

Supplementary data

Efficient preparation of some new 1-carbamato-alkyl-2-naphthols using *N*-halo reagents in neutral media

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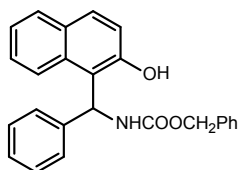
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Khazaei_1326@yahoo.com, moosavizare@yahoo.com

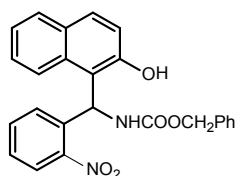
Data of Compounds:

Benzyl (2-hydroxynaphthalen-1-yl)(phenyl)methylcarbamate 1a.



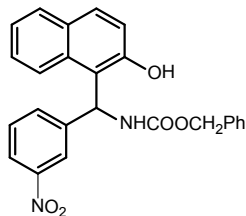
White solid; R_f (EtOAc/n-hexane: 4/6) = 0.60; m.p. 180–182 °C (lit. [27] mp 180–182 °C); ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 5.09 (2H), 6.94 (d, 1H), 6.99–7.83 (16H), 7.99 (d, 1H, NH), 10.13 (s, 1H, OH); ^{13}C NMR (22.5 MHz, DMSO- d_6): δ (ppm) 50.67, 65.67, 118.59, 118.79, 122.49, 122.81, 126.00, 126.34, 126.48, 127.62, 128.01, 128.22, 128.46, 129.26, 132.10, 136.95, 142.35, 152.86, 155.87; IR (KBr, cm^{-1}): 3424, 3200, 3062, 3030, 1676, 1581, 1516, 1439, 1327, 1262, 1220, 1130, 1040, 943, 806, 745, 697.

Benzyl (2-hydroxynaphthalen-1-yl)(2-nitrophenyl)methylcarbamate 2a.



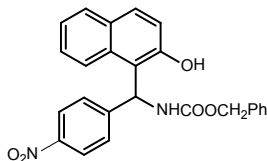
Yellow solid; R_f (EtOAc/n-hexane: 4/6) = 0.35; m.p. 198–200 °C (lit. [26] mp 208–210 °C); ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 5.09 (2H), 7.04–8.15 (17H), 9.84 (s, 1H, OH); ^{13}C NMR (22.5 MHz, DMSO- d_6): δ (ppm) 48.09, 65.55, 116.26, 118.51, 122.45, 123.90, 126.51, 127.28, 127.60, 127.75, 128.20, 129.1, 129.89, 131.95, 132.83, 136.18, 137.03, 148.81, 153.66, 155.71; IR (KBr, cm^{-1}): 3424, 3251, 3062, 1701, 1629, 1579, 1528, 1510, 1453, 1437, 1364, 1334, 1274, 1185, 1130, 1045, 963, 939, 834, 752, 738, 658.

Benzyl (2-hydroxynaphthalen-1-yl)(3-nitrophenyl)methylcarbamate 3a.



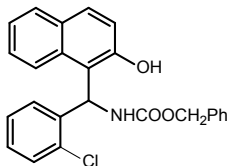
Yellow solid; R_f (EtOAc/n-hexane: 4/6) = 0.50; m.p. 196-197 °C (lit. [26] mp 200-202 °C); ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 5.09 (2H), 6.94-8.12 (17H), 10.23 (s, 1H, OH); ^{13}C NMR (22.5 MHz, DMSO- d_6): δ (ppm) 50.26, 65.82, 117.80, 118.46, 120.50, 121.38, 122.60, 126.82, 127.57, 127.67, 128.24, 128.61, 129.57, 129.86, 131.91, 132.71, 144.99, 153.11, 156.28; IR (KBr, cm^{-1}): 3389, 3352, 1694, 1629, 1527, 1514, 1438, 1348, 1333, 1276, 1233, 1142, 1048, 1016, 922, 830, 810, 743, 624.

Benzyl (2-hydroxynaphthalen-1-yl)(4-nitrophenyl)methylcarbamate 4a.



