Electronic Supplementary Material (ESI) for RSC Advances

Oxidative ortho-Amino-methylation of phenols via C─H and C─C bond cleavage

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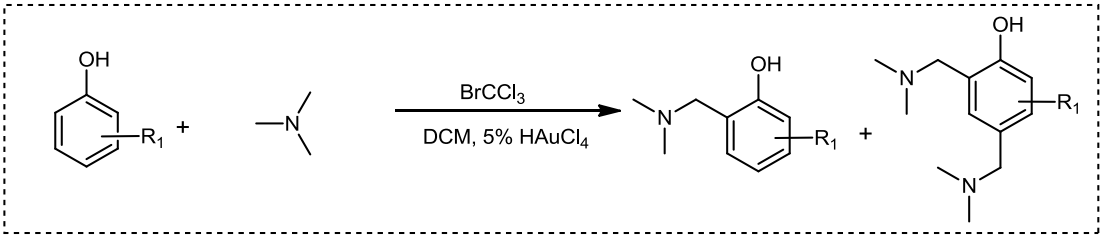
Table of Contents

Materials and methods

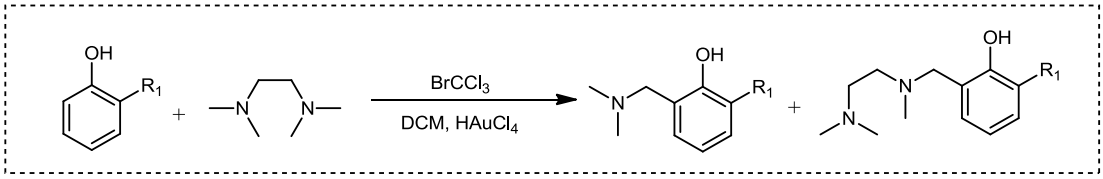
All reactions were carried out under a nitrogen atmosphere with dry solvents under anhydrous conditions, unless otherwise noted. All the chemicals were purchased commercially, and used without further purification. Anhydrous THF and toluene were distilled from sodium-benzophenone, and 1,2-dichloroethane, dichloromethane, acetonitrile, and tetramethylethylenediamine (TMEDA) were distilled from calcium hydride. Thin-layer chromatography (TLC) was conducted with 0.25 mm Tsingdaosilica gel plates (60F-254) and visualized by exposure to UV light (254 nm) or stained with potassium permanganate. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040–0.063 mm). Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. IR spectra were measured on an IR Prestige-21 series spectrometer in dry film (KBr) and were reported in terms of frequency (cm–1) and intensity of absorption. 1H NMR spectra were recorded on Bruker spectrometers (at 300, 400 or 500 MHz) and were reported relative to deuterated solvent signals. Data for 1H NMR spectra were reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. 13C NMR spectra were recorded on Bruker Spectrometers (at 75, 100 or 125 MHz). Data for 13C NMR spectra were reported in terms of chemical shift. Mass spectrometric data were obtained using Bruker Apex IV RTMS. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad.

General procedure for the CDC reaction between phenols and trimethylamine



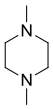
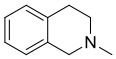
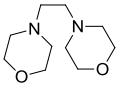
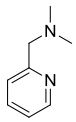
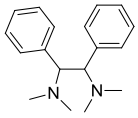
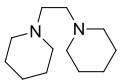
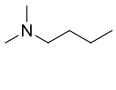
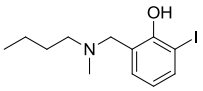
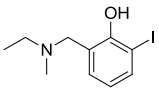
An oven-dried round bottom flask (25 mL) was equipped with a magnetic stir bar and charged with phenol (1 mmol, 1.0 equiv), $HAuCl_4.3H_2O$ (0.05 mmol, 0.05equiv), $BrCCl_3$ (3 mmol, 3equiv) and DCM(10.0 mL). The mixture was then bubbled with trimethylamine gas at room temperature. The reaction was monitored by TLC until the starting material was consumed. Then the solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography to afford the product.

General procedure for the reaction between phenols and TMEDA



An oven-dried round bottom flask (25 mL) was equipped with a magnetic stir bar and charged with phenol (1 mmol, 1.0 equiv), TMEDA 2 (10 mmol, 10.0 equiv), $HAuCl_4.3H_2O$ (0.05 mmol, 0.05equiv), $BrCCl_3$(3 mmol, 3equiv) and DCM(10.0 mL). The mixture was then stirred under a balloon nitrogen atmosphere at room temperature until the starting material disappeared from the TLC. After that the solvent was removed under reduced pressure, the residue was purified by silica gel column chromatography to afford the desired pure product.

SI Table 1: Amine[1,2,3] screened in this reaction with phenol

	no desired product		no desired product
	no desired product		no desired product
	no desired product		no desired product
	no desired product		no desired product
	 10% yield		
	 10% yield		

Typical procedure for the N1,N1,N2,N2-tetramethyl-1,2-diphenylethane-1,2-diamine synthesis

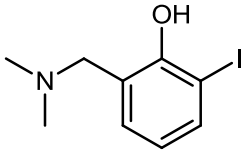
To 10.24 g. (0.2 mol) of 90% formic acid in a 500-ml round-bottomed flask, cooled in running tap water, is added slowly 2.12 g. (0.02 mol) of 1,2-diphenylethane-1,2-diamine. To the resulting clear solution are added 45 ml. (0.24mol) of formaldehyde solution (concentration, 37%) and a small boiling stone. The flask is connected to a reflux condenser and is placed in an oil bath which has been heated to 90–100°. A vigorous evolution of carbon dioxide begins after 2–3 minutes, at which time the flask is removed from the bath until the gas evolution notably subsides (15–20 minutes); then it is returned to the bath and heated at 95–100° for 8 hours.

After the solution has been cooled, 100 ml. of 4 *N* hydrochloric acid is added and the solution is evaporated to dryness under reduced pressure (water pump) from a water bath; the receiver is cooled in an ice bath. The pale yellow syrupy residue (or crystalline solid) is dissolved in 60–75 ml. of water, and the organic base is liberated by the addition of 50 ml. of 18 *N*sodium hydroxide solution. The upper (organic) phase is separated, and the lower (aqueous) phase is extracted with two 30-ml. portions of benzene. The combined organic base and benzene extracts are dried over 10 g. of anhydrous granular potassium carbonate. After the benzene has been distilled slowly under slightly reduced pressure from a 125-ml. Claisen flask, the pressure is lowered further, and the product is purified by high flash chromatography to get N^1, N^1, N^2, N^2-tetramethyl-1, 2-diphenylethane-1, 2-diamine 268mg(50% yield). And the other methylated diamine were synthesized following the same procedure.

Typical procedure for the 1,2-di(piperidin-1-yl)ethane synthesis

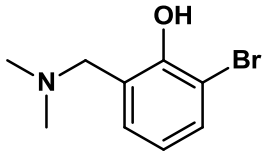
To a mixture of 17 g (0.2 mol) of piperidine, heated to 100°C, was added 4.9 g (0.05 mol) of 1,2-dichloroethane. The reaction mixture was heated at the same temperature on an oil bath for 5 h. After that, the reaction product was cooled and the precipitate was several times washed with dry ethyl ether. The solvent was evaporated and the reaction product was subjected to vacuum distillation and get 7.9 g 1,2-di(piperidin-1-yl)ethane (80%). 1,2-Dimorpholinoethane was synthesized following the same procedure.

^{1}H NMR and ^{13}C NMR Spectra data of CDC and CAC products



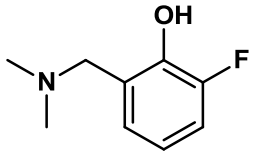
2-((dimethylamino)methyl)-6-iodophenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.64 (d, *J*=7.9, 1H), 6.94 (d, *J*=7.3, 1H), 6.54 (t, *J*=7.6, 1H), 3.63 (s, 2H), 2.34 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.40, 137.94, 128.28, 122.06, 120.48, 84.88, 62.67, 44.19; HRMS calculated forC9H13INO (M + H^+): 278.0042, found: 230.0037; IR (KBr)3614, 2951, 2926, 2831, 2789, 1724, 1597, 1546, 1448, 1427, 1400, 1298, 1257, 1168, 1099, 1072, 1016, 873, 840, 804, 759, 636 cm^{-1}.



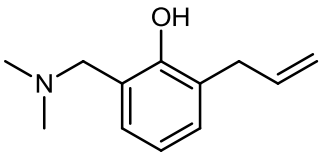
2-bromo-6-((dimethylamino)methyl)phenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42 (d, *J*=8.0, 1H), 6.91 (d, *J*=7.4, 1H), 6.66 (t, *J*=7.7, 1H), 3.66 (s, 2H), 2.35 (d, *J*=1.2, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 155.00, 131.99, 127.26, 122.99, 119.65, 110.13, 62.62, 44.24; HRMS calculated for C9H13BrNO (M + H^+): 230.0181, found: 230.0177; IR (KBr) 3645, 3051, 2954, 2886, 2858, 2833, 2775, 1728, 1645, 1604, 1452, 1429, 1398, 1357, 1300, 1257, 1180, 1122, 1101, 1072, 1041, 1016, 879, 815, 761, 729, 644 cm^{-1}.



2-((dimethylamino)methyl)-6-fluorophenol:

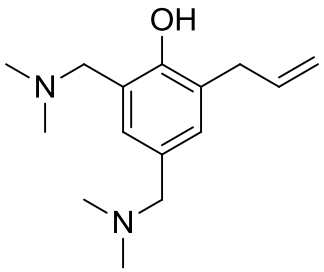
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.08 – 6.94 (m, 1H), 6.79 – 6.60 (m, 2H), 3.70 (s, 2H), 2.36 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 152.48, 150.06, 146.21, 124.06, 123.18, 118.34, 115.30, 62.48, 44.41; HRMS calculated for C9H13FNO (M + H^+): 170.0981, found: 170.0975; IR (KBr) 3564, 2960, 2916, 2848, 1722, 1699, 1633, 1471, 1462, 1409, 1340, 1290, 1259, 1093, 1022, 862, 801, 729, 702, 659 cm^{-1}.



2-allyl-6-((dimethylamino)methyl)phenol:

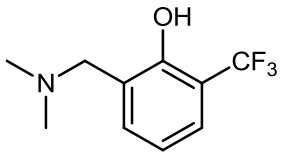
^{1}H NMR (400 MHz, $CDCl_3$) 7.07 (d, *J*=7.5, 1H), 6.87 (d, *J*=6.9, 1H), 6.75 (t, *J*=7.5, 1H), 6.11 – 6.01 (m, 1H), 5.17 – 5.02 (m, 2H), 3.66 (s, 2H), 3.41 – 3.38 (d, *J*=3, 2H),2.35 (d, *J*=8.4, 6H);^{13}C NMR (75 MHz, $CDCl_3$) δ 173.40, 155.77, 144.95, 137.19, 135.85, 128.96, 127.81, 126.33, 125.73, 121.45, 120.35, 118.53, 115.71, 115.03, 99.91,75.59, 62.78, 55.30, 44.31, 43.52, 33.91, 32.72; HRMS calculated for C12H18NO (M + H^+): 192.1388, found: 192.1384; IR (KBr) 3643, 3072, 3049, 2978,

2953, 2912, 2858, 2829, 2783, 2727, 2638, 1732, 1637, 1624, 1595, 1469, 1429, 1402, 1359, 1282, 1257, 1224, 1178, 1099, 1078, 1041, 1020, 981, 966, 910, 844, 802, 779, 750, 704, 640, 601, 518 cm^{-1}.



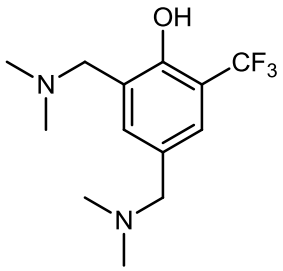
2-allyl-4,6-bis((dimethylamino)methyl)phenol:

^{1}H NMR (300 MHz, $CDCl_3$) δ = 6.95 (d, *J*=1.7, 1H), 6.82 (s, 1H), 6.05 (dd, *J*=16.5, 10.6, 1H), 5.08 (dd, *J*=11.8, 6.3, 2H), 3.63 (s, 2H), 3.41 – 3.38 (d, *J*=3, 2H), 3.31 (s, 2H), 2.32(s, 6H), 2.23(s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.85, 136.16, 129.37, 127.85, 125.76, 121.03, 115.48, 63.87, 62.58, 45.42, 43.30, 33.53, 30.02; HRMS calculated for C15H25N2O (M + H^+): 249.1967, found: 249.1960; IR (KBr)2960, 2912, 2848, 2814, 2777, 1732, 1637, 1610, 1460, 1406, 1363, 1257, 1097, 1024, 1020, 966, 864, 802, 719, 702 cm^{-1}.



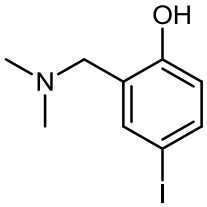
2-((dimethylamino)methyl)-6-(trifluoromethyl)phenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.46 (d, *J*=7.8, 1H), 7.14 (d, *J*=7.4, 1H), 6.82 (t, *J*=7.7, 1H), 3.73 (s, 2H), 2.37 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.00, 131.69, 125.90, 125.42, 122.81, 117.94, 117.12, 116.82, 62.41, 44.20; HRMS calculated for C10H13F3NO (M + H^+): 220.0949, found: 220.0943; IR (KBr) 3425, 3059, 2960, 2926, 2856, 2791, 2723, 2630, 1726, 1706, 1650, 1606, 1467, 1433, 1409, 1281, 1365, 1328, 1307, 1263, 1236, 1124, 1078, 1041, 1018, 974, 900, 839, 802, 786, 752, 648, 611, 580, 532 cm^{-1}.



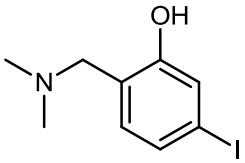
2,4-bis((dimethylamino)methyl)-6-(trifluoromethyl)phenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.35 (s, 1H), 7.14 (s, 1H), 3.71 (s, 2H)), 3.34 (s, 2H), 2.38 (d, *J*=11.8, 6H), 2.25 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 155.95, 132.30, 128.49, 126.41, 122.87, 63.42, 62.33, 45.15, 44.21; IR (KBr) 3624, 2958, 2929, 2856, 2818, 2777, 2732, 1728, 1630, 1595, 1490, 1476, 1431, 1408, 1375, 1352, 1300, 1282, 1255, 1236, 1213, 1178, 1091, 1043, 1018, 959, 867, 844, 821, 771, 744, 684, 609 cm^{-1}.



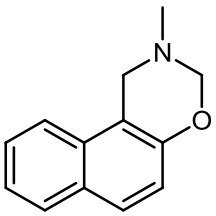
2-((dimethylamino)methyl)-4-iodophenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.44 (dd, *J*=8.5, 2.1, 1H), 7.31 (2, 1H), 6.62 (d, *J*=8.5, 1H), 3.60 (s, 2H), 2.34 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 158.09, 137.38, 136.65, 124.54, 118.48, 80.16, 62.09, 44.43; HRMS calculated for C9H13INO (M + H^+): 278.0042, found: 278.0034; IR (KBr) 3473, 2958, 2916, 2848, 2760, 1718, 1690, 1653, 1635, 1473, 1411, 1261, 1231, 1176, 1095, 1043, 1018, 961, 866, 844, 798, 763, 742, 683, 607 cm^{-1}.



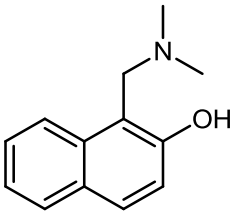
2-((dimethylamino)methyl)-5-iodophenol:

^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.21 (d, *J*=1.5, 1H), 7.11 (dd, *J*=7.9, 1.5, 1H), 6.68 (d, *J*=7.9, 1H), 3.61 (s, 2H), 2.32 (d, *J*=15.7, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 158.95, 129.58, 127.90, 125.11, 121.54, 93.24, 62.35, 44.23;HRMS calculated for C9H13INO (M + H^+): 278.0042, found:278.0037; IR (KBr) 2958, 2926, 2852, 2829, 2816, 2785, 1734, 1647, 1595, 1481, 1476, 1375, 1348, 1300, 1263, 1234, 1178, 1099, 1043, 1018, 974, 881, 846, 800 cm^{-1}.



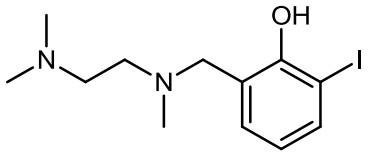
2-methyl-2,3-dihydro-1H-naphtho[1,2-e][1,3]oxazine:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.80 (d, *J*=8.1, 1H), 7.66 (dd, *J*=17.5, 8.7, 1H), 7.51 (ddd, *J*=8.3, 6.9, 1.2, 1H), 7.43 – 7.34 (m, 1H), 7.29 (s, 1H), 7.07 (d, *J*=8.9, 1H) , 4.89 (s, 2H), 4.32 (s, 2H), 2.70 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 151.32, 131.84, 128.94, 128.57, 127.93, 126.42, 123.38, 120.93, 118.43, 111.40, 83.47, 49.69, 40.24; HRMS calculated for C13H14NO (M + H^+): 200.1075, found: 200.1074; IR (KBr) 3548, 3061, 2960, 2926, 2850, 2780, 1724, 1625, 1598, 1516, 1471, 1400, 1263, 1228, 1168, 1082, 950, 902, 858, 810, 744, 694, 678 cm^{-1}.



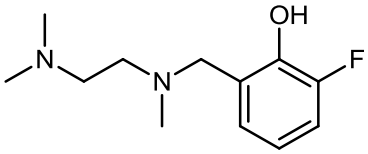
1-((dimethylamino)methyl)naphthalen-2-ol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.81 (dd, *J*=23.4, 8.3, 2H), 7.71 (d, *J*=8.9, 1H), 7.51 – 7.41 (m, 1H), 7.35 – 7.26 (m, 1H), 7.13 (d, *J*=8.8, 1H), 4.13 (s, 2H), 2.45 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 156.76, 132.52, 129.13, 128.84, 128.41, 126.23, 122.31, 120.89, 119.21, 111.38, 57.86, 44.68; HRMS calculated for C13H16NO (M + H^+): 202.1232, found: 202.1228; IR (KBr) 3520, 3053, 2956, 2924, 2856, 2783, 1732, 1714, 1622, 1598, 1494, 1415, 1367, 1332, 1265, 1204, 1165, 1099, 1039, 1018, 993, 858, 804, 742, 711, 665, 632, 532, 520 cm^{-1}.



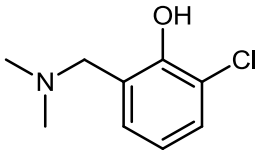
2-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)-6-iodophenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.65 (dd, *J*=7.9, 1.3, 1H), 6.95 (d, *J*=7.3, 1H), 6.54 (t, *J*=7.6, 1H) , 3.65 (s, 2H), 2.69 – 2.59 (t, *J*=7.4, 2H), 2.54 (dd, *J*=7.4, 5.8, 2H), 2.29 (s, 3H), 2.26 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.11, 138.03, 128.85, 122.68, 120.35, 85.18, 60.18, 56.57, 54.27, 45.40, 41.78; HRMS calculated for C12H20IN2O (M + H^+): 335.0620, found: 335.0620; IR (KBr) 3500, 3055, 2956, 2854, 2819, 2792, 2775, 2717, 1732, 1666, 1593, 1583, 1450, 1421, 1365, 1300, 1261, 1230, 1159, 1111, 1072, 1026, 925, 871, 806, 731, 640, 497 cm^{-1}.



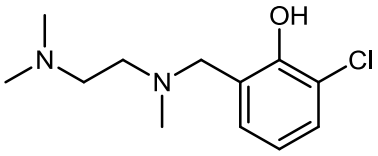
2-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)-6-fluorophenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.07 – 6.95 (m, 1H), 6.77 (d, *J*=7.5, 1H), 6.69 (dd, *J*=7.8, 4.7, 1H), 3.61 (s, 2H), 2.60 (m, 4H), 2.29 (d, *J*=12.3, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 152.94, 150.53, 145.90, 125.46, 124.12, 117.88, 115.28, 58.09, 56.19, 53.87, 44.98, 42.19, 29.65; HRMS calculated for C12H20FN2O (M + H^+): 227.1560, found: 227.1556; IR (KBr) 3477, 2975, 2932, 2854, 2821, 2792, 2713, 1672, 1632, 1585, 1477, 1454, 1423, 1402, 1365, 1303, 1259, 1234, 1153, 1122, 1097, 1070, 1026, 840, 800, 781, 729, 704 cm^{-1}.

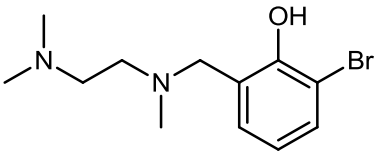


2-chloro-6-((dimethylamino)methyl)phenol:

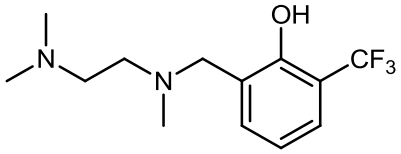
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.31 – 7.22 (m, 1H), 6.88 (d, *J*=7.4, 1H), 6.72 (t, *J*=7.7, 1H), 3.68 (s, 2H), 2.36 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 154.04, 129.04, 126.50, 123.04, 120.72, 119.08, 62.58, 44.29; HRMS calculated for C9H13ClNO (M + H^+): 186.0686, found: 186.0678; IR (KBr)3068, 2954, 2924, 2856, 2833, 2789, 2717, 1733, 1666, 1604, 1581, 1450, 1429, 1398, 1384, 1359, 1300, 1257, 1170, 1136, 1101, 1074, 1018, 972, 891, 842, 827, 769, 731, 655, 594 cm^{-1}.



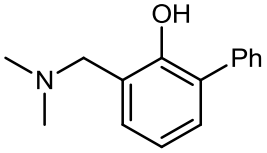
2-chloro-6-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)phenol:
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.31 – 7.22 (m, 1H), 6.92 – 6.85 (m, 1H), 6.70 (t, *J*=7.7, 1H), 3.65 (s, 2H), 2.64 (dd, *J*=9.9, 3.9, 2H), 2.53 (dd, *J*=9.9, 3.9, 2H), 2.28 (d, *J*=1.8, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 153.78, 129.12, 127.24, 123.95, 120.98, 118.88, 59.46, 56.48, 54.22, 45.29, 41.93;. HRMS calculated for C12H20ClN2O (M + H^+): 243.1264, found: 243.1264; IR (KBr) 3469, 2962, 2924, 2850, 2821, 1728, 1660, 1602, 1568, 1456, 1404, 1365, 1261, 1159, 1097, 1076, 1022, 927, 864, 826, 731, 659 cm^{-1}.



2-bromo-6-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)phenol:
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.41 (dd, *J*=8.0, 1.4, 1H), 6.92 (d, *J*=7.4, 1H), 6.64 (t, *J*=7.7, 1H), 3.65 (s, 2H), 2.71 – 2.42 (m, 4H), 2.39 – 2.23 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ = ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.88, 132.24, 128.09, 119.65, 110.59, 77.63, 77.21, 76.79, 59.92, 56.68, 54.41, 45.51, 42.03; HRMS calculated for C12H20BrN2O (M + H^+): 287.0759, found: 287.0752; IR (KBr) 3066, 2968, 2918, 2848, 2819, 2773, 2715, 1600, 1568, 1455, 1403, 1363, 1301, 1261, 1228, 1159, 1124, 1072, 1024, 925, 877, 813, 763, 731, 646 cm^{-1}.

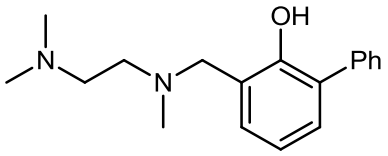


2-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)-6-(trifluoromethyl)phenol:
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 (d, *J*=7.8, 1H), 7.15 (d, *J*=7.4, 1H), 6.81 (t, *J*=7.6, 1H), 3.72 (s, 2H), 2.70 – 2.60 (m, 4H), 2.55 (dd, *J*=7.3, 5.7, 2H), 2.33 (s, 3H), 2.27 (s, 6H); HRMS calculated for C13H20F3N2O (M + H^+): 277.1528, found: 277.1525; IR (KBr)3477, 2958, 2918, 2848, 2821, 2775, 2716, 1672, 1604, 1469, 1265, 1263, 1161, 1128, 1078, 1028, 929, 839, 800, 786, 752, 651, 613 cm^{-1}.



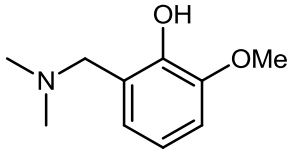
3-((dimethylamino)methyl)-[1,1'-biphenyl]-2-ol:
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.68 – 7.61 (m, 2H), 7.45 (dd, *J*=10.5, 4.8, 2H), 7.38 – 7.26 (m, 2H), 7.03 – 6.96 (m, 1H), 6.88 (t, *J*=7.5, 1H), 3.74 (s, 2H), 2.37 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 155.27, 138.56, 129.72, 129.36, 128.76, 127.98, 127.60, 126.68, 122.22, 118.84, 62.96, 44.30; HRMS calculated for C15H18NO (M +

H^+): 228.1388, found: 228.1382; IR (KBr)3472, 2960, 2924, 2854, 2789, 1737, 1681, 1633, 1469, 1435, 1298, 1261, 1168, 1095, 1028, 800, 731, 699, 665 cm^{-1}.



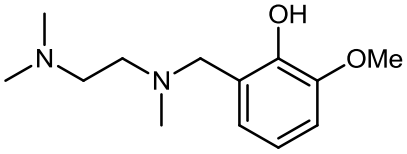
3-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)-[1,1'-biphenyl]-2-ol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.65 (d, *J*=7.4, 2H), 7.44 (t, *J*=7.5, 2H), 7.36 – 7.21 (m, 2H), 7.00 (d, *J*=6.9, 1H), 6.87 (t, *J*=7.5, 1H), 3.78 (s, 2H), 2.77 – 2.62 (m, 2H), 2.62 – 2.45 (m, 2H), 2.45 – 2.28 (m, 3H), 2.25 (s, 6H); HRMS calculated for C18H25N2O (M + H^+): 285.1967, found: 285.1964; IR (KBr) 3431, 3055, 3034, 3020, 2960, 2924, 2854, 2818, 2789, 2773, 2721, 1722, 1698, 1591, 1498, 1463, 1431, 1363, 1300, 1261, 1244, 1230, 1157, 1133, 1072, 1028, 974, 927, 829, 788, 756, 700, 630, 582, 563 cm^{-1}.



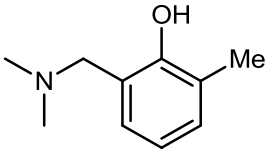
2-((dimethylamino)methyl)-6-methoxyphenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.99 – 6.78 (m, 1H), 6.75 (m, 1H), 6.61 (d, *J*=7.5, 1H), 3.89 (t, *J*=3.2, 3H), 3.67 (s, 2H), 2.35 (d, *J*=2.5, 6H);^{13}C NMR (75 MHz, $CDCl_3$) δ = 147.81, 147.29, 122.06, 120.28, 118.43, 110.83, 62.56, 55.78, 44.41;HRMS calculated forC10H16NO2 (M + H^+): 182.1181, found: 182.1176; IR (KBr) 3053, 2956, 2916, 2848, 2831, 2783, 1724, 1589, 1475, 1458, 1431, 1406, 1357, 1292, 1262, 1238, 1180, 1078, 1041, 1016, 974, 833, 800, 771, 734, 711cm^{-1}.



2-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)-6-methoxyphenol:

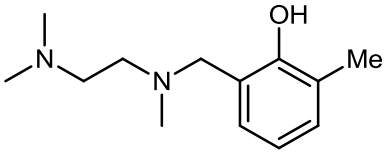
^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.81 (d, *J*=8.1, 1H), 6.72 (t, *J*=7.8, 1H), 6.61 (d, *J*=7.5, 1H), 3.93 – 3.80 (m, 3H), 3.63 (d, *J*=10.6, 2H), 2.63 – 2.55 (m, 2H), 2.52 (t, *J*=6.7, 2H), 2.25 (t, *J*=6.6, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 148.01, 146.97, 122.84, 120.99, 118.20, 110.79, 59.25, 56.47, 55.73, 54.26, 45.20, 41.97; HRMS calculated forC13H23N2O2 (M + H^+): 239.1760, found: 239.1752; IR (KBr) 3051, 2943, 2852, 2818, 2787, 2773, 2721, 1726, 1585, 1498, 1421, 1402, 1365, 1301, 1284, 1261, 1186, 1157, 1128, 1078, 1028, 975, 935, 835, 813, 771, 732, 713, 617 cm^{-1}.



2-((dimethylamino)methyl)-6-methylphenol:

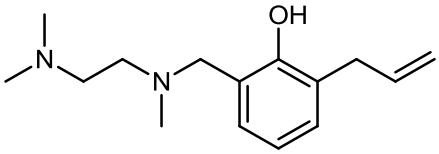
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.06 (d, *J*=7.3, 1H), 6.99 – 6.73 (m, 1H), 6.69 (d, *J*=7.4, 1H), 3.64 (s, 2H), 2.37 – 2.15 (m, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ =155.69,

129.73, 125.79, 124.13, 120.19 118.33, 62.78, 44.34, 29.64;HRMS calculated forC10H16NO (M + H^+): 166.1232, found: 166.1225; IR (KBr)3489, 2960, 2916, 2848, 1731, 1698, 1606, 1462, 1402, 1261, 1093, 1020, 864, 801, 740, 704 cm^{-1}.



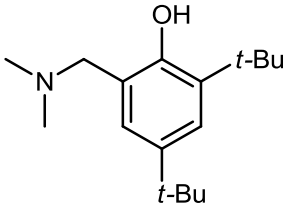
2-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)-6-methylphenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.06 (d, *J*=7.3, 1H), 6.84 (d, *J*=7.2, 1H), 6.70 (t, *J*=7.4, 1H), 3.67 (s, 2H), 2.63 (t, *J*=6.6, 3H), 2.53 (t, *J*=6.6, 3H), 2.40 – 2.22 (m, 6H; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 155.85, 129.79, 126.26, 124.87, 121.47, 118.28, 60.52, 56.79, 54.54, 45.46, 41.76, 29.63, 15.73;HRMS calculated forC13H23N2O (M + H^+): 223.1810, found: 223.1805; IR (KBr) 3458, 2960, 2924, 2852, 2818, 2791, 2773, 1728, 1693, 1597, 1469, 1454, 1365, 1300, 1261, 1232, 1085, 1022, 974, 927, 802, 763, 740, 692 cm^{-1}.



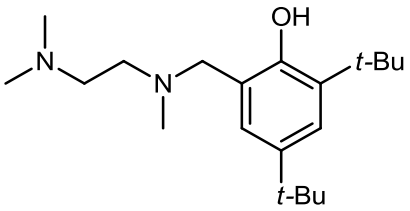
2-allyl-6-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)phenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.06 (d, *J*=7.5, 1H), 6.89 – 6.83 (m, 1H), 6.74 (td, *J*=7.4, 3.0, 1H), 6.06 (m, 1H), 5.16 – 4.95 (m, 2H), 3.69 (s, 2H), 3.42 (d, *J*=6.6, 2H), 2.65 – 2.57 (m, 2H), 2.52 (dd, *J*=7.7, 5.6, 2H), 2.34 (s, 3H), 2.31 (s, 3H), 2.29 (d, *J*=4.6, 3H), 2.26 (s, 3H); HRMS calculated forC15H25N2O (M + H^+): 249.1967, found:249.1967; IR (KBr) 3440, 3070, 2960, 2924, 2854, 1732, 1643, 1614, 1597, 1462, 1435, 1377, 1261, 1095, 1026, 864, 800, 721, 704, 661 cm^{-1}.

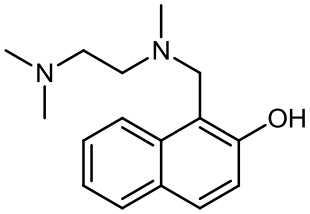


2,4-di-tert-butyl-6-((dimethylamino)methyl)phenol:

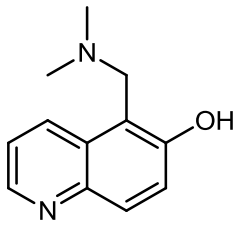
^{1}H NMR (300 MHz, $CDCl_3$) δ = 7.23 (s, 1H), 6.84 (s, 1H), 3.63 (s, 2H), 2.33 (s, 6H), 1.43 (s, 9H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ = 154.38, 140.21, 123.04, 122.71, 122.23, 115.81, 63.50, 44.22, 34.76, 34.05, 31.62, 31.55, 29.63, 29.57, 29.52;HRMS calculated forC17H30NO (M + H^+): 264.2327, found:264.4262; IR (KBr)2954, 2866, 2827, 2786, 1723, 1645, 1606, 1456, 1463, 1411, 1361, 1303, 1234, 1043, 1018, 977, 879, 840, 798, 723 cm^{-1}.



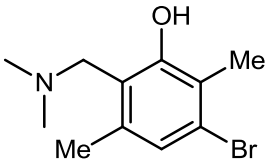
2,4-di-tert-butyl-6-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)phenol:
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.23 (d, *J*=2.4, 1H), 6.85 (d, *J*=2.3, 1H), 3.70 (s, 2H), 2.61 (dd, *J*=8.2, 5.5, 2H), 2.56 – 2.46 (m, 2H), 2.35 (d, *J*=4.2, 3H), 2.25 (s, 6H), 1.45 (s, 9H), 1.32 (d, *J*=5.3, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 154.29, 140.29, 135.47, 123.37, 122.78, 121.35, 62.15, 57.06, 54.31, 45.72, 41.83, 34.83, 34.09, 31.69, 29.60; HRMS calculated forC20H37N2O (M + H^+): 321.2906, found:321.2906; IR (KBr) 3441, 2962, 2927, 2908, 2858, 2819, 1722, 1653, 1633, 1481, 1463, 1411, 1361, 1261, 1097, 1020, 871, 801, 727, 696, 661 cm^{-1}.



1-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)naphthalen-2-ol:
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.88 (d, *J*=8.5, 1H), 7.78 (d, *J*=7.7, 1H), 7.70 (d, *J*=8.8, 1H), 7.46 (dd, *J*=11.2, 4.1, 1H), 7.28 (d, *J*=8.8, 1H), 7.14 (d, *J*=8.8, 1H), 4.13 (s, 2H), 2.75 (t, *J*=6.7, 2H), 2.62 (t, *J*=6.7, 2H), 2.39 (s, 3H), 2.32 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 156.43, 132.89, 129.13, 128.81, 128.42, 126.22, 122.29, 121.09, 119.36, 112.18, 56.53, 54.77, 54.42, 45.26, 44.65, 42.21, 29.70; HRMS calculated forC16H23N2O (M + H^+): 259.1810, found:259.1805; IR (KBr)3471, 2962, 2924, 2854, 1732, 1666, 1645, 1504, 1469, 1367, 1286, 1263, 1097, 1018, 862, 800, 729, 682 cm^{-1}.

