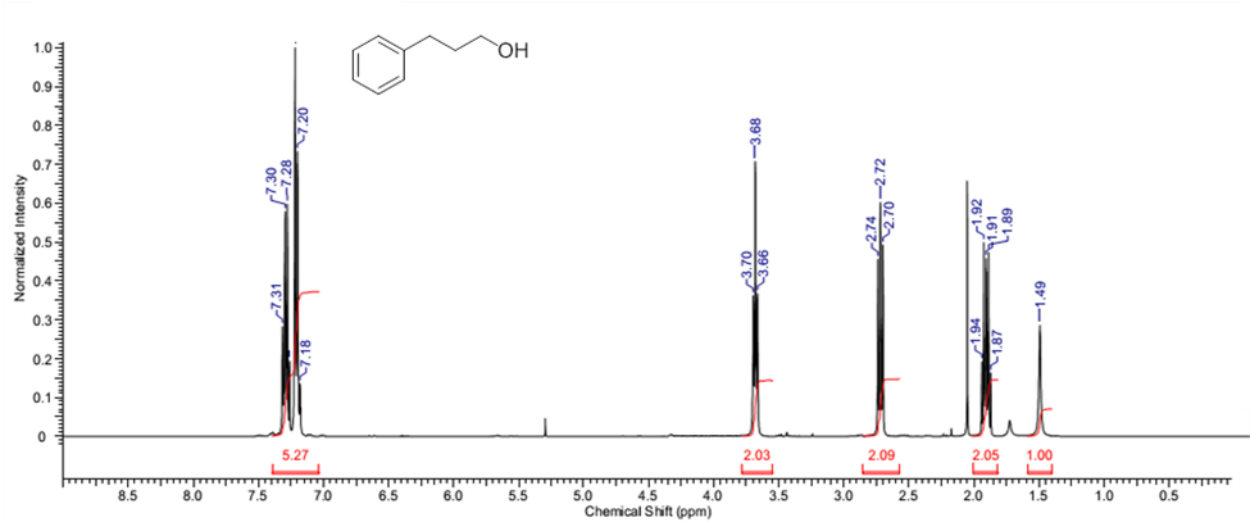


COSY-NMR (25 °C).

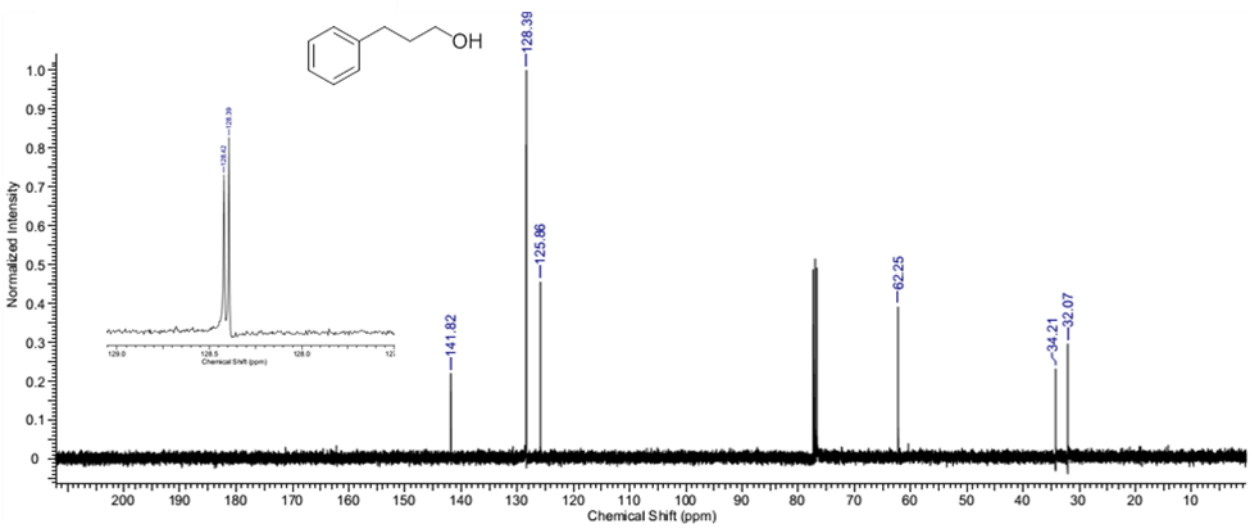


3-Phenyl propanol (33).

^{1}H-NMR (25 °C)

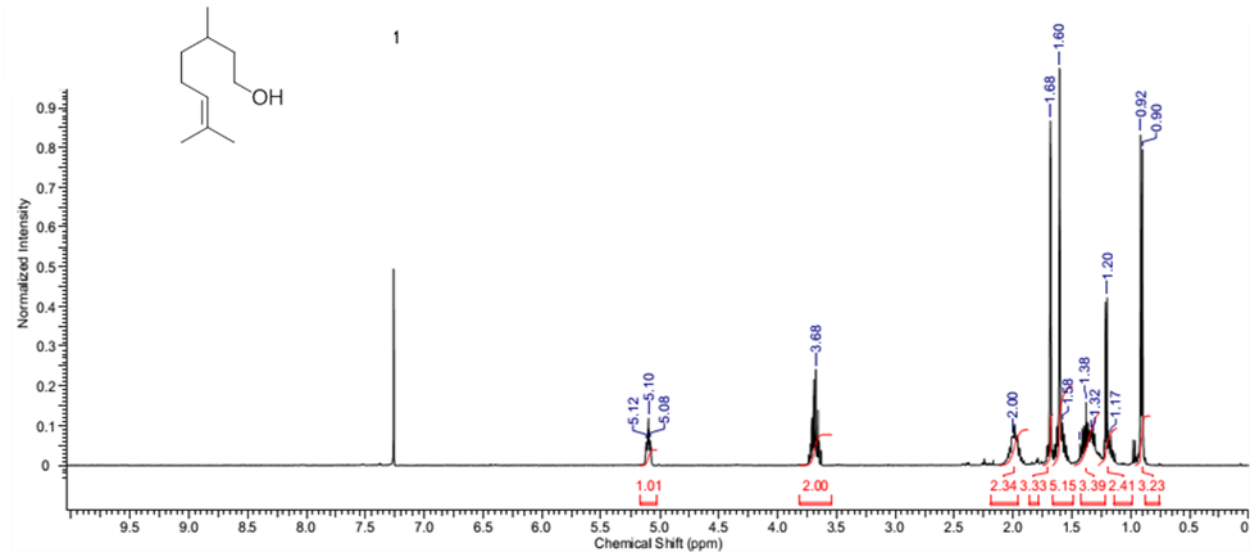


^{13}C-NMR (25 °C)

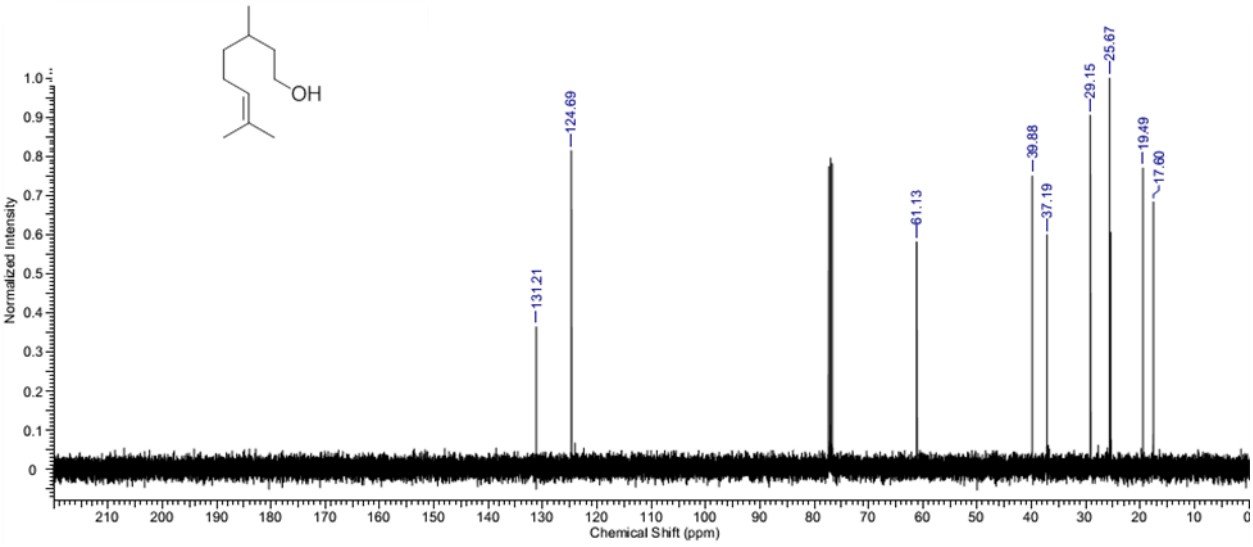


(±)Citronellol (34).

