Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supplementary Information

An efficient method for regioselective ring opening of epoxides by amines under microwave irradiation using $Bi(NO_3)_3.5H_2O$ as catalyst

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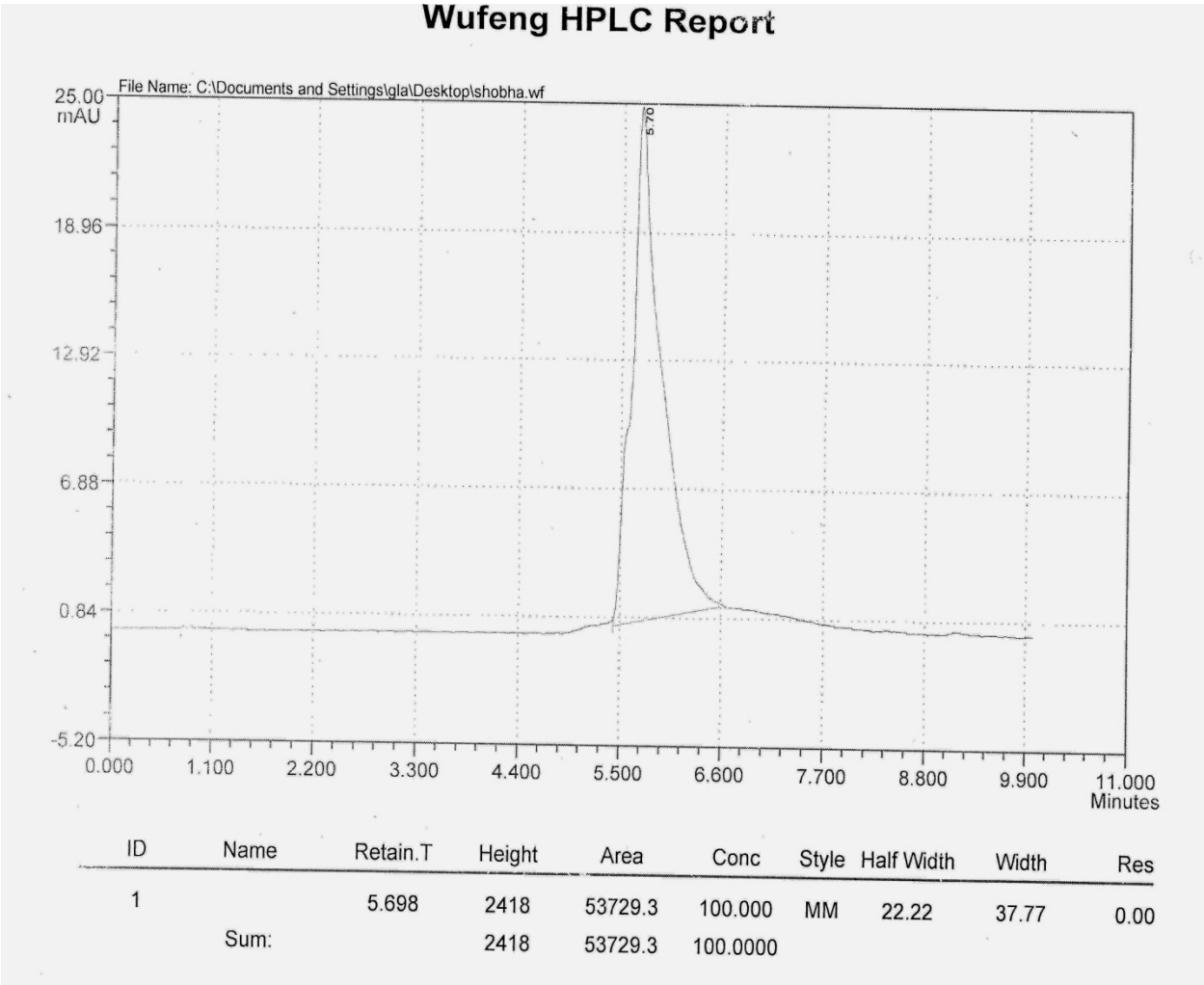

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Figure S1. HPLC spectrum of 2-(1H-imidazol-1-yl)-2-phenylethanol) Scheme 3