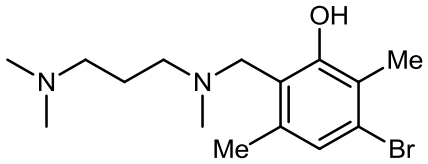


5-((dimethylamino)methyl)quinolin-6-ol:
^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.73 (dd, *J*=4.1, 1.5, 1H), 8.16 (dd, *J*=8.6, 0.6, 1H), 7.96 (d, *J*=9.1, 1H), 7.38 – 7.26 (m, 2H), 4.07 (s, 2H), 2.43 (d, *J*=1.6, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.02, 146.60, 143.74, 130.47, 129.11, 127.47, 122.75, 120.90, 110.90, 57.32, 44.64; HRMS calculated forC12H15N2O (M + H^+): 203.1184, found:203.1181; IR (KBr)3439, 3066, 2960, 2926, 2873, 2854, 2787, 1726, 1624, 1512, 1504, 1479, 1415, 1325, 1288, 1265, 1230, 1151, 1099, 1041, 1020, 991, 923, 831, 801, 717, 549 cm^{-1}.



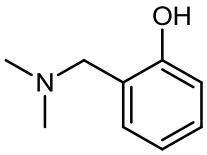
3-bromo-6-((dimethylamino)methyl)-2,5-dimethylphenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.88 (s, 1H), 3.61 (s, 2H), 2.34 (s, 6H), 2.29 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.34, 134.44, 124.17, 123.92, 122.96, 118.29, 58.37, 44.24, 19.24, 15.15; HRMS calculated forC11H17BrNO (M + H^+): 258.0494, found:258.0489; IR (KBr) 2960, 2924, 2866, 2835, 2785, 2733, 1728, 1606, 1568, 1456, 1398, 1296, 1261, 1207, 1178, 1099, 1089, 1066, 1041, 958, 800, 786, 732, 702, 582 cm^{-1}.



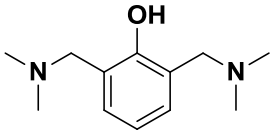
3-bromo-6-(((3-(dimethylamino)propyl)(methyl)amino)methyl)-2,5-dimethylphenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.88 (s, 1H), 3.65 (s, 2H), 2.64 – 2.59 (m, 2H), 2.56 – 2.47 (m, 2H), 2.34 (s, 3H), 2.30 (s, 3H), 2.29 (s, 3H), 2.26 (s, 3H), 2.19 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.04, 134.55, 124.06, 123.85, 122.99, 118.59, 56.71, 56.09 \, 54.50, 45.50, 41.59, 19.27, 15.20; HRMS calculated for C14H24BrN2O (M + H^+): 315.1072, found:315.1062; IR (KBr) 2962, 2926, 2854, 1734, 1674, 1564, 1456, 1400, 1261, 1095, 1041, 864, 801, 731, 702, 661 cm^{-1}.



2-((dimethylamino)methyl)phenol:

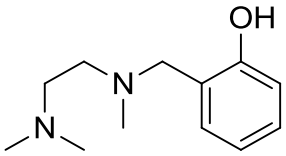
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.19 (dd, *J*=11.2, 4.2, 1H), 6.99 (d, *J*=7.3, 1H), 6.90 – 6.74 (m, 2H), 3.67 (s, 2H), 2.35 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 157.97, 128.64, 128.23, 121.82, 118.85, 115.95, 77.28, 76.96, 76.64, 62.73, 44.37, 29.62;HRMS calculated forC9H14NO (M + H^+): 152.1075, found:152.1069; IR (KBr)3450, 2962, 2926, 2854, 1718, 1635,1448, 1498, 1409, 1261, 1093, 1026, 866, 801, 704, 667 cm^{-1}.



2,6-bis((dimethylamino)methyl)phenol:

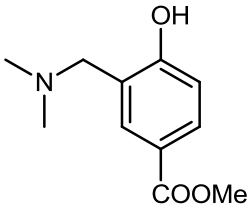
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.03 (d, *J*=7.4, 2H), 6.74(t, *J*=7.4, 1H), 3.56 (s, 4H), 2.30 (s, 12H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 156.69, 128.84, 123.14, 118.15, 60.28, 44.74; HRMS calculated forC12H21N2O (M + H^+): 209.1576, found:209.1640; IR

(KBr)2928, 2855, 2816, 2777, 1597, 1458,1406, 1286, 1177, 1078, 1041, 1018, 844, 769, 752 cm^{-1}.



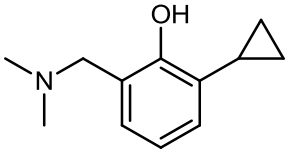
2-(((3-(dimethylamino)propyl)(methyl)amino)methyl)phenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.20 – 7.18 (m, 1H), 7.03 – 6.96 (m, 1H), 6.86 – 6.84 (m, 1H), 6.79-6.76(m, 1H), 3.63 (d, *J*=8.7, 2H), 2.63 (dd, *J*=9.8, 3.9, 2H), 2.53 (dd, *J*=9.9, 4.0, 2H), 2.29 (d, *J*=3.1, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.69, 128.87, 128.65, 128.60, 122.58, 118.68, 116.22, 62.78, 59.55, 56.48, 54.22, 45.22, 44.40, 41.95;HRMS calculated forC12H21N2O (M + H^+): 209.1654, found:209.1652; IR (KBr) 3045, 2958, 2918, 2848, 2818, 2791, 1726, 1651, 1614, 1589, 1487, 1458, 1383, 1294, 1259, 1180, 1122, 1099, 1026, 933, 800, 752, 721 cm^{-1}.



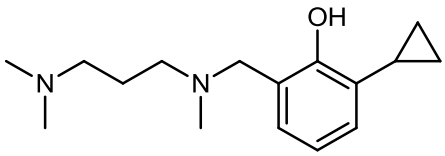
methyl 3-((dimethylamino)methyl)-4-hydroxybenzoate:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.89 (dd, *J*=8.5, 2.1, 1H), 7.72 (s, 1H), 6.85 (d, *J*=8.5, 1H), 3.89 (s, 3H), 3.71 (s, 2H), 2.35 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 167.02, 162.84, 130.87, 130.19, 121.40, 120.73, 115.95, 62.41, 51.72, 44.34, 29.65; HRMS calculated for C12H18NO (M + H^+): 192.1388, found:192.1384; IR (KBr) 2956, 2916, 2848, 2789, 1714, 1614, 1595, 1494, 1469, 1435, 1292, 1265, 1228, 1192, 1180, 1101, 1041, 1022, 993, 800, 771, 746, 684, 651, 632 cm^{-1}.



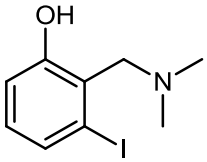
2-cyclopropyl-6-((dimethylamino)methyl)phenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.79 (dd, *J*=9.4, 6.7, 1H), 6.74 – 6.67 (m, 2H), 3.66 (s, 2H), 2.32 (d, *J*=32.5, 6H), 2.20 (m, 1H), 0.98 – 0.82 (m, 2H), 0.68 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 156.66, 130.04, 125.43, 124.15, 121.02, 118.51, 62.70, 44.30, 9.13, 7.31; HRMS calculated for C12H18NO (M + H^+): 192.1388, found:192.1384; IR (KBr)3080, 3047, 2999, 2981, 2960, 2926, 2858, 2827, 2785, 2736, 2648, 1728, 1595, 1498, 1463, 1431, 1408, 1375, 1352, 1300, 1282, 1255, 1236, 1213, 1178, 1098, 1043, 1018, 956, 867, 844, 821, 771, 744, 684, 609, 563 cm^{-1}.



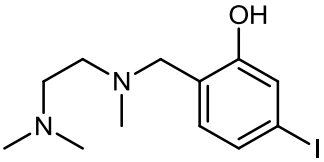
2-cyclopropyl-6-(((3-(dimethylamino)propyl)(methyl)amino)methyl)phenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.90 – 6.62 (m, 3H), 3.67 (s, 2H), 2.74 – 2.60 (m, 2H), 2.60 – 2.45 (m, 2H), 2.45 – 2.13 (m, 6H), 0.97 – 0.88 (m, 2H), 0.65 (dd, *J*=5.4, 4.1, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 156.41, 130.17, 129.53, 125.73, 123.95, 121.36, 118.46, 115.29, 60.75, 56.83, 54.58, 45.50, 41.73, 29.62, 9.10, 7.46;HRMS calculated for C12H18NO ($M + H^+$): 192.1388, found:192.1384; IR (KBr) 3080, 3055, 2981, 2926, 2848, 2821, 2794, 1734, 1676, 1458, 1406, 1375, 1352, 1261, 1236, 1091, 1018, 866, 798, 738, 704 cm^{-1}.



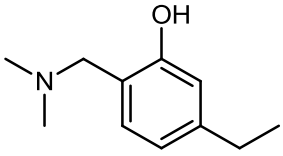
2-((dimethylamino)methyl)-3-iodophenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45 – 7.26 (m, 1H), 7.26 – 6.78 (m, 2H), 3.86 (s, 2H), 2.38 (s, 6H);^{13}C NMR (100 MHz, $CDCl_3$) δ = 158.42, 129.88 , 123.55, 116.80, 98.95, 67.22, 44.15; HRMS calculated forC9H13INO ($M + H^+$): 278.0042, found:278.0037; IR (KBr) 3585, 2956, 2916, 2848, 2787, 2713, 1732, 1595, 1568, 1463, 1431, 1384, 1362, 1310, 1271, 1180, 1099, 1041, 1018, 968, 867, 794, 771, 709 cm^{-1}.



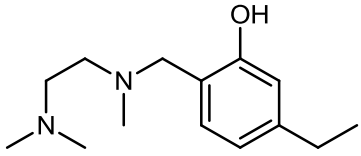
2-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)-5-iodophenol:

^{1}H NMR (500 MHz, $CDCl_3$) δ = 7.44 (d, *J*=6.7, 1H), 7.21 – 6.95 (m, 2H), 6.86 (d, *J*=7.6, 1H), 4.28 (s, 1H), 3.58 (t, *J*=35.8, 2H), 3.58 (t, *J*=35.8, 2H), 2.89 (s, 6H), 2.74 (s, 3H));^{13}C NMR (75 MHz, $CDCl_3$) δ = 155.16, 131.88, 124.89, 120.79, 111.02, 99.91, 60.52, 51.90, 49.33, 42.81, 40.67, 29.38;HRMS calculated forC12H20IN2O ($M + H^+$): 335.0620, found:335.0614; IR (KBr) 3429, 2960, 2928, 2848, 1687, 1668, 1645, 1581, 1444, 1409, 1373, 1255, 1199, 1172, 1099, 1018, 864, 844, 800, 719cm^{-1}.



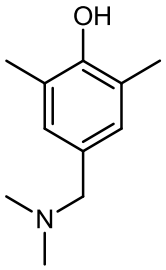
2-((dimethylamino)methyl)-5-ethylphenol:

^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.90 (d, *J*=7.6, 1H), 6.72 (d, *J*=1.2, 1H), 6.65 (dd, *J*=7.6, 1.5,1H), 3.63 (s, 2H), 2.62 (q, *J*=7.6, 2H), 2.36 (d, *J*=8.9, 6H), 1.24 (dd, *J*=20.4, 12.8, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 157.86, 145.09, 128.04, 119.11, 118.37, 115.34, 62.55, 44.40, 28.53, 15.49; HRMS calculated forC11H18NO ($M + H^+$): 180.1388, found:180.1386; IR (KBr) 3442, 2962, 2924, 2854, 1730, 1633, 1469, 1446, 1402, 1261, 1095, 1026, 862, 800, 727, 704 cm^{-1}.



2-(((2-(dimethylamino)ethyl)(methyl)amino)methyl)-5-ethylphenol:

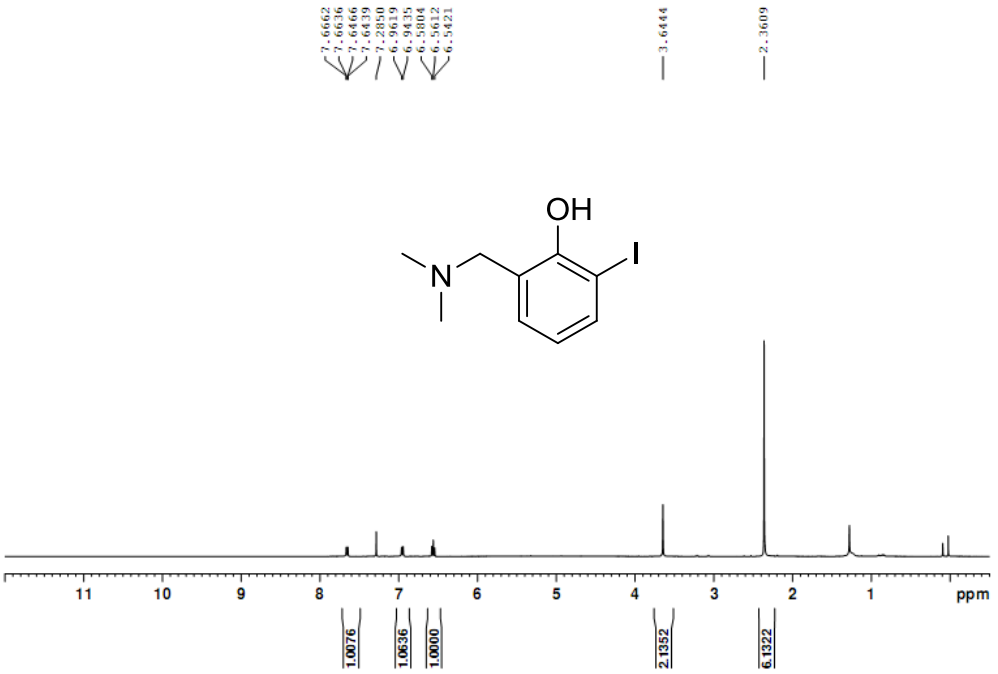
δ_H (400 MHz, $CDCl_3$) 6.89 (1 H, dd, *J* 7.5, 5.3), 6.80 – 6.59 (2 H, m), 3.62 (2 H, d, *J* 6.8), 2.90 – 2.46 (6 H, m), 2.41 – 2.21 (9 H, m), 1.47 – 0.97 (3 H, m); δ_C (100MHz, $CDCl_3$) 157.83, 145.12, 128.68, 119.75, 118.25, 115.61, 62.55, 59.40, 56.51, 54.22, 45.23, 41.93, 28.51, 15.34; HRMS calculated forC14H25N2O ($M + H^+$): 237.1967, found:237.1964;IR (KBr) 2958, 2929, 2854, 2821, 2785, 1728, 1624, 1581, 1500, 1458, 1382, 1373, 1261, 1155, 1097, 1043, 1018, 931, 866, 844, 800, 729 cm^{-1}.



4-((dimethylamino)methyl)-2,6-dimethylphenol:

^1H NMR (400 MHz, $CDCl_3$) δ = 6.91 (s, 2H), 3.32 (s, 2H), 2.24 (d, *J*=5.3, 6H);^{13}C NMR (75 MHz, $CDCl_3$) δ = 150.13, 129.67, 129.42, 125.63, 63.67, 45.04, 15.87; HRMS calculated forC11H18NO ($M + H^+$): 180.1388, found:180.1383; IR (KBr)3624, 2962, 2916, 2848, 1718, 1653, 1473, 1400, 1261, 1093, 1024, 864, 798, 732, 700cm^{-1}.

Copies of ^1H NMR and ^{13}C NMR Spectra of CDC and CAC products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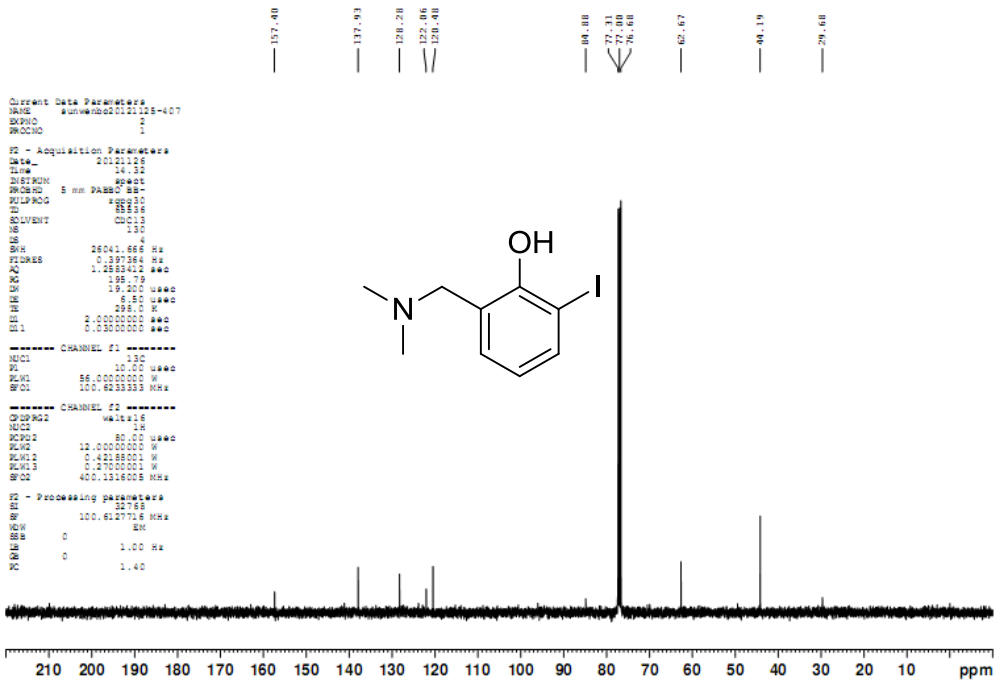
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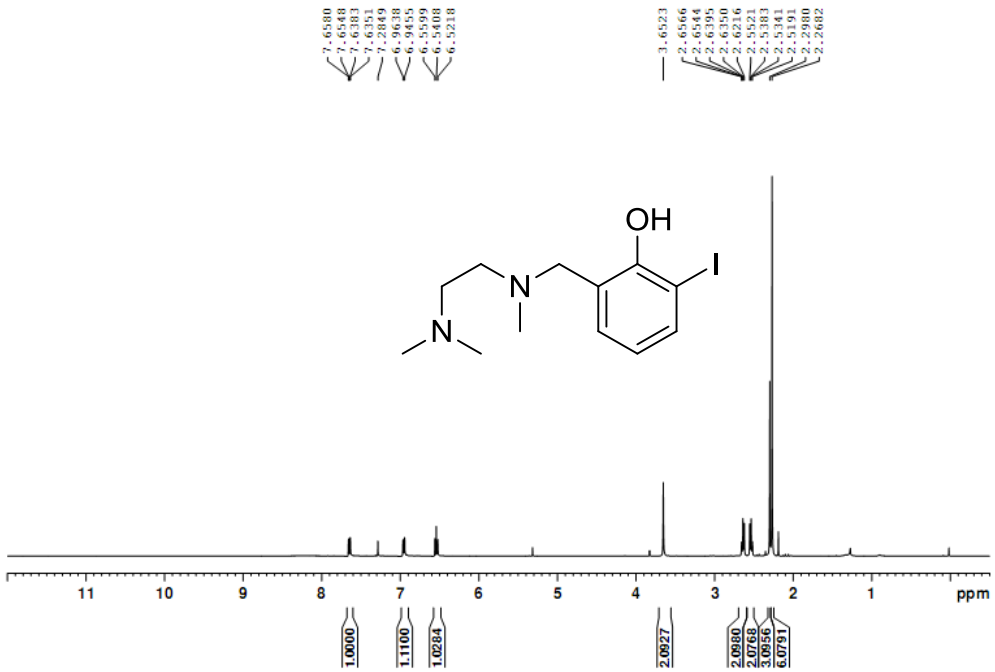
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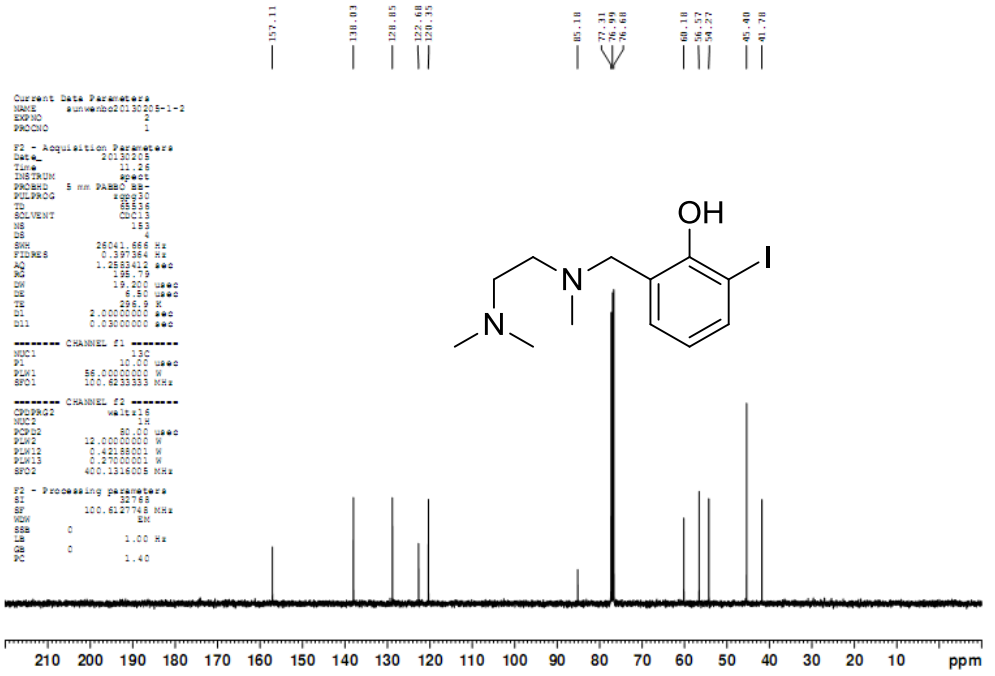
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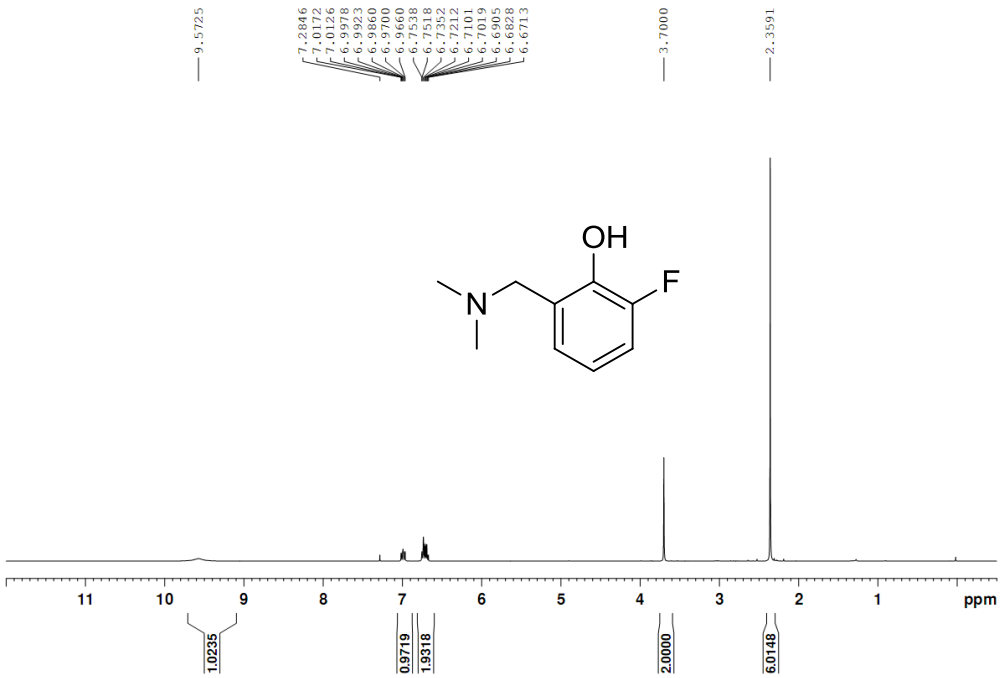

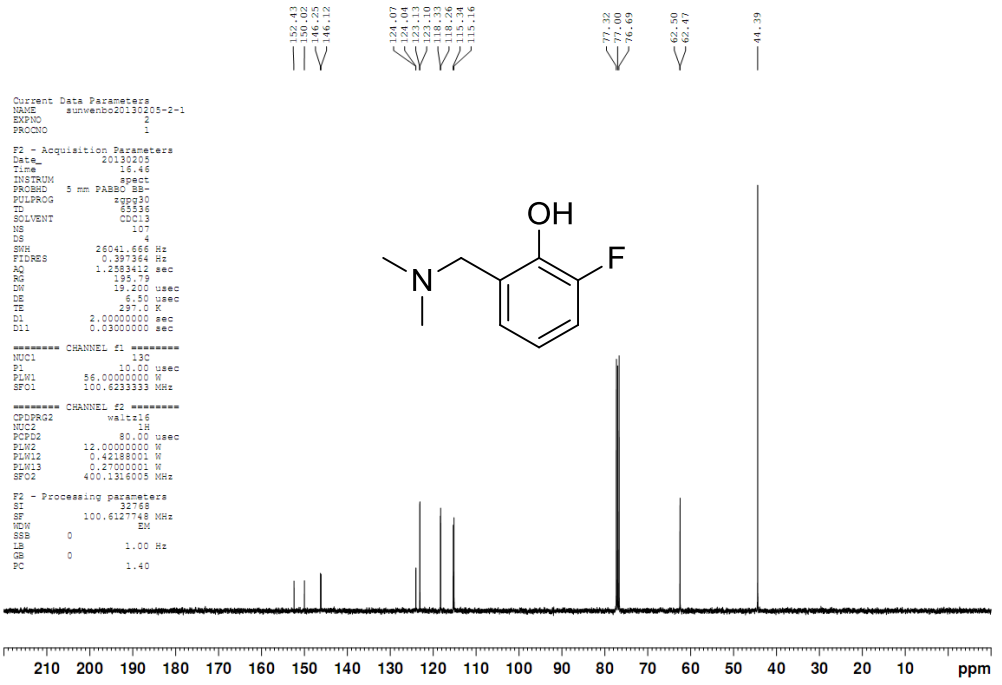


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PLW1             56.00000000 W
SFO1             100.6233333 MHz

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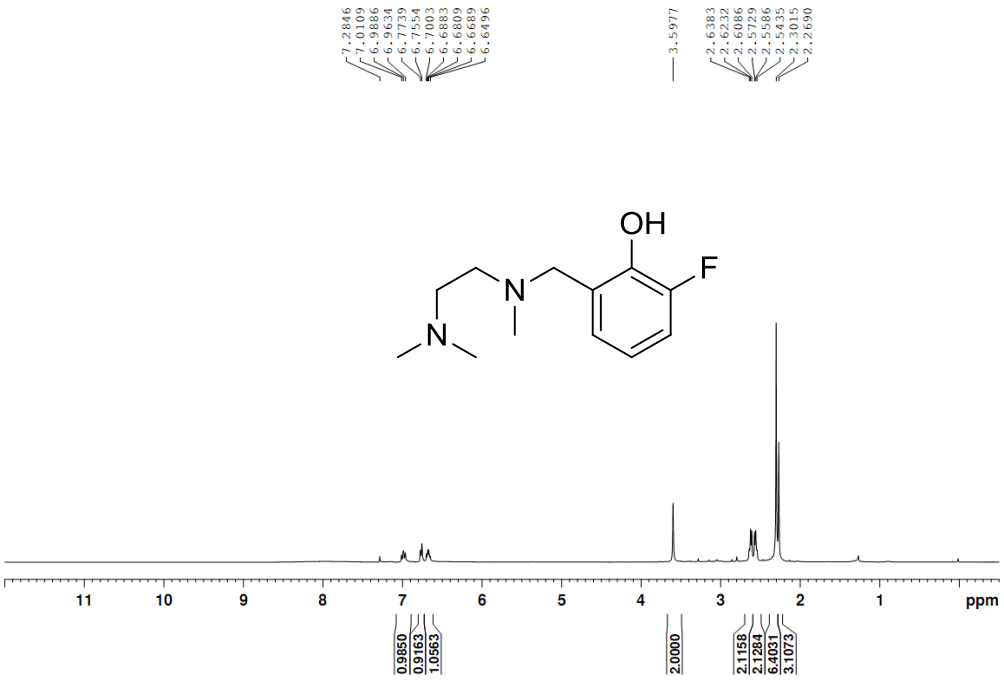