^{1}H-NMR (25 °C)

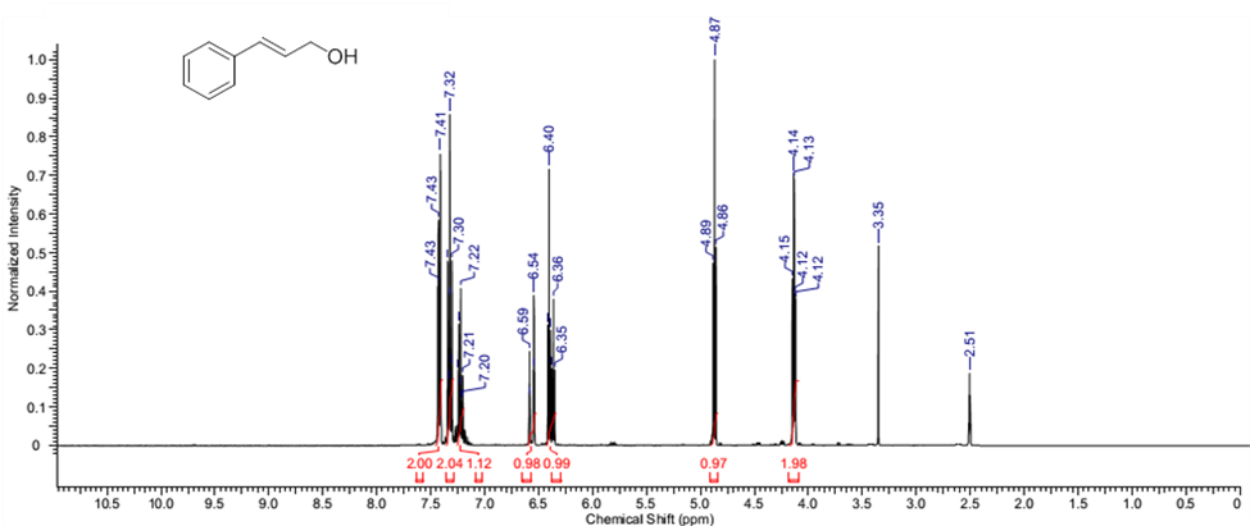


^{13}C-NMR (25 °C)

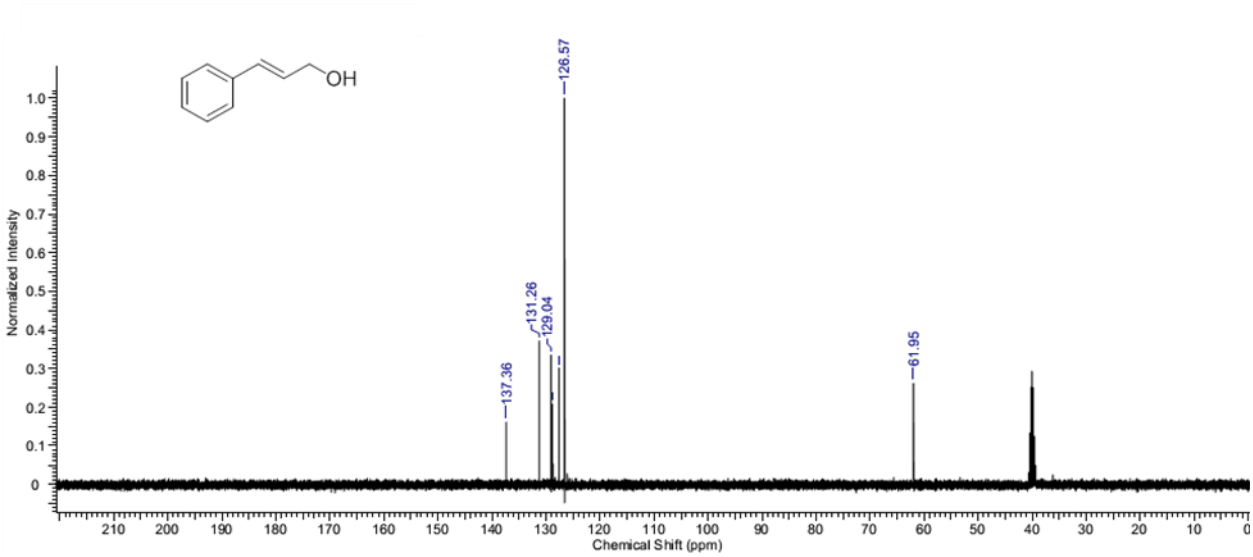


(*E*)-Cinnamyl alcohol (35).

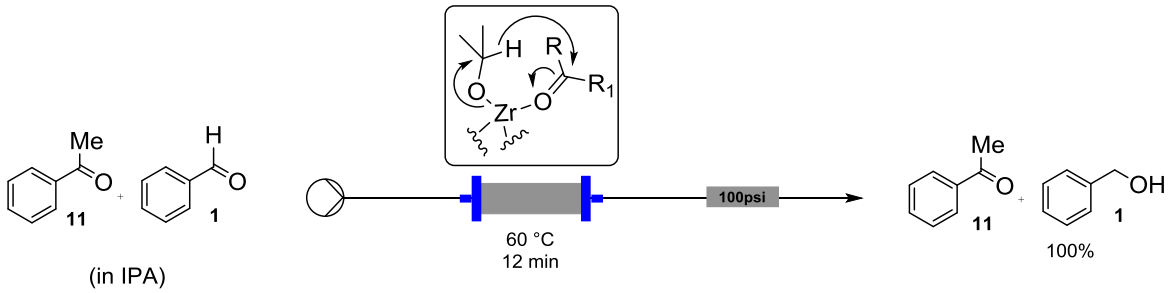
^{1}H-NMR (25 °C)