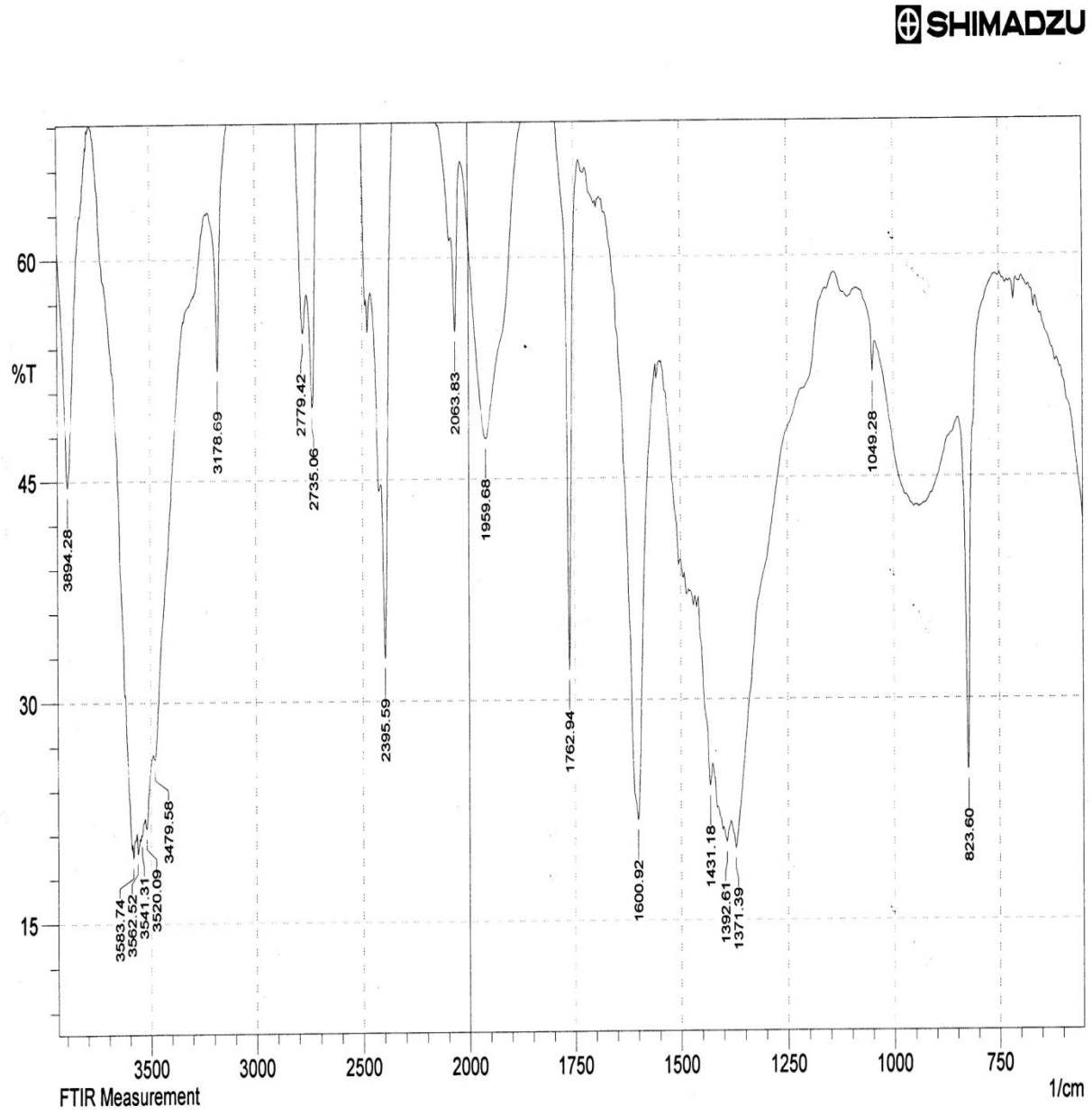

Figure S2. FTIR spectrum of Bismuth(III)nitrate pentahydrate

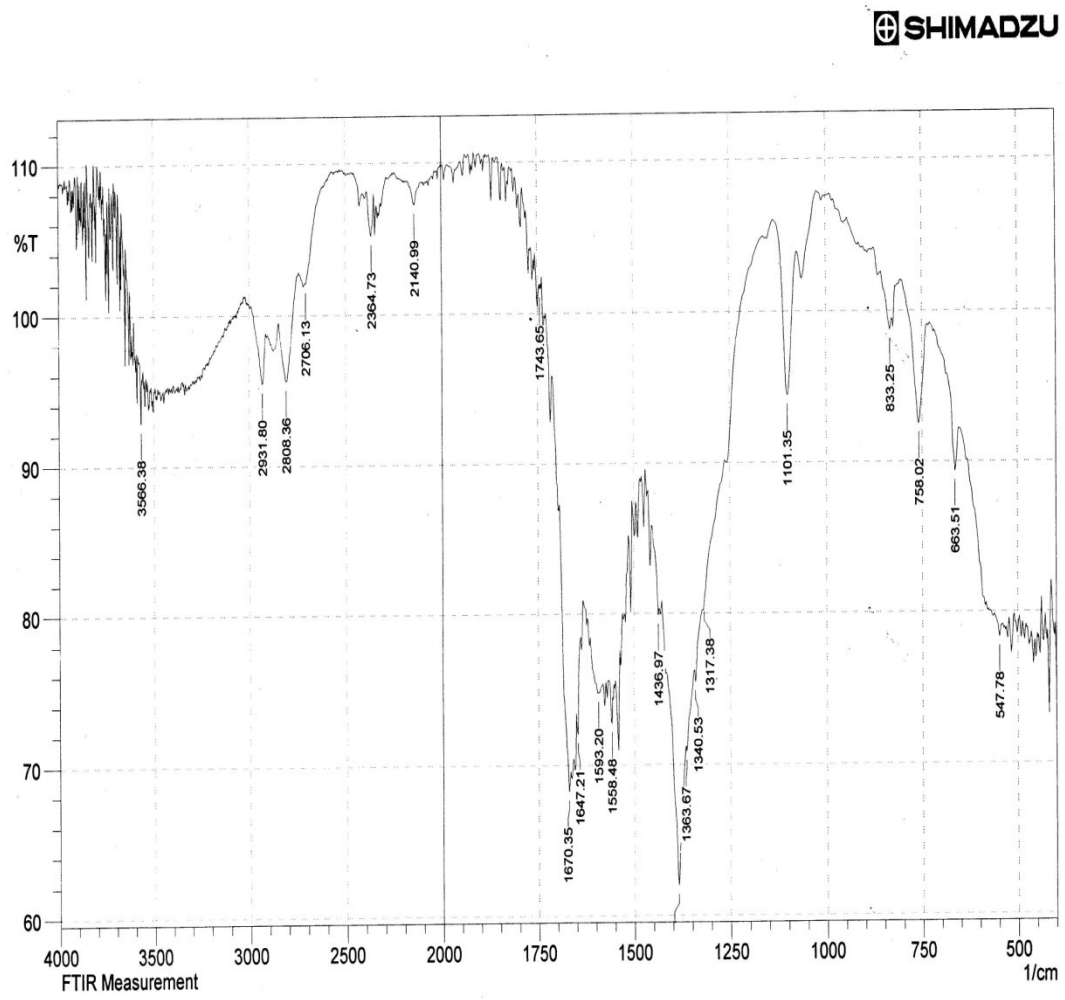

Figure S3. FTIR spectrum of Bismuth nitrate dehydrated (water molecule replaced by DMF molecule)

(2-phenyl-2-(phenylamino)ethanol), (Table 4, entry 1) Colourless Liquid, R_f = 0.46 (2:8 EtOAc/hexane); IR (KBr cm^{-1}): 2932, 2878, 2402, 1949, 1453, 1361, 1216, 1066, 1028, 926, 756. ^{1}H NMR (300 MHz, CDCl3): 7.39-7.05(m, 5H, ArH); 7.12-7.05(m, 2H); 6.60(t, *J*=8.7Hz,1H); 6.50(d, *J*=8.7Hz, 1H); 4.48(dd, *J*=4.2Hz,7.2Hz, 1H); 3.92(dd, *J* = 4.2,11.1Hz, 1H); 3.74(dd, *J* = 6.9Hz,11Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): 147.2, 140.2, 129.4, 127.8, 126.9, 126.0, 118.2, 114.2, 67.4. Mass ESI m/z (%) M^++1=214; 196(90%).

2-(4-methylpiperazine-1-yl)-2-phenylethanol) (Table 4, entry 2) Liquid, R_f = 0.35 (2:8 EtOAc/hexane); IR(KBr, cm^{-1}): 3787, 2944, 2805, 1363, 1215, 1060, 928, 702. ^{1}H NMR (300 MHz, $CDCl_3$):7.28(m, 5H, Ar); 3.95(t, 1H); 3.65(m,2H,CH_2); 2.7(s, -OH); 2.50(m,-CH_2); 2.31(s,CH3) ^{13}C NMR (75MHz, $CDCl_3$): 142, 136, 127, 68, 66, 60, 55, 49, 45. Mass ESI m/z (%) M^++ CH_3COOH= 281.

(2-phenyl-2-(pyrolodin-1-yl)ethanol), (Table 4, entry 3) Viscous Liquid, R_f = 0.56 (4:6 EtOAc/hexane); ^{1}H NMR (300 MHz, $CDCl_3$): 7.42-7.23(m, 3H); 3.93-3.81(dd, 1H); 3.23 (brs, -OH, 1H); 2.87-2.81(dd, 1H); 2.64-2.52(dd, 1H); 2.04-1.71(m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): 142.5, 138.7, 128.7, 128.2, 126.8, 70.7, 70.2, 64.3, 54.1, 51.6, 23.8, 23.3. Mass ESI m/z (%) M^++1= 192.

2-(4-methylpiperidin-1-yl)2-phenylethanol) (Table 4, entry 4) Liquid, R_f=0.38 (2:8 EtOAc/hexane); ^{1}H NMR (300 MHz, $CDCl_3$): 7.27(m, 5H, ArH); 4.7(1H,dd, *J*=4.2,9.9, -CH) ; 3.12(s,-OH); 2.65(t,2H,-CH_2); 2.38(t,2H,-CH_2); 2.0(t,2H,-CH_2); 1.28(-CH_2); 0.90(s,-CH_3). 13 C NMR (75MHz, $CDCl_3$): 142.2, 128.4, 127.9, 126.8, 70.1, 63.9, 46.4, 34.7, 30.9, 22.0. Mass ESI m/z (%) M^+= 220

(2-(diisopropylamino)-2-phenylethanol) (Table 4, entry 5) viscous liquid, R_f =0.46(1:9 EtOAc/hexane); ^{1}H NMR (300 MHz, $CDCl_3$): 7.4-7.2(m, 5H, Ar); 4.7(dd, *J*=2.7Hz,7.8Hz 1H); 3.7-3.3(m, 3H). ^{13}C NMR (75MHz, $CDCl_3$): 140.7, 139.1, 138.1, 128.6, 128.3, 127.8, 126.8, 126.2, 84.6, 83.1, 79.4, 68.4, 67.2. Mass ESI m/z (%) M^++AcOH= 281, M^+= 221(100%).

(2-(dipropylamino)-2-phenylethanol) (Table 4, entry 6) Liquid, R_f = 0.55 (4:6 EtOAc/hexane); IR(KBr, cm^{-1}): 3399, 2962, 2401, 1390, 1216, 1029, 757, 669. ^{1}H NMR (300 MHz, $CDCl_3$): 7.2(m, 5H, ArH); 4.5(brs, OH); 3.9(t,-CH); 3.61(dd, J=2.7, 2H); 2.5(m,-CH_2); 2.20(m, -CH_2); 1.56(m,-CH3). ^{13}C NMR (75MHz, $CDCl_3$): 142.6, 127.9, 69.4, 64.1, 60.5, 56.2, 51.8, 21.0, 12.0; Mass ESI m/z (%) M^++1=222.

(2-(naphthalene-1-ylamino)-2-phenylethanol), (Table 4, entry 7) Liquid, R_f = 0.70 (1:4 EtOAc/hexane); IR (KBr, cm^{-1}): 3410, 3018, 2931, 2402, 1583, 1479, 1407, 1283, 1216, 1067, 759. ^{1}H NMR (300 MHz, $CDCl_3$): 8.0-8.3(m, 1H, ArH); 7.75-7.79 (m, 1H, ArH);7.12-7.50(m, 12H, ArH, NH); 6.36(d, 1H, *J*=8.4Hz); 4.65 (1H, dd, *J*=4.2Hz-6.9Hz, 1H); 4.05(dd, *J*=4.2Hz,11Hz, 1H); 3.87(dd, *J*=6.9Hz,10.9Hz, 1H); 3.45(s,1H,-OH). ^{13}C NMR (75MHz, $CDCl_3$): 142.3, 140.0, 134.4, 128.9, 127.8, 127.2, 126.8, 126.6, 125.9, 125.0, 124.0, 120.2, 118.0, 106.7, 67.7, 60.1. Mass ESI m/z (%) M^++1 = 264.

(2-phenyl-2-(o-tolylamino)ethanol). (Table 4, entry 8) Thick viscous liquid, R_f = ^{1}H NMR (300 MHz, $CDCl_3$): 7.22-7.36(m, 5H, ArH); 7.05(d, *J*=7.8Hz, 1H); 6.9(t, *J*=16.8Hz, 1H); 6.6(t, *J*= 15.6Hz, 1H); 6.37(d, *J*=7.8Hz, 1H); 4.5(dd, *J*=4.2Hz,7.2Hz, 1H); 3.9(dd, *J*=4.2Hz,6Hz, 1H), 3.7(dd, *J*=6.9Hz,12Hz, 1H); 2.2(s, 3H); 2.0(brs, 1H, OH). ^{13}C NMR (75MHz, $CDCl_3$): 144.9, 140.1, 130.3, 129.0, 127.8,

127.1, 126.9, 122.9, 117.9, 112.0, 67.5, 60.2, 17.8; Mass ESI m/z (%) $M^+ = 108$; $M^+ + 2 = 110$.

(2-(butyl(methyl)amino)-2-phenylethanol). (Table 4, entry 9) Thick viscous liquid, R_f =0.41(1:9 EtOAc/hexane); ^{1}H NMR (300 MHz, $CDCl_3$): 7.28(m, 5H, ArH); 3.94(t, 1H); 3.78(d, 2H); 3.65(dd, *J*=3.6Hz,9Hz, 2H); 3.27(s, -OH); 2.43(m, 2H); 2.16(s, 3H). ^{13}C NMR (75MHz, $CDCl_3$): 142.5, 131.2, 126.0, 69.1, 60.6, 57.7, 41.9, 29.5, 20.6, 14.2; Mass ESI m/z (%) $M^+ + 1 = 208$.