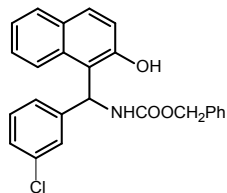
Yellow solid; R_f (EtOAc/n-hexane: 4/6) = 0.50; m.p. 198-200 °C (lit. [26] mp 202-204 °C); ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 5.09 (2H), 7.05-8.18 (17H), 10.20 (s, 1H, OH); ^{13}C NMR (22.5 MHz, DMSO- d_6): δ (ppm) 50.93, 66.32, 118.26, 118.90, 123.061, 123.65, 127.23, 127.59, 128.09, 128.69, 128.85, 129.07, 130.30, 132.43, 146.97, 151.48, 153.56, 156.45; IR (KBr, cm^{-1}): 3424, 3171, 1683, 1629, 1583, 1519, 1439, 1346, 1326, 1274, 1228, 1178, 1134, 1068, 1048, 1004, 949, 854, 827, 743.

Benzyl (2-chlorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate 5a.



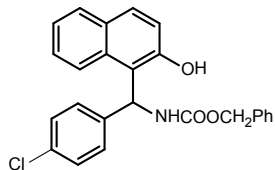
White solid; R_f (EtOAc/n-hexane: 4/6) = 0.50; m.p. 214-216 °C (lit. [26] mp 211-213 °C); ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 5.07 (2H), 6.92 (d, 1H), 7.03-8.03 (15H), 8.08 (d, 1H, NH), 9.96 (s, 1H, OH); ^{13}C NMR (22.5 MHz, DMSO- d_6): δ (ppm) 50.01, 65.47, 117.13, 118.70, 122.36, 122.77, 126.41, 127.35, 127.58, 128.20, 128.39, 129.33, 129.49, 129.88, 132.61, 137.12, 139.31, 153.56, 155.49; IR (KBr, cm^{-1}): 3421, 3160, 3060, 3032, 1701, 1627, 1521, 1437, 1336, 1275, 1248, 1050, 820, 753, 733.

Benzyl(3-chlorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate 6a.



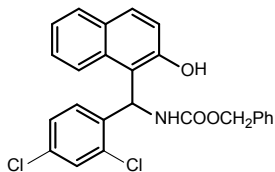
White solid; R_f (EtOAc/n-hexane: 4/6) = 0.65; m.p. 183-185 °C ; ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 5.09 (2H), 6.88-8.00 (17H), 10.19 (s, 1H, OH); ^{13}C NMR (22.5 MHz, DMSO- d_6): δ (ppm) 50.35, 65.82, 118.27, 118.52, 122.61, 124.77, 125.83, 126.34, 126.73, 127.55, 128.24, 128.45, 128.58, 129.59, 129.86, 131.99, 132.98, 136.89, 145.10, 153.00, 155.96; IR (KBr, cm^{-1}): 3435, 3241, 1675, 1629, 1575, 1507, 1439, 1327, 1271, 1227, 1064, 947, 892, 813, 784, 754, 702; CHN analysis: Anal. Calcd for $\text{C}_{25}\text{H}_{20}\text{ClNO}_3$: C, 71.85; H, 4.82; N, 3.35. Found: C, 71.97; H, 4.91; N, 3.18.

Benzyl (4-chlorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate 7a.



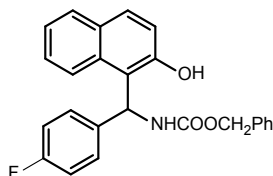
White solid; R_f (EtOAc/n-hexane: 4/6) = 0.60; m.p. 176-177 °C (lit. [26] mp 178-180 °C); ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 5.08 (2H), 6.84-7.94 (17H), 10.16 (s, 1H, OH); ^{13}C NMR (22.5 MHz, DMSO- d_6): δ (ppm) 50.23, 65.79, 118.53, 122.57, 126.64, 127.93, 128.24, 128.56, 129.49, 131.04, 132.00, 136.91, 141.45, 152.96, 155.93; IR (KBr, cm^{-1}): 3426, 3167, 3050, 1679, 1519, 1488, 1438, 1328, 1273, 1227, 1178, 1134, 1068, 1012, 937, 851, 819, 806, 722, 695.