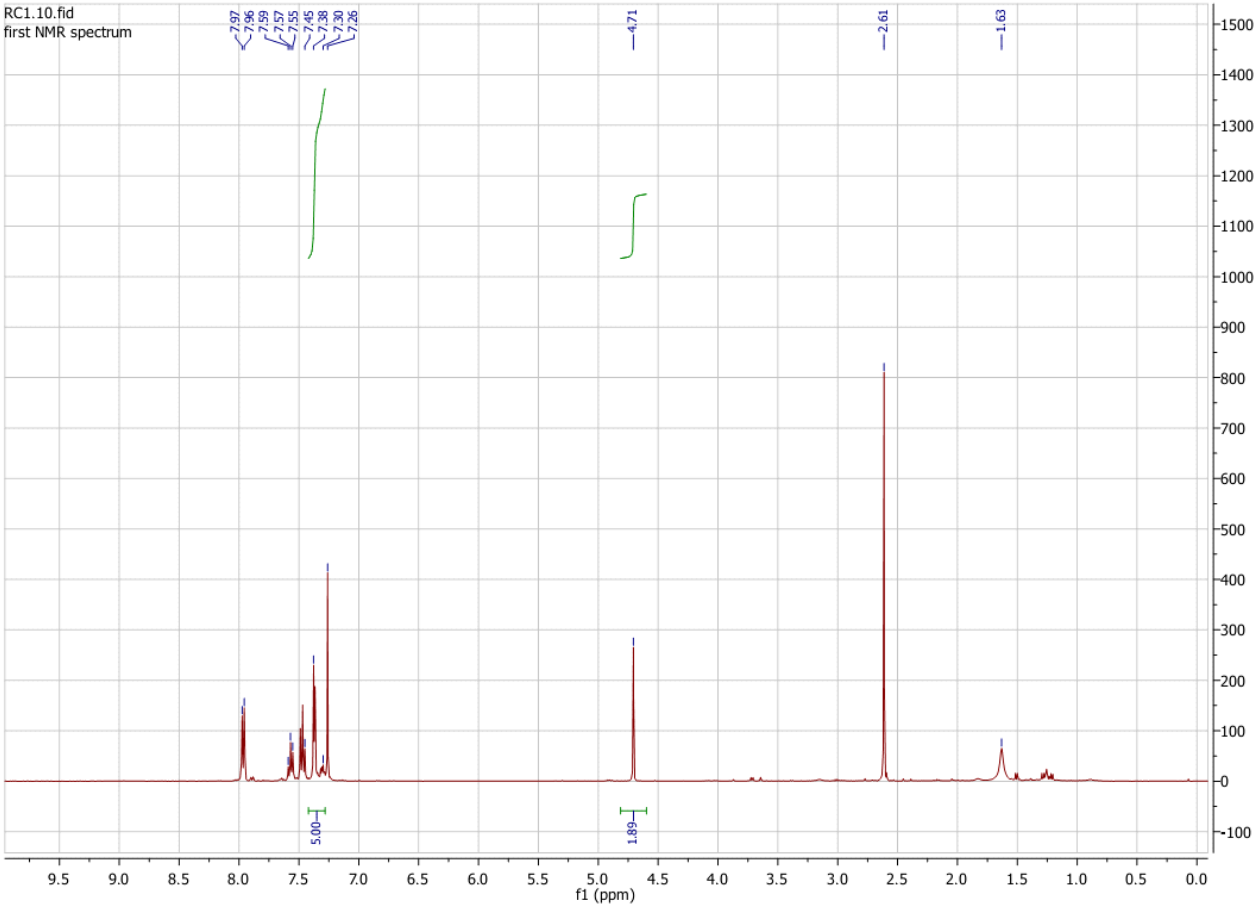


^{13}C-NMR (25 °C)



Selective reduction of benzaldehyde over acetophenone (^{1}H-NMR).





Mercury porosimetry measurements. Mercury porosimetry intrusion measurements were collected using an AutoporeIV instrument (Micromeritics).[7]

Summary Report

Penetrometer parameters

Penetrometer:	0166 - (14) 3 Bulb, 0.412 Stem, Powder		
Pen. Constant:	10.790 µL/pF	Pen. Weight:	62.7371 g
Stem Volume:	0.4120 mL	Max. Head Pressure:	4.6800 psia
Pen. Volume:	3.1233 mL	Assembly Weight:	98.5182 g

Hg Parameters

Adv. Contact Angle:	130.000 degrees	Rec. Contact Angle:	130.000 degrees
Hg Surface Tension:	485.000 dynes/cm	Hg Density:	13.5335 g/mL

User Parameters

Param 1:	0.000	Param 2:	0.000	Param 3:	0.000

Low Pressure:

Evacuation Pressure:	50 µmHg
Evacuation Time:	5 mins
Mercury Filling Pressure:	1.02 psia
Equilibration Time:	40 secs

High Pressure:

Equilibration Time:	40 secs

Blank Correction Sample: C:\9500\DATA\BLANKS\BLNK0166.SMP
Blank Correction ID: No Sample

(From Pressure 0.10 to 30000.00 psia)

Intrusion Data Summary

Total Intrusion Volume =	0.7448	mL/g
Total Pore Area =	21.508	m²/g
Median Pore Diameter (Volume) =	230.9	nm
Median Pore Diameter (Area) =	48.9	nm
Average Pore Diameter (4V/A) =	138.5	nm
Bulk Density at 1.02 psia =	0.9512	g/mL
Apparent (skeletal) Density =	3.2629	g/mL
Porosity =	70.8469	%
Stem Volume Used =	89	%

Pore Structure Summary

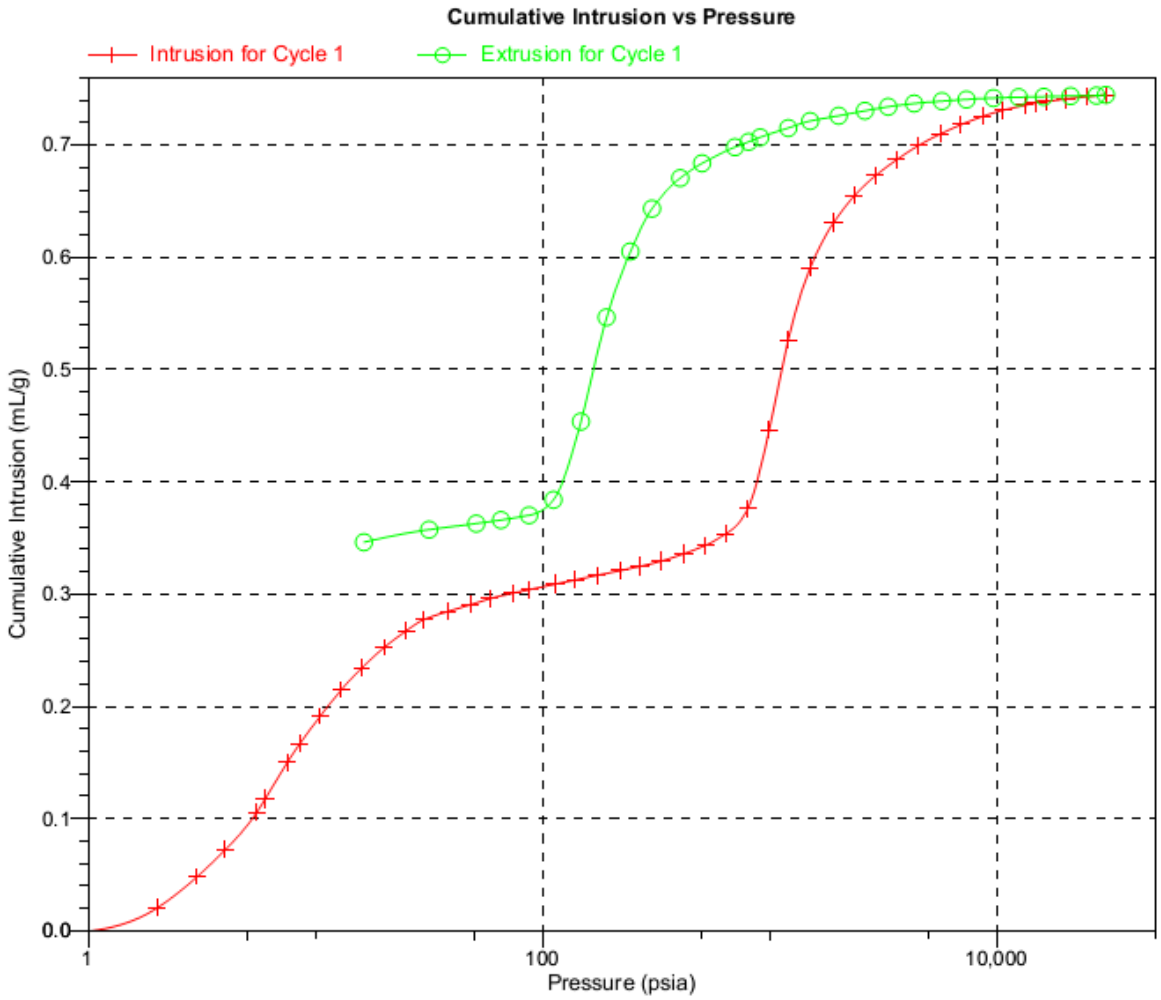
Threshold Pressure:	2.39	psia (Calculated)
Characteristic length =	75728.9	nm
Conductivity formation factor =	0.031	
Permeability constant =	0.00442	
Permeability =	796.7810	mdarcy
BET Surface Area =	230.0000	m²/g
Pore shape exponent =	1.00	
Tortuosity factor =	1.678	
Tortuosity =	3.5896	
Percolation Fractal dimension =	2.931	
Backbone Fractal dimension =	2.864	