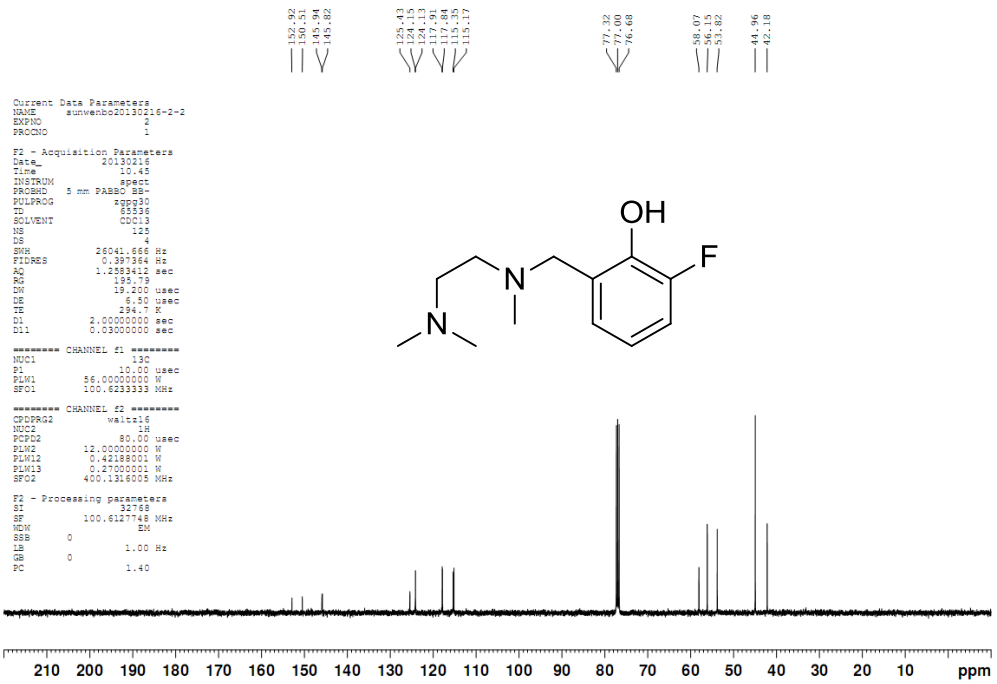


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F2 - Processing parameters
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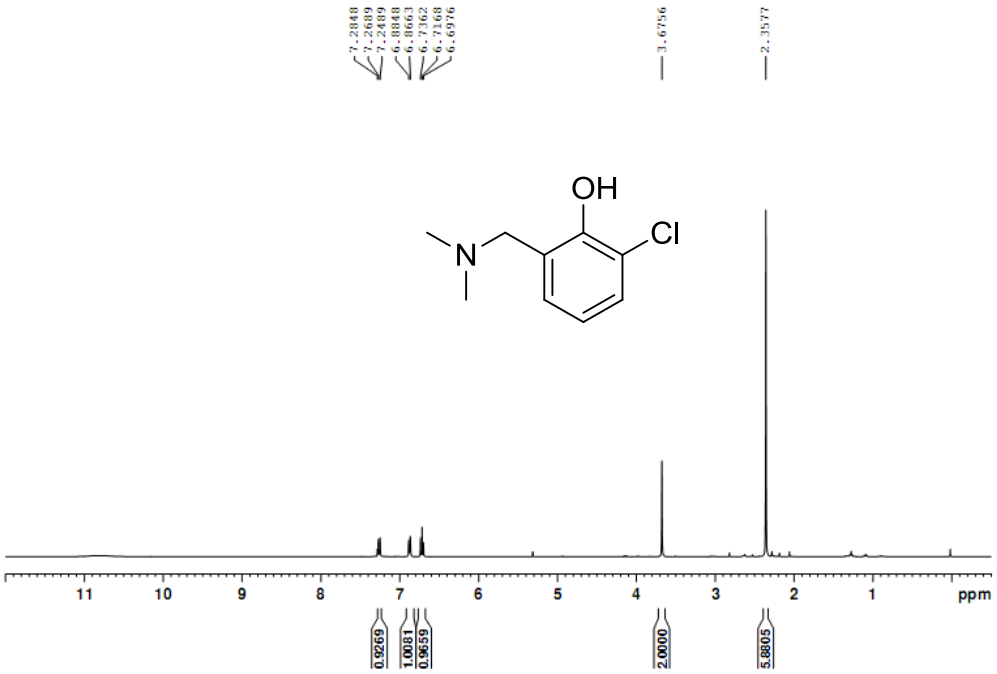
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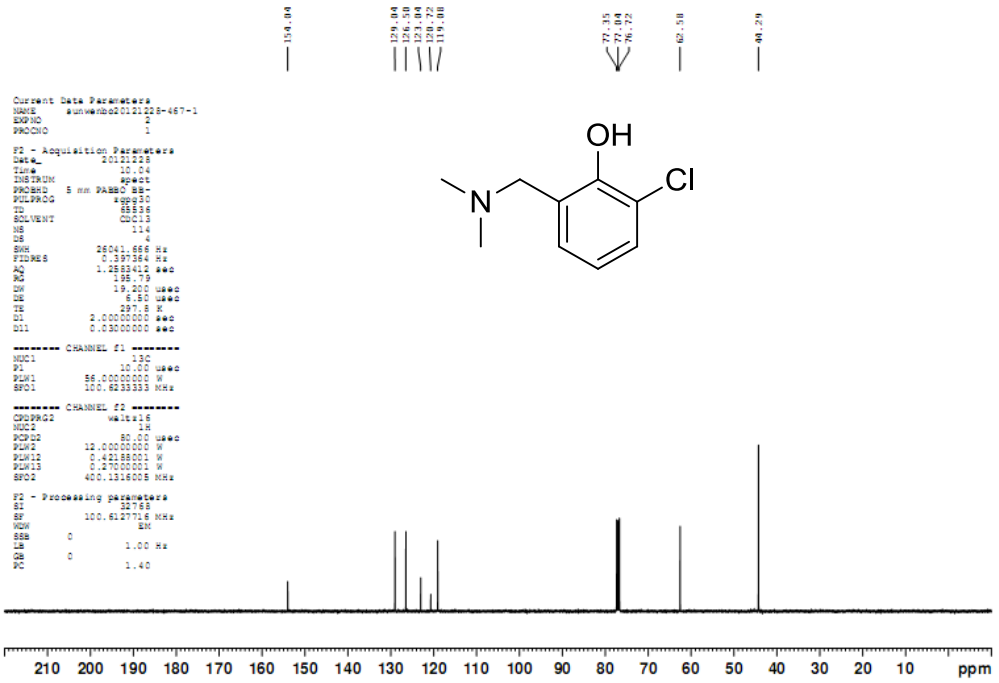
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D11 0.03000000 sec
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PLW13 0.27000001 W
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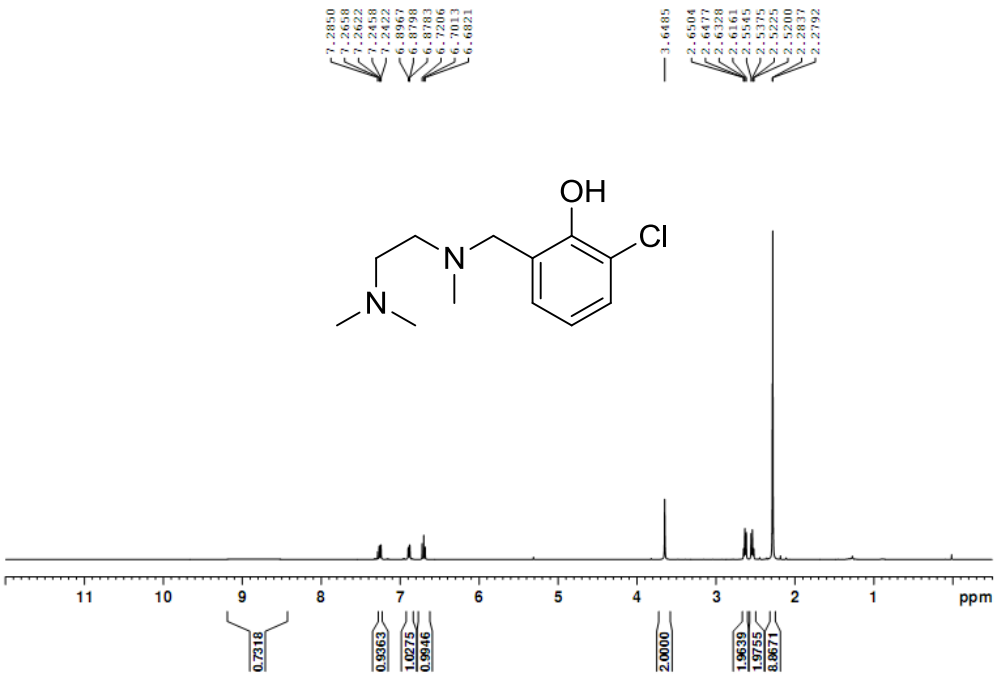
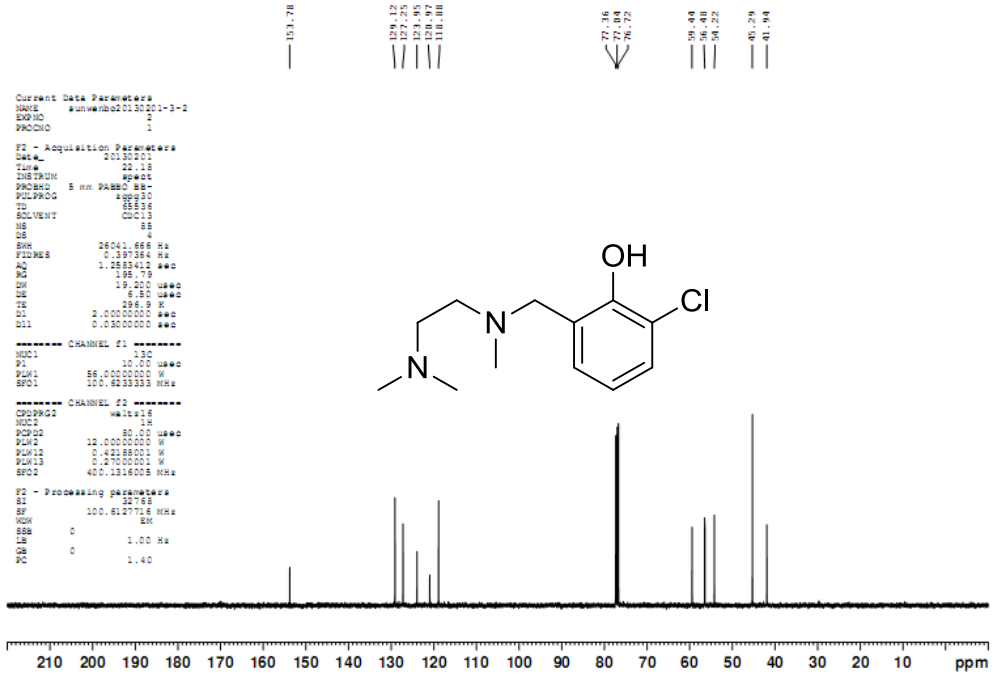
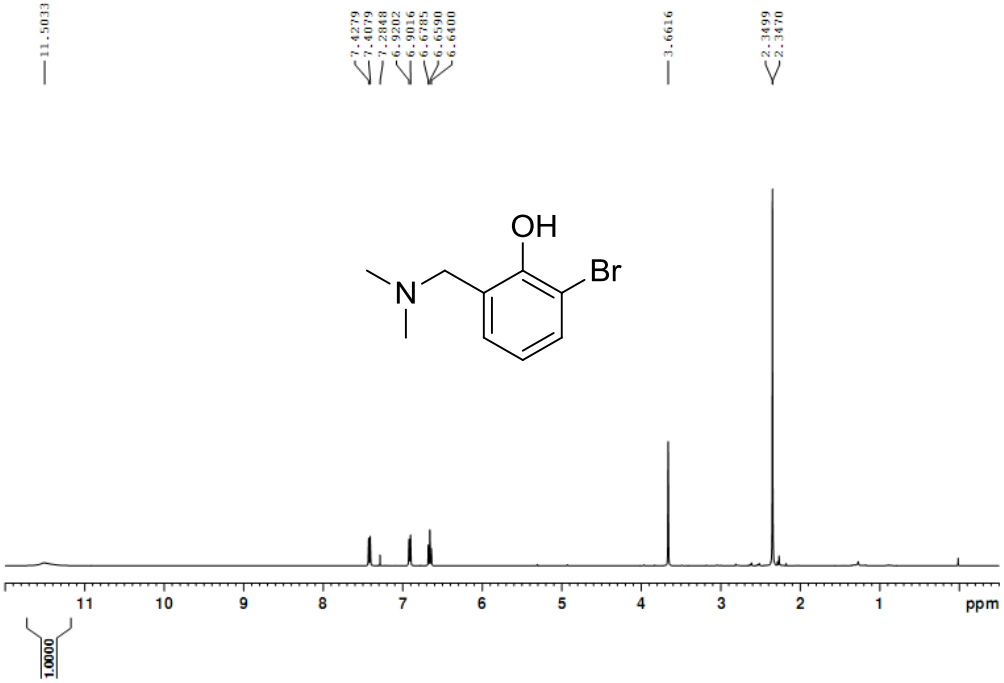
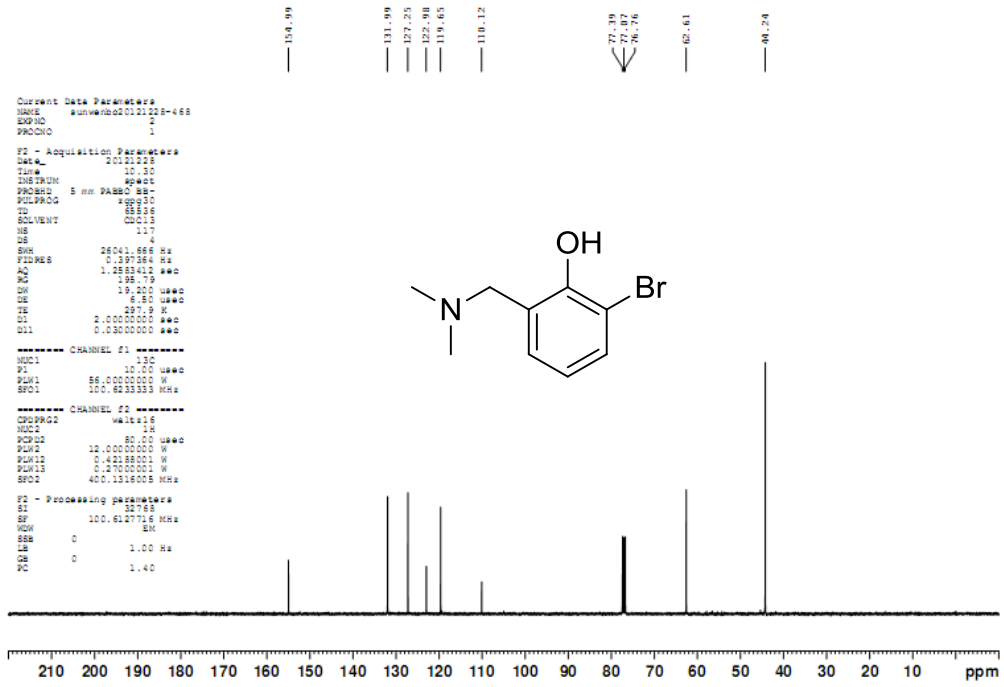
SF 100.6127716 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

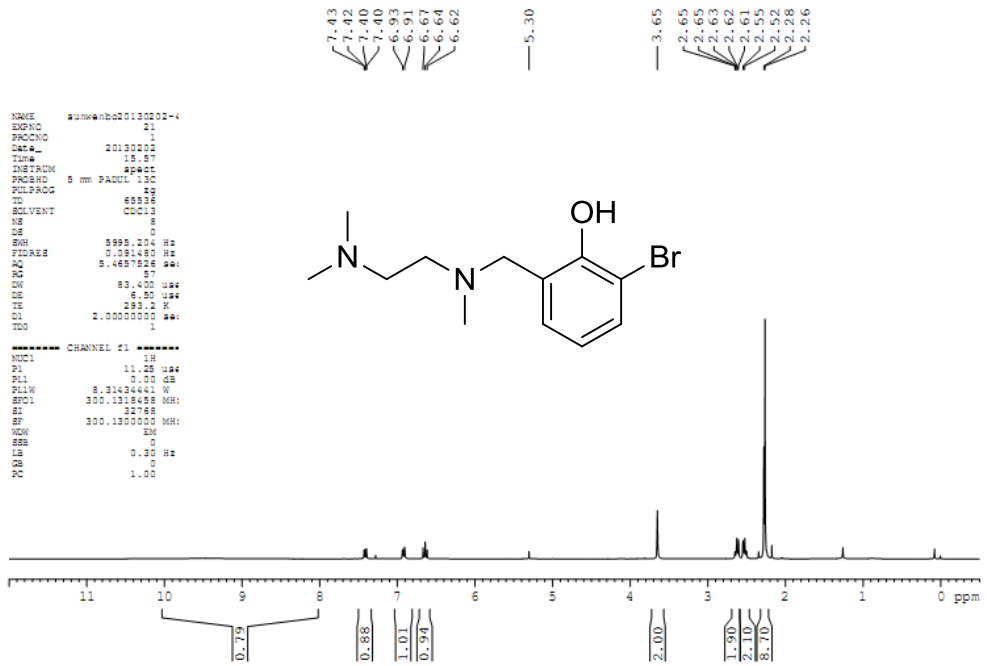

7.2850
7.2658
7.2622
7.2458
7.2422
6.8967
6.8798
6.8783
6.7206
6.7013
6.6821
3.6485
2.6504
2.6477
2.6328
2.6161
2.5545
2.5375
2.5225
2.5200
2.2837
2.2792
OH
Cl
N
N
11
10
9
8
7
6
5
4
3
2
1
ppm
0.7318
0.9363
1.0275
0.9946
2.0000
1.9639
1.9755
8.8671

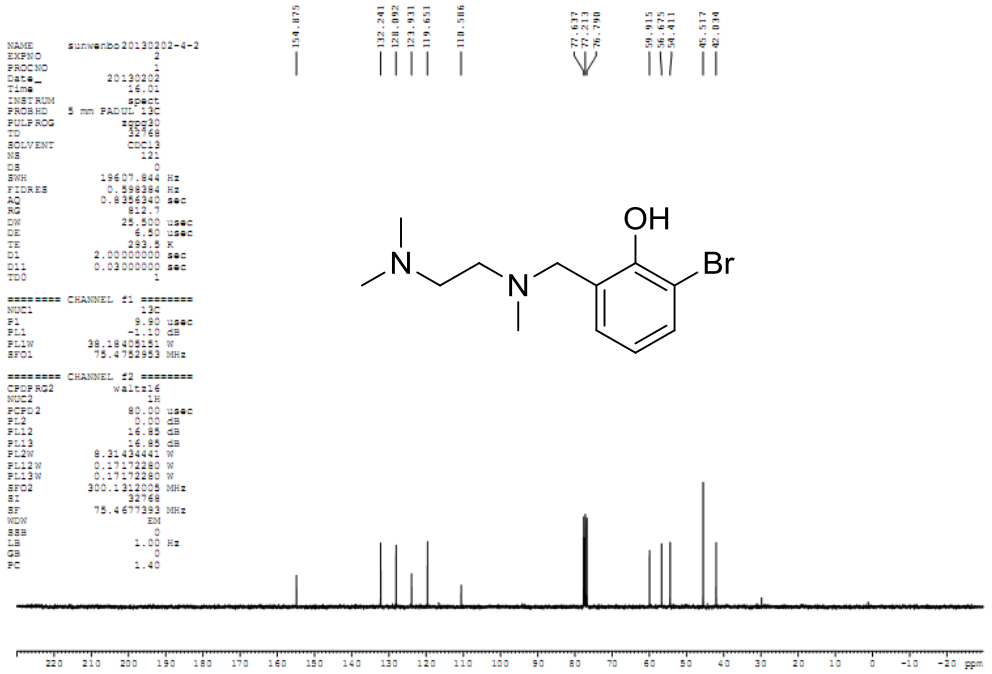

153.78
129.12
127.25
123.95
120.97
118.88
77.36
77.04
76.72
59.44
56.48
54.22
45.29
41.94
OH
Cl
N
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

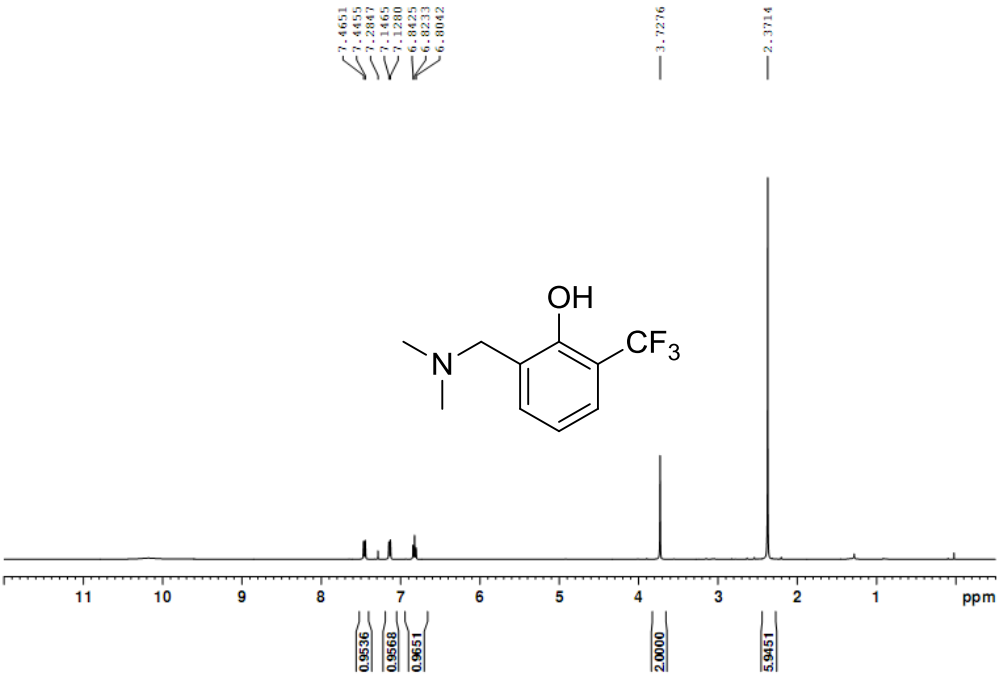


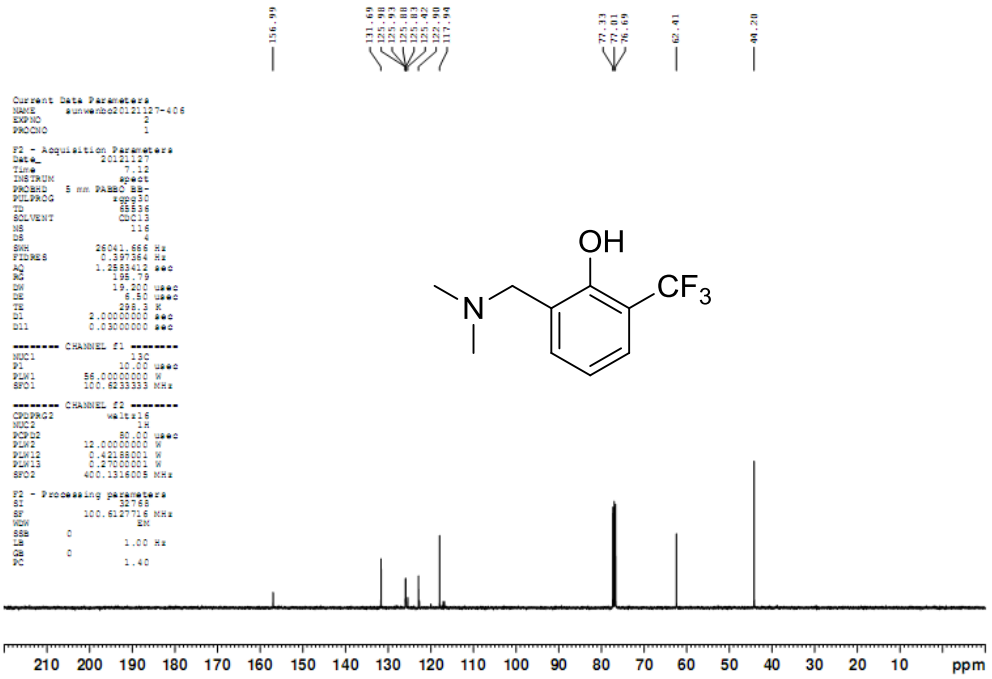


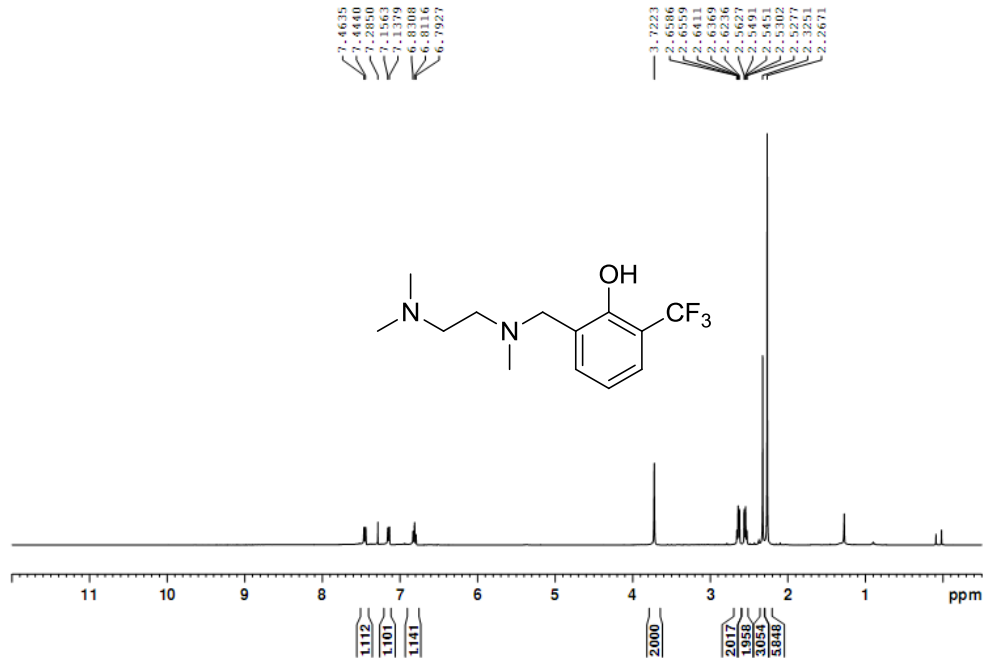

```
Current Data Parameters
NAME      sunwenbo20121228-468
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20121228
Time                   10.30
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                  65536
SOLVENT               CDCl3
NS                      117
DS                        4
SWH               26041.666 Hz
FIDRES            0.397364 Hz
AQ                1.2583412 sec
RG                    195.79
DW                  19.200 usec
DE                   6.50 usec
TE                   297.9 K
D1              2.00000000 sec
D11             0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PLW1          56.00000000 W
SFO1          100.6233333 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PLW2          12.00000000 W
PLW12          0.42188001 W
PLW13          0.27000001 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF            100.6127716 MHz
WDW                       EM
SSB                    0
LB                    1.00 Hz
GB                     0
PC                    1.40
```

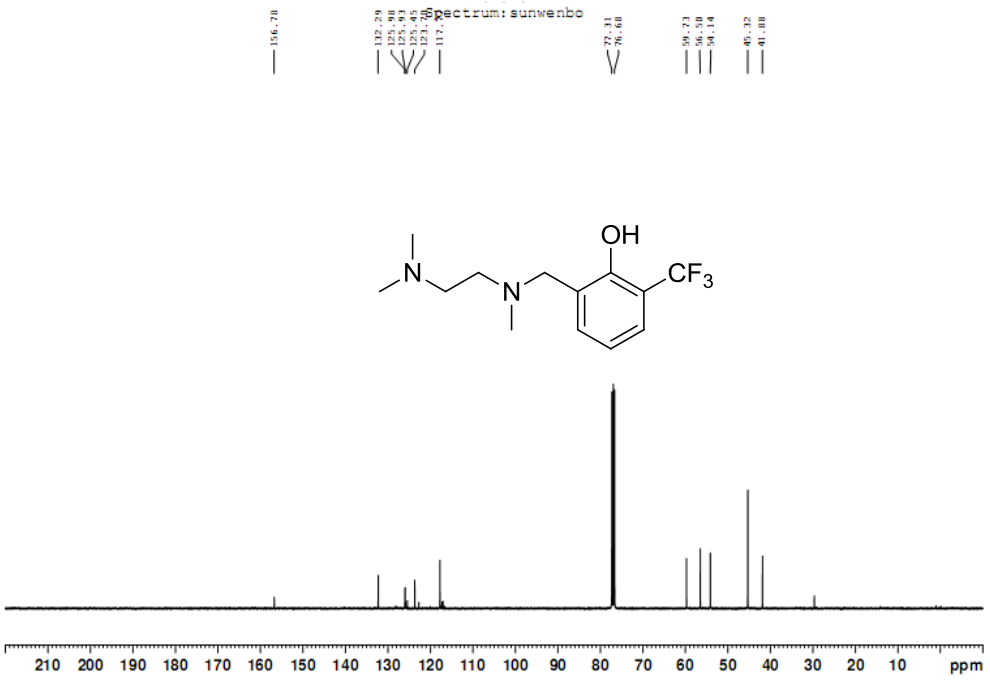

7.43
7.42
7.40
7.40
6.93
6.91
6.67
6.64
6.62
5.30
3.65
2.65
2.65
2.63
2.62
2.61
2.55
2.52
2.28
2.26
OH
Br
N
N
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.79
0.88
1.01
0.94
2.00
1.90
2.10
8.70

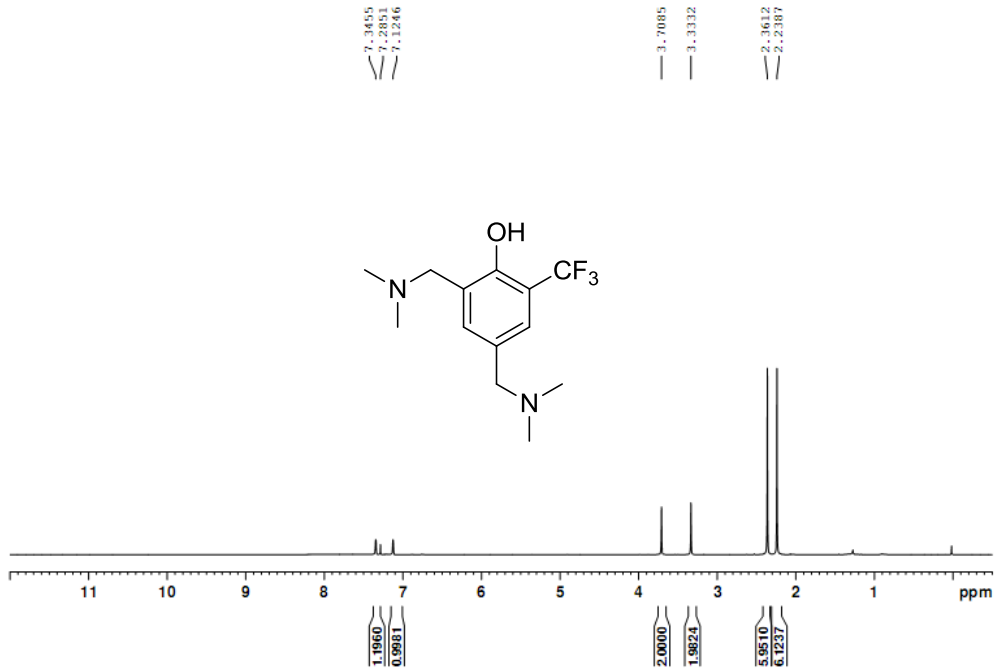

154.875
132.241
128.892
123.931
119.651
110.586
77.637
77.213
76.798
59.915
56.675
54.411
45.517
42.894
OH
Br
N
N
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20 ppm

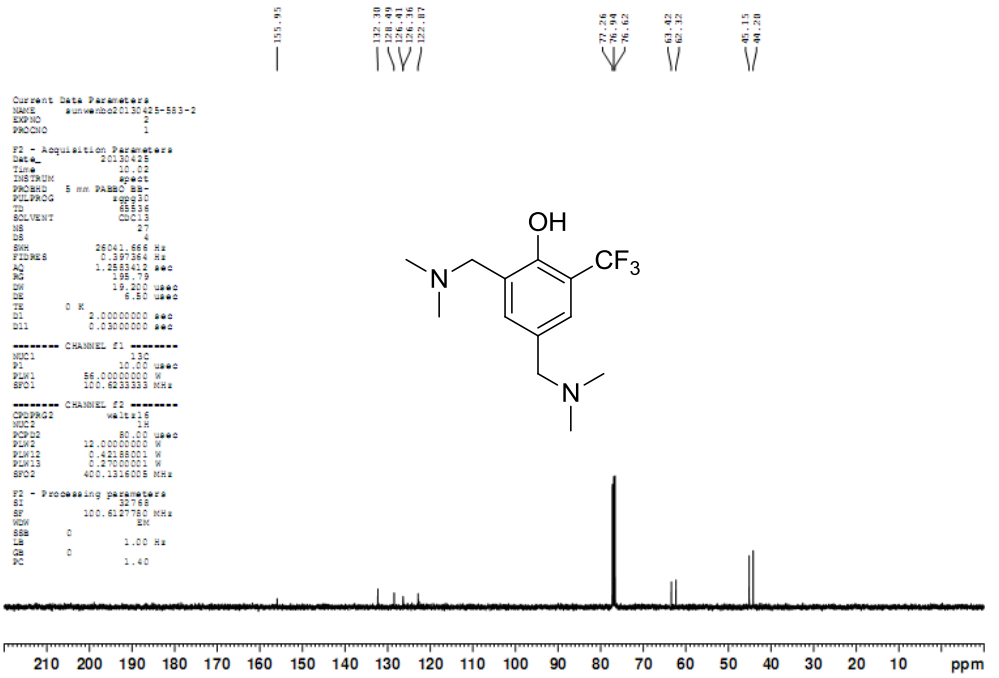

7.4651
7.4455
7.2847
7.1465
7.1260
6.8625
6.8233
6.8042
3.7276
2.3714
OH
CF3
N
11
10
9
8
7
6
5
4
3
2
1
ppm
0.9536
0.9568
0.9651
2.0000
5.9451


156.99
131.69
125.96
125.93
125.88
125.83
125.42
122.98
117.94
77.33
77.01
76.69
62.41
44.20
Current Data Parameters
NAME sunwenbo20121127-406
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121127
Time 7.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 116
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 198.79
DW 19.200 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
======= CHANNEL f1 =======
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======= CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127716 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
CF3
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


7.4635
7.4440
7.2850
7.1563
7.1379
6.8308
6.8116
6.7927
3.7223
2.6586
2.6559
2.6411
2.6369
2.6236
2.5627
2.5491
2.5451
2.5302
2.5277
2.3251
2.2671
OH
CF3
N
N
11
10
9
8
7
6
5
4
3
2
1
ppm
1112
1101
1141
2000
2017
1958
3054
5848


156.78
132.29
125.98
125.93
125.45
123.70
117.76
77.31
76.68
59.73
56.50
54.14
45.32
41.88
OH
CF3
N
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm





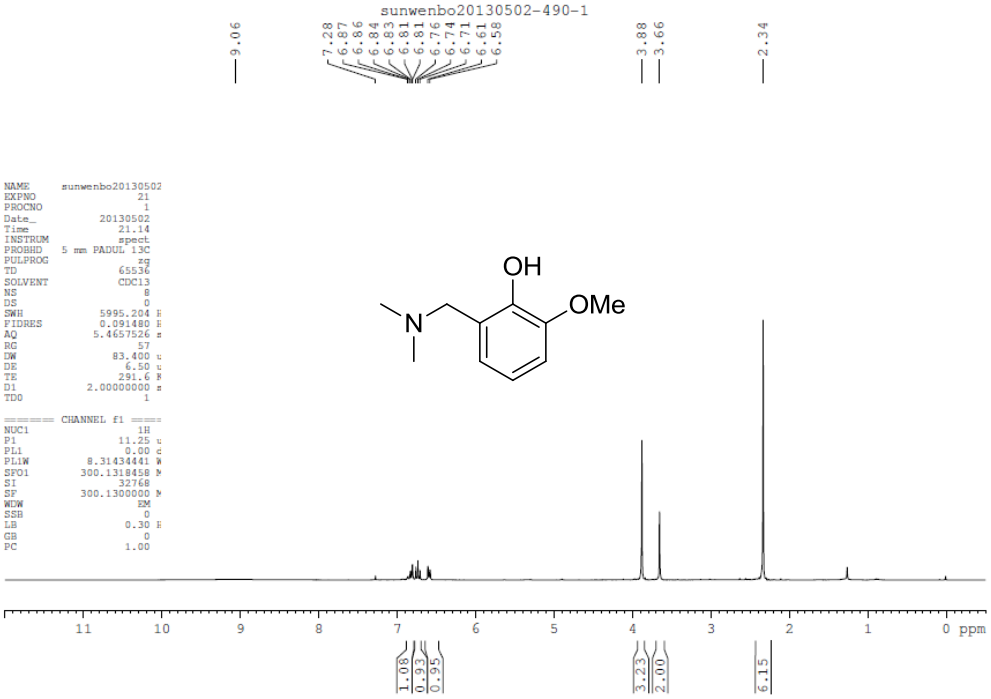
Current Data Parameters
NAME sunwenbo20130425-583-2
EXPNO 2
PROCNO 1

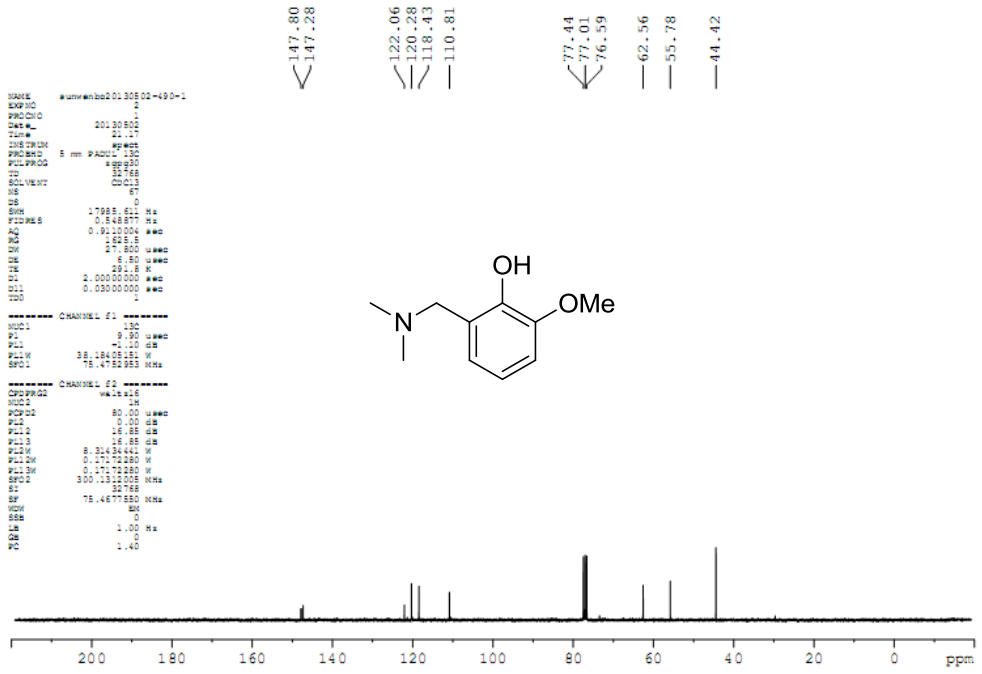
F2 - Acquisition Parameters
Date_ 20130425
Time 10.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 27
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583413 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 0 K
D1 2.00000000 sec
D11 0.03000000 sec