(1-chloro-3-(phenylamino)propan-2-ol). (Table 4, entry 10) Light Yellow solid, mp 69°-70°C; IR (KBr, cm^{-1}): 3523, 3388, 3267, 3055, 1436, 1255, 602, 502. ^{1}H NMR (300MHz, $CDCl_3$): 7.17-7.25(m, 2H) 4.77(dd, *J*=4.2*Hz*,13.0*Hz,* 2H) 3.65(dd, *J*=6.9*Hz*,13.3*Hz,* 1H) 2.99(brs, 1H); ^{13}C NMR (75MHz, $CDCl_3$): 147.5, 129.6, 118.6, 113.6, 70.0, 47.8, 47.4. Mass ESI m/z (%): (M+1)=186; $(M^+ + 3)$188.

(1-chloro-3-(p-tolylamino)propan-2-ol). (Table 4, entry 11) Crystalline Solid, mp 68-70°C; R_f =0.63 (1:4 EtOAc/hexane); IR (KBr, cm^{-1}): 3336, 2958, 2850, 1616, 1517, 1450, 1288, 707, 543. ^{1}H NMR (300 MHz, $CDCl_3$): 3.18(dd, *J*= 7.2 Hz,15Hz, 1H); 3.34(dd, *J*=4.2Hz,13.3Hz, 1H); 3.63(m, *J*=3.6Hz,12.0Hz, 2H); 4.0(m, 1H). ^{13}C NMR (75MHz, $CDCl_3$): 145.6, 130.0, 127.8, 113.7, 76.8, 47.9, 20.5. Mass ESI m/z (%) $(M^+ + 1) = 200$.

(1-chloro-3-(o-tolylamino)propan-2-ol). (Table 4, entry 12) Viscous Liquid, ^{1}H NMR (300MHz, $CDCl_3$): 3.27(dd, *J*=7.2Hz,13.2Hz, 1H); 3.43(dd, *J*=4.2Hz,13.2Hz, 1H); 3.65(dd, *J*=4.5Hz,12Hz, 2H); 4.13-4.17(m, 1H);

6.70(dd, *J*=7.2Hz,16.5Hz, 2H); 7.06-7.16(m, 2H); ^{13}C NMR (75MHz, $CDCl_3$): 145.6, 130.6, 127.4, 123.1, 118.3, 110.6, 77.6, 69.9, 48.0.

(1-chloro-3-(naphthalen-1-ylamino)propan-2-ol) (Table 4, entry 13) Liquid, R_f=0.48 (1:4 EtOAc/hexane) ^{1}H NMR (300 MHz, $CDCl_3$):3.39(dd, *J*=7.2Hz,12.9Hz, 1H); 3.53(brs, 1H, OH); 3.47 (m, *J*=2.4Hz,4.8Hz, 1H); 3.7(m, 2H); 4.2(1H, NH); ^{13}C NMR (75MHz, CDCl3): 142.8, 134.5, 128.9, 126.5, 126.1, 125.3, 124.0, 120.1, 118.9, 105.7, 69.8.

2-(1H-imidazol-1-yl)-2-phenylethanol) (Scheme 3) Liquid, R_f = 0.59 (Pure EtOAc); ^{1}H NMR (300 MHz, $CDCl_3$): 7.43(m, 5H, Ar); 7.16(s, 1H); 6.9(s, 1H); 6.9(s, 1H); 4.9(s, 1H); 4.1(2H,CH_2); 2.6(s,-OH). 13 C NMR (75MHz, $CDCl_3$): 141.0, 129.2, 128.9, 128.7, 128.5, 127.0, 126.0, 120.0. Mass ESI m/z (%) M^+=188; M^++2=189.

(1-chloro-3-(1H-imidazo-1-yl)propan-2-ol), (Scheme 3) Viscous Liquid, R_f = 0.59 (Pure EtOAc) IR (KBr cm^{-1}): 3685, 3467, 2400, 1671, 1513, 1476, 1215, 1079. ^{1}H NMR (300 MHz, $CDCl_3$): 7.55(d, 1H); 6.97(m, 1H); 4.07(m, 2H); 3.55(dd, *J* = 4.5,12.6, 1H), 3.48-3.36(m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): 136.5, 128.7, 121.0, 70.4, 50.3, 45.7. Mass ESI m/z (%) M^++1=161; M^++3= 163(42%).

Figure S4. ^{1}H and ^{13}C Spectrum of 2-phenyl-2-(phenylamino)ethanol (Table 4, entry 1)

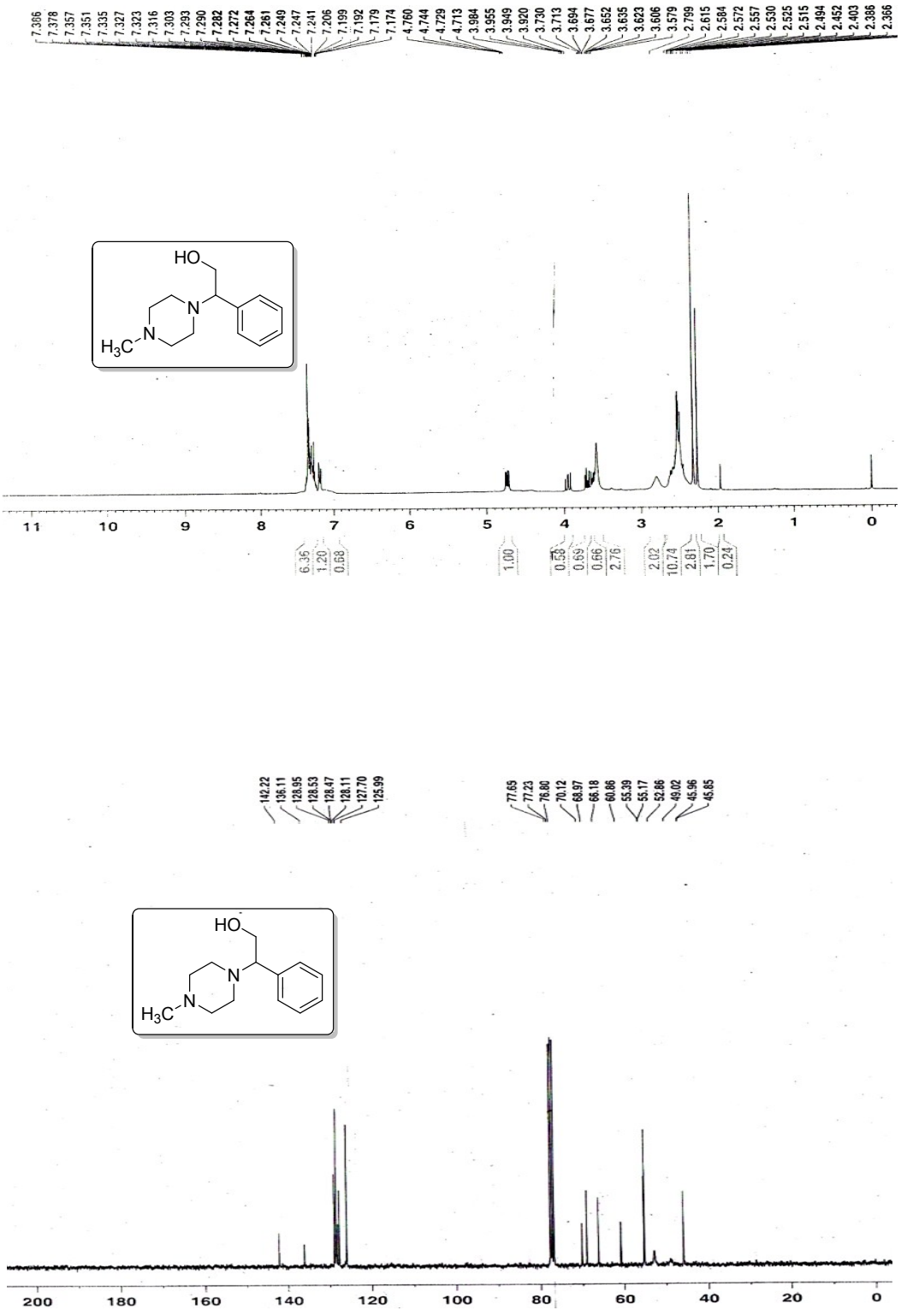

Figure S5. ^{1}H and ^{13}C Spectrum of *2-(4-methylpiperizine-1-yl)2-phenylethano* (Table 4, entry 2)