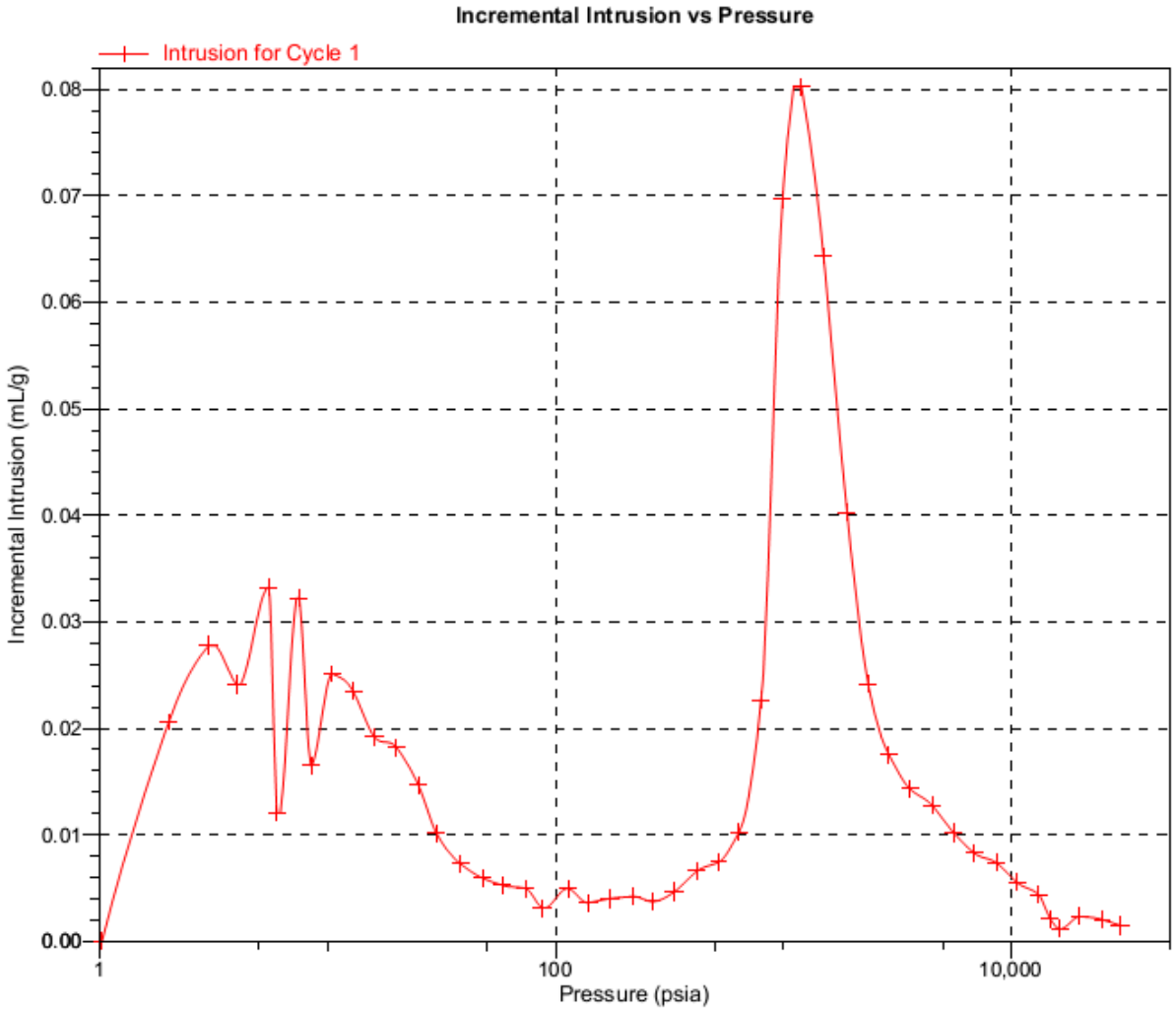
Mayer Stowe Summary

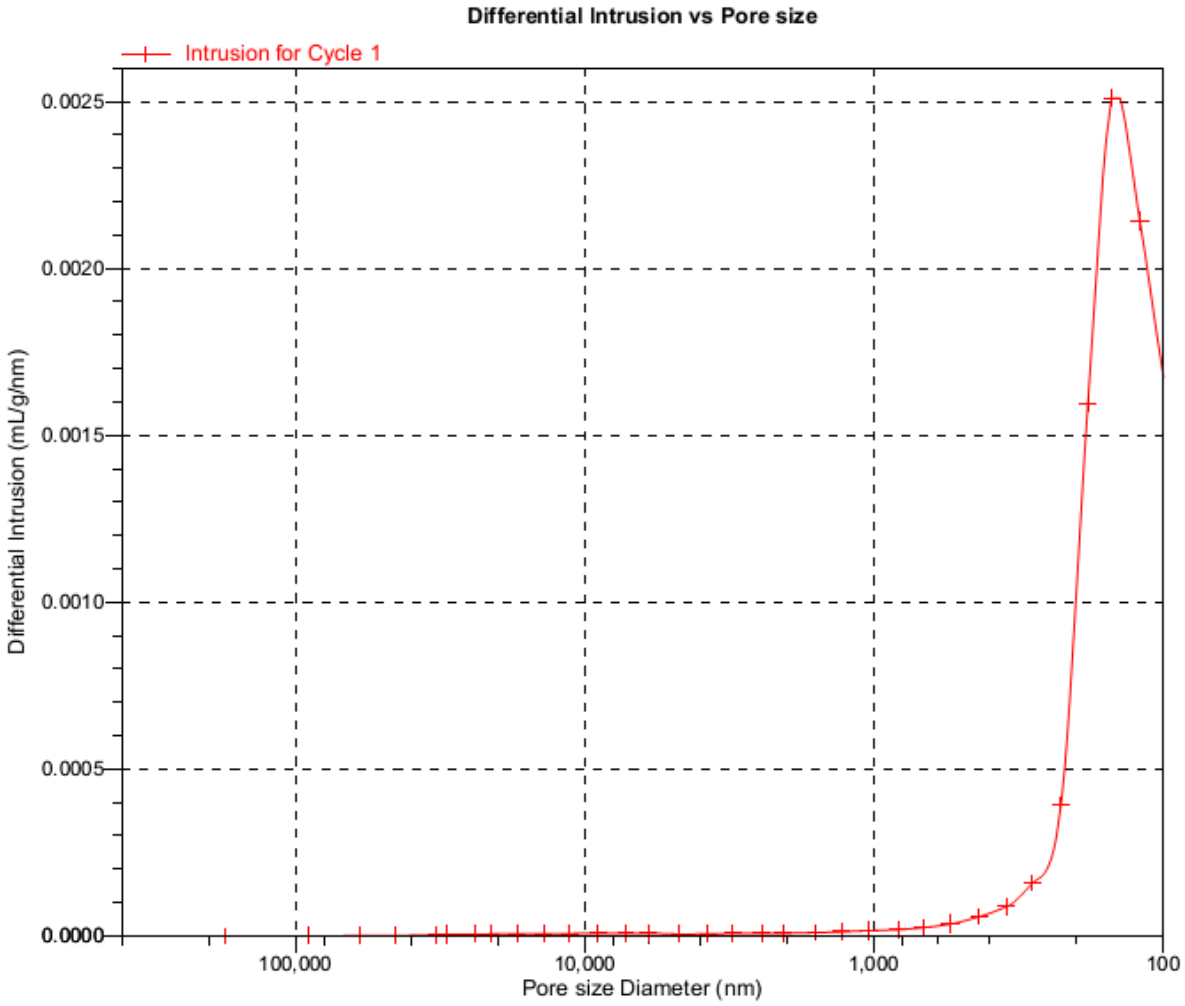
Interstitial porosity =	47.6300	%
Breakthrough pressure ratio =	3.3512	