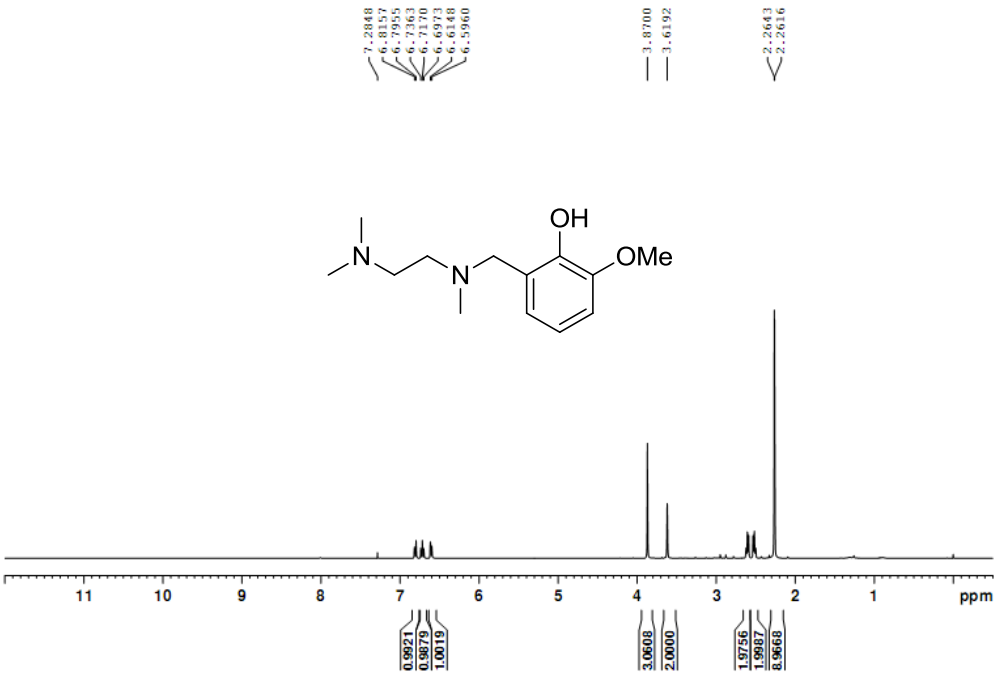
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz

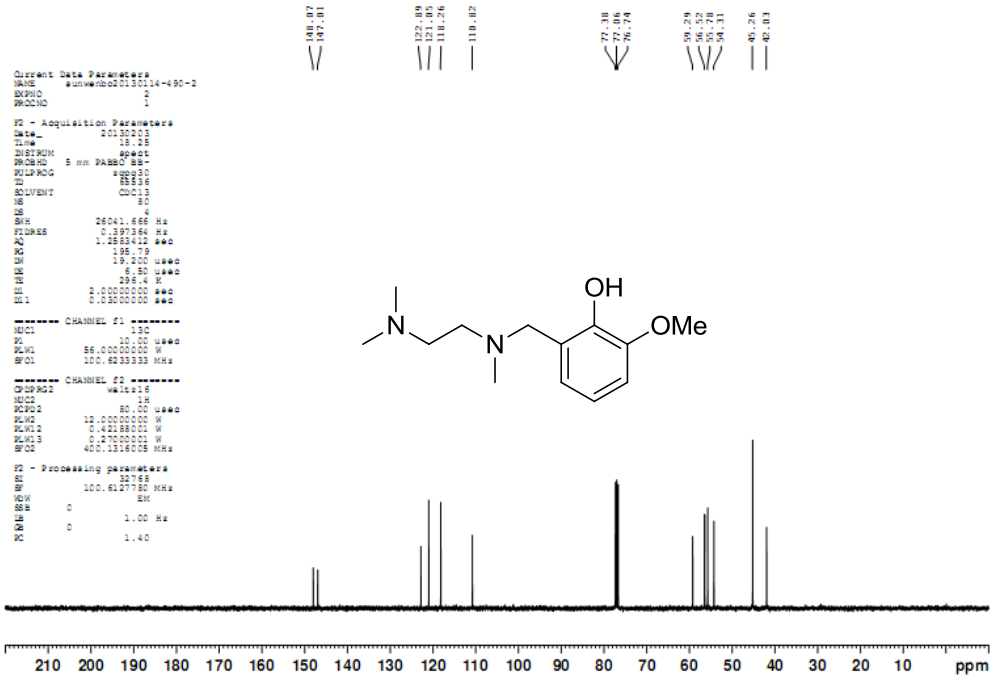
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42185501 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz

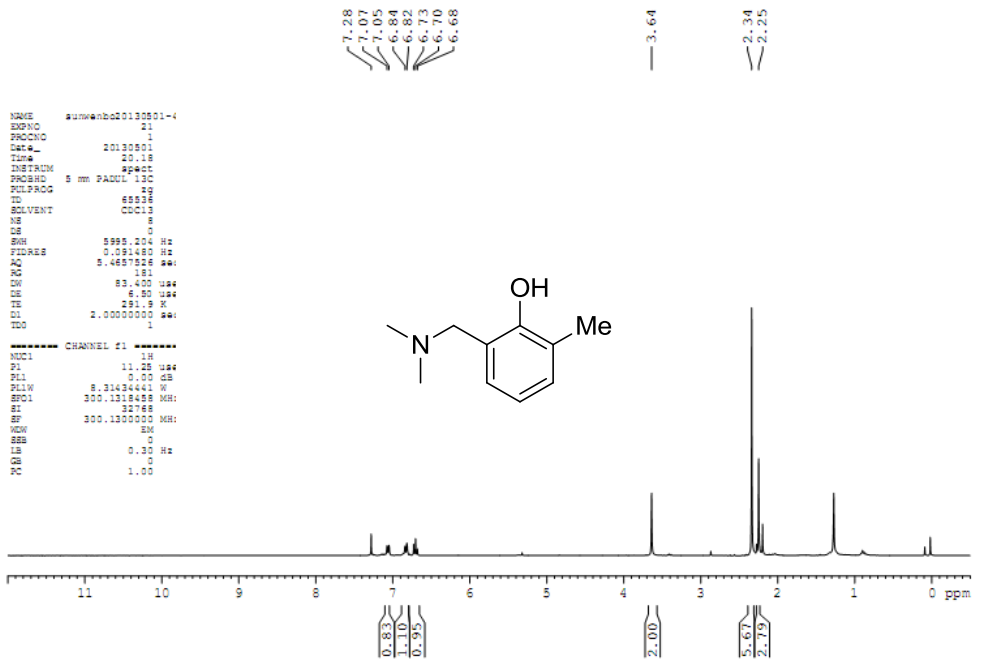
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

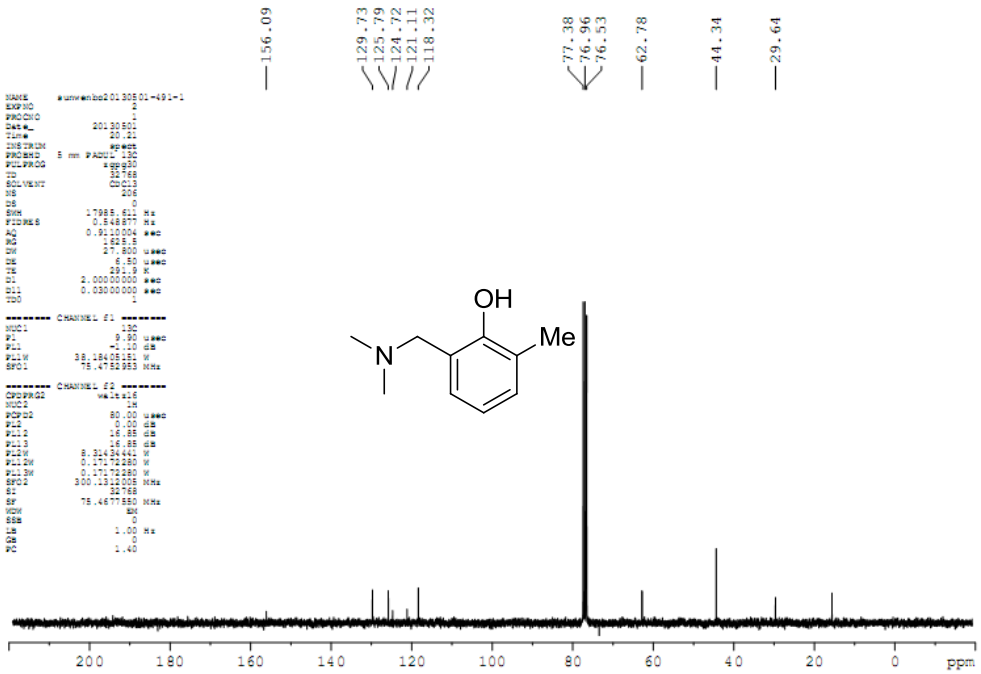

sunwenbo20130502-490-1
9.06
7.28
6.87
6.86
6.84
6.83
6.81
6.81
6.76
6.74
6.71
6.61
6.58
3.88
3.66
2.34
OH
OMe
N
ppm
1.08
0.93
0.95
3.23
2.00
6.15

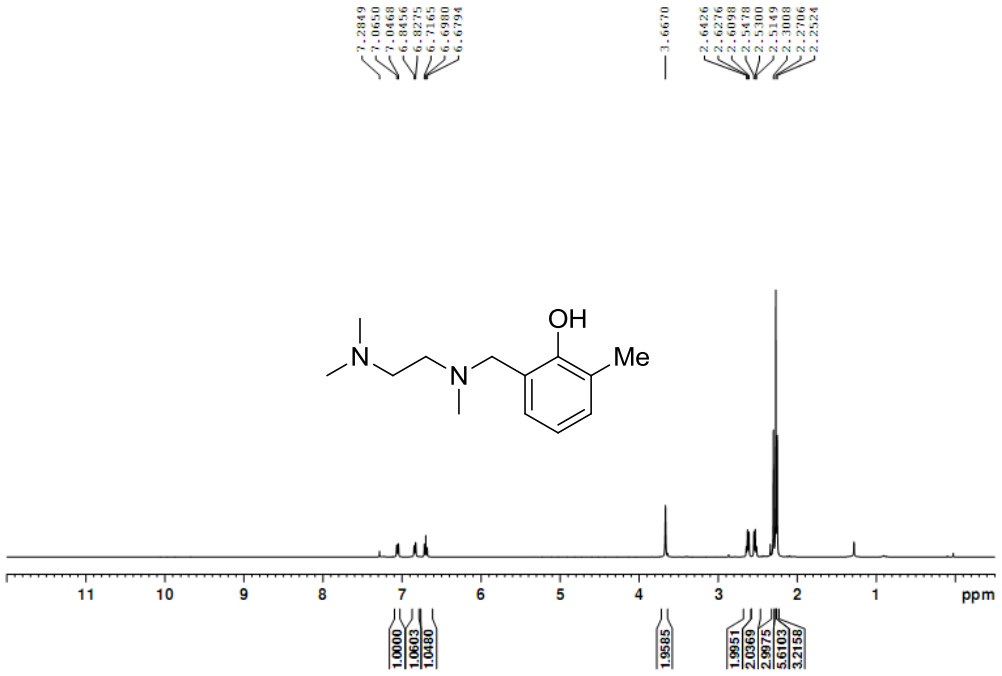

147.80
147.28
122.06
120.28
118.43
110.81
77.44
77.01
76.59
62.56
55.78
44.42
OH
OMe
N
ppm

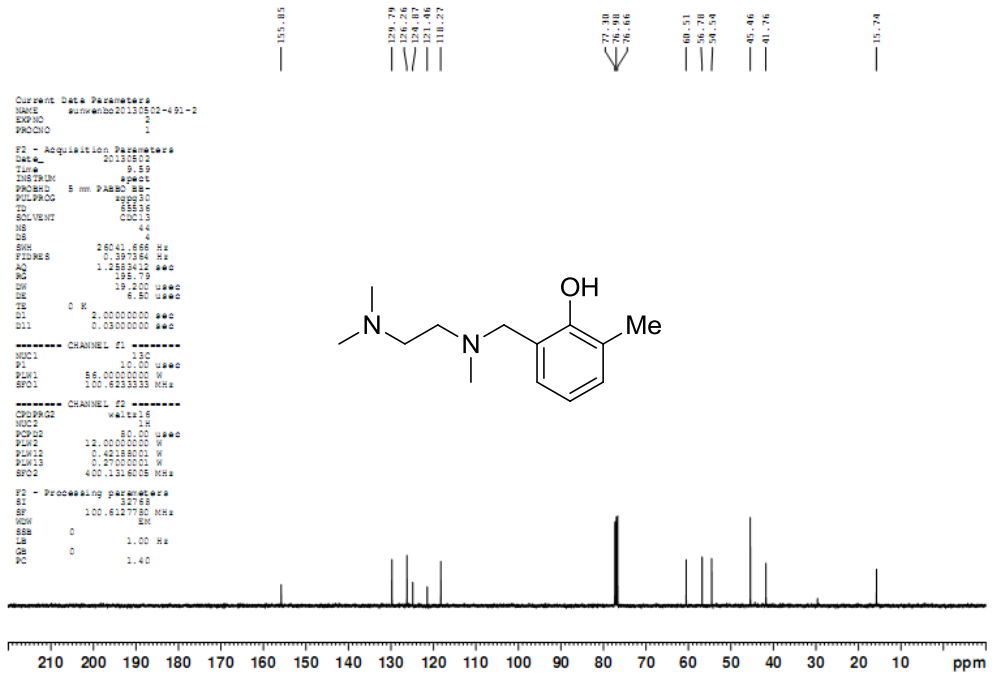

7.2848
6.8157
6.7955
6.7363
6.7170
6.6973
6.6148
6.5960
3.8700
3.6192
2.2643
2.2616
OH
N
N
OMe
11
10
9
8
7
6
5
4
3
2
1
ppm
0.9921
0.9879
1.0019
3.0608
2.0000
1.9756
1.9987
8.9668

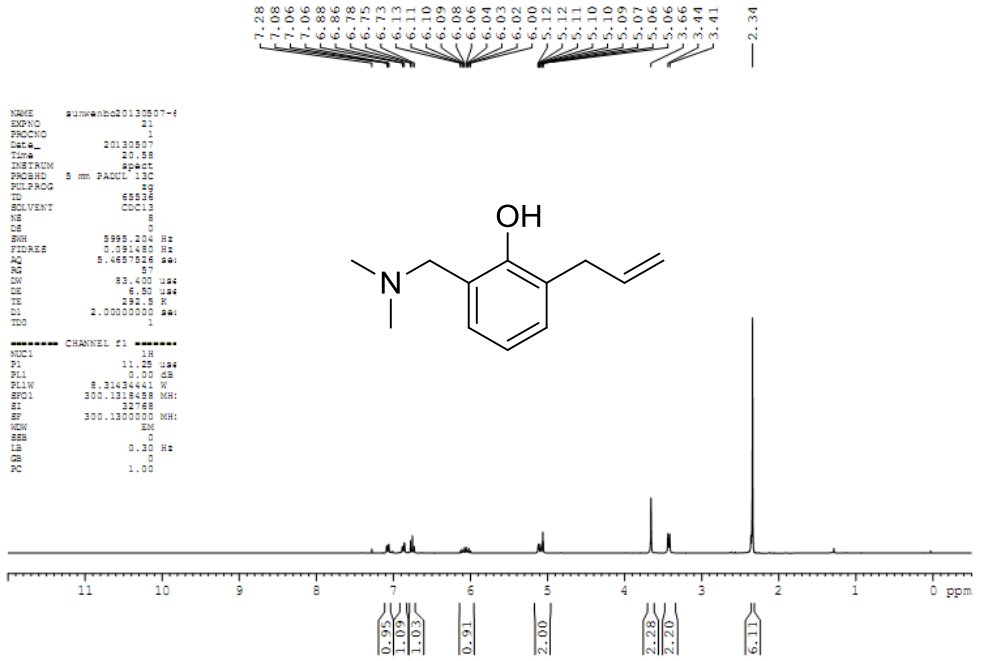

148.07
147.01
122.89
121.85
118.26
110.82
72.38
72.06
76.74
59.29
56.52
55.78
54.31
45.26
42.83
OH
N
N
OMe
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

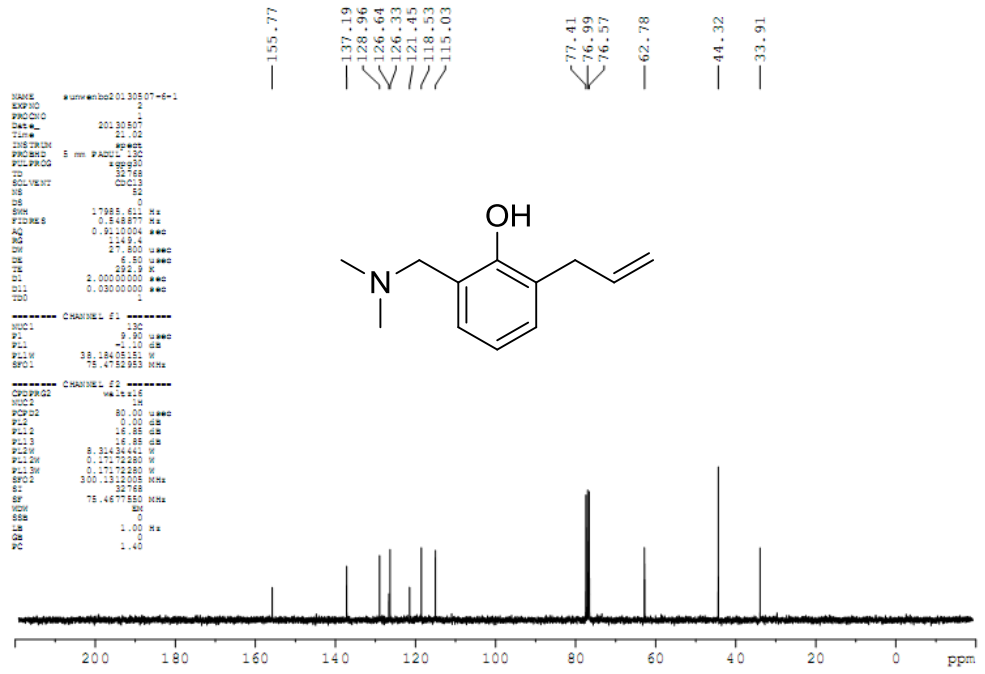

7.28
7.07
7.05
6.84
6.82
6.73
6.70
6.68
3.64
2.34
2.25
OH
Me
N
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.83
1.10
0.95
2.00
5.67
2.79

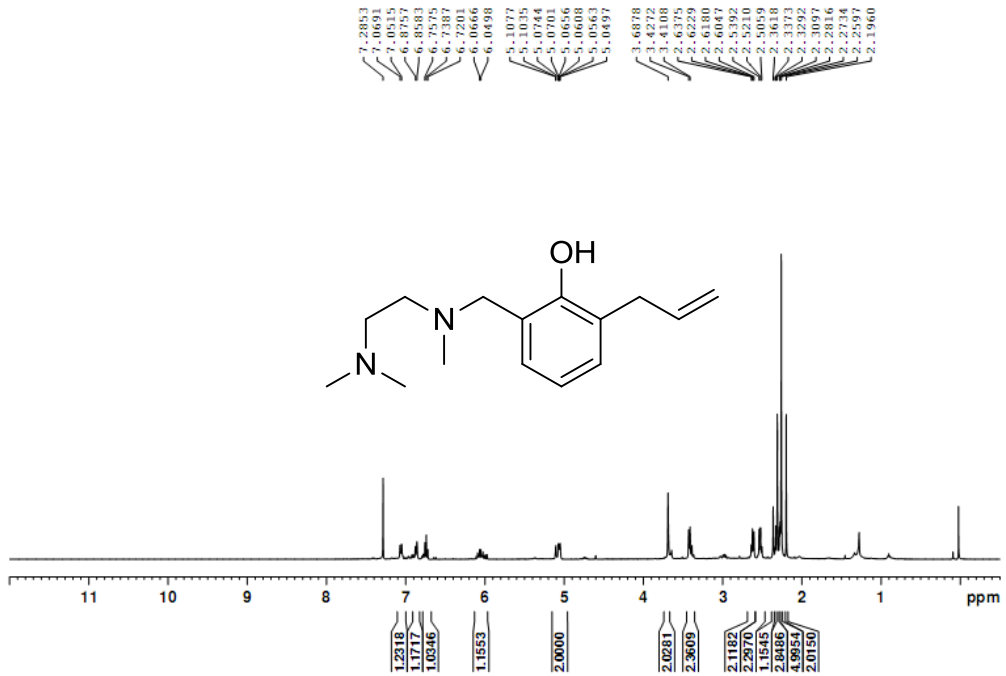

156.09
129.73
125.79
124.72
121.11
118.32
77.38
76.96
76.53
62.78
44.34
29.64
OH
Me
N
200
180
160
140
120
100
80
60
40
20
0
ppm

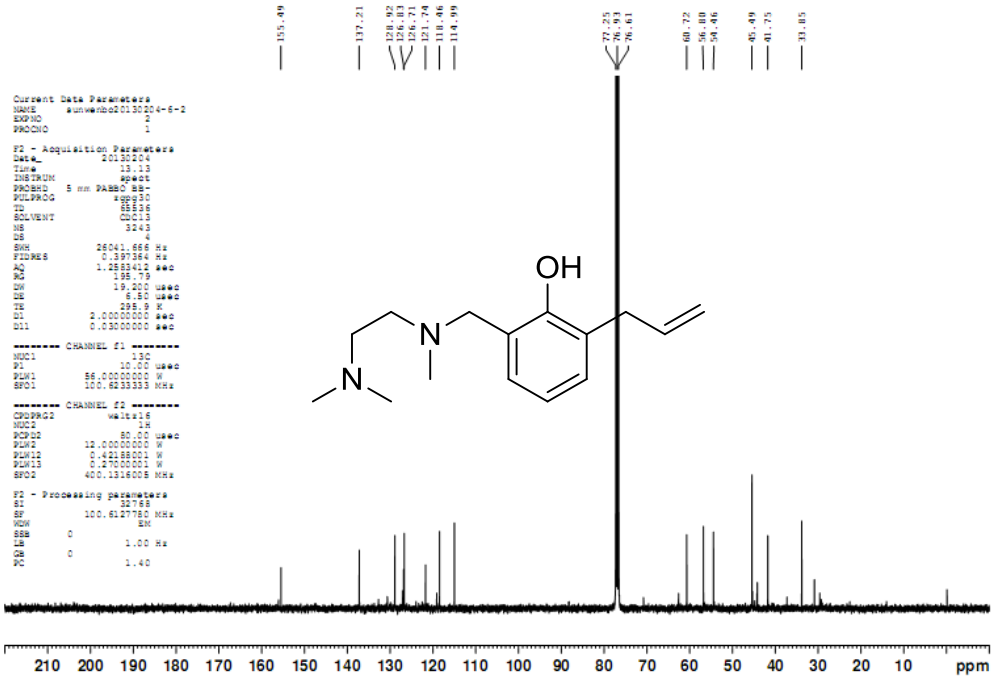

7.2849
7.0650
7.0468
6.8456
6.8275
6.7165
6.6980
6.6794
3.6670
2.6426
2.6276
2.6098
2.5478
2.5300
2.5149
2.3008
2.2706
2.2524
OH
N
N
Me
11
10
9
8
7
6
5
4
3
2
1
ppm
1.0000
1.0603
1.0480
1.9585
1.9951
2.0369
2.9975
5.6103
3.2158

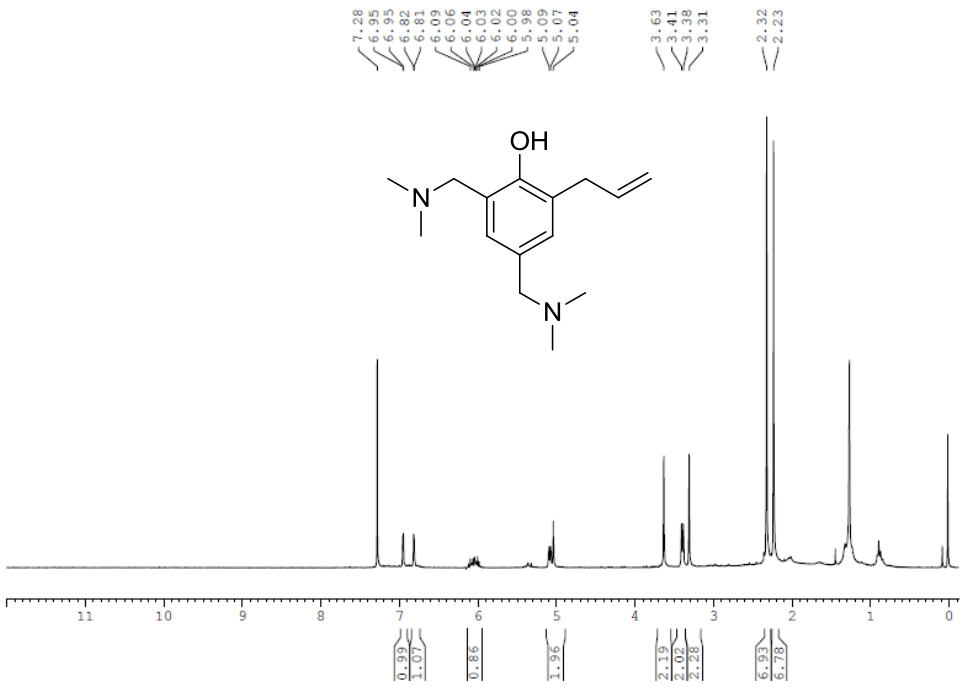

155.85
129.79
126.26
124.87
121.96
118.27
77.30
76.98
76.66
68.51
56.78
54.54
45.46
41.76
15.74
Current Data Parameters
NAME sunwenbo20130802-491-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130802
Time 9.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 44
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 0 K
D1 2.00000000 sec
D11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
N
N
Me
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

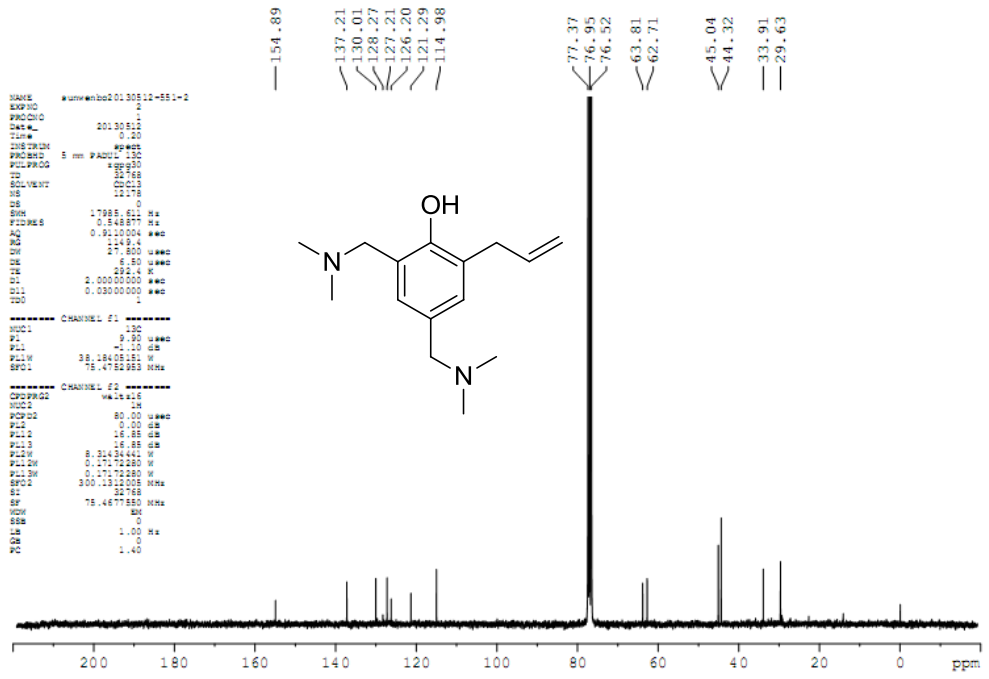

7.28
7.08
7.06
7.06
6.88
6.86
6.78
6.75
6.73
6.13
6.11
6.10
6.09
6.08
6.06
6.04
6.03
6.02
6.00
5.12
5.12
5.11
5.10
5.09
5.07
5.06
5.06
3.66
3.44
3.41
2.34
OH
N
11
10
9
8
7
6
5
4
3
2
1
0 ppm
0.95
1.09
1.03
0.91
2.00
2.28
2.20
6.11

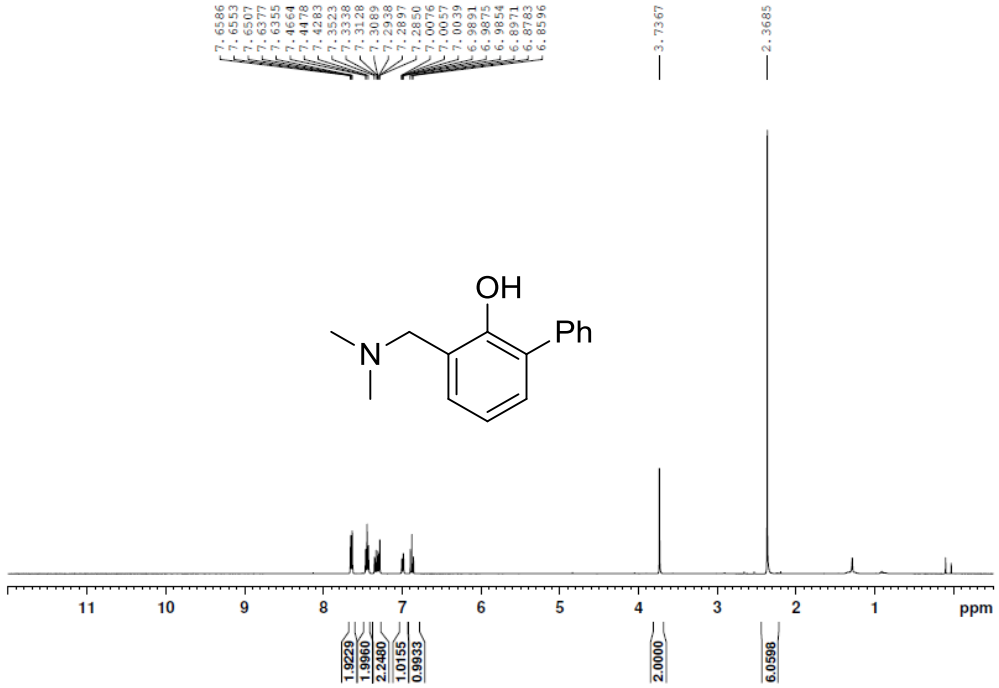

155.77
137.19
128.96
126.64
126.33
121.45
118.53
115.03
77.41
76.99
76.57
62.78
44.32
33.91
OH
N
200
180
160
140
120
100
80
60
40
20
0
ppm

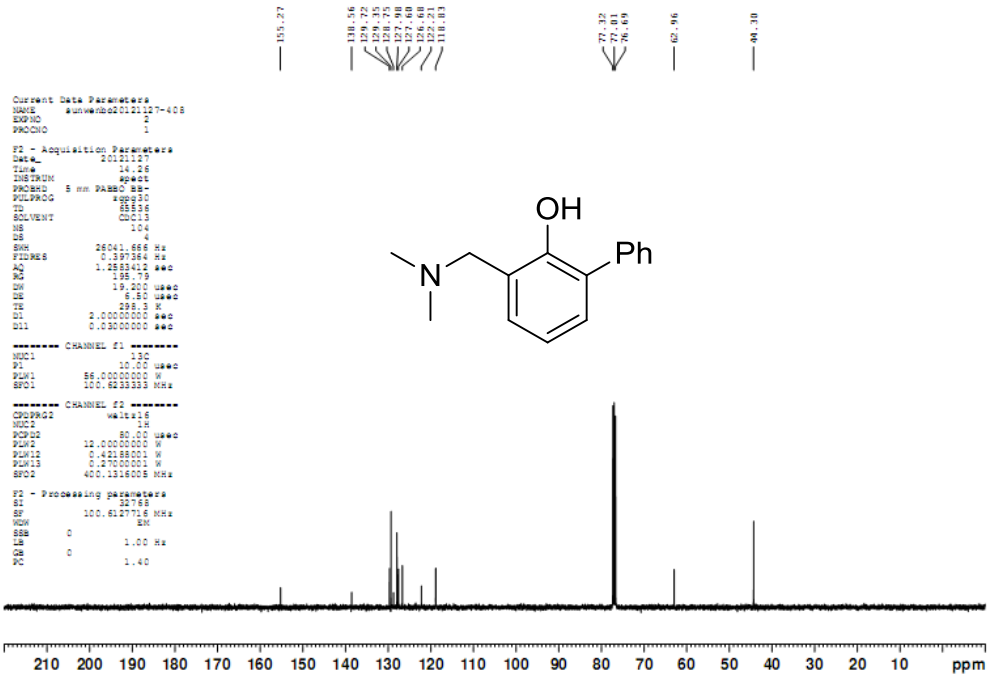

7.2853
7.0691
7.0515
6.8757
6.8583
6.7575
6.7387
6.7201
6.0666
6.0498
5.1077
5.1035
5.0744
5.0701
5.0656
5.0608
5.0563
5.0497
3.6878
3.4272
3.4108
2.6375
2.6229
2.6180
2.6047
2.5392
2.5210
2.5059
2.3618
2.3373
2.3292
2.3097
2.2816
2.2734
2.2597
2.1960
OH
N
N
11
10
9
8
7
6
5
4
3
2
1
ppm
1.2318
1.1717
1.0346
1.1553
2.0000
2.0281
2.3609
2.1182
2.2970
1.1545
2.9486
4.9954
2.0150