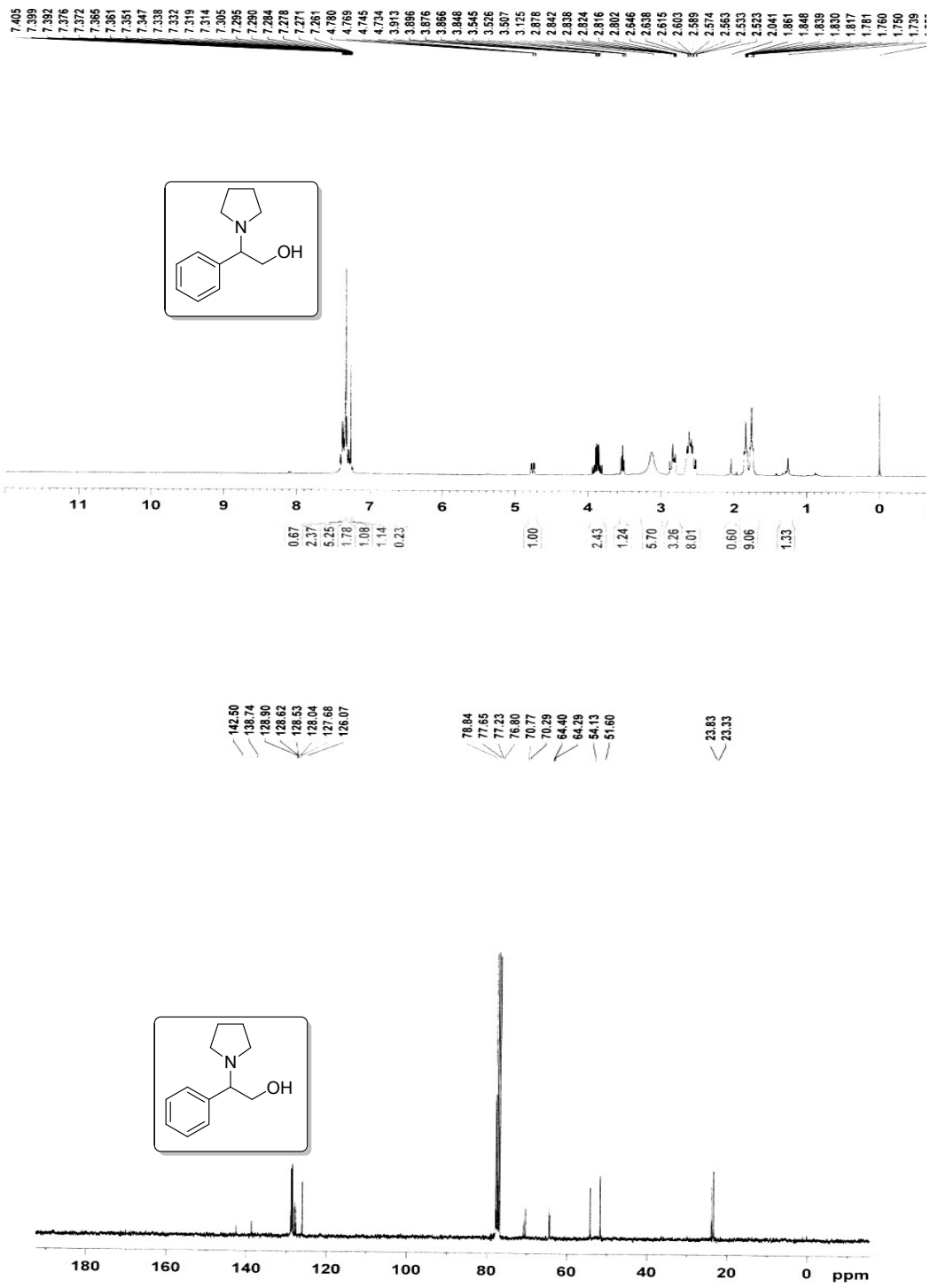

Figure S6. ^{1}H and ^{13}C Spectrum of (2-phenyl-2-(pyrolodin-1-yl)ethanol) (Table 4, entry 3)

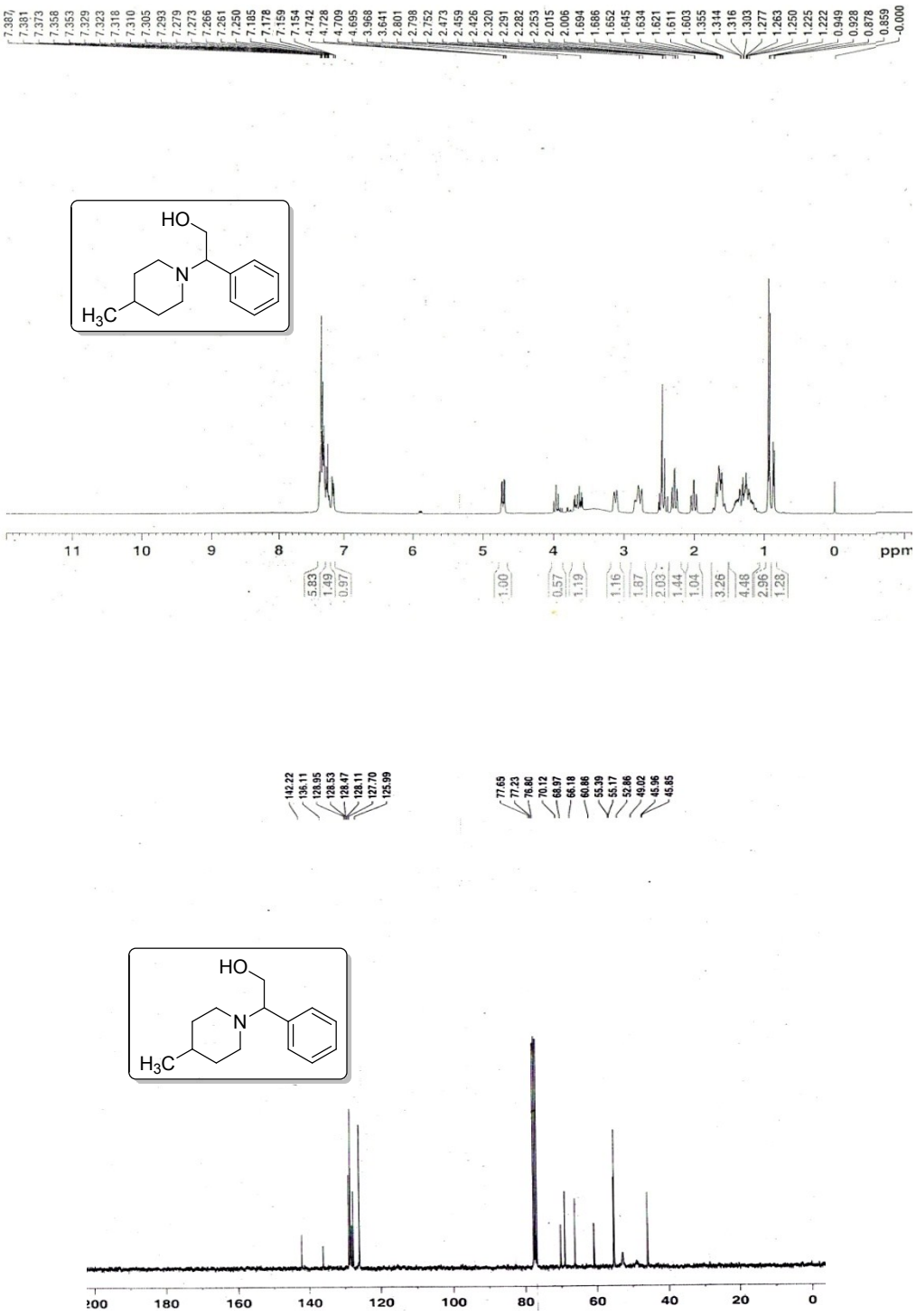

Figure S7. ^{1}H and ^{13}C Spectrum of 2-(4-methylpiperidin-1-yl)2-phenylethanol (Table 4, entry 4)