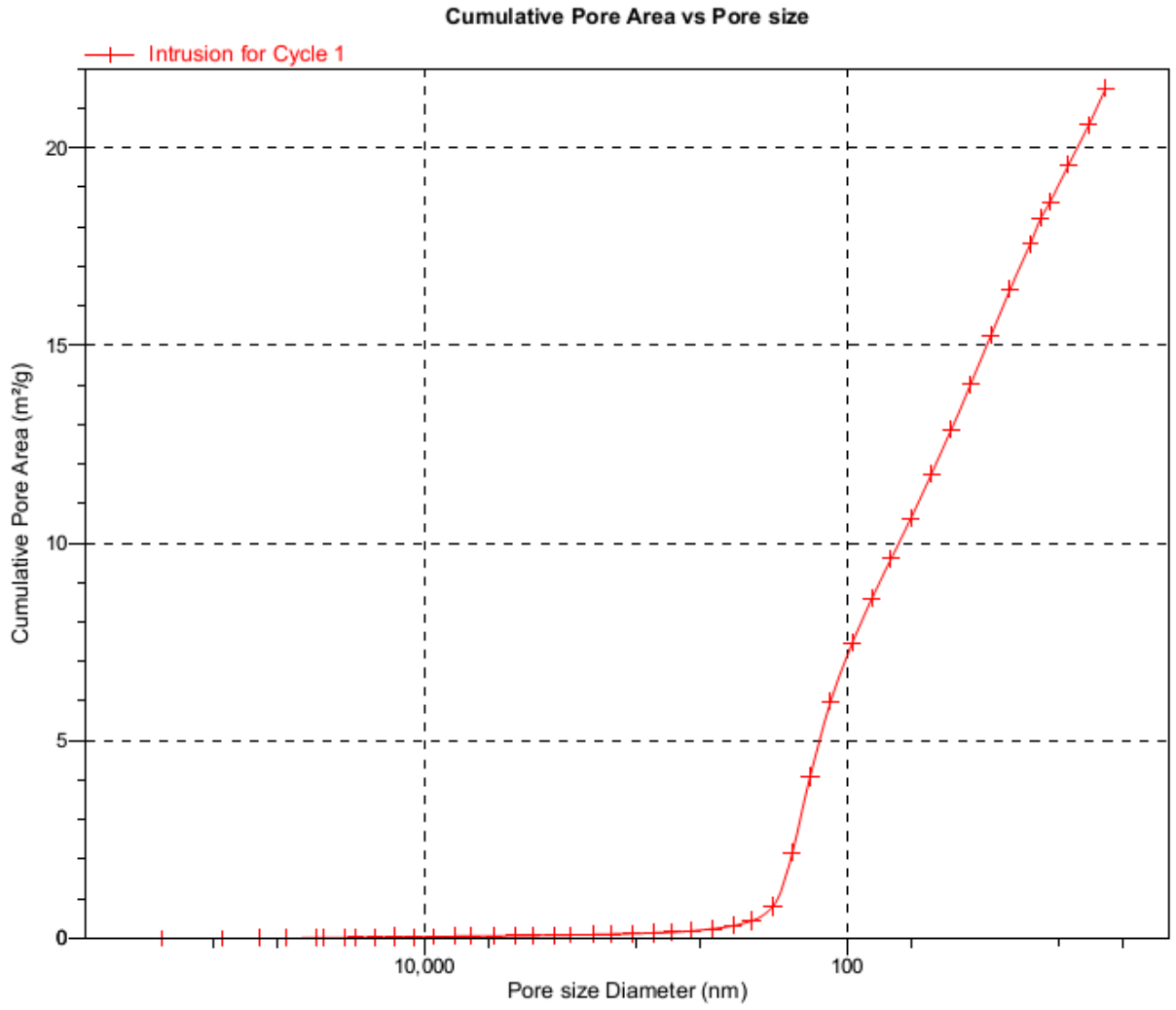
Material Compressibility

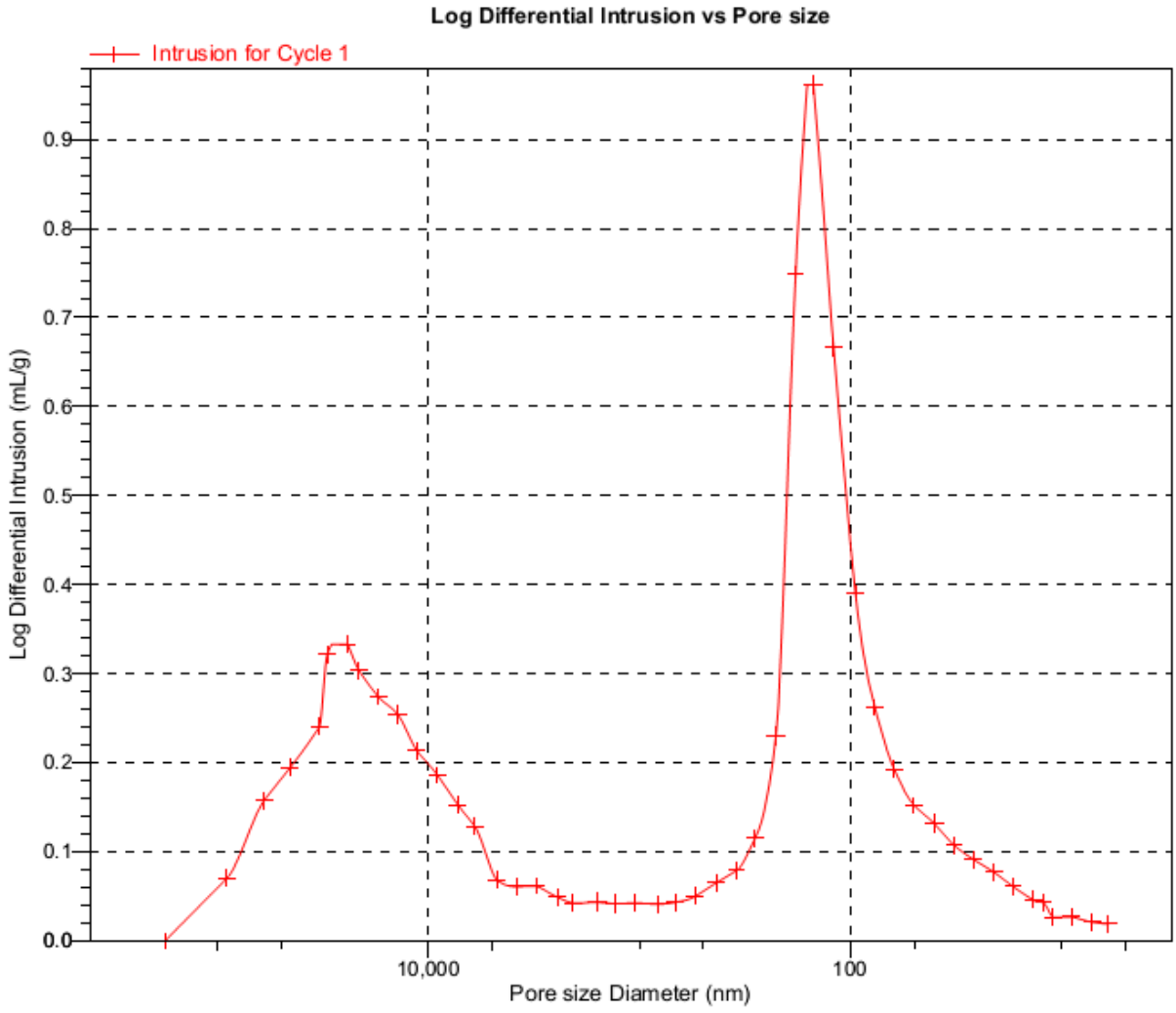
Linear Coefficient =	-8.8650e-02	1/psia
Quadratic Coefficient =	2.0642e-03	1/psia²