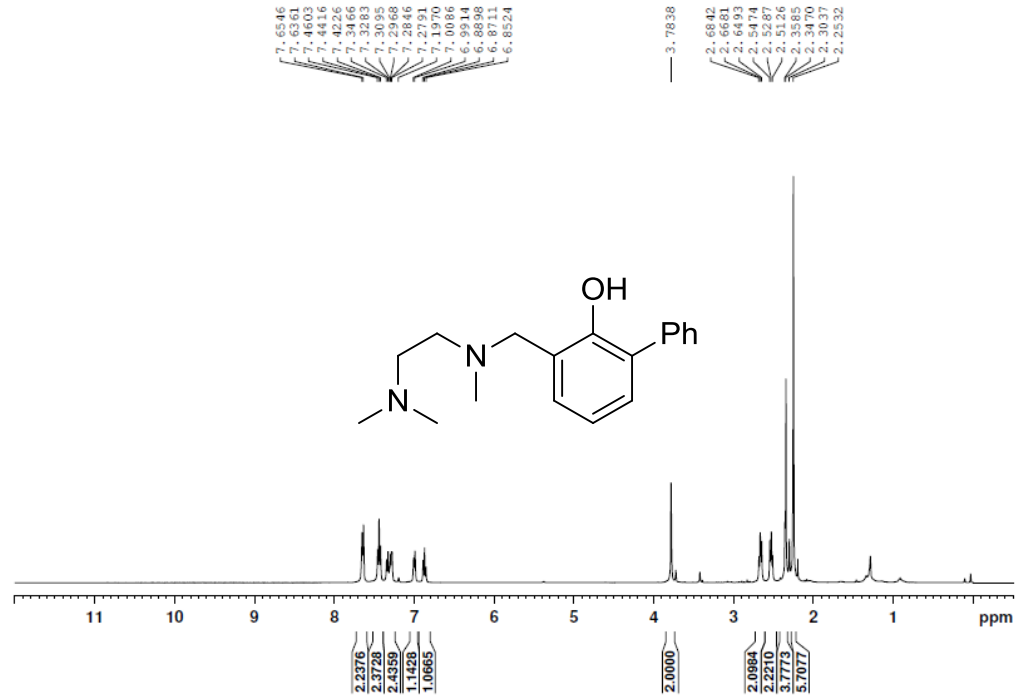

155.49
137.21
128.92
126.83
126.71
121.74
118.46
114.99
77.25
76.93
76.61
68.72
56.80
54.46
45.49
41.75
33.85
OH
N
N
Current Data Parameters
NAME sunwenbo20130204-6-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130204
Time 13.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3243
DS 4
SWH 26041.666 Hz
FIDRES 0.397384 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

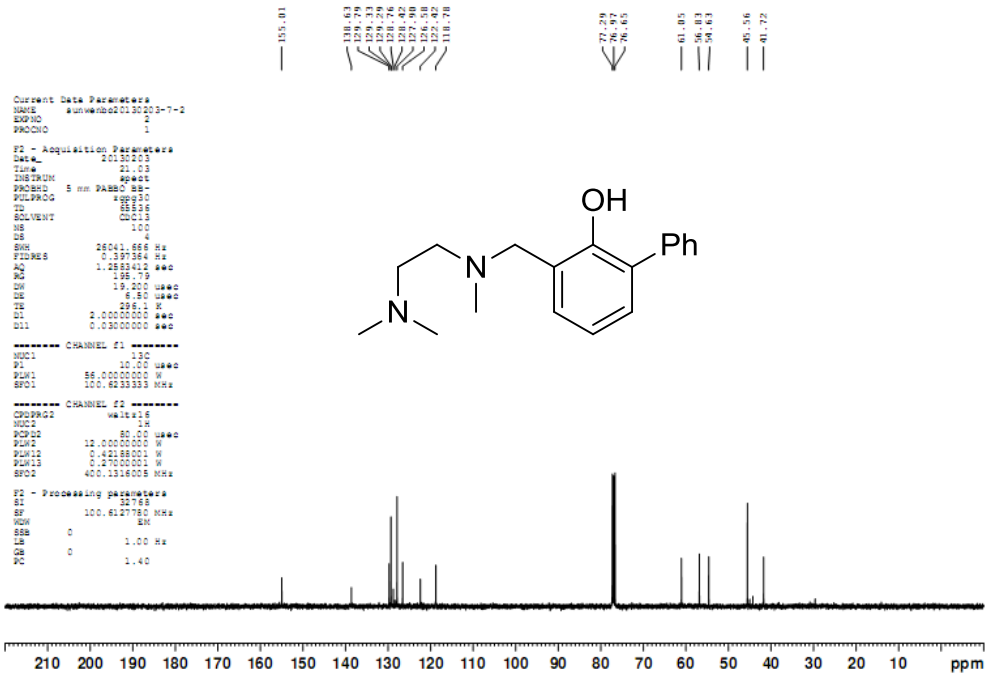

7.28
6.95
6.82
6.81
6.09
6.06
6.04
6.03
6.02
6.00
5.98
5.09
5.07
5.04
3.63
3.41
3.38
3.31
2.32
2.23
OH
N
N
0.99
1.07
0.86
1.96
2.19
2.02
2.28
6.93
6.78


154.89
137.21
130.01
128.27
127.21
126.20
121.29
114.98
77.37
76.95
76.52
63.81
62.71
45.04
44.32
33.91
29.63
OH
N
N
ppm

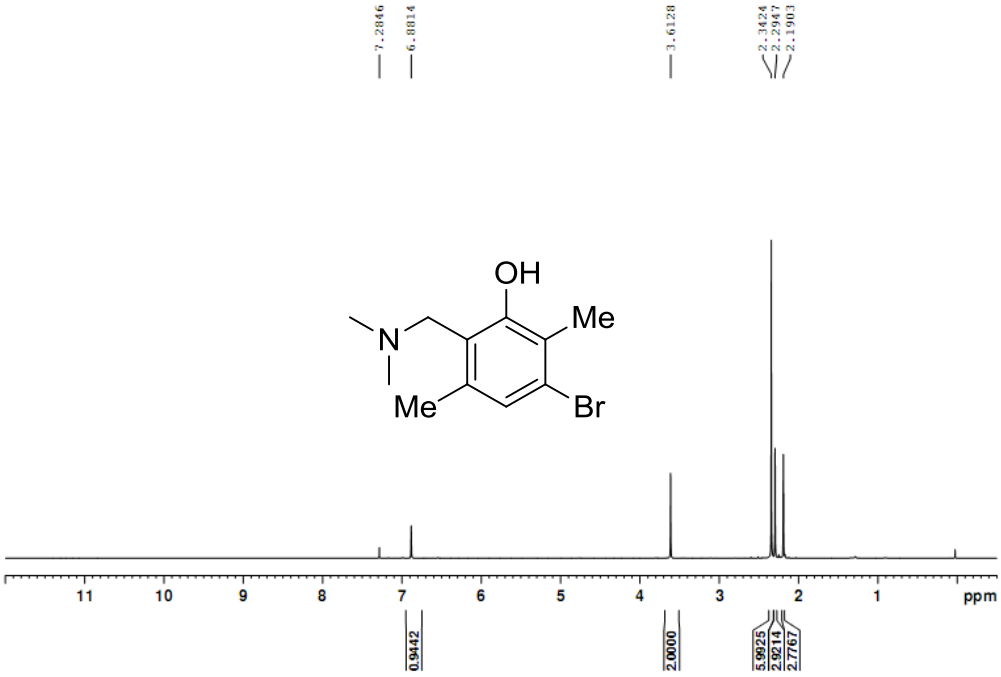

7.6586
7.6553
7.6507
7.6377
7.6355
7.4664
7.4478
7.4283
7.3523
7.3338
7.3128
7.3089
7.2938
7.2897
7.2850
7.0076
7.0057
7.0039
6.9891
6.9875
6.9854
6.8971
6.8783
6.8596
3.7367
2.3685
OH
Ph
N
11
10
9
8
7
6
5
4
3
2
1
ppm
1.9229
1.9960
2.2480
1.0155
0.9933
2.0000
6.0598

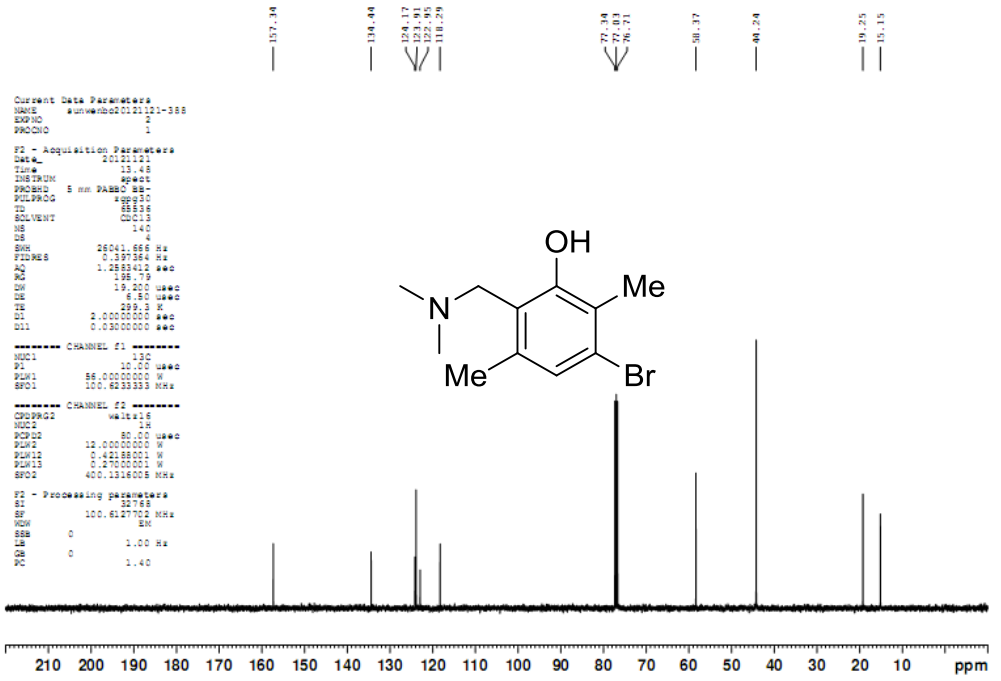

155.27
138.56
129.72
129.35
128.75
127.98
127.68
126.68
122.21
118.83
77.32
77.01
76.69
62.96
44.30
OH
Ph
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

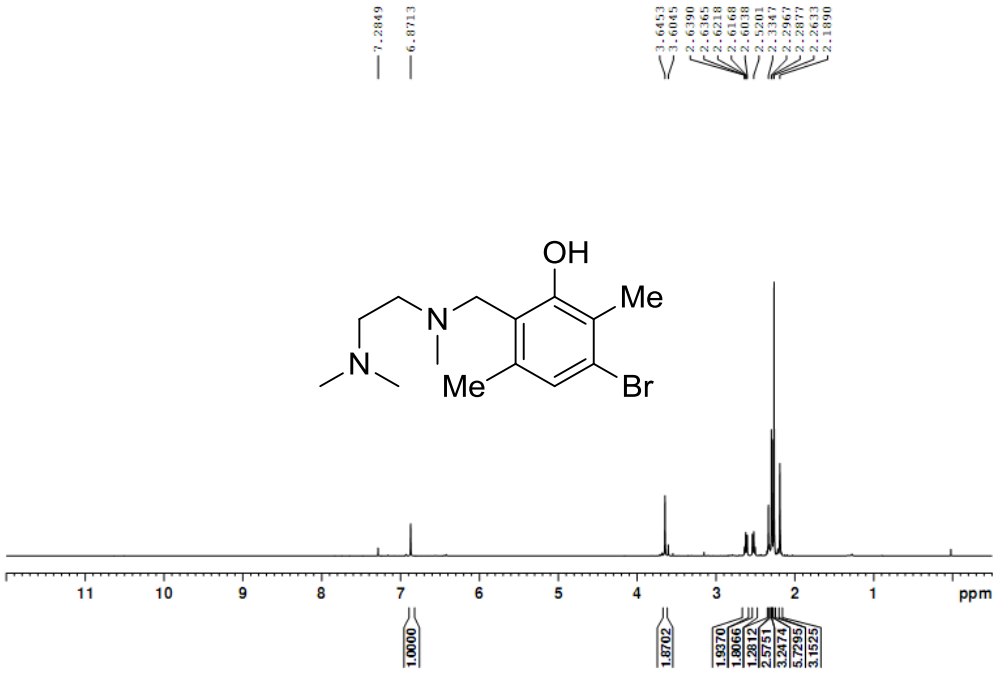


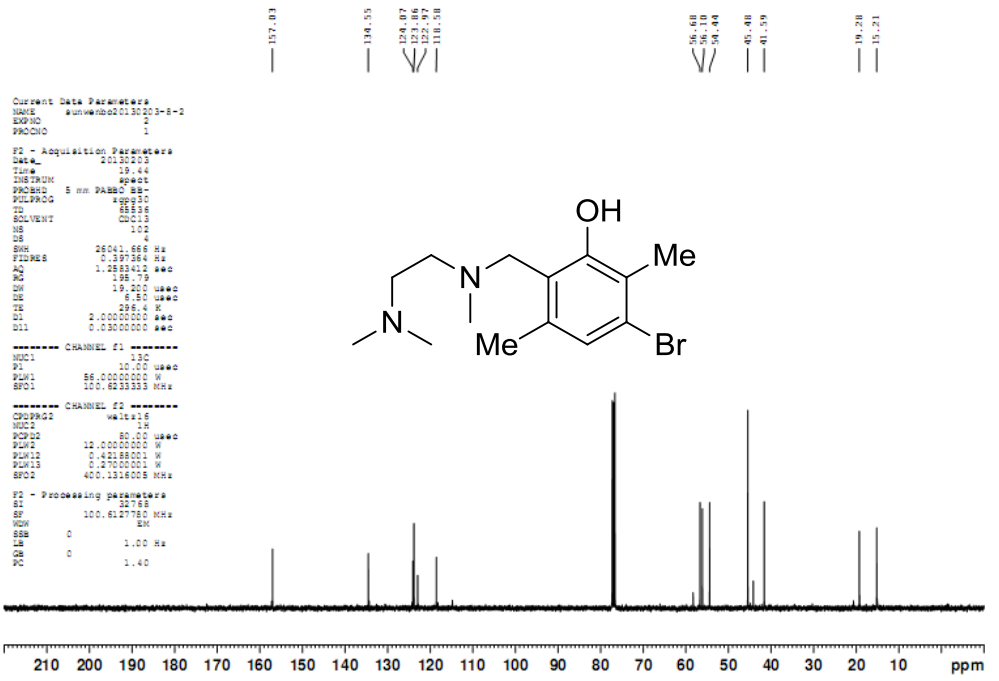


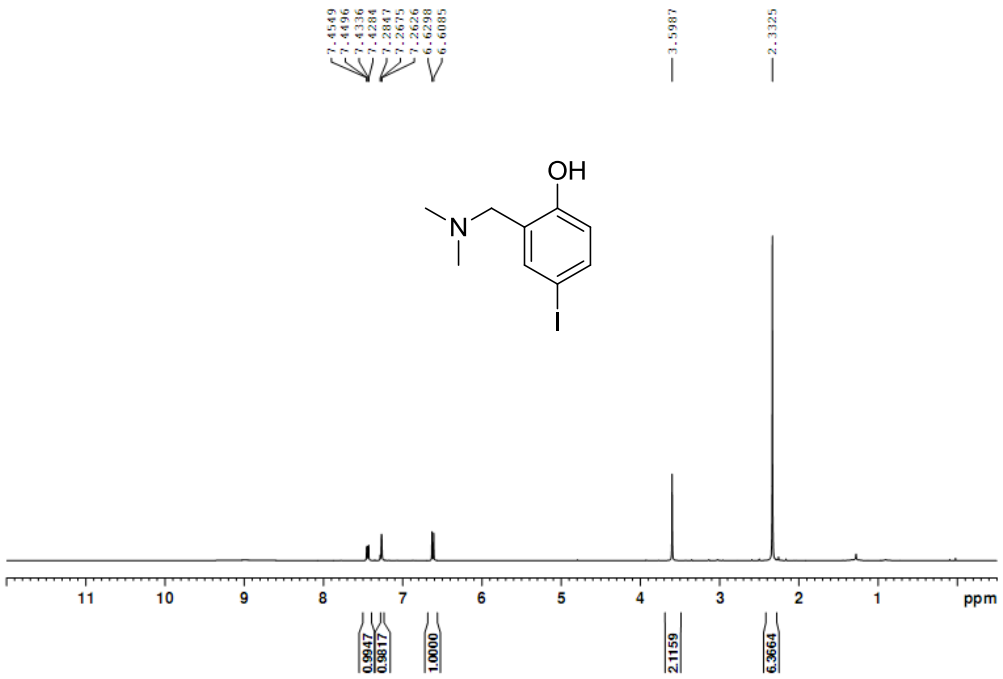

```
Current Data Parameters
NAME      sunwenbo20130203-7-2
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20130203
Time                  21.03
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                  65536
SOLVENT               CDCl3
NS                      100
DS                        4
SWH               26041.666 Hz
FIDRES            0.397354 Hz
AQ                1.2583412 sec
RG                   196.79
DW                  19.200 usec
DE                   6.50 usec
TE                   296.1 K
D1                2.00000000 sec
D11               0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PLW1          56.00000000 W
SFO1          100.6233333 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PLW2          12.00000000 W
PLW12          0.42188001 W
PLW13          0.27100001 W
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF            100.6127780 MHz
WDW                      EM
SSB                   0
LB                    1.00 Hz
GB                    0
PC                    1.40
```

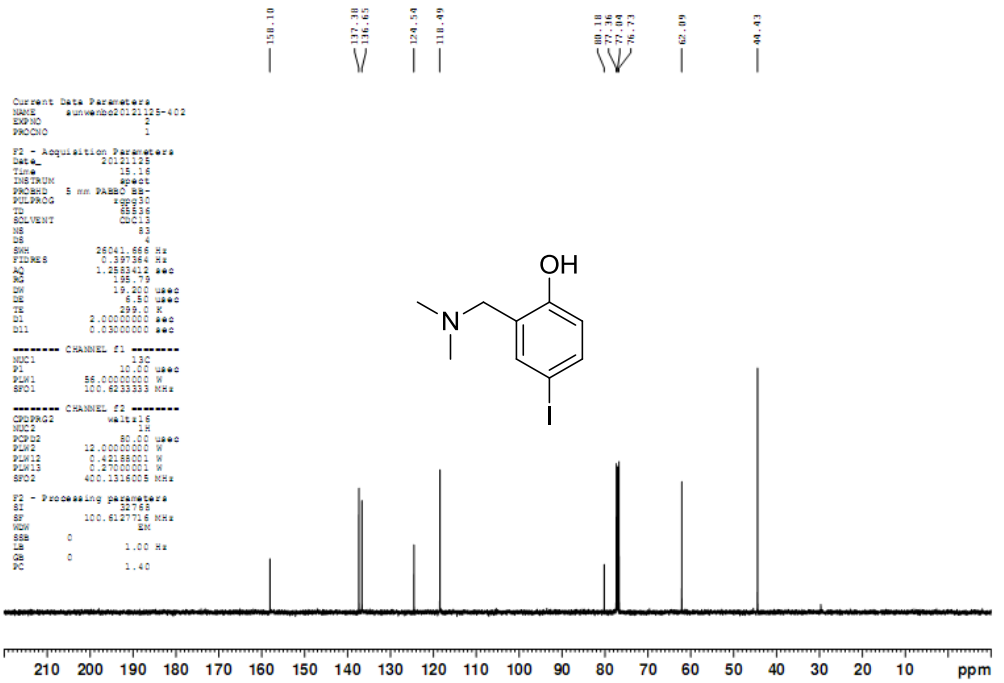

7.2846
6.8814
3.6128
2.3424
2.2947
2.1903
OH
Me
N
Me
Br
11
10
9
8
7
6
5
4
3
2
1
ppm
0.9442
2.0000
5.9925
2.9214
2.7767


157.34
134.48
124.17
123.91
122.95
118.29
77.34
77.03
76.71
58.37
40.24
19.25
15.15
OH
Me
N
Me
Br
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

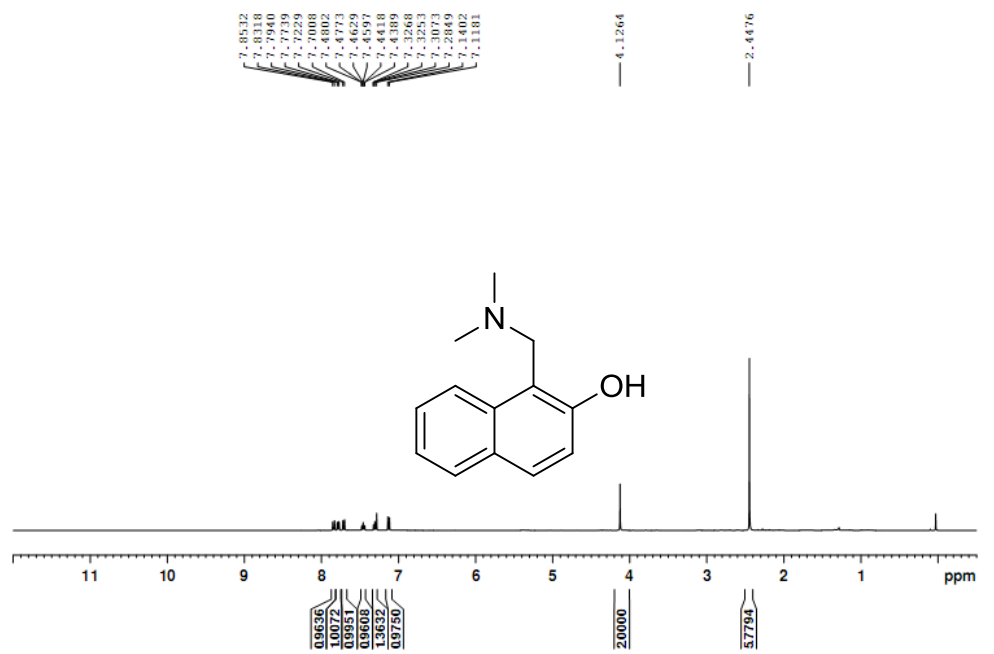


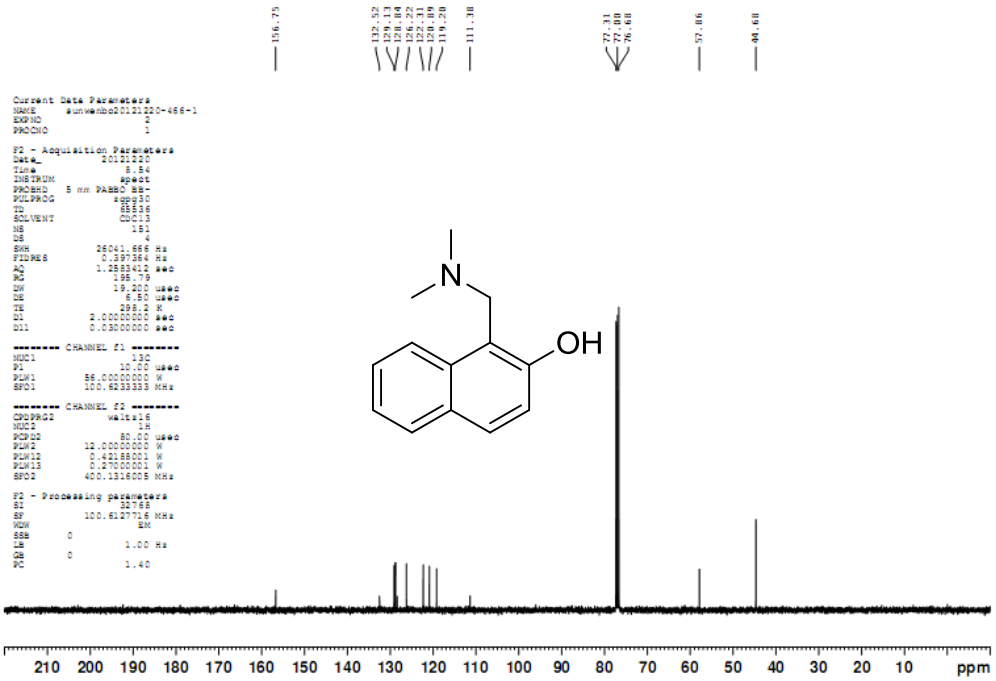




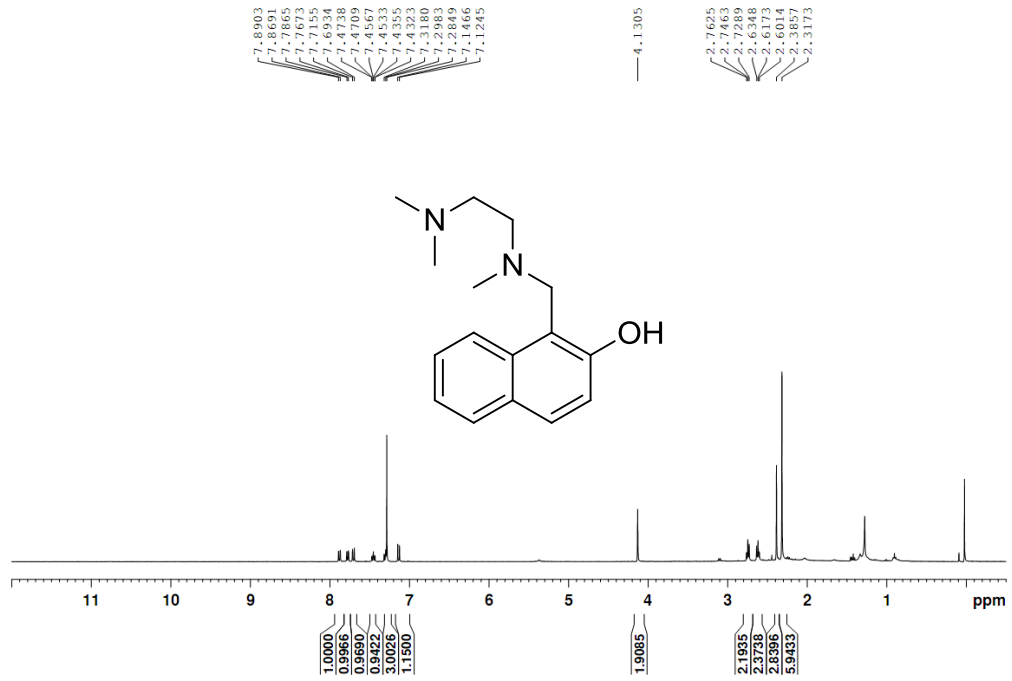


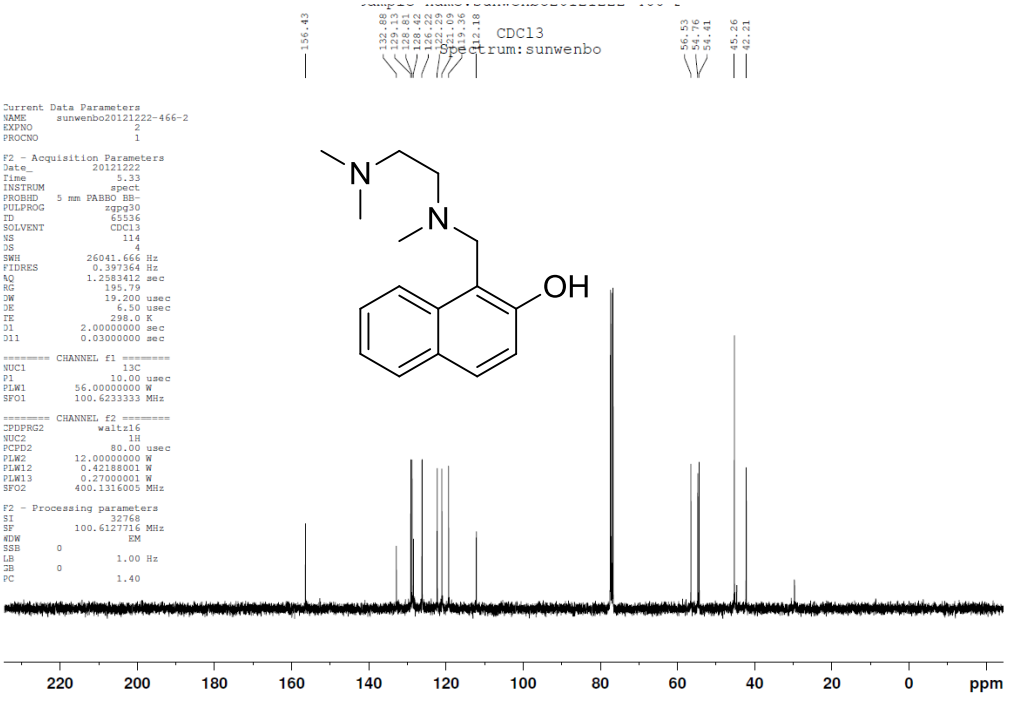

```
Current Data Parameters
NAME      sunwenbo20121125-402
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_          20121125
Time              15.16
INSTRUM           spect
PROBHD  5 mm PABBO BB-
PULPROG         zgpg30
TD              65536
SOLVENT           CDCl3
NS                  83
DS                   4
SWH           26041.666 Hz
FIDRES        0.397364 Hz
AQ            1.2583412 sec
RG                1.95.79
DW               19.200 usec
DE                6.50 usec
TE                299.0 K
D1          2.00000000 sec
D11         0.03000000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               10.00 usec
PLW1         56.00000000 W
SFO1        100.6233333 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PLW2         12.00000000 W
PLW12         0.42188001 W
PLW13         0.27000001 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI               32768
SF          100.6127716 MHz
WDW                 EM
SSB          0
LB               1.00 Hz
GB           0
PC               1.40
```