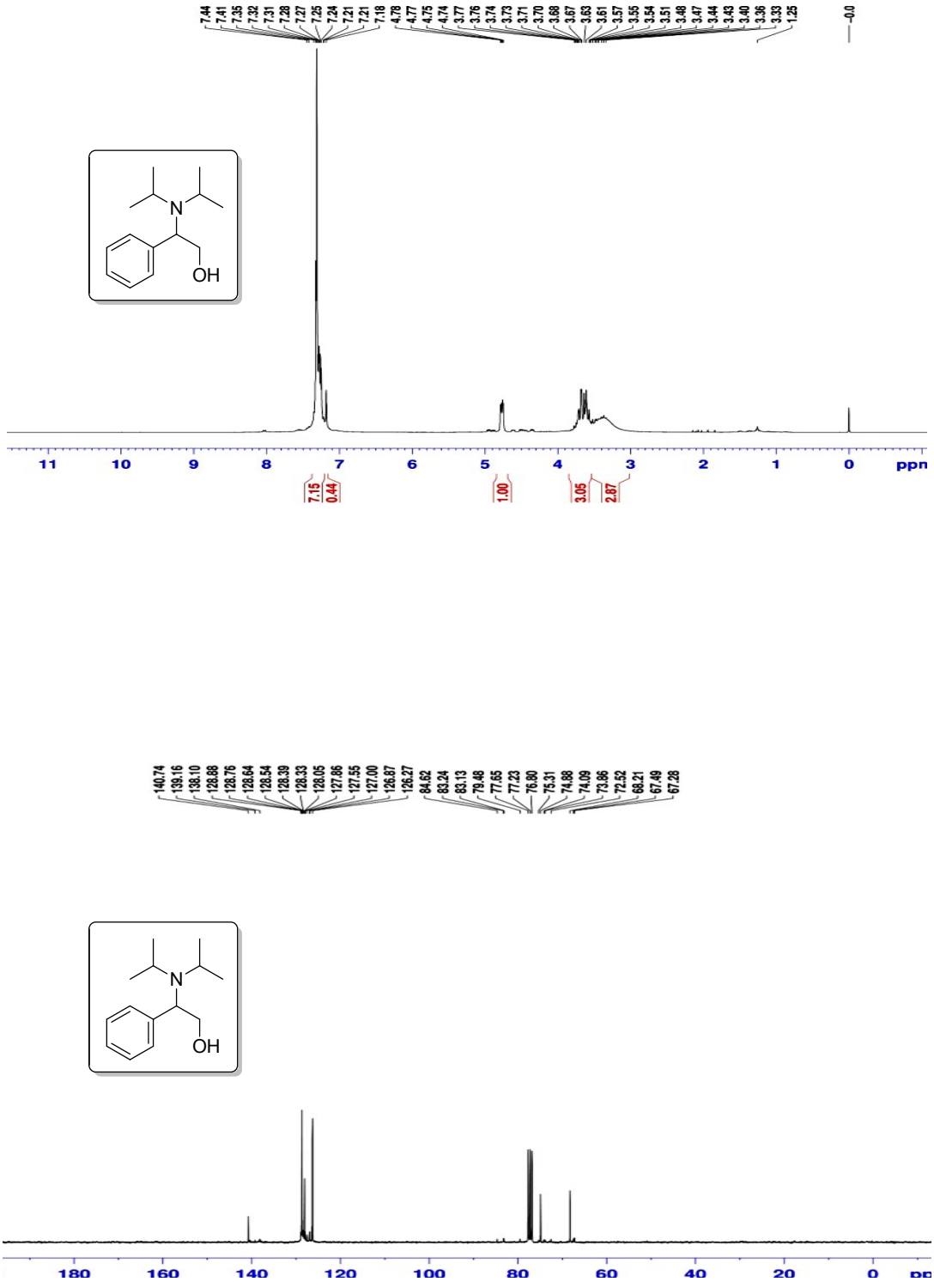

Figure S8. ^{1}H and ^{13}C Spectrum of (2-(dipropylamino)-2-phenylethanol) (Table 4, entry 5)

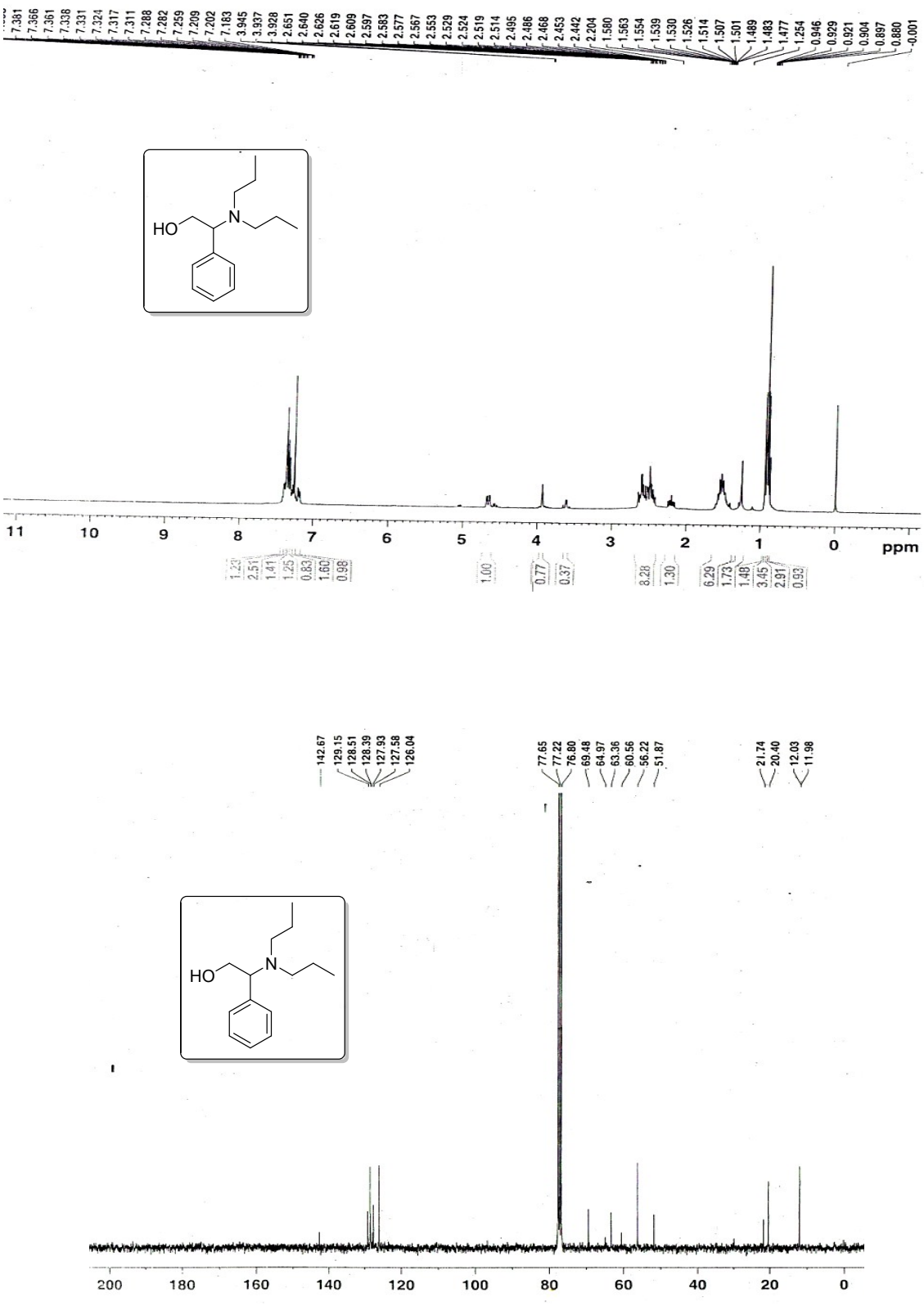

Figure S9. ^{1}H and ^{13}C Spectrum of *2-(dipropylamino)-2-phenylethanol* (Table 4, entry 6)

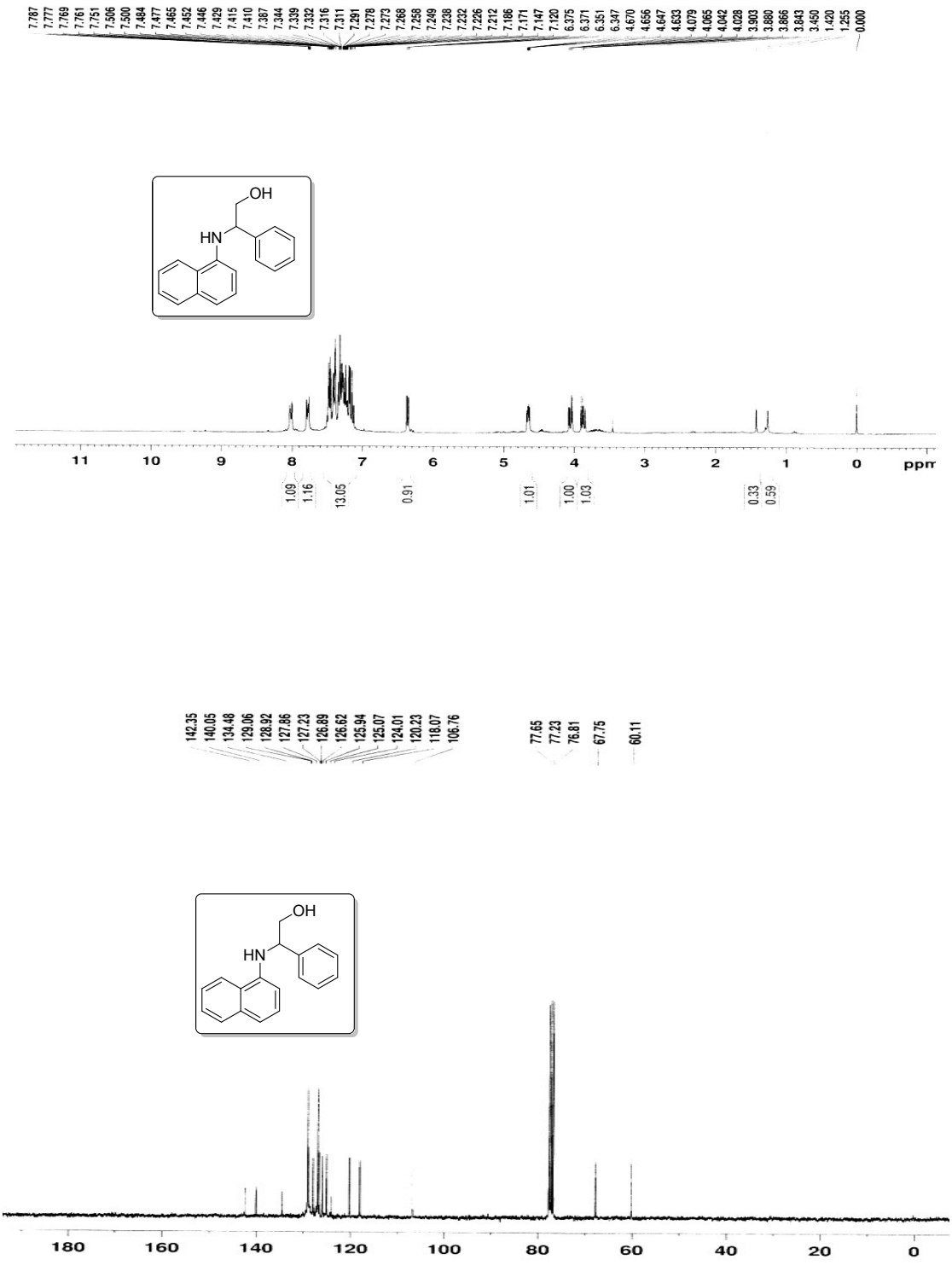

Figure S10. ^{1}H and ^{13}C Spectrum of 2-(naphthalene-1-ylamino)-2-phenylethanol (Table 4, entry 7)