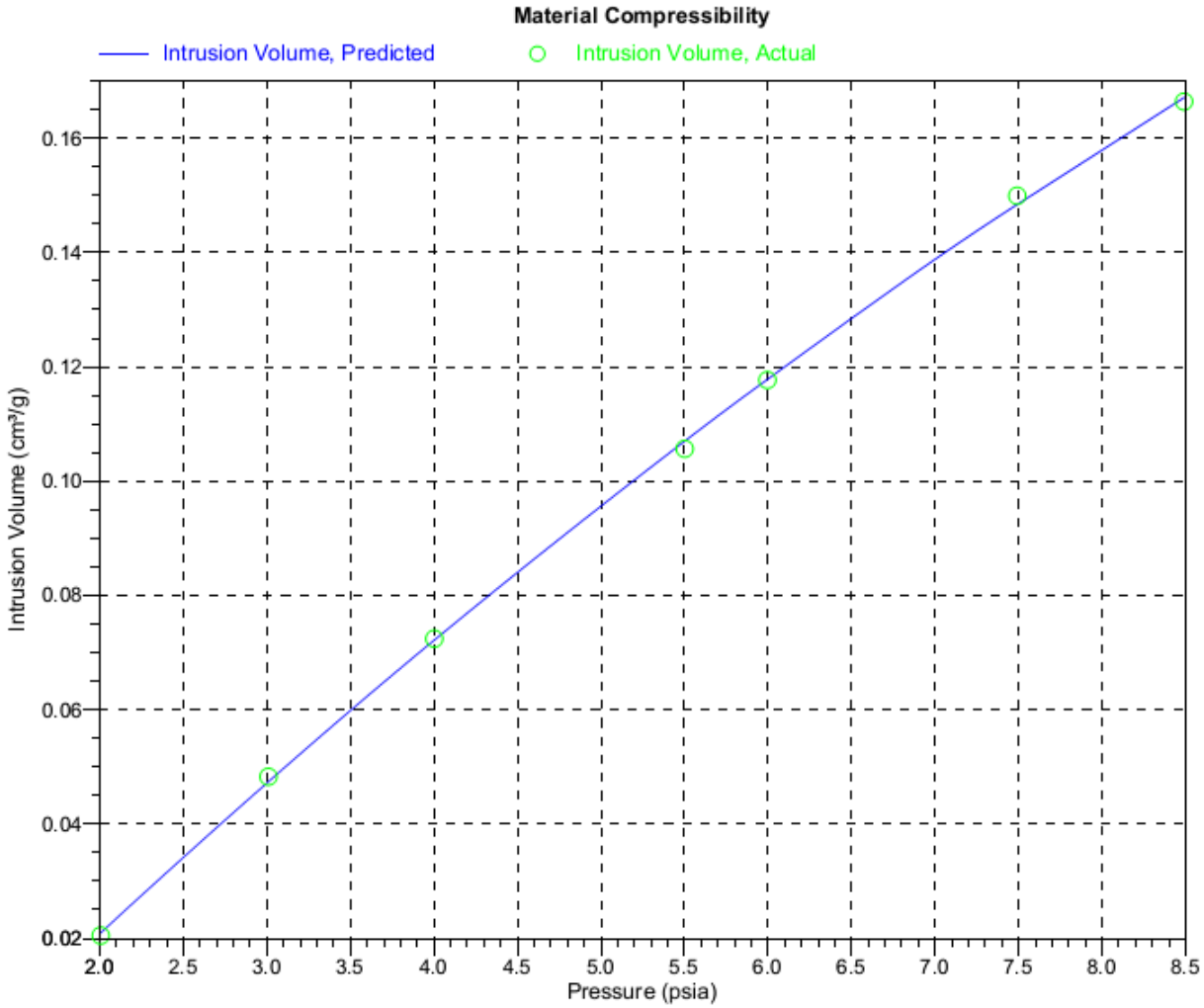

Cumulative Intrusion vs Pressure
Intrusion for Cycle 1
Extrusion for Cycle 1
Cumulative Intrusion (mL/g)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
1
100
10,000
Pressure (psia)


Incremental Intrusion vs Pressure
Intrusion for Cycle 1
Incremental Intrusion (mL/g)
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0.00
1
100
10,000
Pressure (psia)


Differential Intrusion vs Pore size
Intrusion for Cycle 1
Differential Intrusion (mL/g/nm)
0.0025
0.0020
0.0015
0.0010
0.0005
0.0000
100,000
10,000
1,000
100
Pore size Diameter (nm)


Cumulative Pore Area vs Pore size
Intrusion for Cycle 1
Cumulative Pore Area (m²/g)
20
15
10
5
0
10,000
100
Pore size Diameter (nm)


Log Differential Intrusion vs Pore size
Intrusion for Cycle 1
Log Differential Intrusion (mL/g)
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
10,000
100
Pore size Diameter (nm)


Material Compressibility
Intrusion Volume, Predicted
Intrusion Volume, Actual
Intrusion Volume (cm³/g)
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
Pressure (psia)

BET measurements. BET measurements were collected using a Tristar 3000 apparatus (Micromeritics).[7]

Summary Report

Surface Area

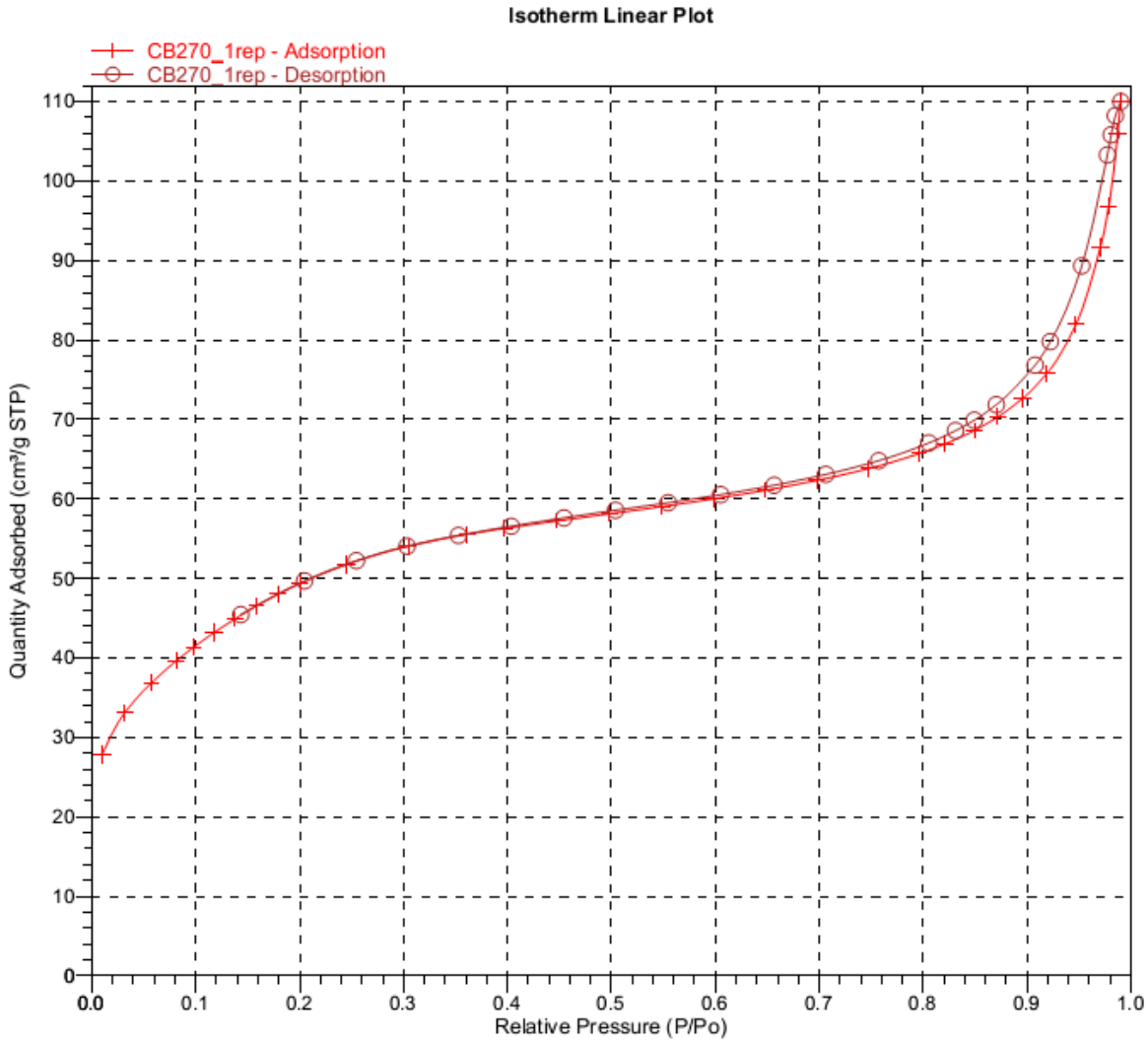
Single point surface area at P/Po = 0.200184774:	171.8769 m²/g
BET Surface Area:	177.1550 m²/g
Langmuir Surface Area:	228.2472 m²/g
t-Plot Micropore Area:	9.3770 m²/g
t-Plot External Surface Area:	167.7780 m²/g
BJH Adsorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm diameter:	98.4617 m²/g
BJH Desorption cumulative surface area of pores between 1.7000 nm and 300.0000 nm diameter:	96.3523 m²/g