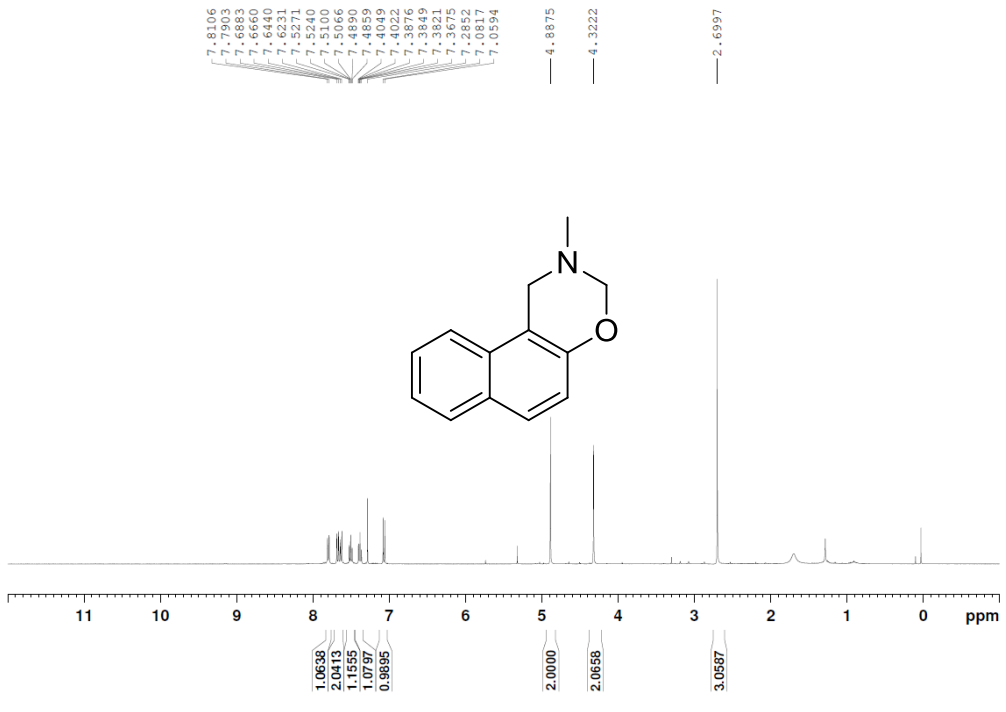


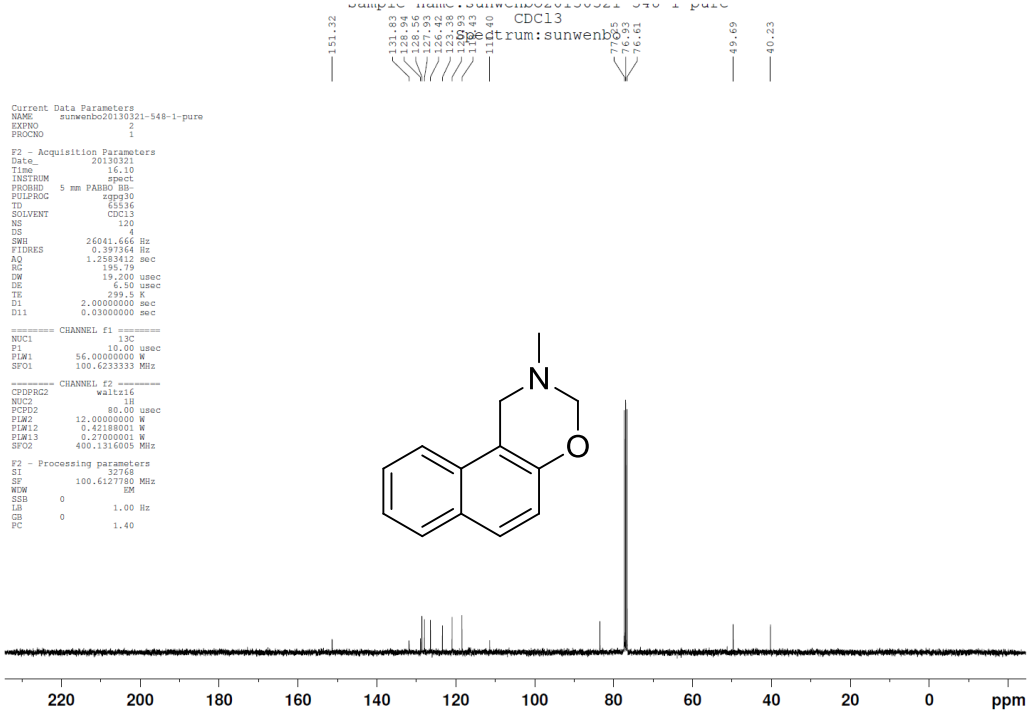


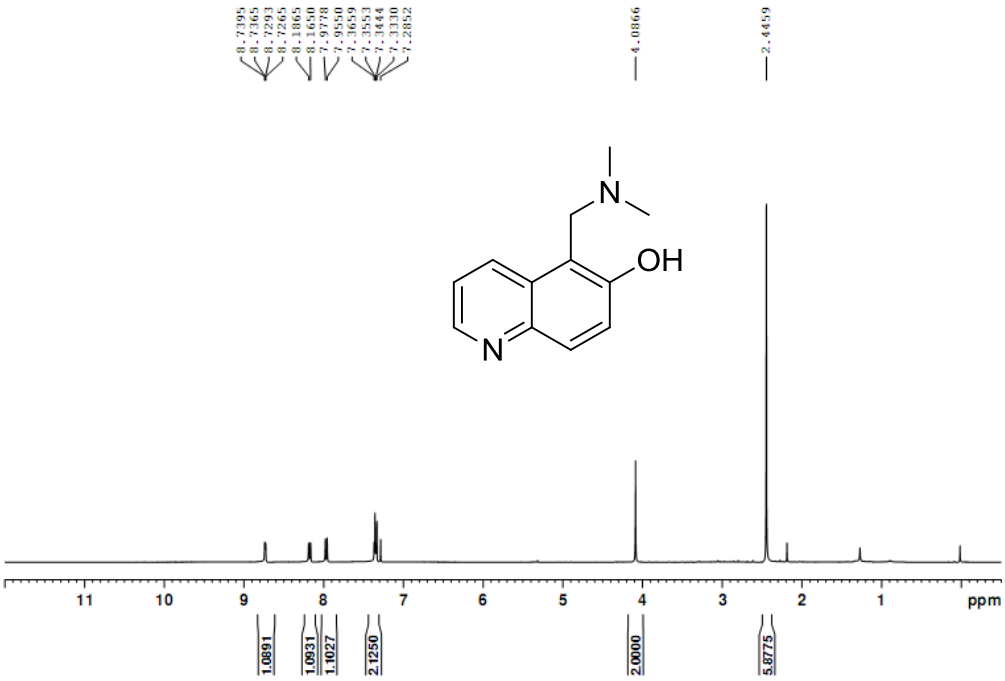

```
Current Data Parameters
NAME      sunwenbo20121220-466-1
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20121220
Time               8.54
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  151
DS                    4
SWH           26041.666 Hz
FIDRES        0.397364 Hz
AQ            1.258342 sec
RG                195.79
DW               19.200 usec
DE                6.50 usec
TE               298.2 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               10.00 usec
PLW1         56.00000000 W
SFO1        100.6233333 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            80.00 usec
PLW2         12.00000000 W
PLW12         0.42186001 W
PLW13         0.17700001 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127716 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

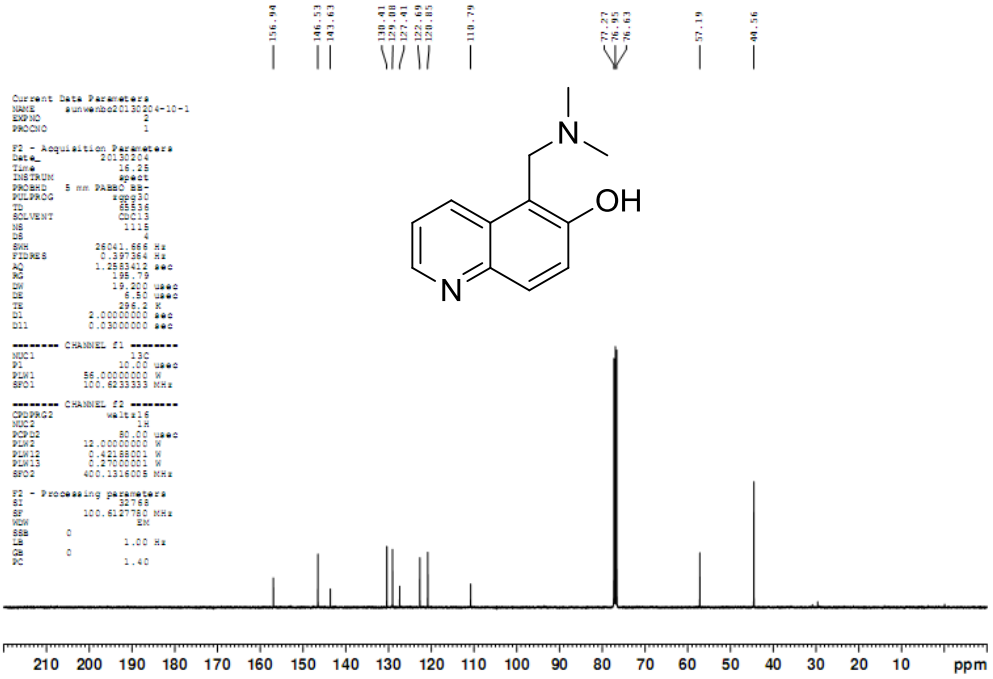

7.8903
7.8691
7.7665
7.7673
7.7155
7.6934
7.4738
7.4709
7.4567
7.4453
7.4355
7.4323
7.4180
7.2983
7.2849
7.1466
7.1245
4.1305
2.7625
2.7463
2.7289
2.6348
2.6173
2.6014
2.3857
2.3173
N
N
OH
11
10
9
8
7
6
5
4
3
2
1
ppm
1.0000
0.9966
0.9690
0.9422
3.0026
1.1500
1.9085
2.1935
2.3738
2.8396
5.9433

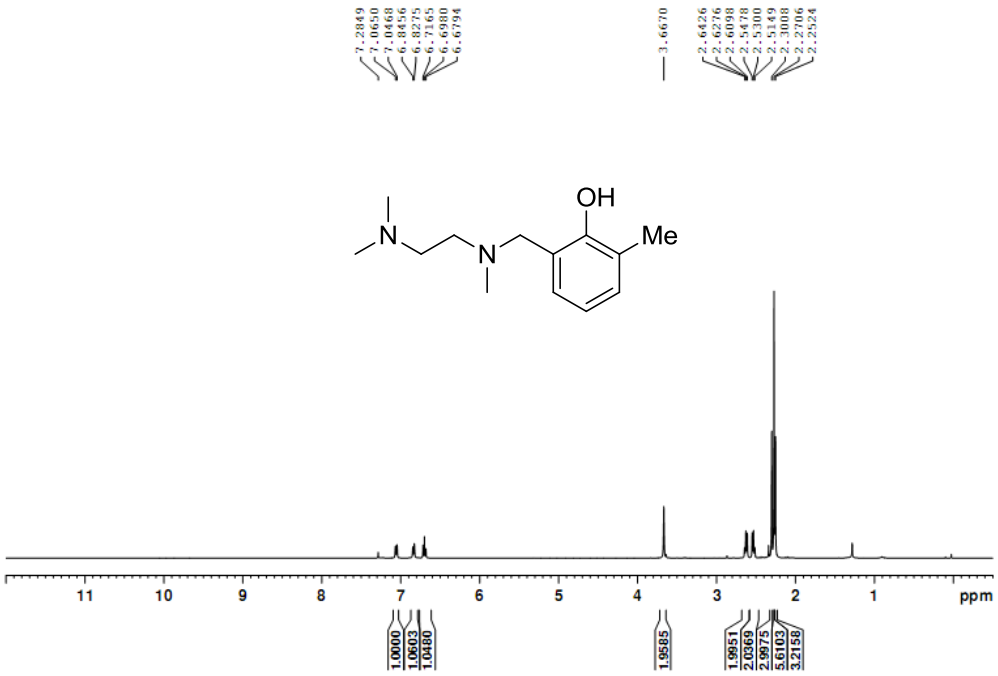

156.43
132.88
129.13
128.81
128.42
126.22
122.69
121.63
119.36
112.18
CDCl3
Spectrum:sunwenbo
56.53
54.76
54.41
45.26
42.21
Current Data Parameters
NAME sunwenbo20121222-466-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20121222
Time 5.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 114
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127716 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
N
OH
220
200
180
160
140
120
100
80
60
40
20
0
ppm

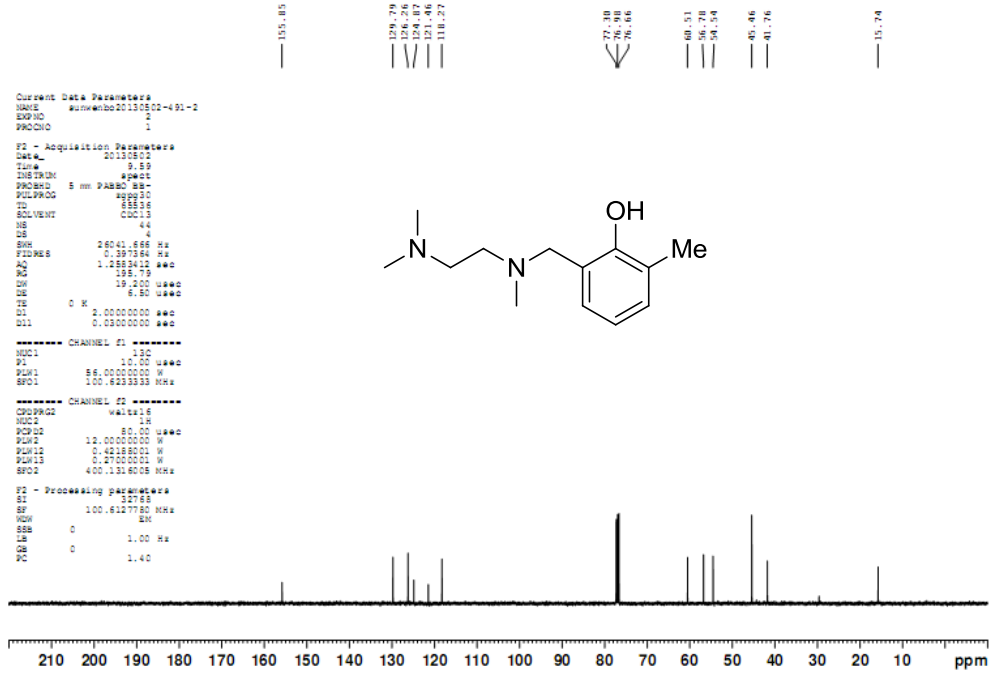

7.8106
7.7903
7.6883
7.6660
7.6440
7.6231
7.5271
7.5240
7.5100
7.5066
7.4890
7.4859
7.4049
7.4022
7.3876
7.3849
7.3821
7.3675
7.2852
7.0817
7.0594
4.8875
4.3222
2.6997
N
O
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.0638
2.0413
1.1555
1.0797
0.9895
2.0000
2.0658
3.0587

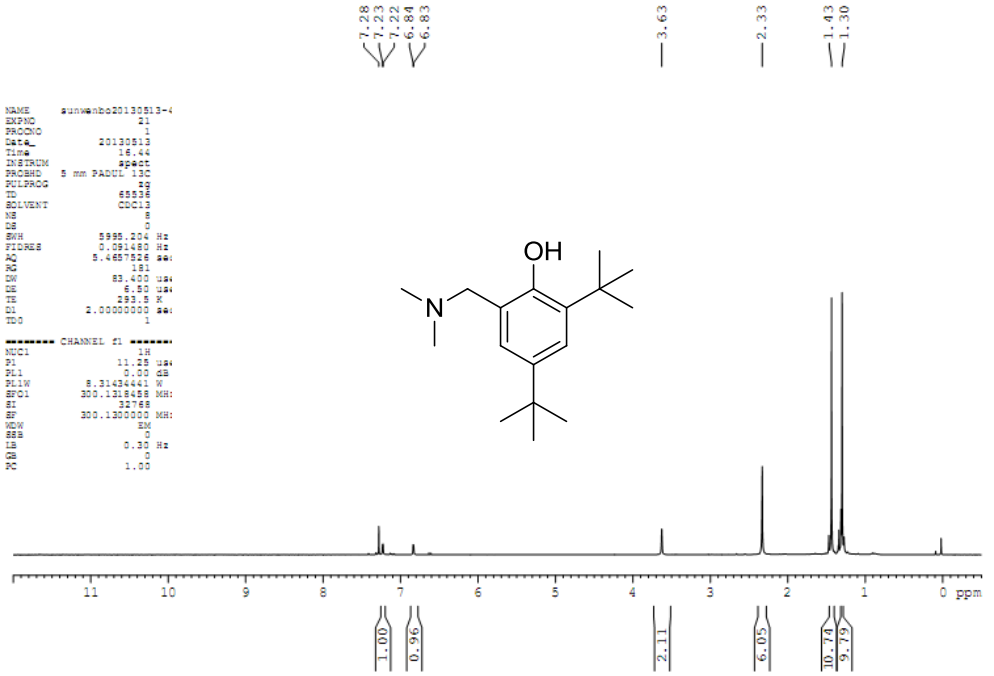

CDCl3
Spectrum:sunwenbo
151.32
131.83
128.94
128.56
128.38
127.42
123.38
122.93
118.43
111.40
77.05
76.93
76.61
49.69
40.23
Current Data Parameters
NAME sunwenbo20130321-548-1-pure
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130321
Time 16.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 120
DS 4
SWH 26041.666 Hz
FIDRES 0.39736 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 299.5 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
O
220 200 180 160 140 120 100 80 60 40 20 0 ppm

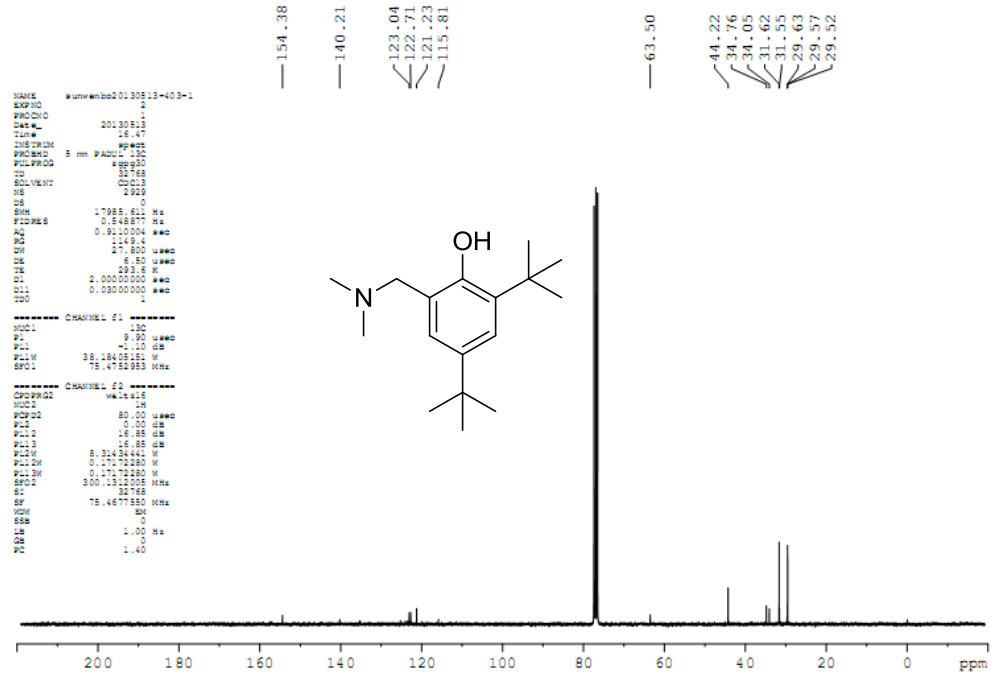

8.7395
8.7365
8.7293
8.7265
8.1865
8.1630
7.9778
7.9550
7.3659
7.3553
7.3444
7.3330
7.2852
4.0866
2.4459
N
OH
N
11
10
9
8
7
6
5
4
3
2
1
ppm
1.0891
1.0931
1.1027
2.1250
2.0000
5.8775

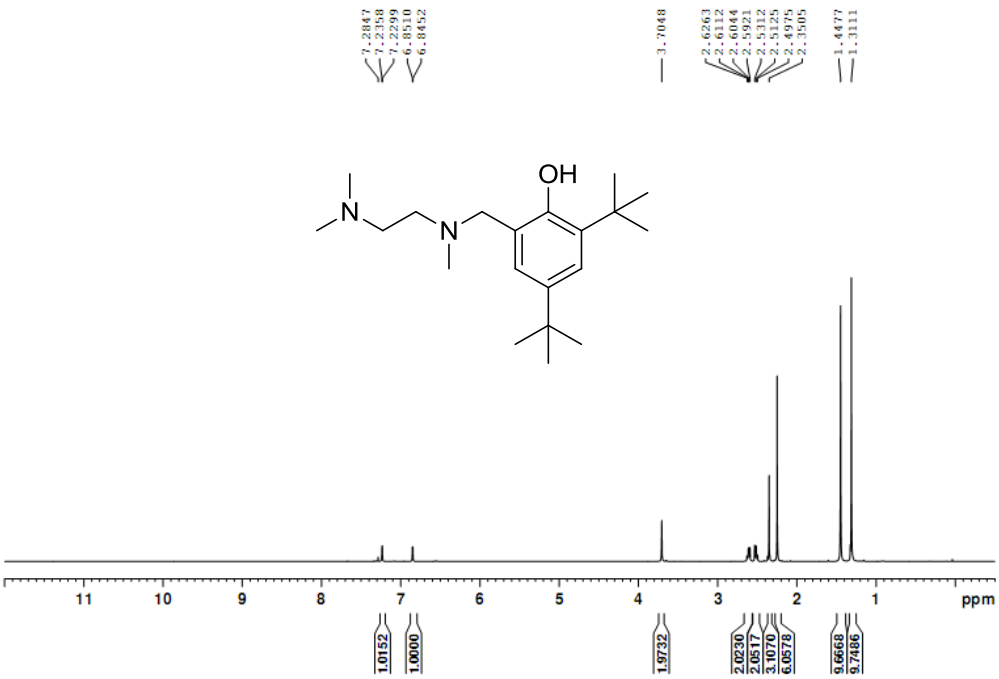

156.94
146.53
143.63
130.41
129.80
127.41
122.69
120.85
110.79
77.27
76.95
76.63
57.19
44.56
Current Data Parameters
NAME sunwenbo20130204-10-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130204
Time 16.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1115
DS 4
SWH 26041.666 Hz
FIDRES 0.397384 Hz
AQ 1.258342 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27700001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
OH
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

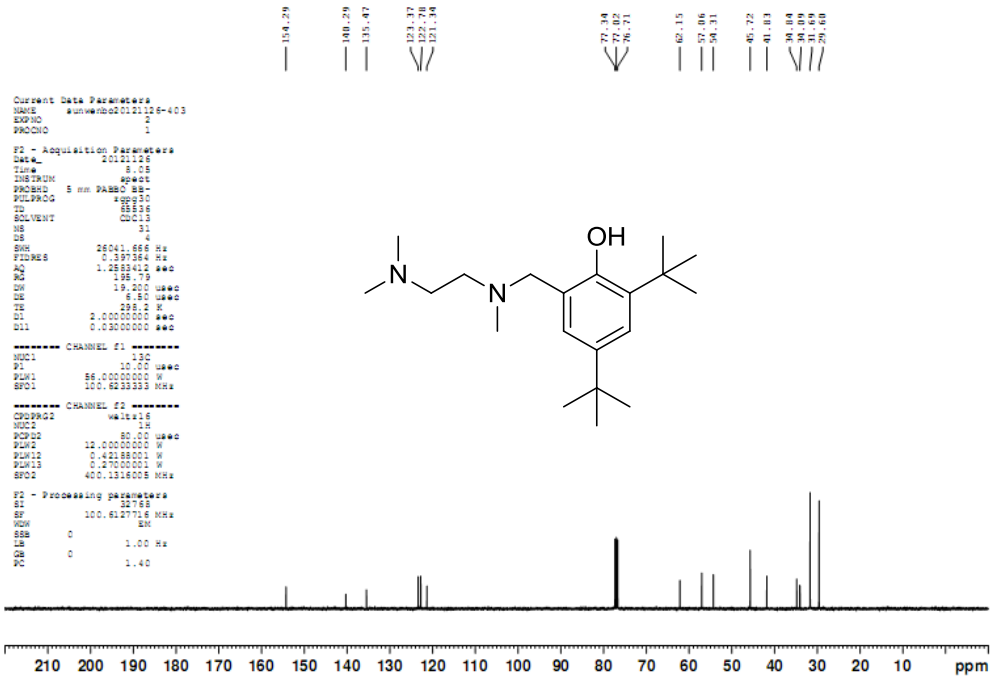

7.2849
7.0650
7.0468
6.8456
6.8275
6.7165
6.6980
6.6794
3.6670
2.6426
2.6276
2.6098
2.5478
2.5300
2.5149
2.3008
2.2706
2.2524
OH
N
N
Me
11
10
9
8
7
6
5
4
3
2
1
ppm
1.0000
1.0603
1.0480
1.9585
1.9951
2.0369
2.9975
5.6103
3.2158


155.85
129.79
126.26
124.87
121.46
118.27
77.38
77.06
76.66
60.51
56.78
54.54
45.46
41.76
15.74
Current Data Parameters
NAME wunwenbo20130502-491-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130502
Time 9.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 44
DS 4
SWH 26041.666 Hz
FIDRES 0.397354 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
N
N
Me
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

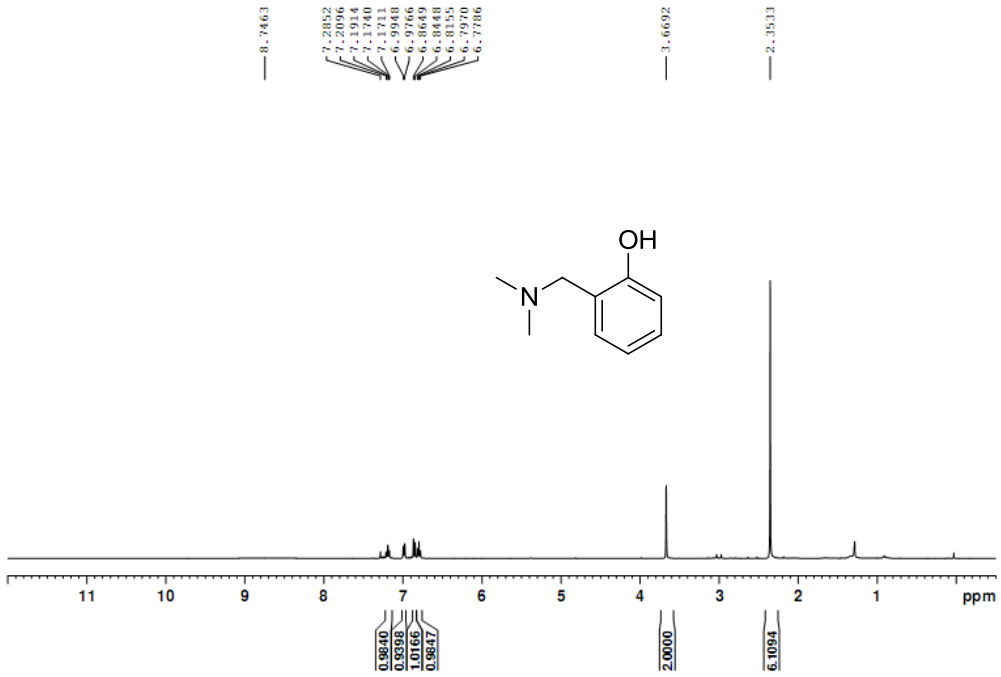

7.28
7.23
7.22
6.84
6.83
3.63
2.33
1.43
1.30
OH
N
11
10
9
8
7
6
5
4
3
2
1
0 ppm
1.00
0.96
2.11
6.05
10.74
9.79

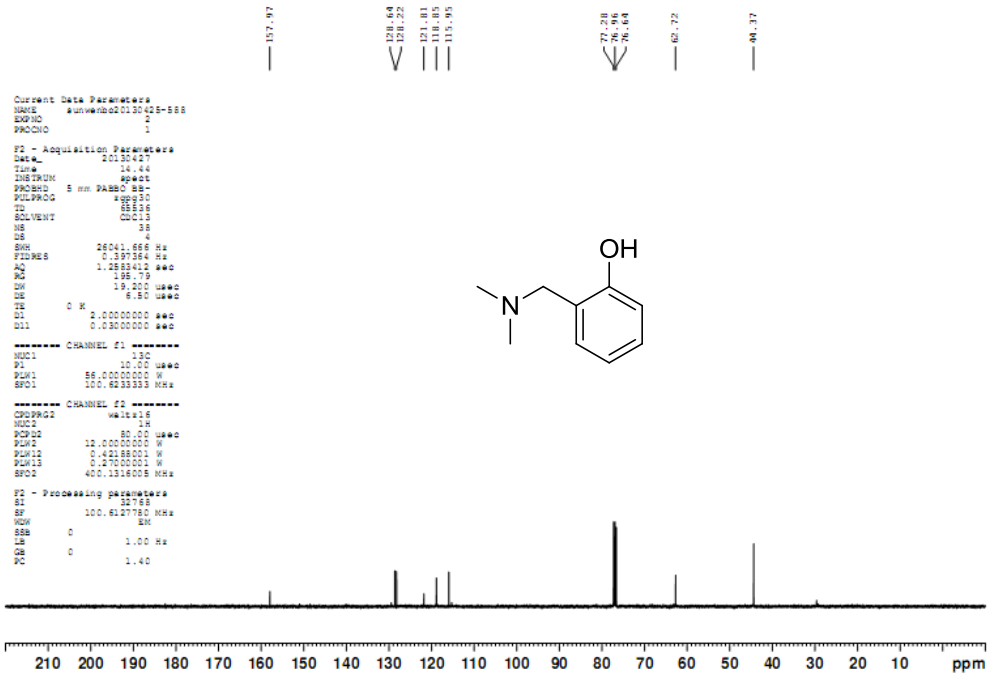

154.38
140.21
123.04
122.71
121.23
115.81
63.50
44.22
34.76
34.05
31.62
31.55
29.63
29.57
29.52
OH
N
200
180
160
140
120
100
80
60
40
20
0
ppm



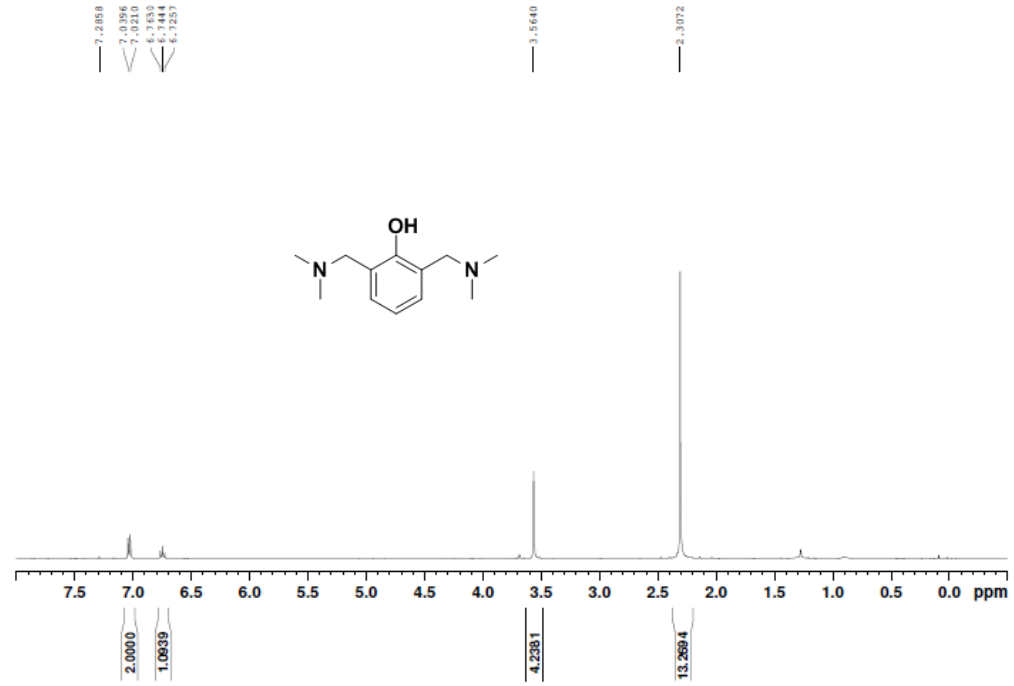


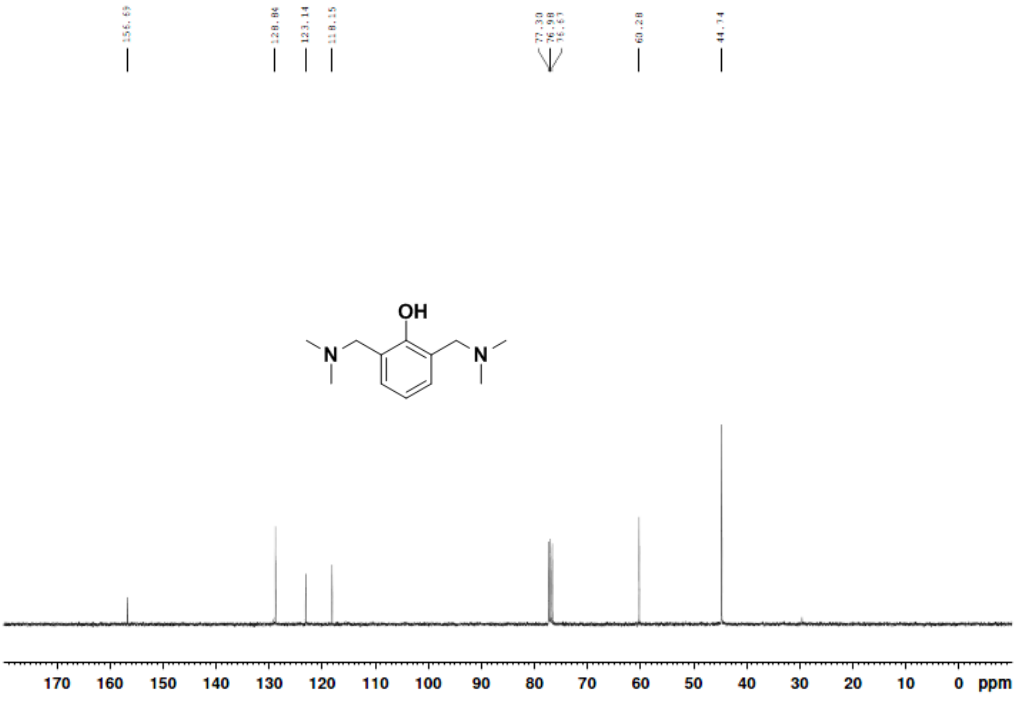

```
Current Data Parameters
NAME      sunwenbo20121126-403
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_           20121126
Time              8.05
INSTRUM           spect
PROBHD    5 mm PABBO BB-
PULPROG         zgpg30
TD              65536
SOLVENT           CDCl3
NS                  31
DS                   4
SWH           26041.666 Hz
FIDRES        0.397364 Hz
AQ            1.2583412 sec
RG                195.79
DW               19.200 usec
DE                6.50 usec
TE              298.2 K
D1          2.00000000 sec
D11         0.03000000 sec

======== CHANNEL f1 ========
NUC1               13C
P1               10.60 usec
PLW1        56.00000000 W
SFO1       100.6233333 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            80.00 usec
PLW2        12.00000000 W
PLW12        0.42188001 W
PLW13        0.27000001 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI              32768
SF         100.6127716 MHz
WDW                EM
SSB         0
LB                1.00 Hz
GB          0
PC                1.40
```

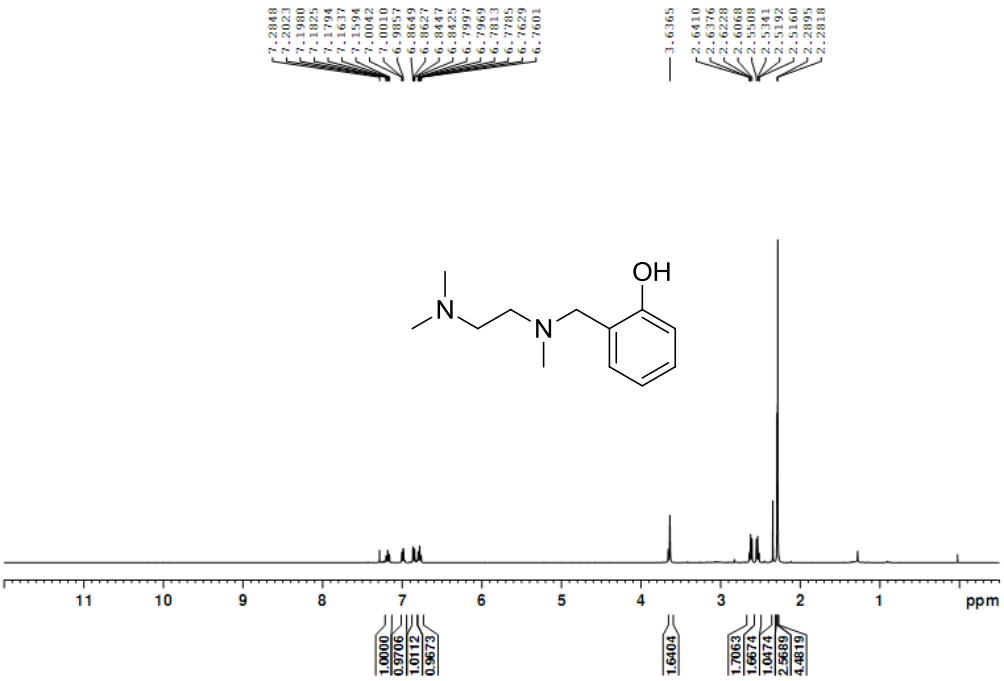


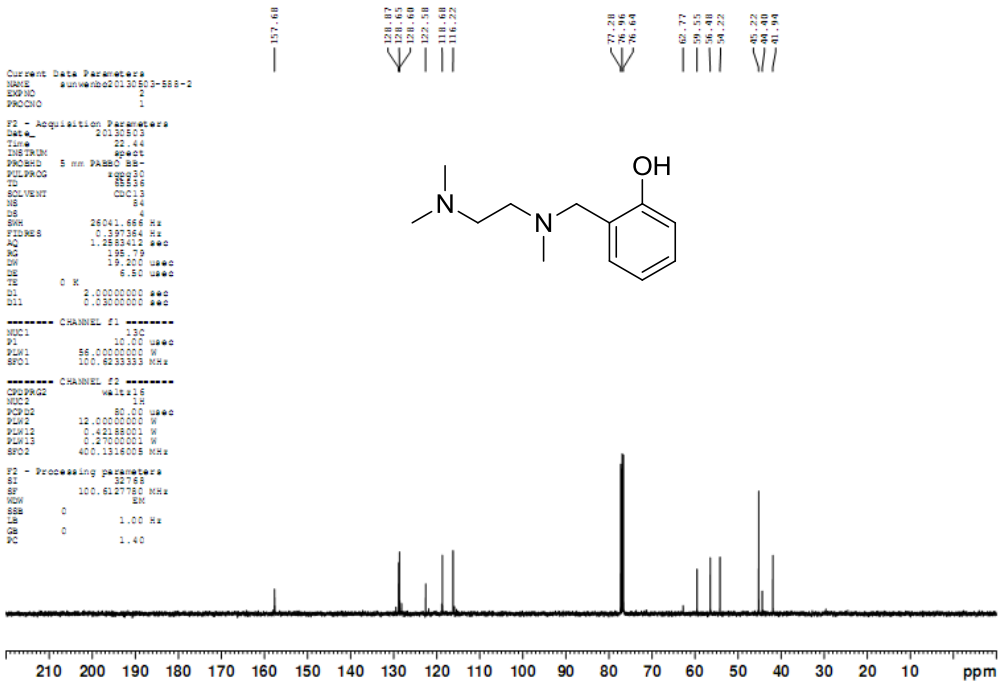


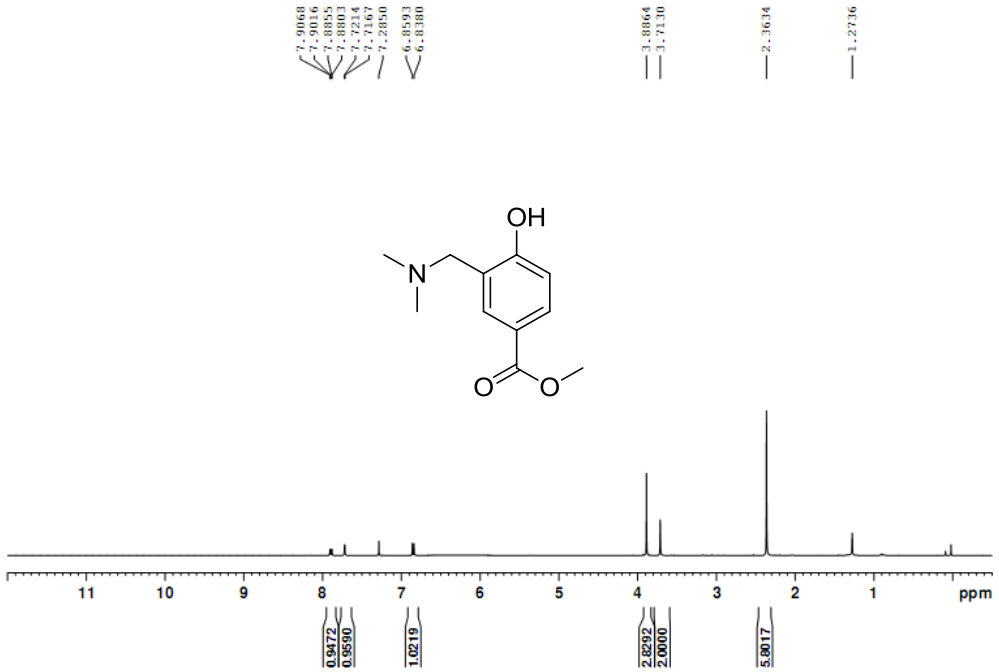