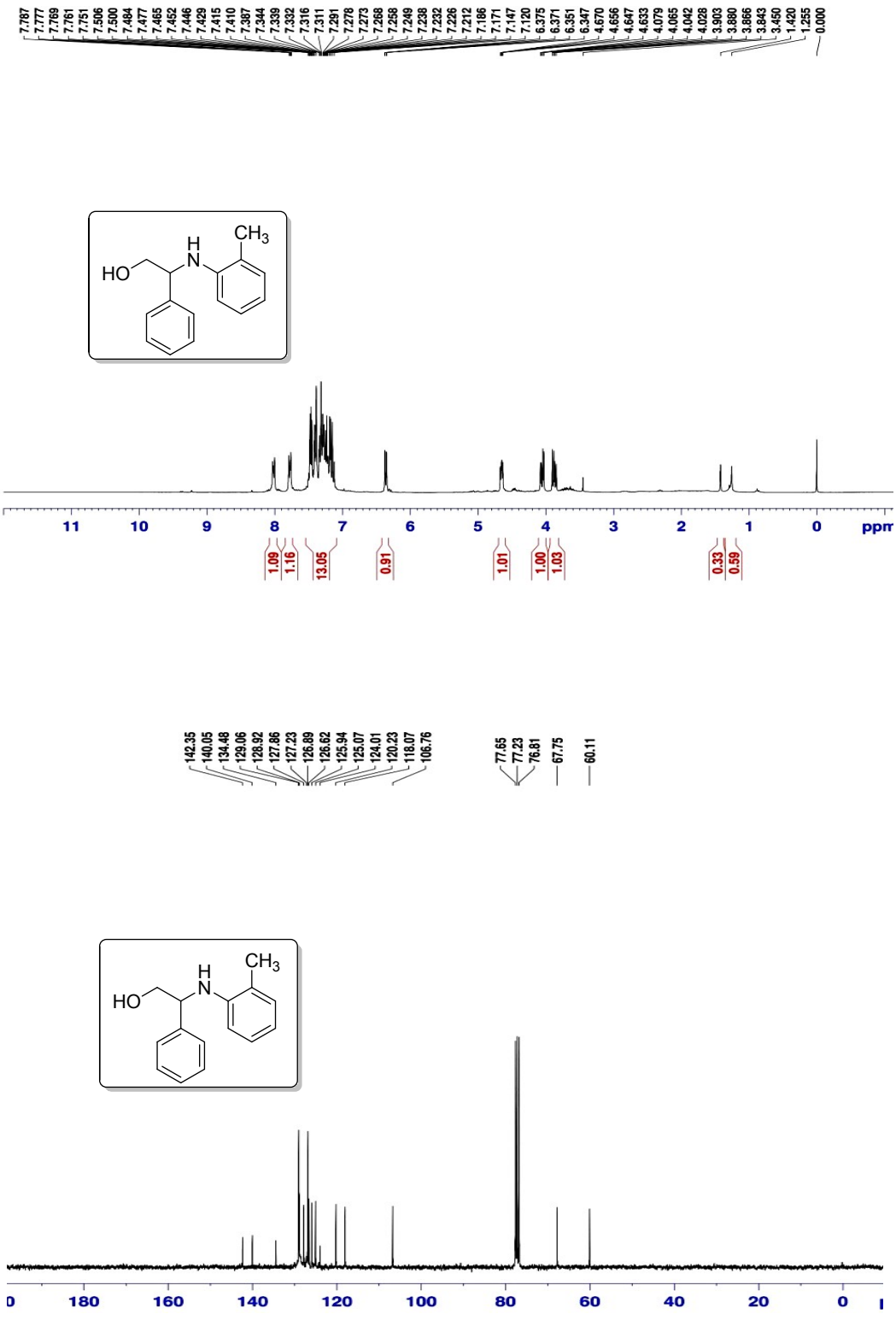

Figure S11. ^{1}H and ^{13}C Spectrum of (2-phenyl-2-(o-tolylamino)ethanol(Table 4, entry 8)

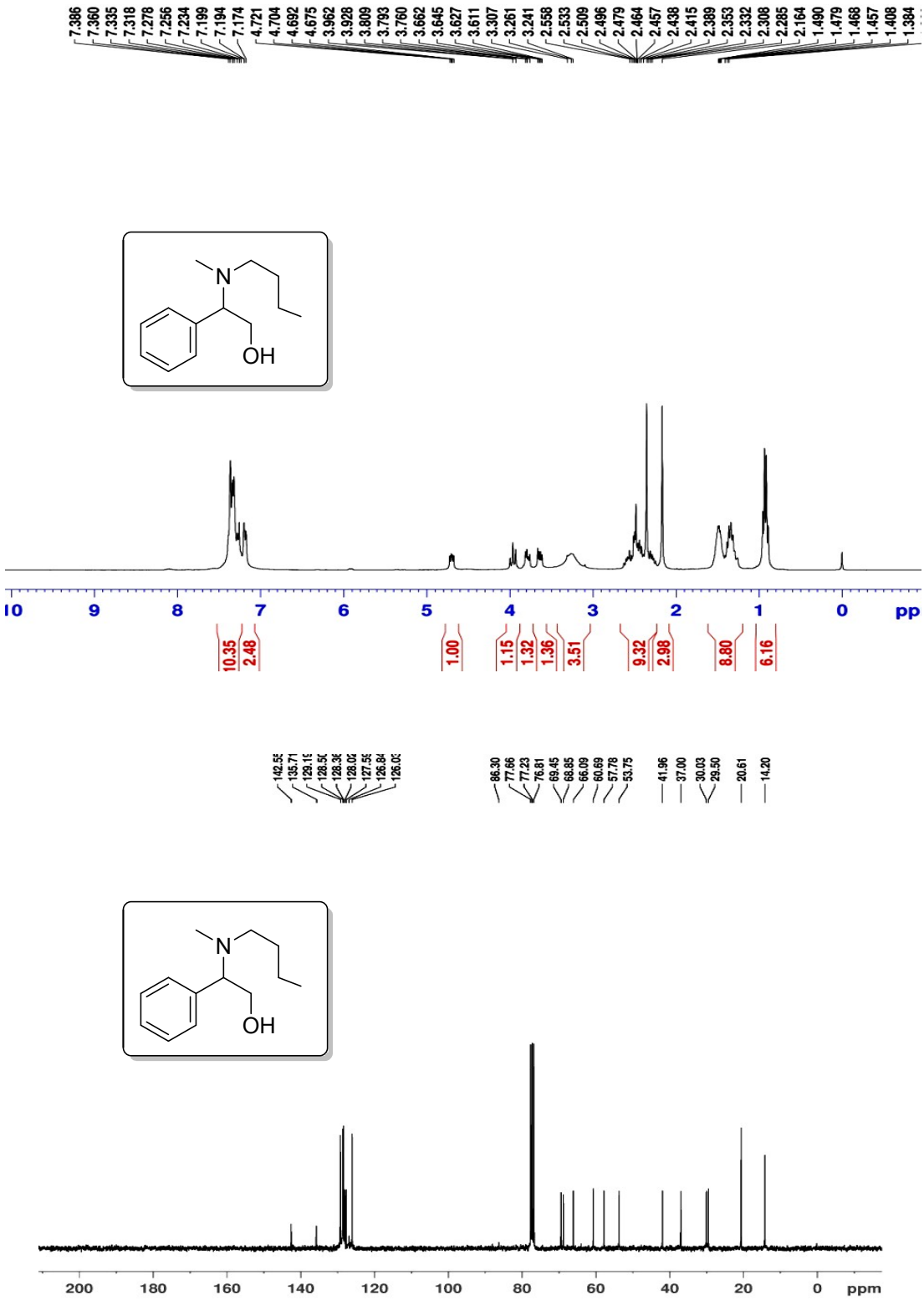

Figure S12. ^{1}H and ^{13}C Spectrum of *2-butyl (methyl) amino-2-phenylethanol)* (Table 4, entry 9)