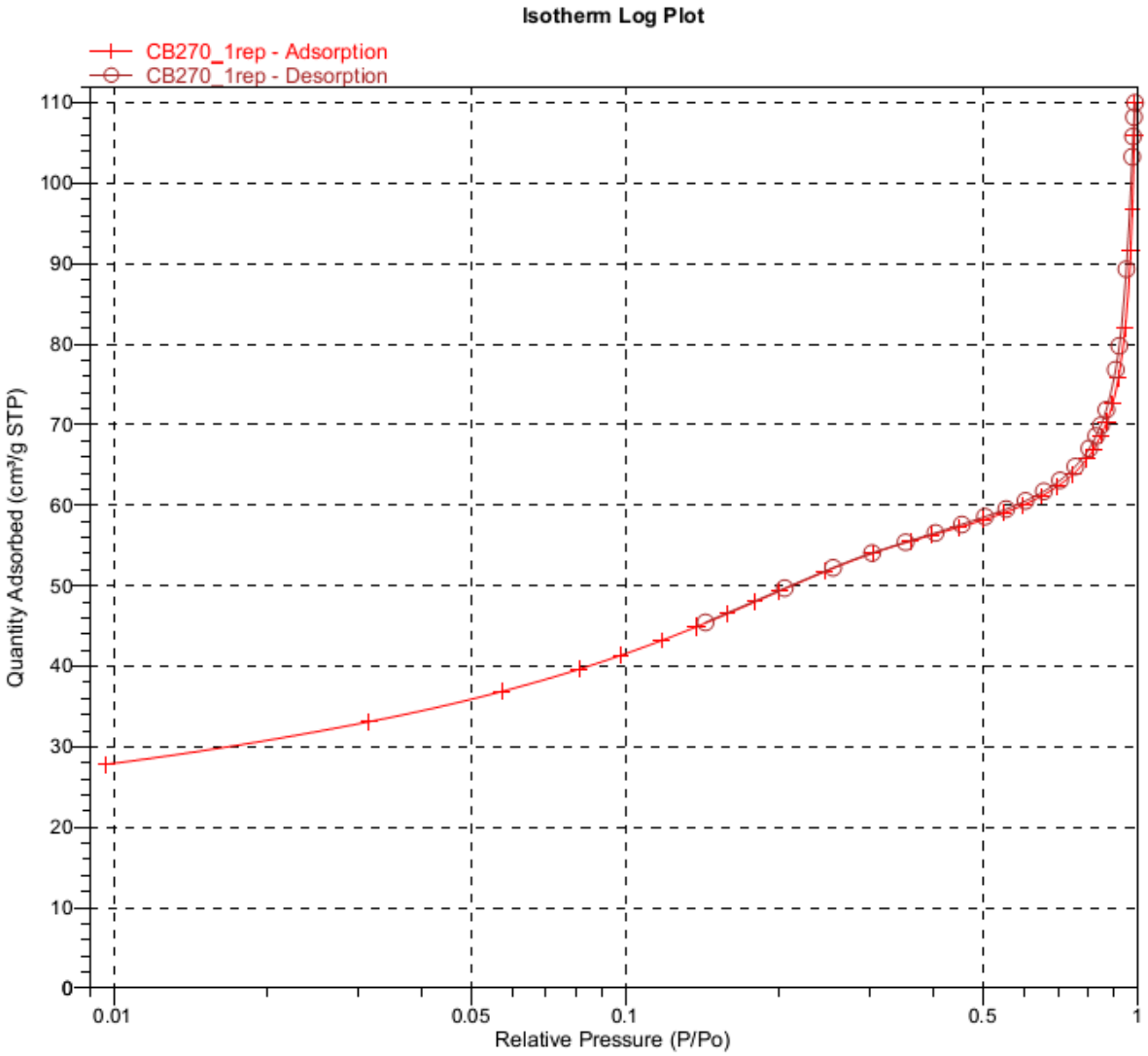
Pore Volume

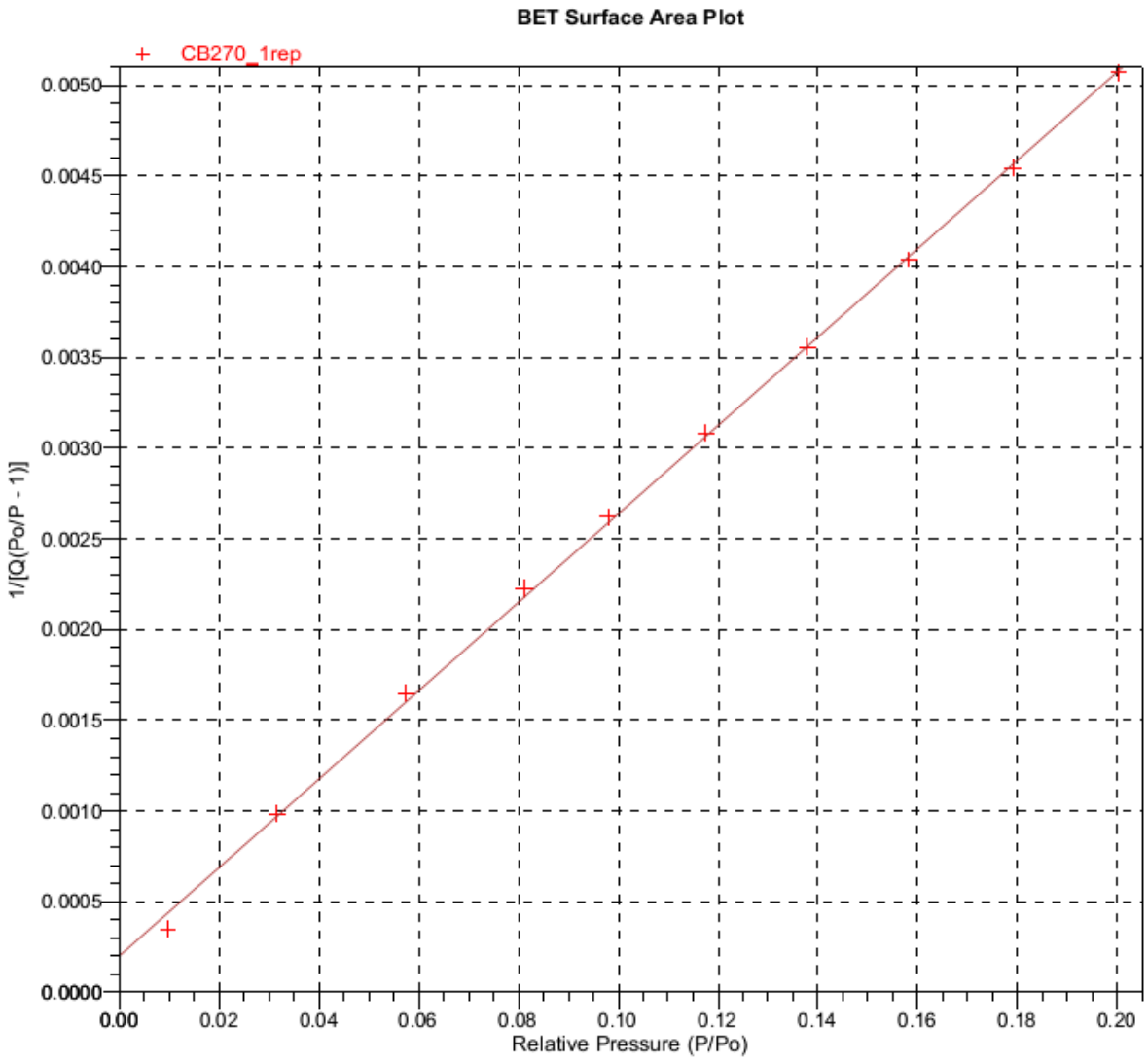
Single point adsorption total pore volume of pores less than 67.6006 nm diameter at P/Po = 0.970525083:	0.141804 cm³/g
t-Plot micropore volume:	0.002791 cm³/g
BJH Adsorption cumulative volume of pores between 1.7000 nm and 300.0000 nm diameter:	0.139658 cm³/g
BJH Desorption cumulative volume of pores between 1.7000 nm and 300.0000 nm diameter:	0.138460 cm³/g