Benzyl (2,4-dichlorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate 8a.



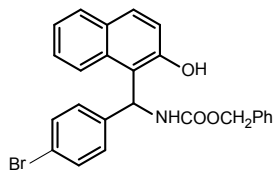
White solid; R_f (EtOAc/n-hexane: 4/6) = 0.60; m.p. 202-204 °C (lit. [26] mp 208-210 °C); ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 5.05 (2H), 6.87-8.06 (16H), 9.94 (s, 1H, OH); ^{13}C NMR (22.5 MHz, DMSO- d_6): δ (ppm) 49.66, 65.53, 116.46, 118.66, 122.51, 126.50, 127.36, 127.59, 128.20, 128.56, 129.67, 131.19, 132.05, 132.55, 133.29, 137.06, 138.76, 153.59, 155.47; IR (KBr, cm^{-1}): 3417, 3343, 3031, 1678, 1628, 1585, 1513, 1432, 1341, 1271, 1139, 1054, 936, 878, 844, 744.

Benzyl (4-fluorophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate 9a.



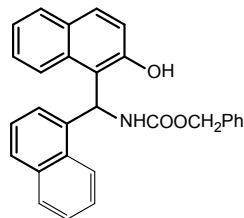
White solid; R_f (EtOAc/n-hexane: 4/6) = 0.60; m.p. 202-203 °C; ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 5.07 (2H), 6.84-7.96 (17H), 10.14 (s, 1H, OH); ^{13}C NMR (22.5 MHz, DMSO- d_6): δ (ppm) 50.29, 65.76, 114.20, 115.16, 118.61, 122.59, 126.62, 127.63, 128.20, 128.49, 129.39, 132.03, 136.92, 138.40, 138.52, 152.92, 155.90, 166.36; IR (KBr, cm^{-1}): 3425, 3251, 1677, 1629, 1582, 1507, 1440, 1328, 1274, 1220, 1161, 1066, 1039, 944, 854, 755, 704; CHN analysis: Anal. Calcd for $\text{C}_{25}\text{H}_{20}\text{FNO}_3$: C, 74.80; H, 5.02; N, 3.49. Found: C, 74.93; H, 4.89; N, 3.61.

Benzyl (4-bromophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate 10a.



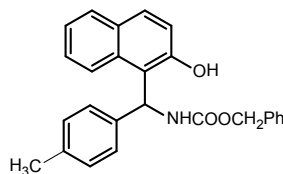
White solid; R_f (EtOAc/n-hexane: 4/6) = 0.57; m.p. 180-182 °C; ^1H NMR (400 MHz, DMSO- d_6): δ (ppm) 5.03 (d, $J=12.4$ Hz, 1H), 5.08 (d, $J=12.8$ Hz, 1H), 6.84 (d, $J=8.4$ Hz, 1H), 7.14–7.21 (m, 3H), 7.25–7.45 (m, 9H), 7.75–7.81 (m, 2H), 7.86 (1H), 10.13 (s, 1H, OH); ^{13}C NMR (100 MHz, DMSO- d_6): δ (ppm) 50.44, 66.27, 118.83, 118.91, 119.94, 123.05, 123.45, 127.09, 128.29, 128.79, 129.10, 130.05, 131.45, 132.45, 137.39, 142.38, 153.49, 156.57; IR (KBr, cm^{-1}): 3424, 3172, 2939, 1681, 1628, 1580, 1519, 1484, 1438, 1329, 1273, 1227, 1791, 1136, 1068, 1007, 960, 937, 817, 736; MS (EI, 70 eV): m/z (%) = 463(M $^{+}$); CHN analysis: Anal. Calcd for $\text{C}_{25}\text{H}_{20}\text{BrNO}_3$: C, 64.95; H, 4.36; N, 3.03. Found: C, 64.77; H, 4.46; N, 3.14.

Benzyl (2-hydroxynaphthalen-1-yl)(naphthalen-1-yl)methylcarbamate 11a.