```
Current Data Parameters
NAME      sunwenbo20130425-588
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20130427
Time                  14.44
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                  655336
SOLVENT              CDCl3
NS                      38
DS                       4
SWH              26041.666 Hz
FIDRES           0.397364 Hz
AQ               1.2583412 sec
RG                   195.79
DW                 19.200 usec
DE                   6.50 usec
TE                 0 K
D1               2.00000000 sec
D11              0.03000000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    10.00 usec
PLW1             56.00000000 W
SFO1            100.6233333 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PLW2             12.00000000 W
PLW12             0.42188001 W
PLW13             0.17000001 W
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF              100.6127780 MHz
WDW                       EM
SSB                    0
LB                    1.00 Hz
GB                     0
PC                    1.40
```

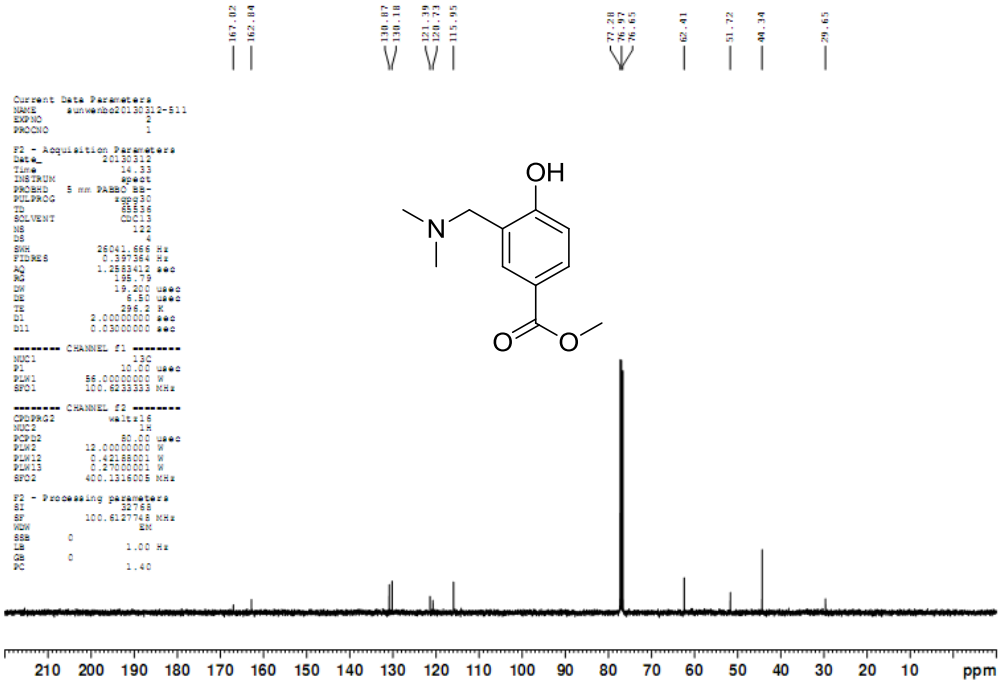





7.2848
7.2023
7.1980
7.1825
7.1794
7.1637
7.1594
7.0042
7.0010
6.9857
6.8649
6.8627
6.8447
6.8425
6.7997
6.7969
6.7813
6.7785
6.7629
6.7601
3.6365
2.6410
2.6376
2.6228
2.6068
2.5508
2.5341
2.5192
2.5160
2.2895
2.2818
OH
N
N
11
10
9
8
7
6
5
4
3
2
1
ppm
1.0000
0.9706
1.0112
0.9673
1.6404
1.7063
1.6674
1.0474
2.5689
4.4819


157.68
128.87
128.65
128.60
122.58
118.68
116.22
77.28
76.96
76.64
62.77
59.55
56.48
54.22
45.22
44.40
41.94
OH
N
N
Current Data Parameters
NAME sunwenbo20130503-588-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130503
Time 22.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 88536
SOLVENT CDCl3
NS 54
DS 4
SWH 26041.666 Hz
FIDRES 0.397354 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm





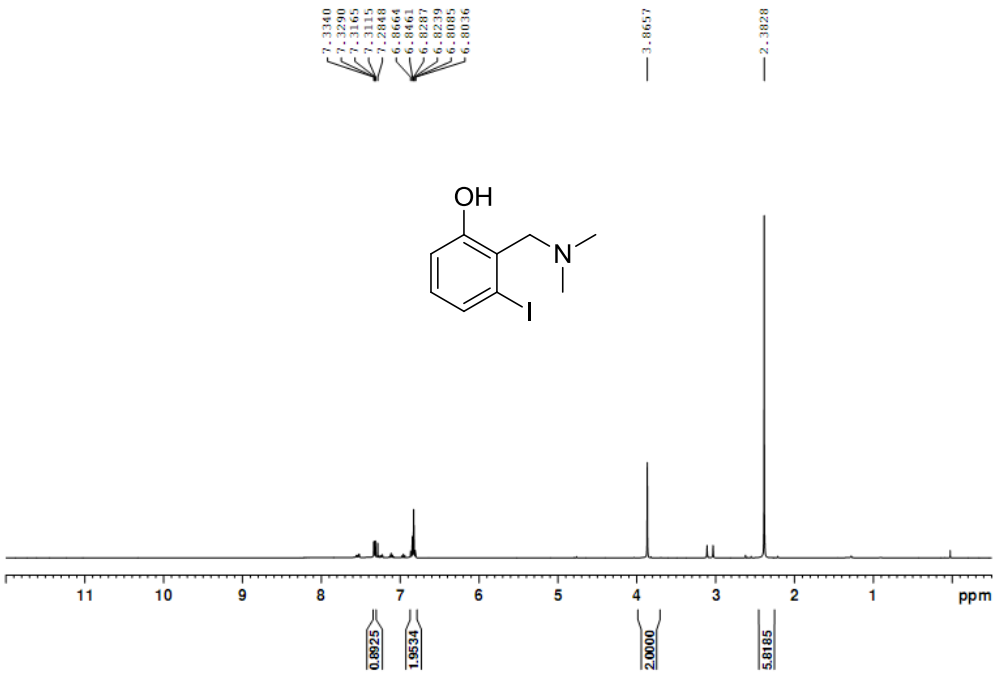
Current Data Parameters
NAME aunwenbo20130312-511
EXPNO 2
PROCNO 1

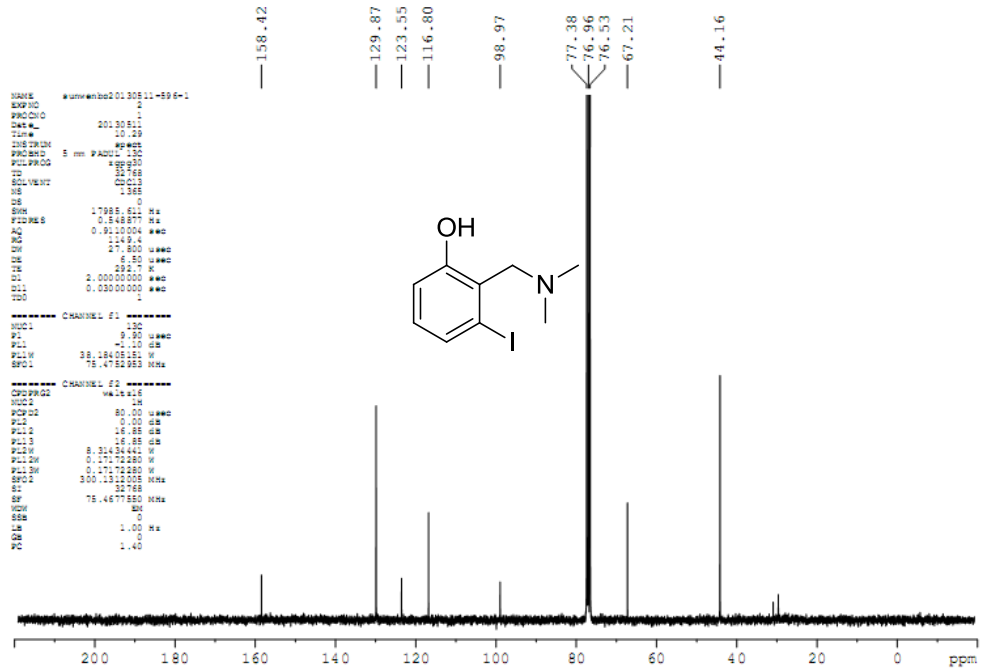
F2 - Acquisition Parameters
Date_ 20130312
Time 14.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 122
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 296.2 K
D1 2.00000000 sec
D11 0.03000000 sec

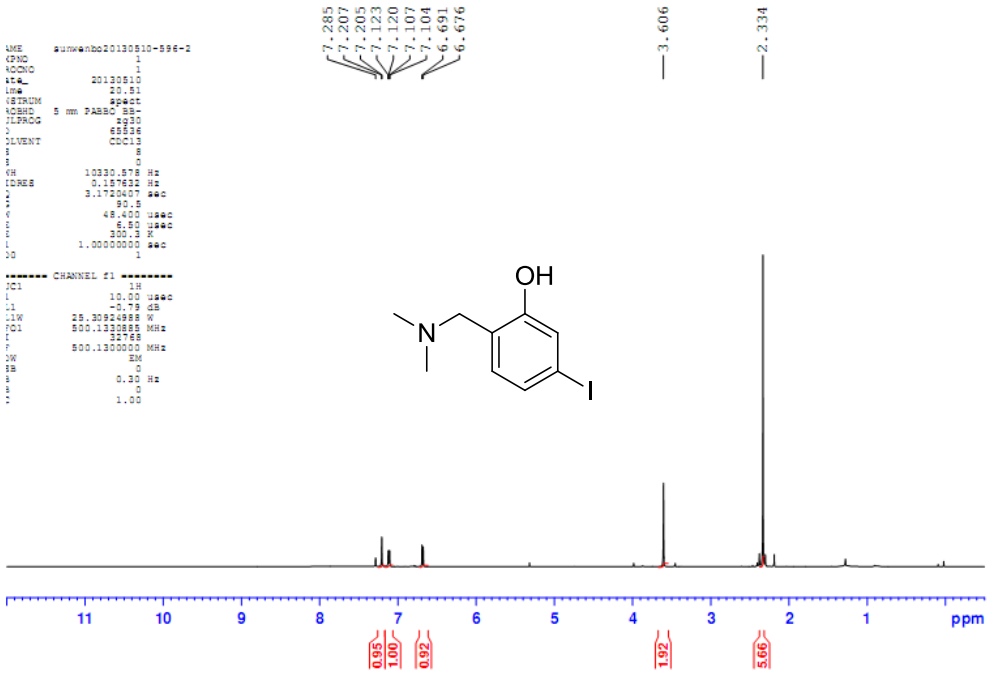
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz

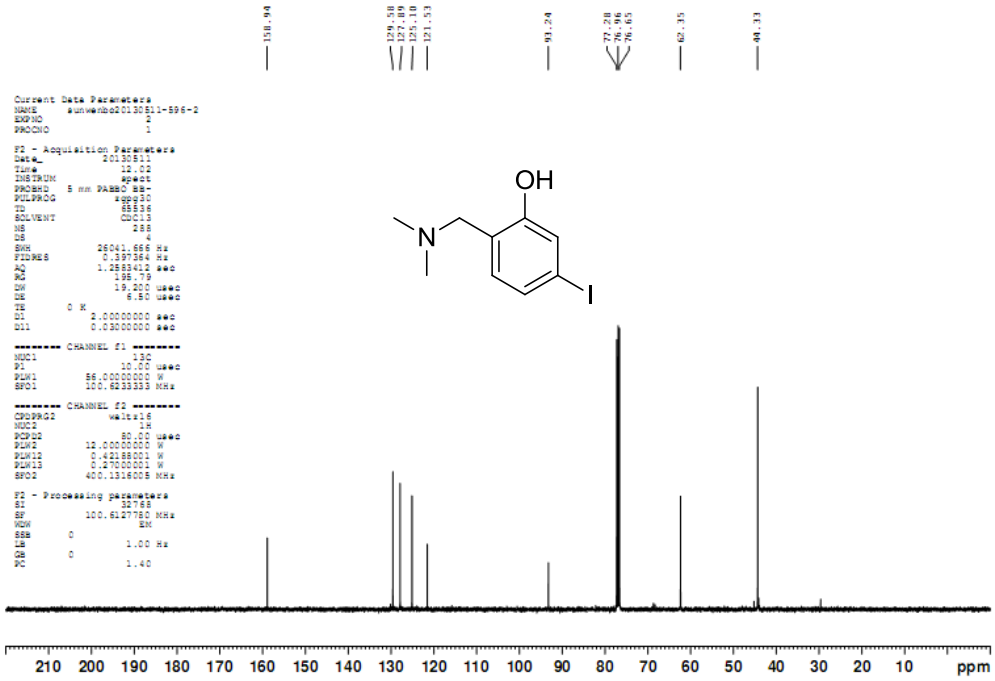
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.17188001 W
PLW13 0.11700001 W
SFO2 400.1316005 MHz

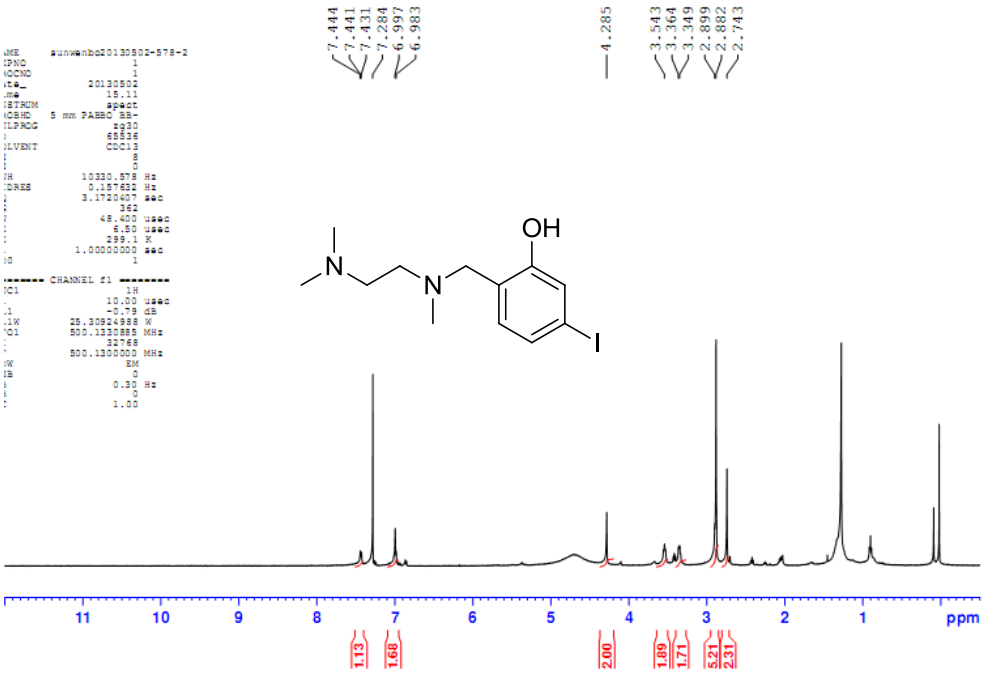
F2 - Processing parameters
SI 32768
SF 100.6127768 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

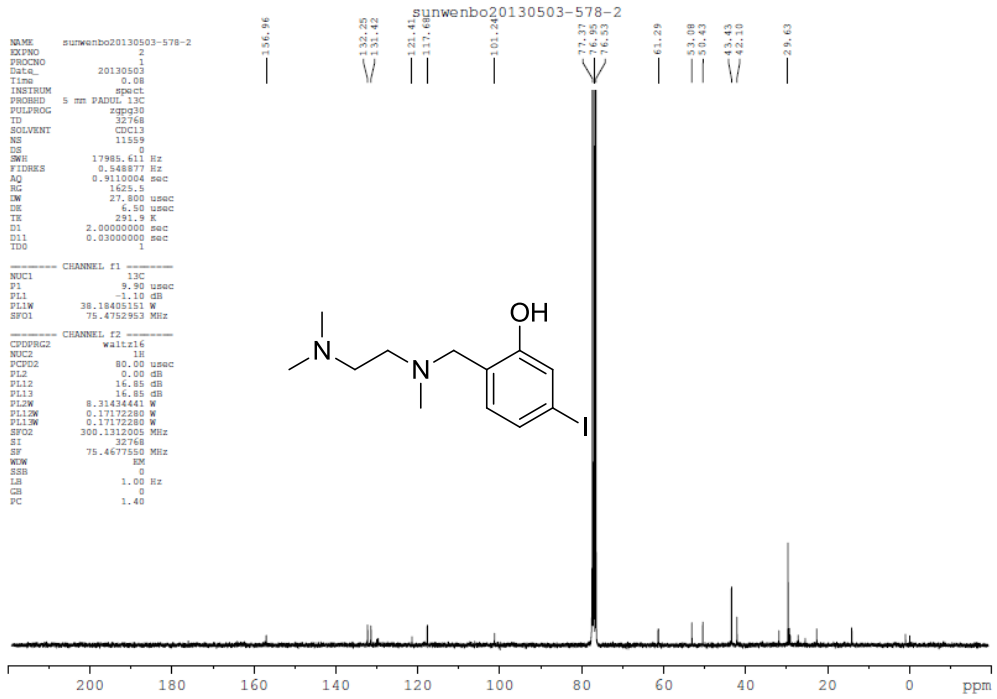

7.3340
7.3290
7.3165
7.3115
7.2848
6.8664
6.8461
6.8287
6.8239
6.8085
6.8036
3.8657
2.3828
OH
N
I
11
10
9
8
7
6
5
4
3
2
1
ppm
0.8925
1.9534
2.0000
5.8185

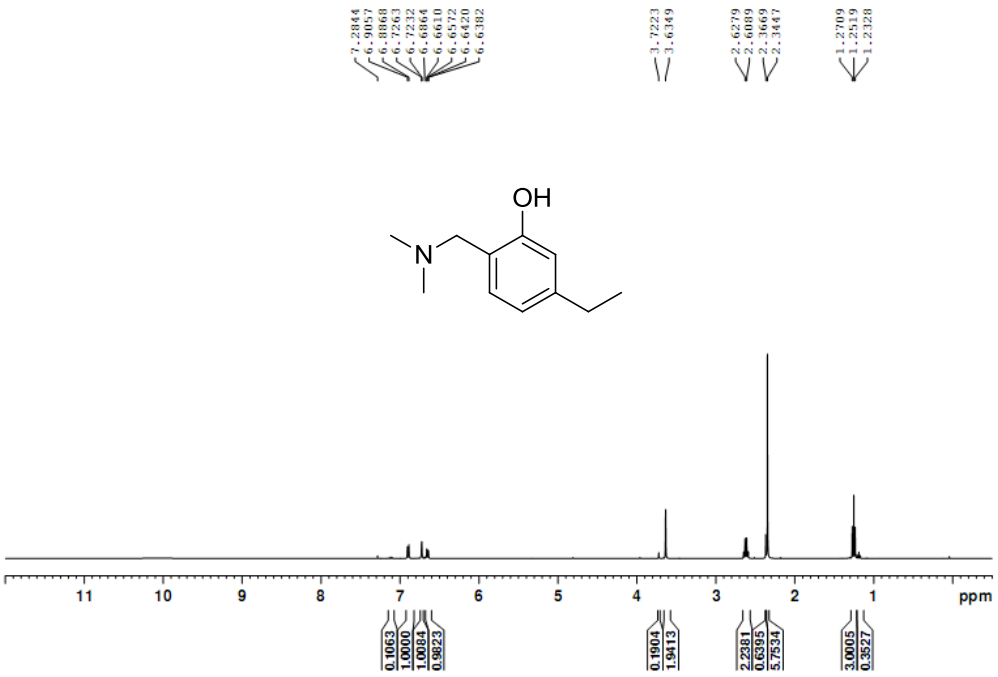

158.42
129.87
123.55
116.80
98.97
77.38
76.96
76.53
67.21
44.16
OH
N
I
200
180
160
140
120
100
80
60
40
20
0
ppm

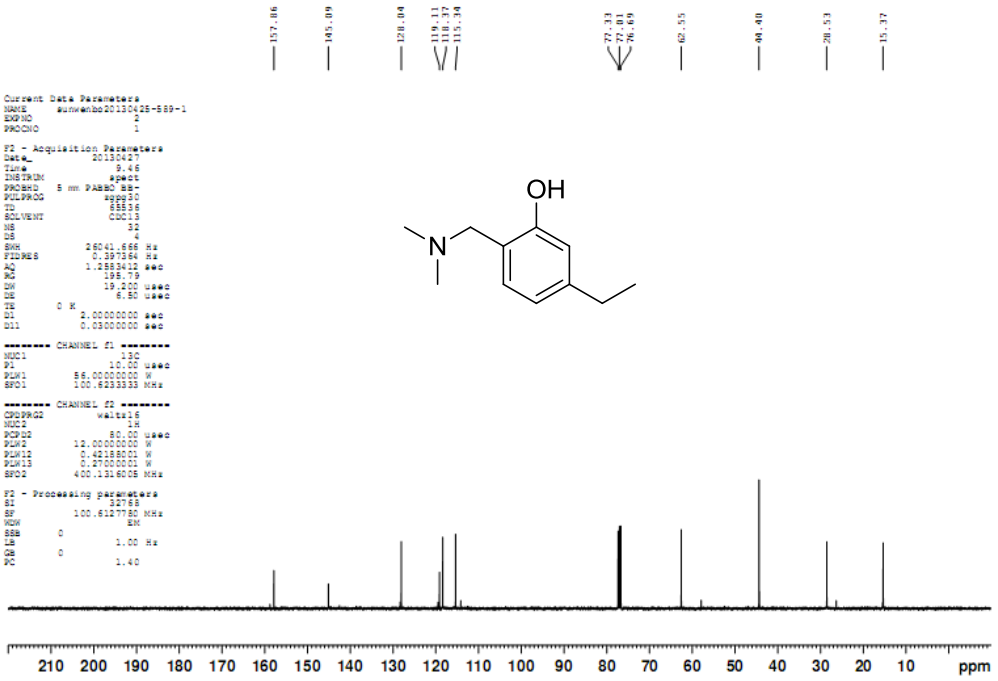

7.285
7.207
7.205
7.123
7.120
7.107
7.104
6.691
6.676
3.606
2.334
OH
N
I
11
10
9
8
7
6
5
4
3
2
1
ppm
0.95
1.00
0.92
1.92
5.66

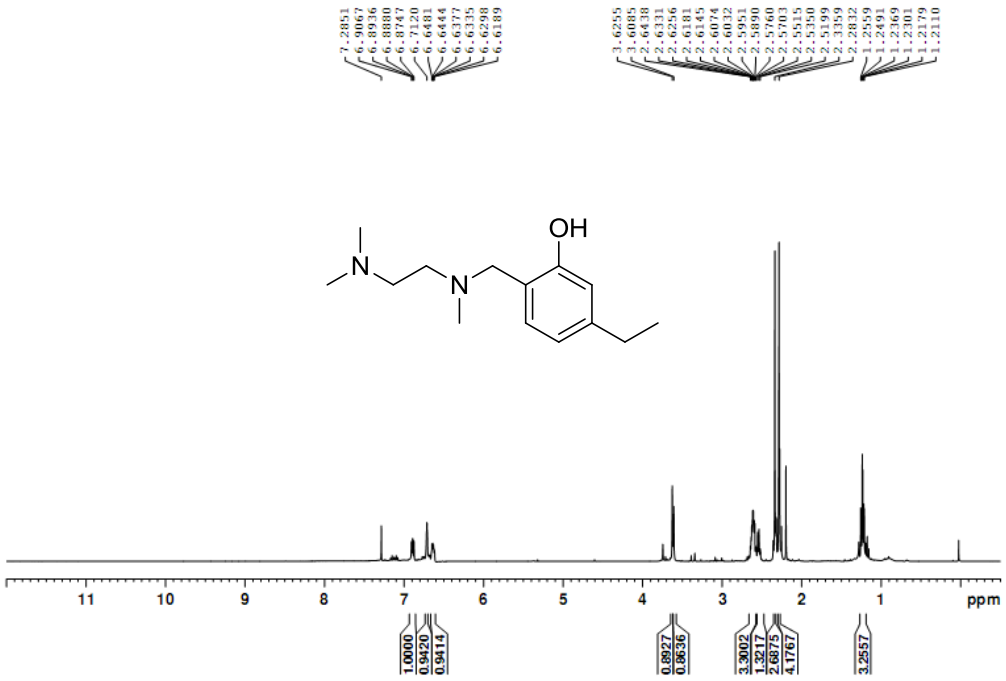

158.94
129.58
127.89
125.10
121.53
93.24
77.28
76.96
76.65
62.35
44.33
OH
N
I
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

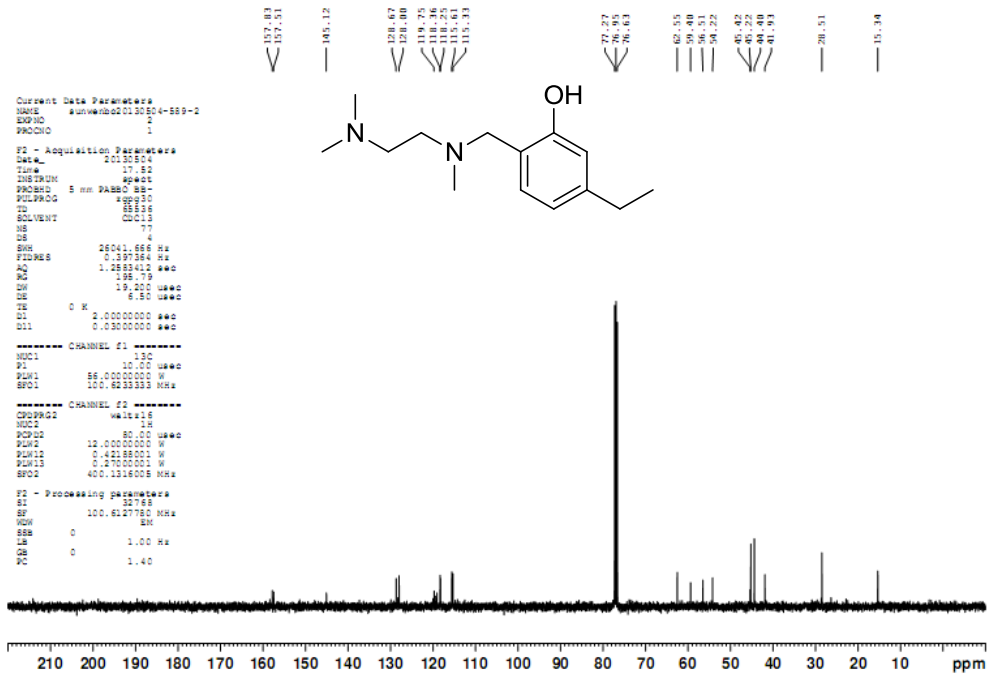

7.444
7.441
7.431
7.284
6.997
6.983
4.285
3.543
3.364
3.349
2.899
2.882
2.743
OH
N
N
I
11
10
9
8
7
6
5
4
3
2
1
ppm
1.13
1.68
2.00
1.89
1.71
5.21
2.31

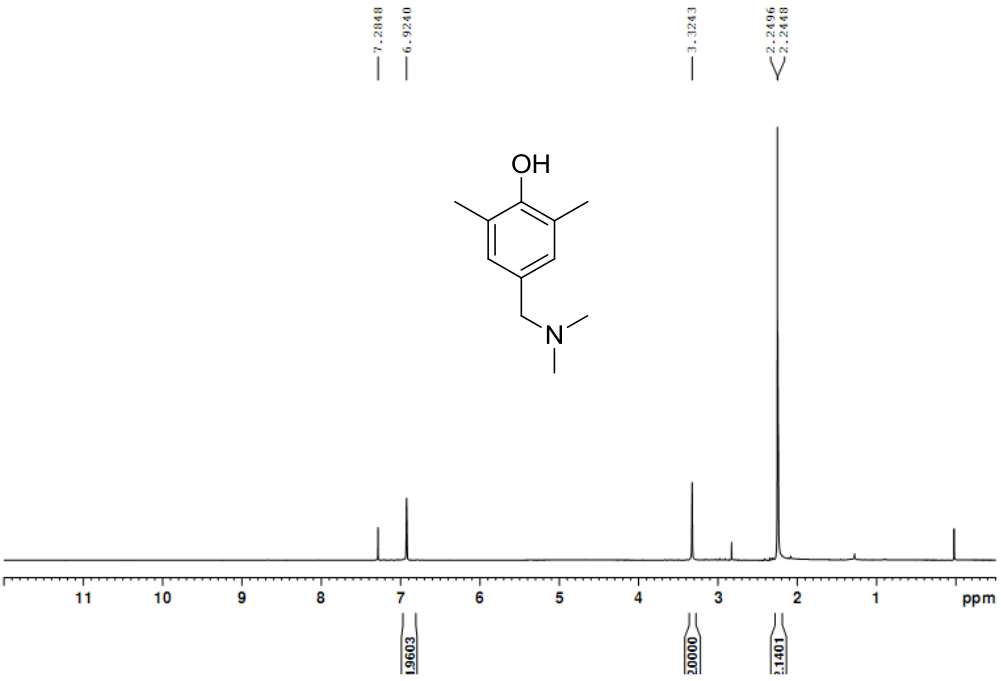

sunwenbo20130503-578-2
156.96
132.25
131.42
121.41
117.68
101.24
77.37
76.95
76.53
61.29
53.08
50.43
43.43
42.10
29.63
OH
N
N
I
200
180
160
140
120
100
80
60
40
20
0
ppm

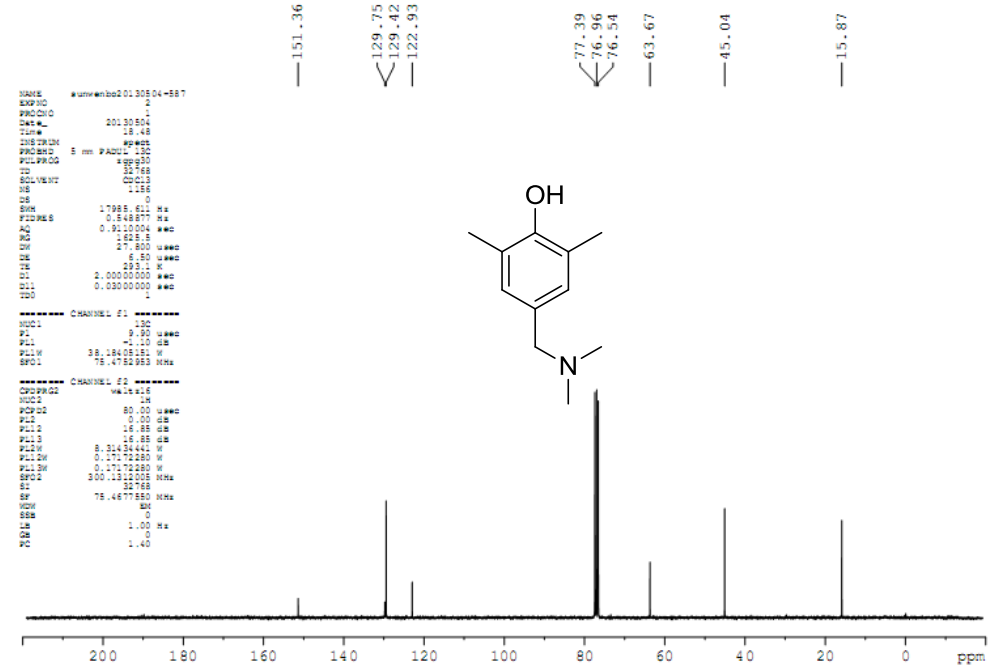

7.2844
6.9057
6.8868
6.7263
6.7232
6.6864
6.6610
6.6572
6.6420
6.6382
3.7223
3.6349
2.6279
2.6089
2.3669
2.3447
1.2709
1.2519
1.2328
OH
N
11
10
9
8
7
6
5
4
3
2
1
ppm
0.1063
1.0000
1.0084
0.9823
0.1904
1.9413
2.2381
0.6395
5.7534
3.0005
0.3527

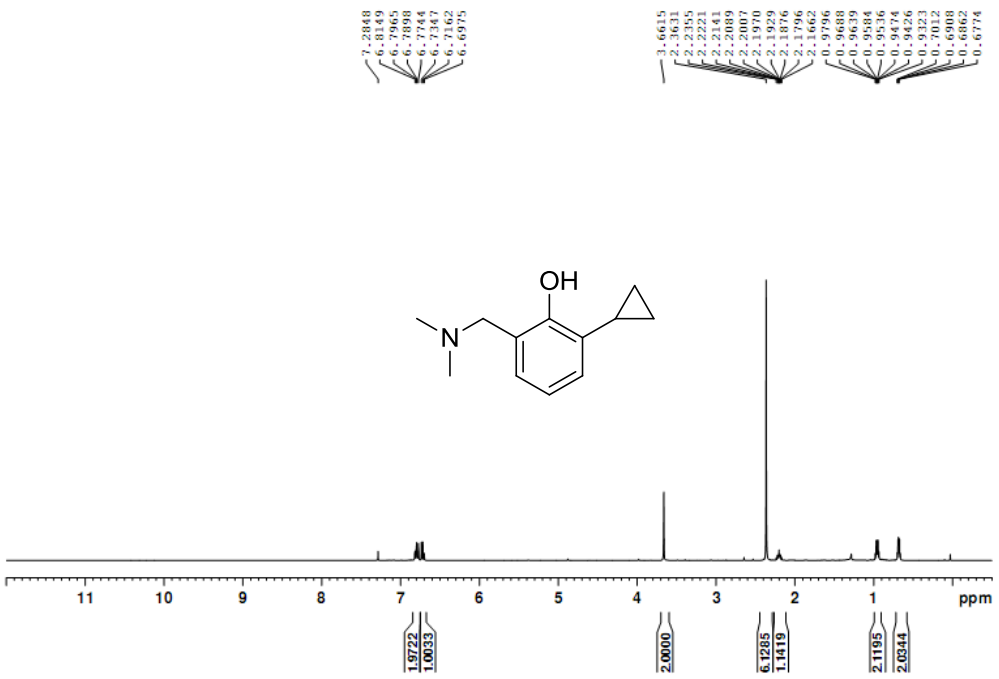

157.86
145.09
128.04
119.11
118.37
115.34
77.33
77.01
76.69
62.55
44.40
28.53
15.37
Current Data Parameters
NAME sunwenbo20130425-589-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130427
Time 9.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 0 K
D1 2.00000000 sec
D11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
OH
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

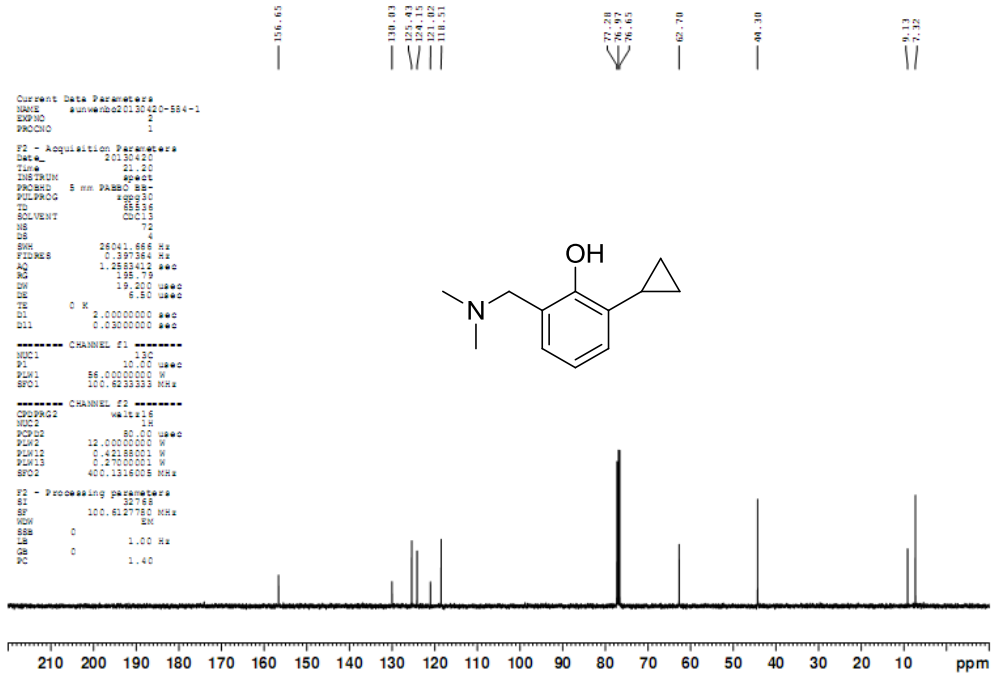

7.2851
6.4067
6.3996
6.8880
6.8747
6.7120
6.6481
6.6444
6.6377
6.6375
6.6298
6.6189
3.6255
3.6085
2.6438
2.6331
2.6256
2.6181
2.6145
2.6074
2.6032
2.5951
2.5890
2.5760
2.5703
2.5515
2.5350
2.5199
2.3359
2.2832
1.2559
1.2491
1.2369
1.2301
1.2179
1.2110
OH
N
N
11
10
9
8
7
6
5
4
3
2
1
ppm
1.0000
0.9420
0.9414
0.0927
0.0636
3.3002
1.3217
2.6875
4.1767
3.2557


157.83
157.51
145.12
128.67
128.00
119.75
118.36
118.25
115.61
115.33
77.27
76.95
76.63
62.55
60.48
58.51
58.22
45.42
45.22
44.88
41.93
29.51
15.34
OH
N
N
Current Data Parameters
NAME sunwenbo20130504-559-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130504
Time 17.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 77
DS 4
SWH 25041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 196.79
DW 19.960 usec
DE 6.50 usec
TE 0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127760 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

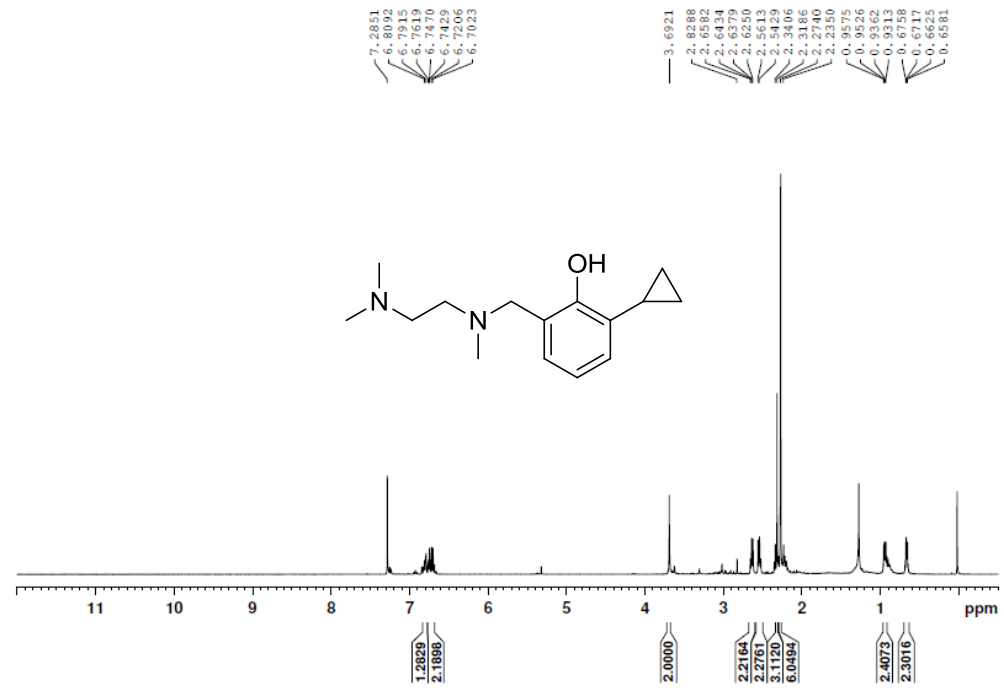

7.2848
6.9240
3.3243
2.2496
2.2448
OH
N
11
10
9
8
7
6
5
4
3
2
1
ppm
1.9603
2.0000
2.1401

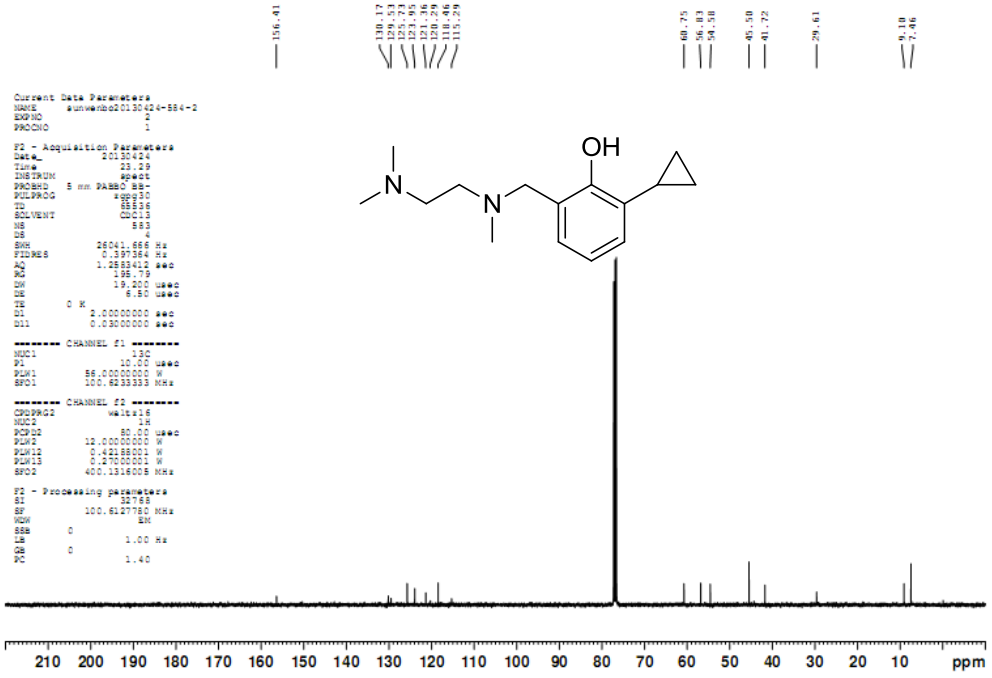

151.36
129.75
129.42
122.93
77.39
76.96
76.54
63.67
45.04
15.87
OH
N
200
180
160
140
120
100
80
60
40
20
0
ppm