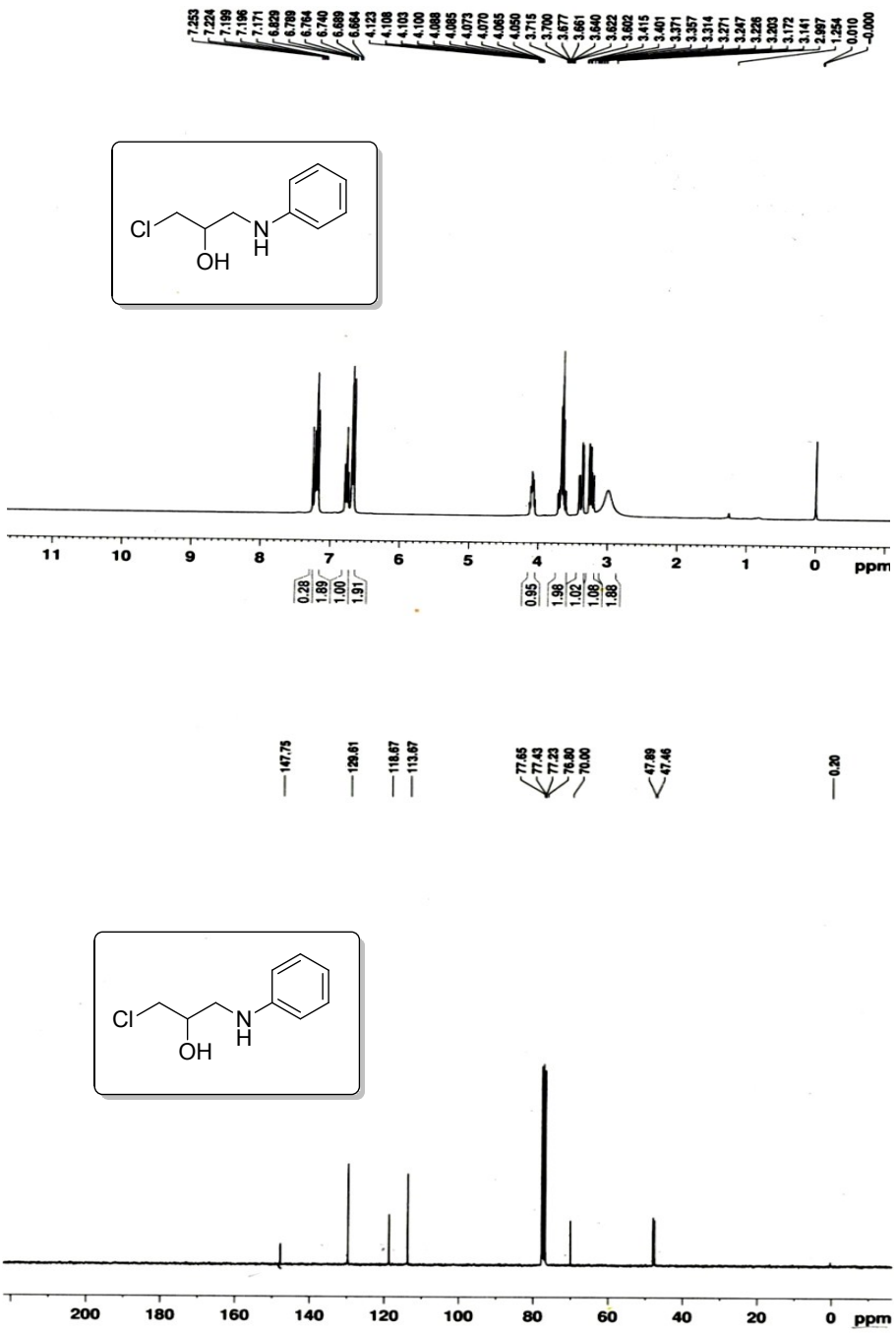

Figure S13. ^{1}H and ^{13}C Spectrum of 1-chloro-3-(phenylamino) propan-2-ol (Table 4, entry 10)

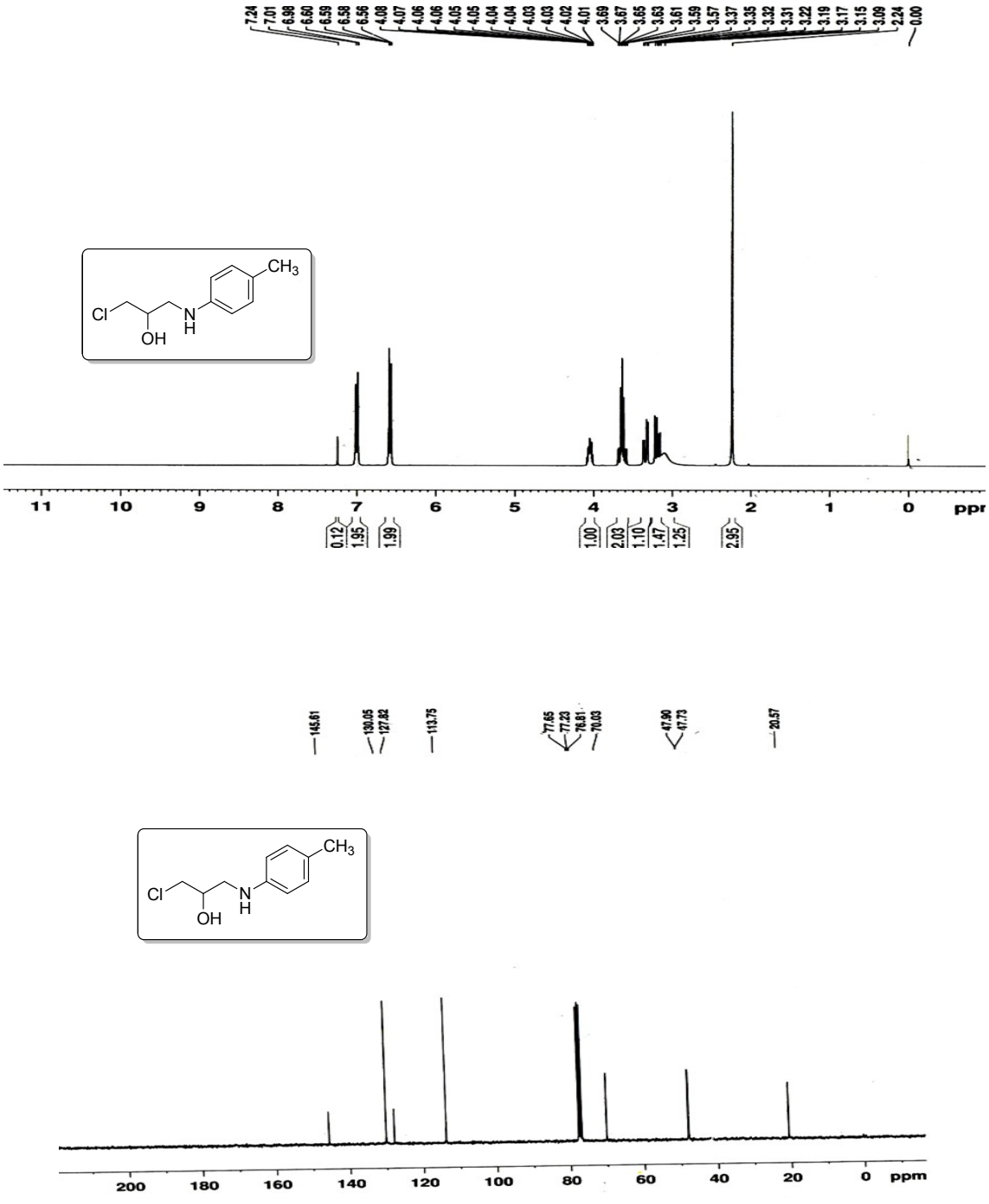

Figure S14. ^{1}H and ^{13}C Spectrum of *1-chloro-3-(p-tolylamino)propan-2*-ol (Table 4, entry 11)