White solid; R_f (EtOAc/n-hexane: 4/6) = 0.55; m. p. 214-217 °C; ^1H NMR (400 MHz, DMSO- d_6): δ (ppm) 4.99 (d, $J=13.3$ Hz, 1H), 5.11 (d, $J=13.2$, 1H), 7.21–7.49 (m, 12H), 7.75–7.80 (m, 3H), 7.92–8.00 (m, 3H), 8.12 (2H), 10.08 (s, 1H, OH); ^{13}C NMR (100 MHz, DMSO- d_6): δ (ppm) 49.62, 65.81, 118.51, 119.02, 122.86, 123.94, 125.64, 125.91, 126.03, 126.66, 127.85, 128.14, 128.25, 128.76, 129.00, 129.06, 129.22, 129.87, 125.91, 126.03, 126.66, 127.85, 128.14, 128.25, 128.76, 129.00, 129.06, 129.22, 129.87, 131.53, 133.18, 134.08, 137.38, 137.72, 153.68, 156.16; IR (KBr, cm^{-1}): 3406, 3373, 3068, 1680, 1628, 1516, 1437, 1338, 1273, 1246, 1178, 1134, 1074, 1048, 885, 803, 755, 736; MS (EI, 70 eV): m/z (%) = 433(M^+); CHN analysis: Anal. Calcd for $\text{C}_{29}\text{H}_{23}\text{NO}_3$: C, 80.35; H, 5.35; N, 3.23. Found: C, 80.49; H, 5.47; N, 3.11.

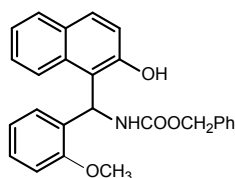
Benzyl (2-hydroxynaphthalen-1-yl)(p-tolyl)methylcarbamate 12a.



White solid; R_f (EtOAc/n-hexane: 4/6) = 0.60; m. p. 152-154 °C; ^1H NMR (90 MHz, DMSO- d_6): δ (ppm) 2.21 (s, 3H, CH_3), 5.02 (d, $J=12.4$ Hz, 2H), 5.08 (d, $J=12.4$ Hz, 1H), 6.85 (d, $J=8\text{Hz}$, 1H), 7.04 (d, $J=7.6$ Hz, 2H), 7.10 (d, $J=7.2$ Hz, 2H), 7.19–7.35 (m, 9H), 7.74–7.80 (m, 3H), 7.90 (1H), 10.07 (s, 1H, OH); ^{13}C NMR (100 MHz, DMSO- d_6): δ (ppm) 21.01, 50.78, 66.17, 118.98, 119.44, 122.97, 123.61, 126.49, 126.94, 127.57, 128.27, 128.80, 128.92, 129.03, 129.16, 129.72, 132.56, 135.89, 137.49, 139.81, 153.36,

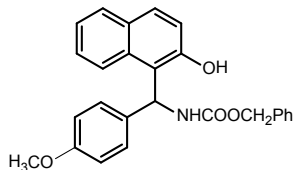
156.50; IR (KBr, cm^{-1}): 3427, 3175, 3010, 1681, 1629, 1579, 1517, 1454, 1437, 1385, 1330, 1273, 1242, 1178, 1135, 1068, 1047, 962, 937, 851, 777, 735, 696; MS (EI, 70 eV): m/z (%) = 397(M^+); CHN analysis: Anal. Calcd for $\text{C}_{26}\text{H}_{23}\text{NO}_3$: C, 78.57; H, 5.83; N, 3.52. Found: C, 78.65; H, 5.69; N, 3.59.

Benzyl (2-hydroxynaphthalen-1-yl)(2-methoxyphenyl)methylcarbamate 13a.