```
Current Data Parameters
NAME      sunwenbo20130420-584-1
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20130420
Time              21.20
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   72
DS                    4
SWH           26041.666 Hz
FIDRES        0.397384 Hz
AQ            1.2583412 sec
RG                195.79
DW               19.200 usec
DE                6.50 usec
TE                298.0 K
D1            2.00000000 sec
D11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1           86.00000000 W
SFO1         100.6233333 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2           12.00000000 W
PL12           0.42158001 W
PL13           0.27000001 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127780 MHz
WDW                  EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```





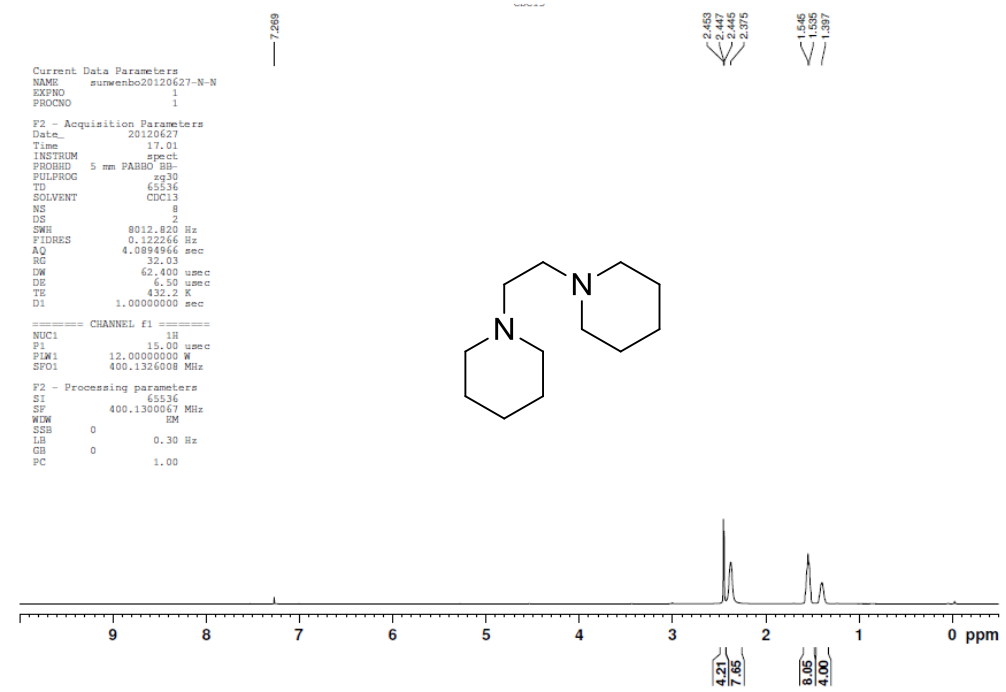
Current Data Parameters
NAME sunwenbo20130424-584-2
EXPNO 2
PROCNO 1

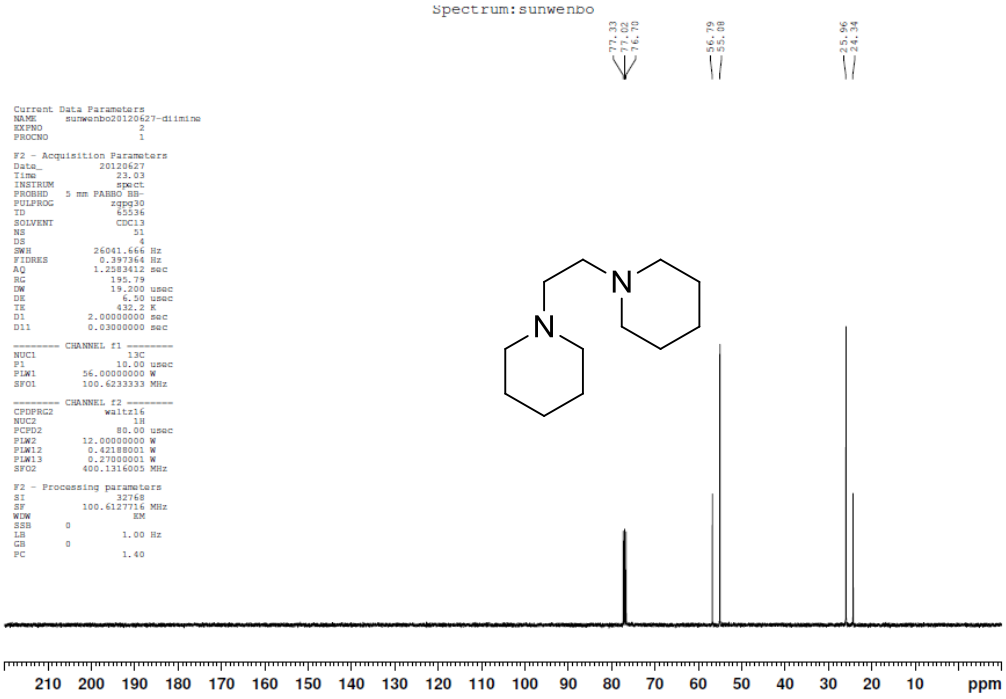
F2 - Acquisition Parameters
Date_ 20130424
Time 23.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 583
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 198.79
DW 19.200 usec
DE 6.50 usec
TE 0 K
D1 2.00000000 sec
D11 0.03000000 sec

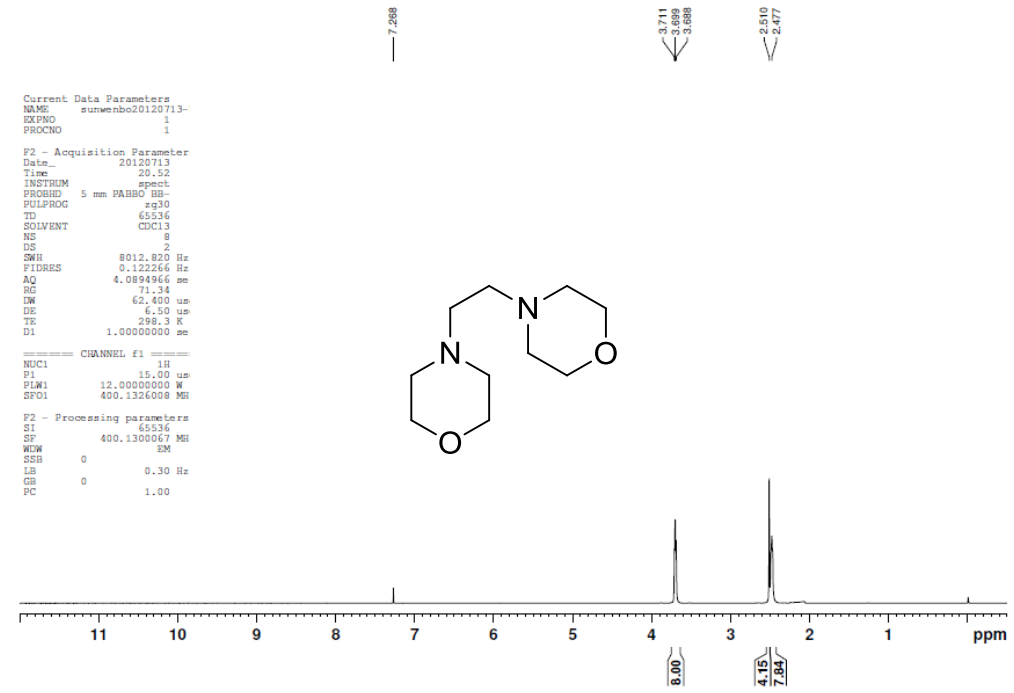
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 56.00000000 W
SFO1 100.6233333 MHz

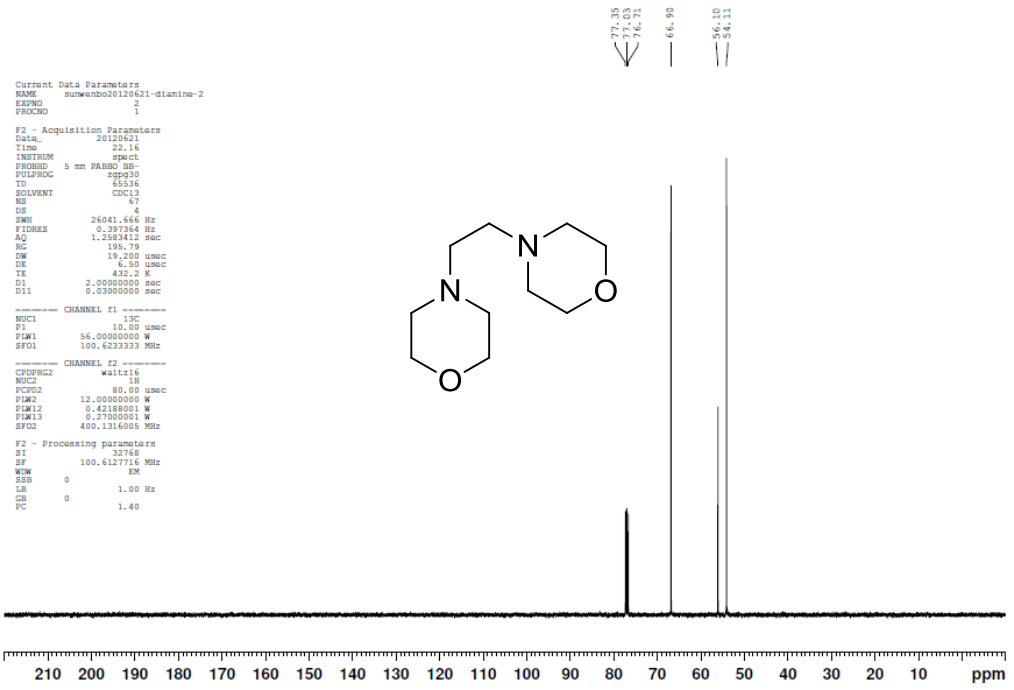
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 12.00000000 W
PL12 0.42188001 W
PL13 0.27000001 W
SFO2 400.1316005 MHz

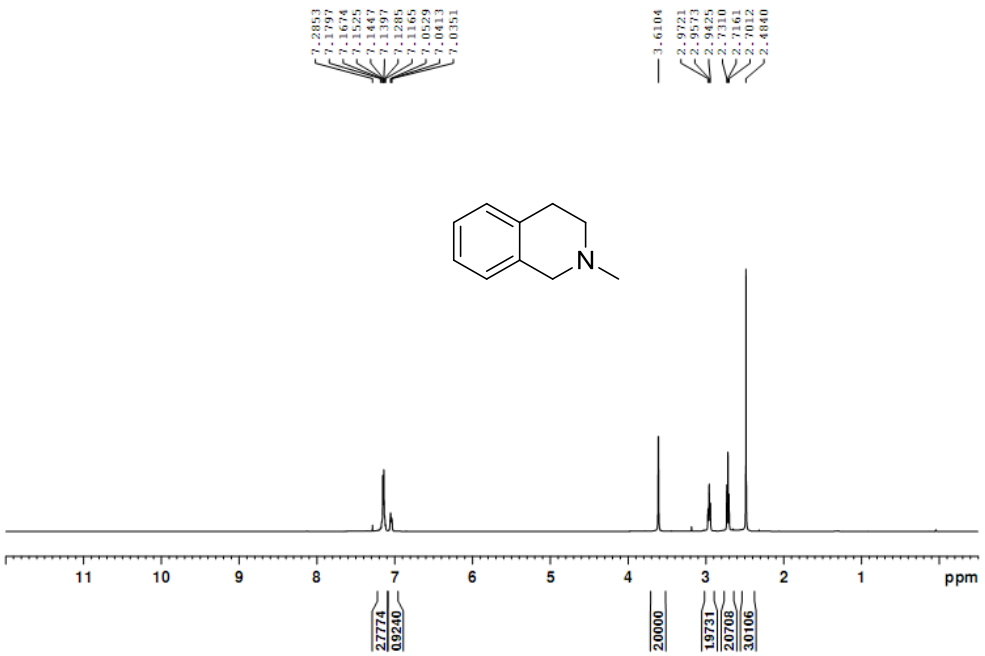
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.269
2.453
2.447
2.445
2.375
1.546
1.535
1.397
Current Data Parameters
NAME sunwenbo20120627-N-N
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120627
Time 17.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 32.03
DW 62.400 usec
DE 6.50 usec
TE 432.2 K
D1 1.00000000 sec
======= CHANNEL f1 =======
NUC1 1H
P1 15.00 usec
PLW1 12.00000000 W
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 65536
SF 400.1300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
N
9
8
7
6
5
4
3
2
1
0 ppm
4.21
7.65
8.05
4.00

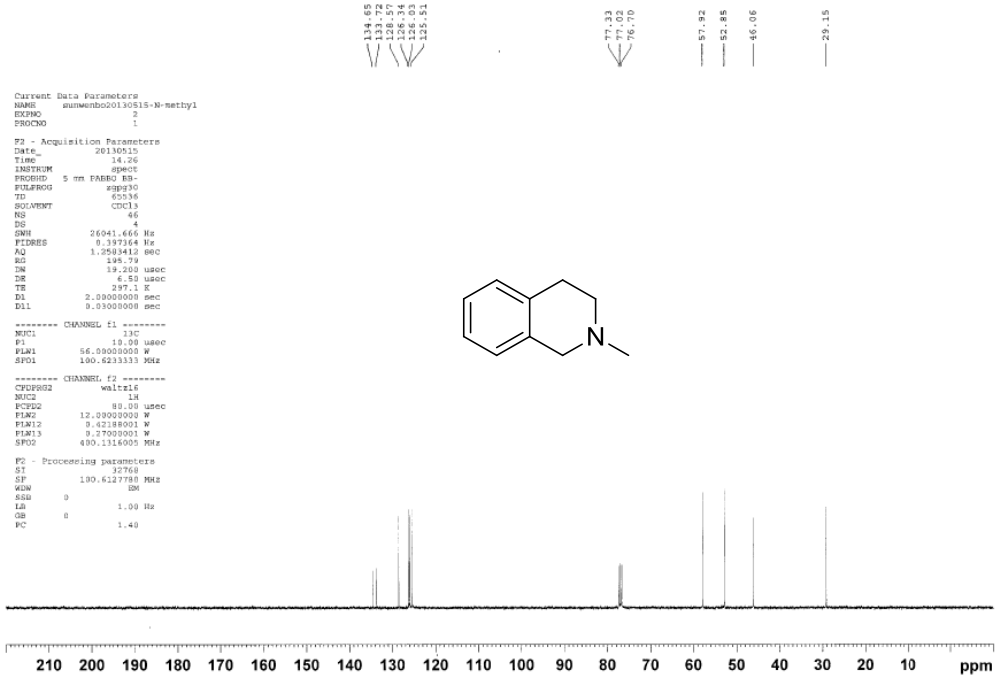

Spectrum:sunwenbo
77.33
77.02
76.70
56.79
55.08
25.96
24.34
Current Data Parameters
NAME sunwenbo20120627-diimine
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120627
Time 23.03
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 432.2 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127716 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

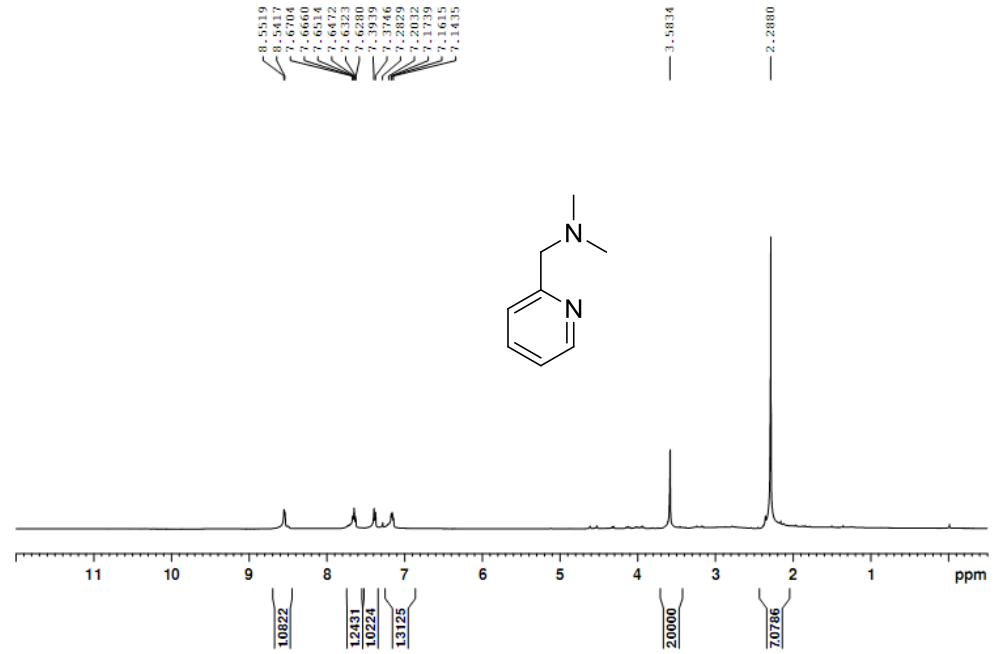


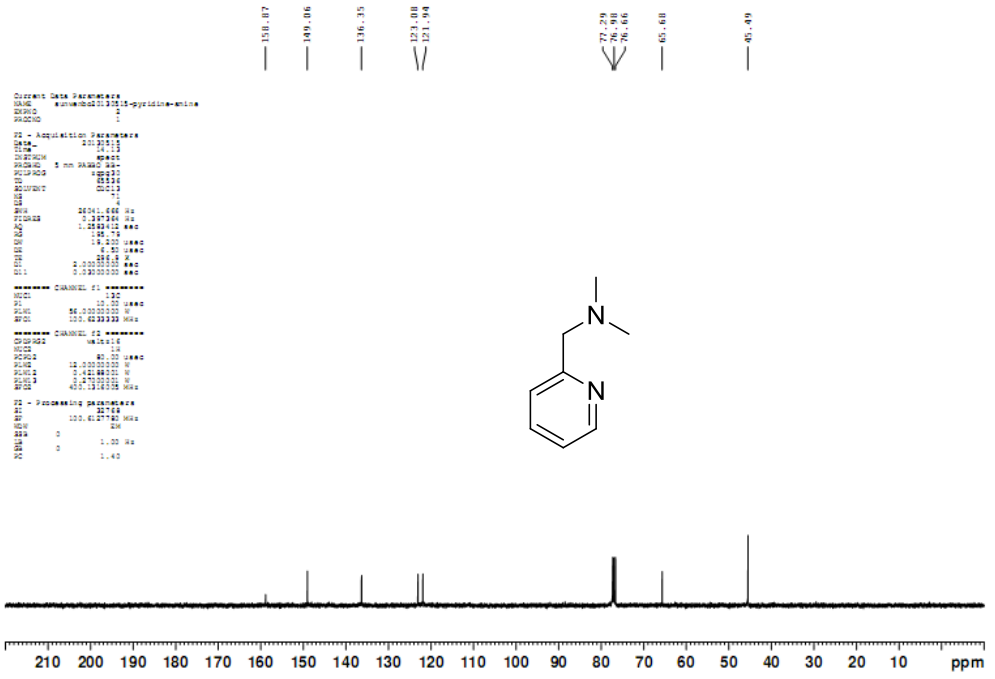


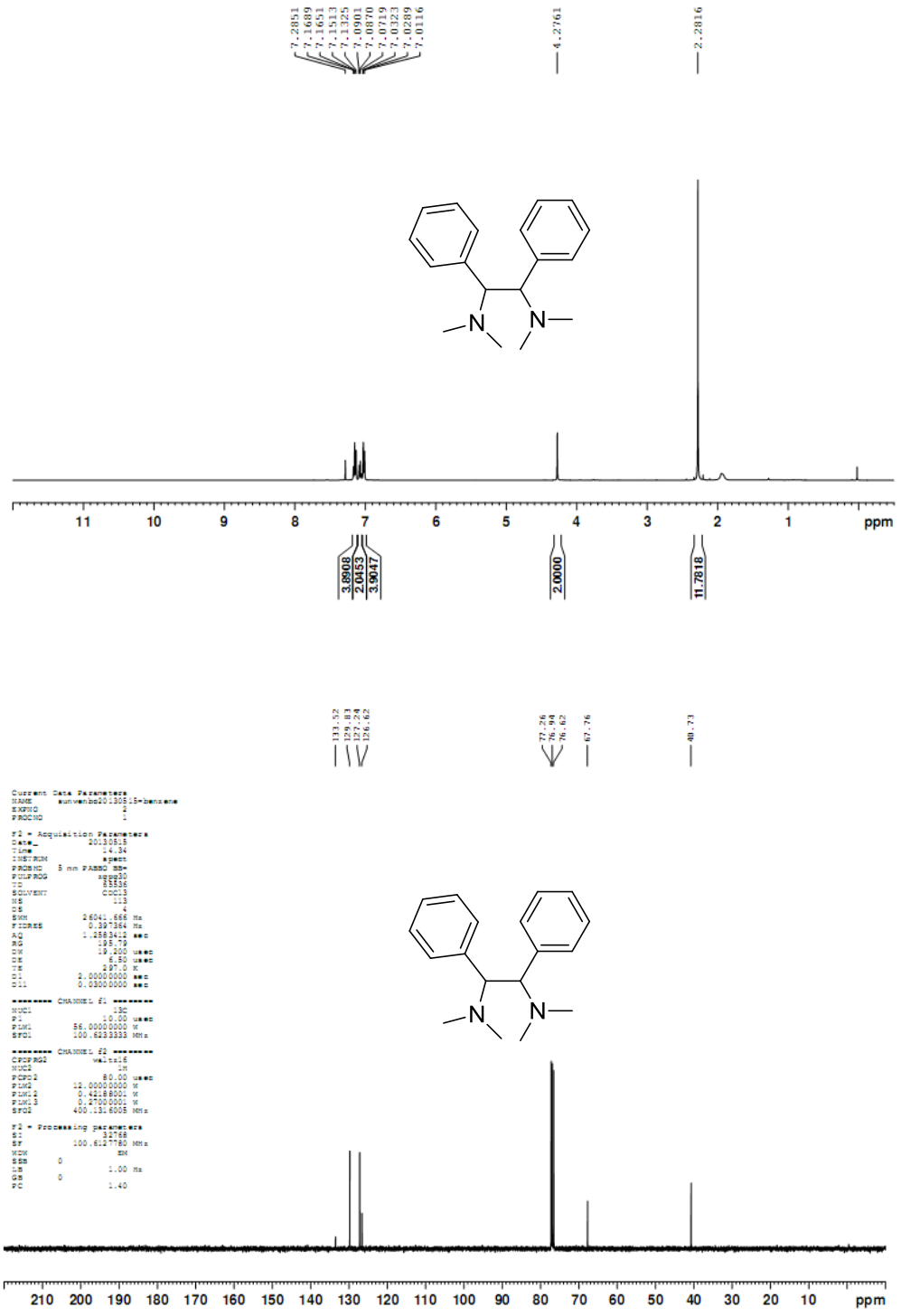

7.2853
7.1997
7.1674
7.1525
7.1447
7.1397
7.1285
7.1165
7.0529
7.0413
7.0351
3.6104
2.9721
2.9573
2.9425
2.7310
2.7161
2.7012
2.4840
N
11
10
9
8
7
6
5
4
3
2
1
ppm
27774
09240
20000
19731
20708
30106

I


134.65
133.72
128.57
126.34
126.03
125.51
77.33
77.02
76.70
57.92
52.85
46.06
29.15
Current Data Parameters
NAME sunwenbo20130515-N-methyl
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130515
Time 14.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 46
DS 4
SWH 26041.666 Hz
FIDRES 0.397364 Hz
AQ 1.2583412 sec
RG 195.79
DW 19.200 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
-------- CHANNEL f1 --------
NUC1 13C
P1 10.00 usec
PLW1 56.00000000 W
SFO1 100.6233333 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 88.00 usec
PLW2 12.00000000 W
PLW12 0.42188001 W
PLW13 0.27000001 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127780 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm


8.5519
8.5417
7.6704
7.6660
7.6514
7.6472
7.6323
7.6280
7.3939
7.3746
7.2829
7.2032
7.1739
7.1615
7.1435
3.5834
2.2880
N
N
11
10
9
8
7
6
5
4
3
2
1
ppm
10822
12431
10224
13125
20000
70786


158.87
149.06
136.35
123.08
121.94
77.29
76.98
76.66
65.68
45.49
N
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



1. Eschweiler, W. *Ber.***1905**, *38*, 880.
2. 2. Clarke, H. T.; Gillespie, H. B.; Weisshaus, S. Z. *J. Am. Chem. Soc.***1933**, *55*, 4571.
3. Gamzaeva, S. A.; Mamedova, **P. Sh.;** Allakhverdieva,K. M. ; Velieva, G. Kh.; Akhundova, M. A.; Allakverdiev, M. A. *Russian Journal of Applied Chemistry*, **2009**,*82*, 1577.