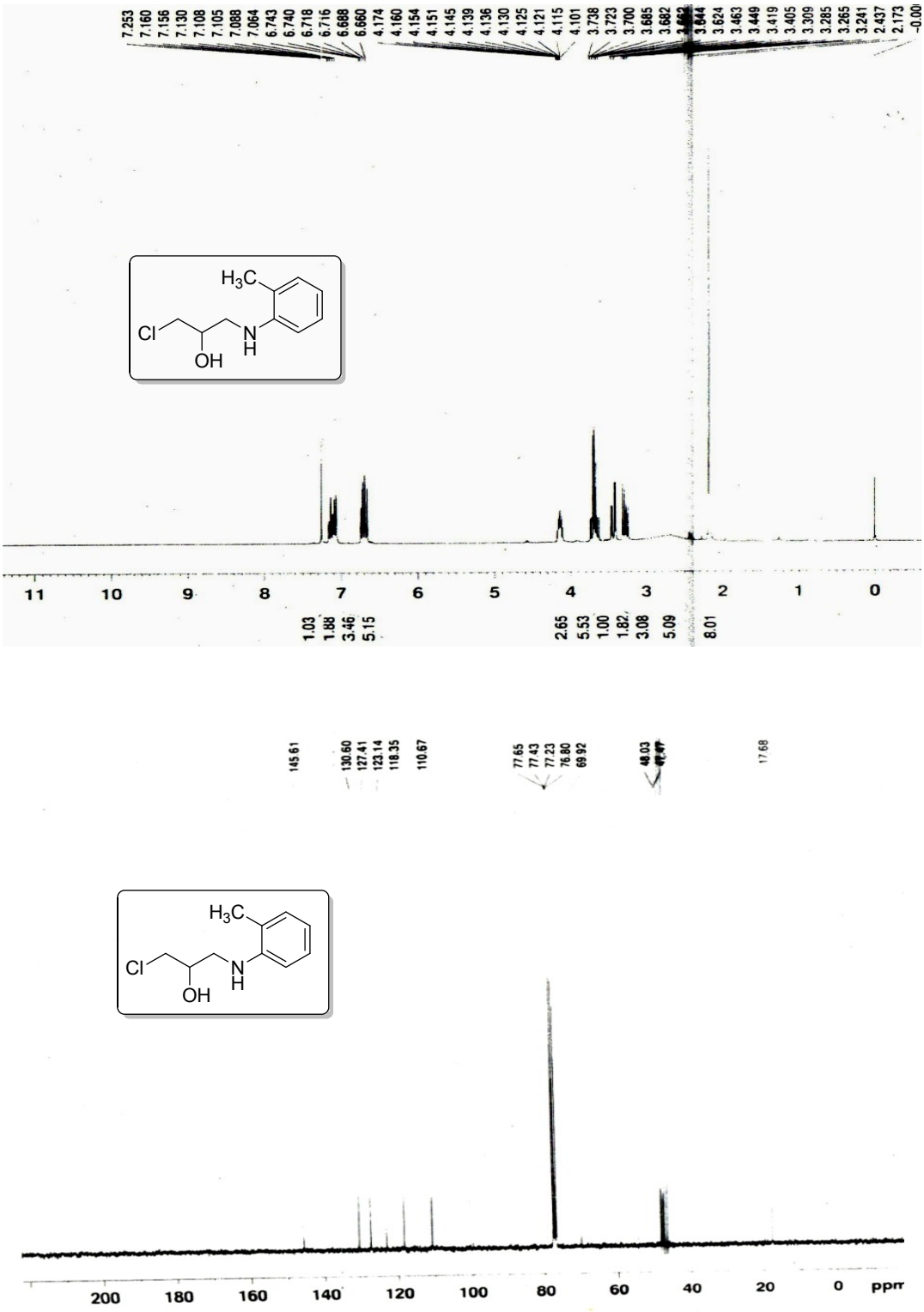

Figure S15. ^{1}H and ^{13}C Spectrum of *(1-chloro-3-(o-tolylamino)propan-2-ol)* (Table 4, entry 12)

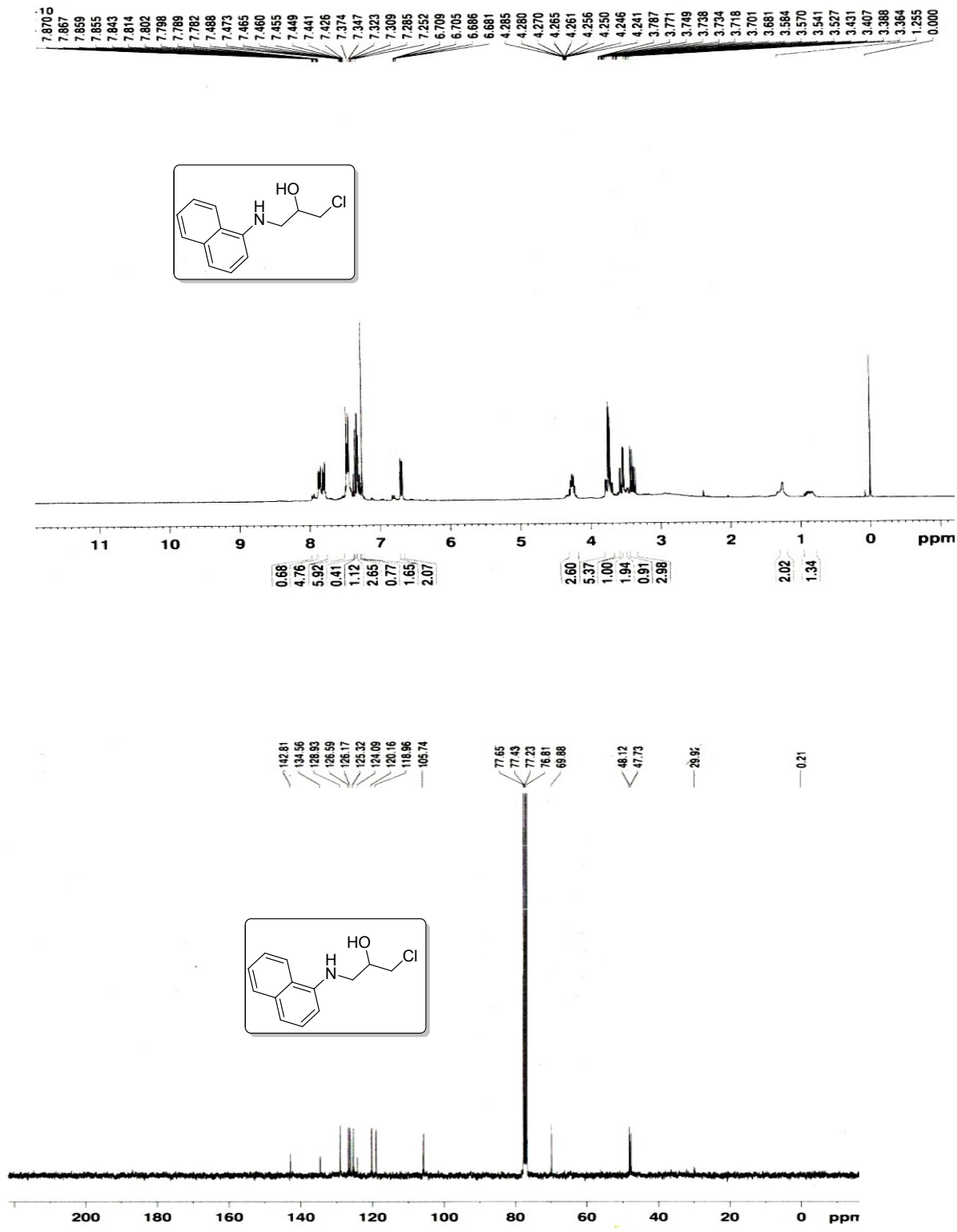

Figure S16. ^{1}H and ^{13}C Spectrum of *1-chloro-3-(naphthalen-1-ylamino) propan-2-ol* (Table 4, entry 13)

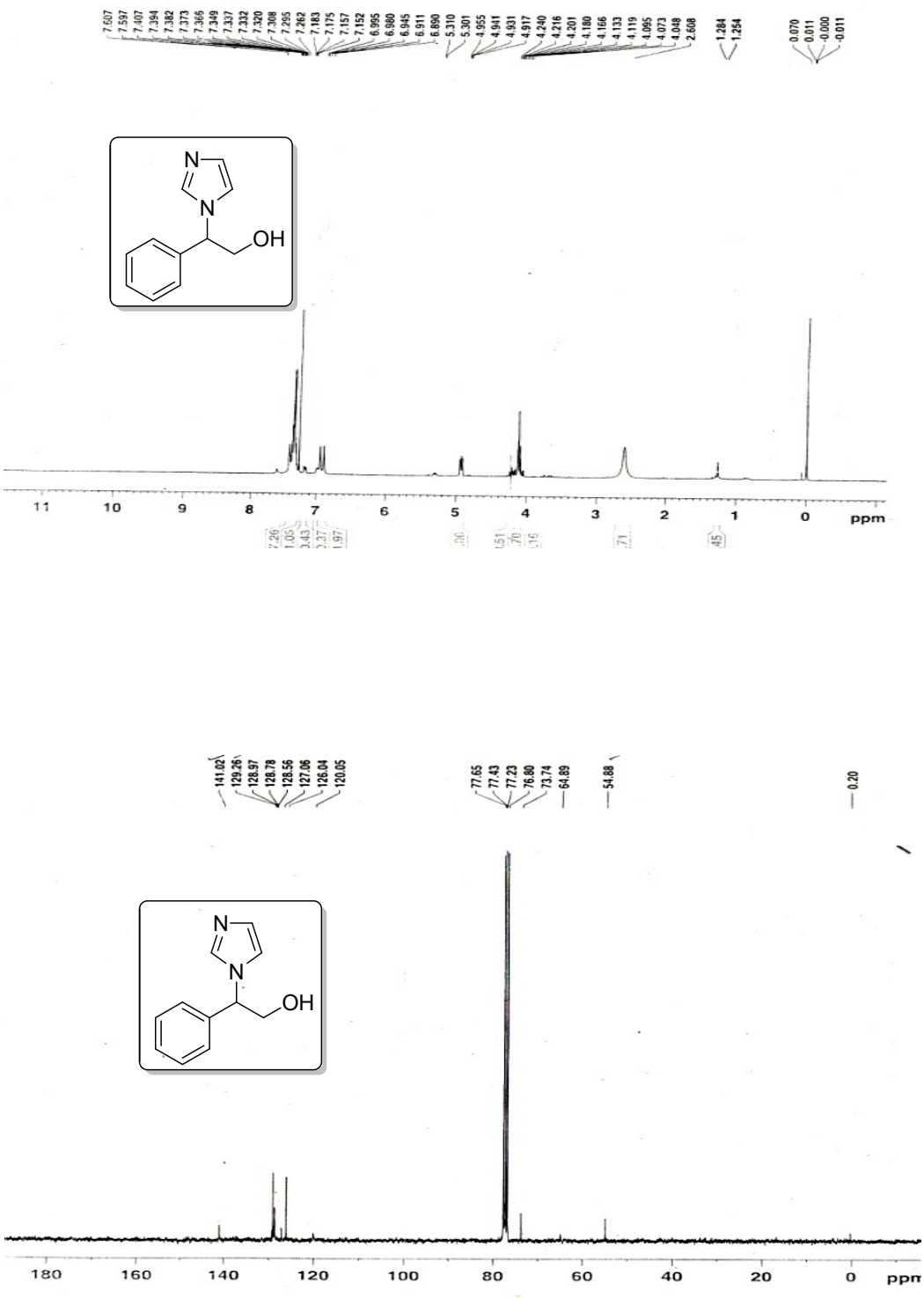

Figure S17. ^{1}H and ^{13}C Spectrum of 2-(1H-imidazol-1-yl)-2-phenylethanol (Scheme 3)