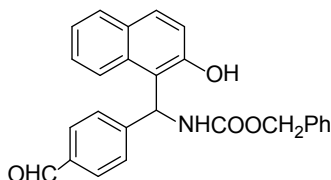
White solid; R_f (EtOAc/n-hexane: 4/6) = 0.50; m. p. 202-204 $^{\circ}\text{C}$; ^1H NMR (400 MHz, DMSO-d_6): δ (ppm) 3.62 (s, 3H, OMe), 4.97 (d, $J=12.4$ Hz, 1H), 5.06 (d, $J=12.4$ Hz, 1H), 6.84 (t, 1H), 6.90 (d, $J=7.6$ Hz, 1H), 7.04 (d, $J=8.4$ Hz, 1H), 7.15–7.32 (m, 10H), 7.6 (1H), 7.71 (d, $J=8.8$ Hz, 1H), 7.77 (d, $J=7.6$ Hz, 1H), 8.2 (d, $J=7.6$ Hz, 1H), 10.00 (s, 1H, OH); ^{13}C NMR (100 MHz, DMSO-d_6): δ (ppm) 47.24, 55.76, 65.82, 111.30, 119.06, 119.15, 120.23, 122.79, 123.82, 126.44, 128.06, 128.15, 128.50, 128.68, 128.75, 128.89, 129.39, 130.18, 132.83, 137.68, 153.57, 155.86, 156.99; IR (KBr, cm^{-1}): 3422, 3159, 2940, 1700, 1626, 1583, 1523, 1437, 1341, 1276, 1245, 1166, 1133, 1046, 967, 938, 822, 752, 733, 671; MS (EI, 70 eV): m/z (%) = 413(M^+); CHN analysis: Anal. Calcd for $\text{C}_{26}\text{H}_{23}\text{NO}_4$: C, 75.53; H, 6.51; N, 3.39. Found: C, 75.41; H, 6.43; N, 3.51.

Benzyl (2-hydroxynaphthalen-1-yl)(4-methoxyphenyl)methylcarbamate 14a.