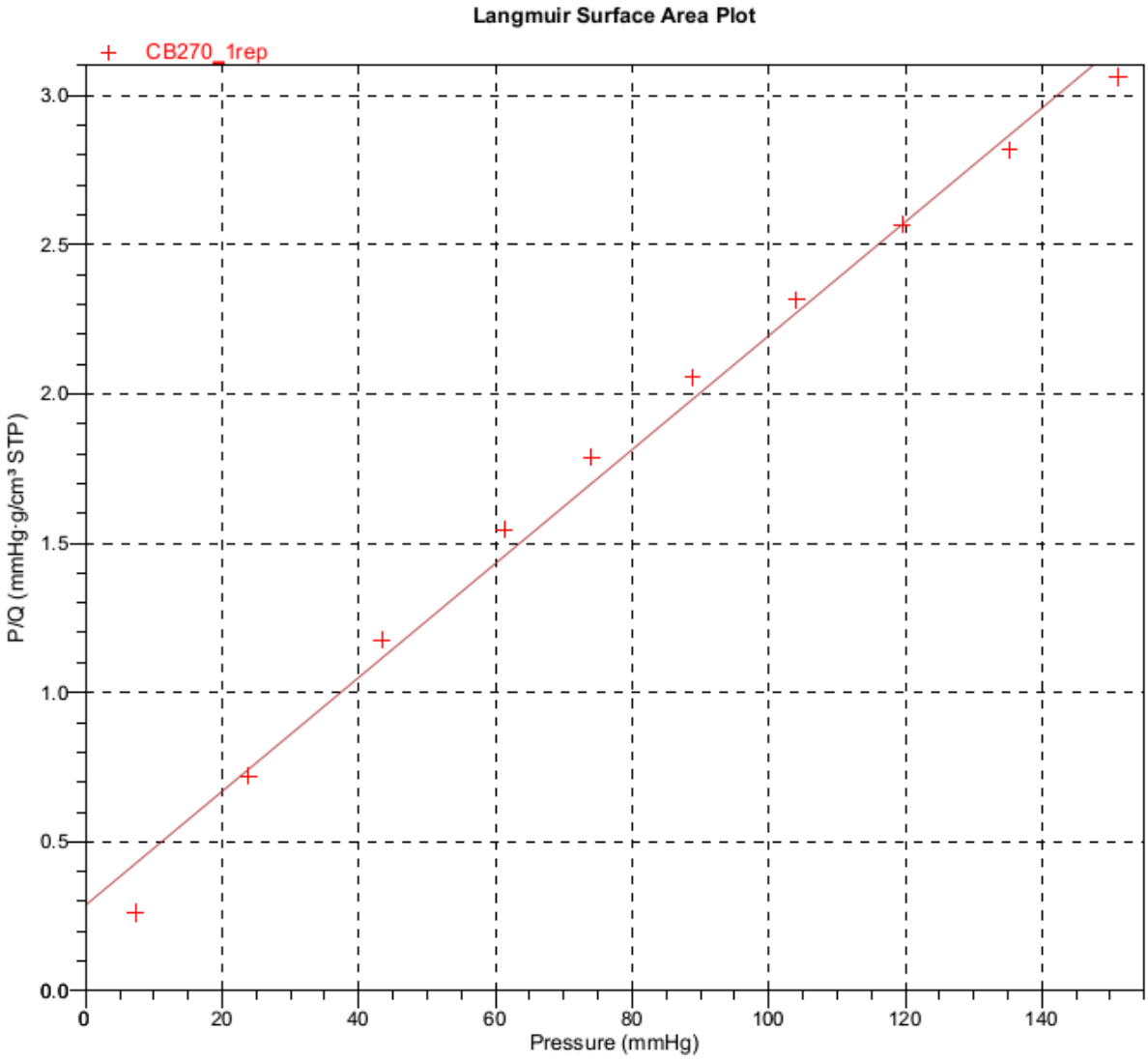
Pore Size

Adsorption average pore width (4V/A by BET):	3.20181 nm
BJH Adsorption average pore diameter (4V/A):	5.6736 nm
BJH Desorption average pore diameter (4V/A):	5.7481 nm


Isotherm Linear Plot
CB270_1rep - Adsorption
CB270_1rep - Desorption
Quantity Adsorbed (cm³/g STP)
Relative Pressure (P/Po)
0
10
20
30
40
50
60
70
80
90
100
110
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0


Isotherm Log Plot
CB270_1rep - Adsorption
CB270_1rep - Desorption
Quantity Adsorbed (cm³/g STP)
Relative Pressure (P/Po)
0
10
20
30
40
50
60
70
80
90
100
110
0.01
0.05
0.1
0.5
1


BET Surface Area Plot
CB270_1rep
1/[Q(Po/P - 1)]
0.0050
0.0045
0.0040
0.0035
0.0030
0.0025
0.0020
0.0015
0.0010
0.0005
0.0000
0.00
0.02
0.04
0.06
0.08
0.10
0.12
0.14
0.16
0.18
0.20
Relative Pressure (P/Po)


Langmuir Surface Area Plot
CB270_1rep
P/Q (mmHg·g/cm³ STP)
Pressure (mmHg)
0.0
0.5
1.0
1.5
2.0
2.5
3.0
0
20
40
60
80
100
120
140

References

1) http://www.vapourtec.co.uk/

2) 2-Propanol (puriss.) was purchased from Sigma-Aldrich (cod. 24137-2.5L). Webpage: http://www.sigmaaldrich.com/catalog/product/sial/24137?lang=en&region=GB

3) PS-TsNHNH$_2$ (benzenesulfonyl hydrazide, polymer-supported, 1.8-2.2 mmol/g on polystyrene) was purchased from Alfa Aesar (cod. L19466).
Webpage: http://www.alfa.com/it/GP100W.pgm?DSSTK=L19466

4) Zirconium hydroxide (zirconium(IV) hydroxide 97%) was purchased from Sigma Aldrich (cod. 464171). Webpage: http://www.sigmaaldrich.com/catalog/product/aldrich/464171?lang=en&region=GB

5) http://www.nist.gov/ncnr/drying-tube-buchi-to-51.cfm

6) Isoborneol and borneol ^{1}H-NMR, ^{13}C-NMR and melting point matched the literature.

7) http://micromeritics2.reachlocal.com/?scid=1229585&kw=7605560&pub_cr_id=6234969370