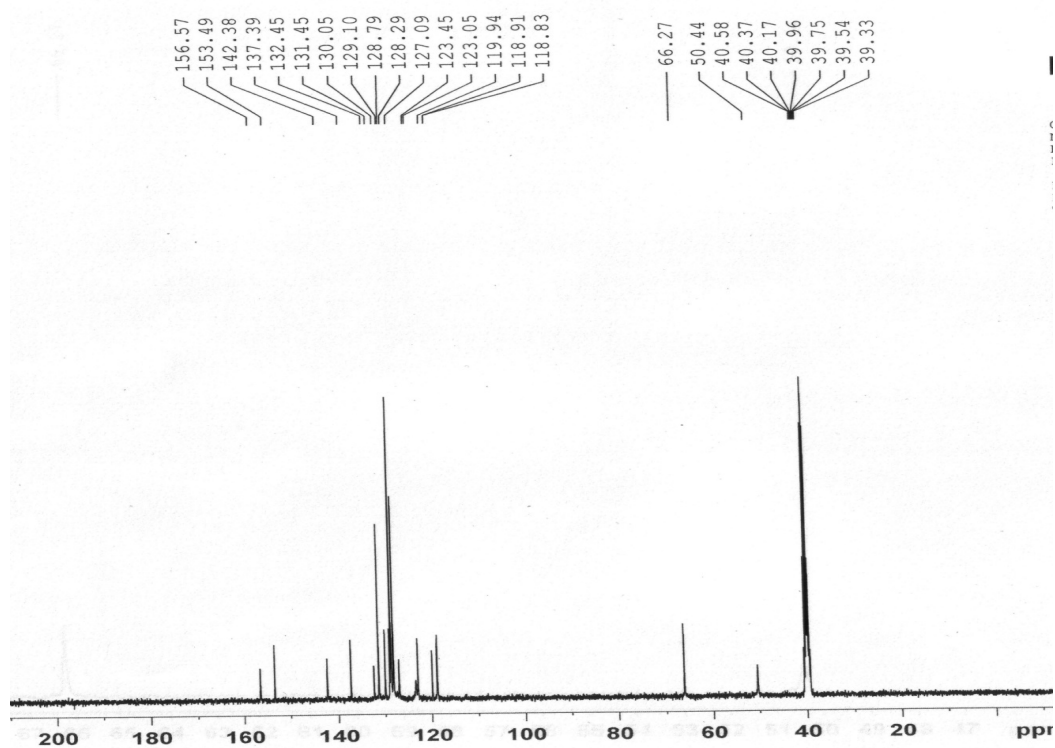
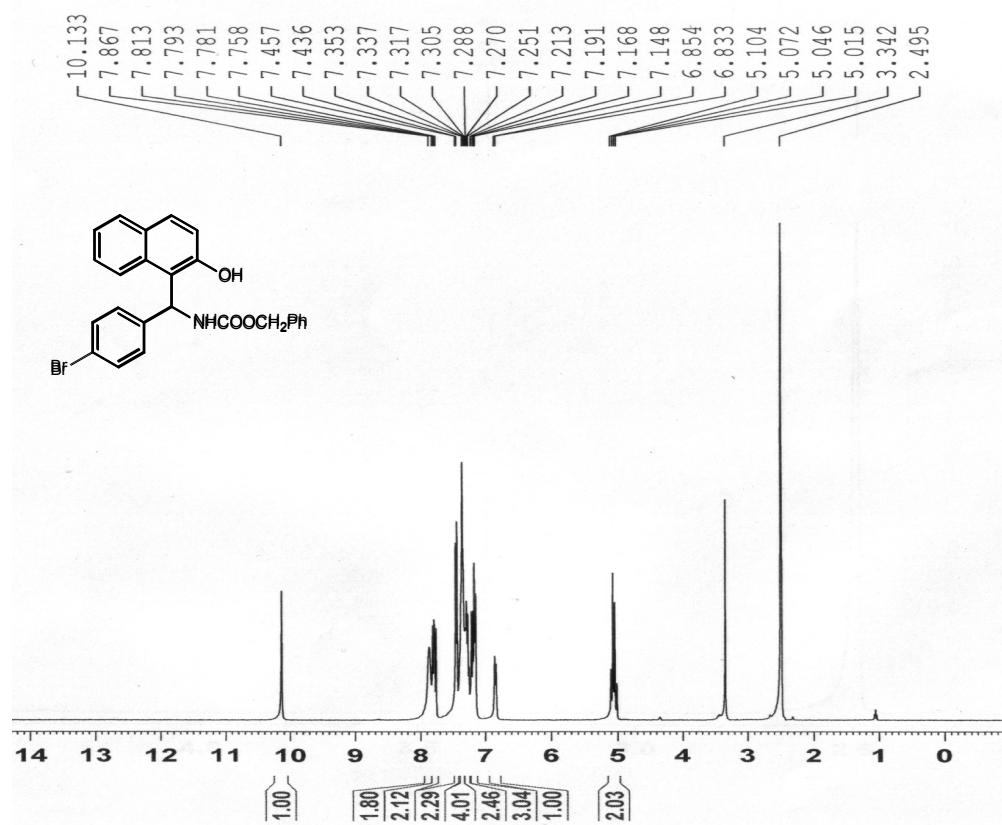
White solid; R_f (EtOAc/n-hexane: 4/6) = 0.52; m. p. 165-167 °C; ^1H NMR (400 MHz, DMSO- d_6): δ (ppm) 3.67 (s, 3H, CH₃), 5.02 (d, J =12.4 Hz, 1H), 5.08 (d, J =12.8 Hz, 1H), 6.81 (d, J =8 Hz, 3H), 7.14–7.34 (m, 9H), 7.74–7.80 (m, 3H), 7.92 (2H), 10.10 (s, 1H, OH); ^{13}C NMR (100 MHz, DMSO- d_6): δ (ppm) 50.62, 55.43, 66.15, 114.00, 119.01, 119.44, 122.99, 123.59, 126.96, 127.79, 128.28, 128.82, 128.92, 129.05, 129.70, 132.53, 134.66, 137.49, 153.33, 156.46, 158.37; IR (KBr, cm^{-1}): 3427, 3175, 3010, 1681, 1629, 1579, 1517, 1454, 1437, 1385, 1330, 1273, 1242, 1178, 1135, 1068, 1047, 962, 937, 851, 777, 735, 696; MS (EI, 70 eV): m/z (%) =413(M^+); CHN analysis: Anal. Calcd for $\text{C}_{26}\text{H}_{23}\text{NO}_4$: C, 75.53; H, 6.51; N, 3.39. Found: C, 75.67; H, 6.41; N, 3.41.

Benzyl (4-formylphenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate 1b.

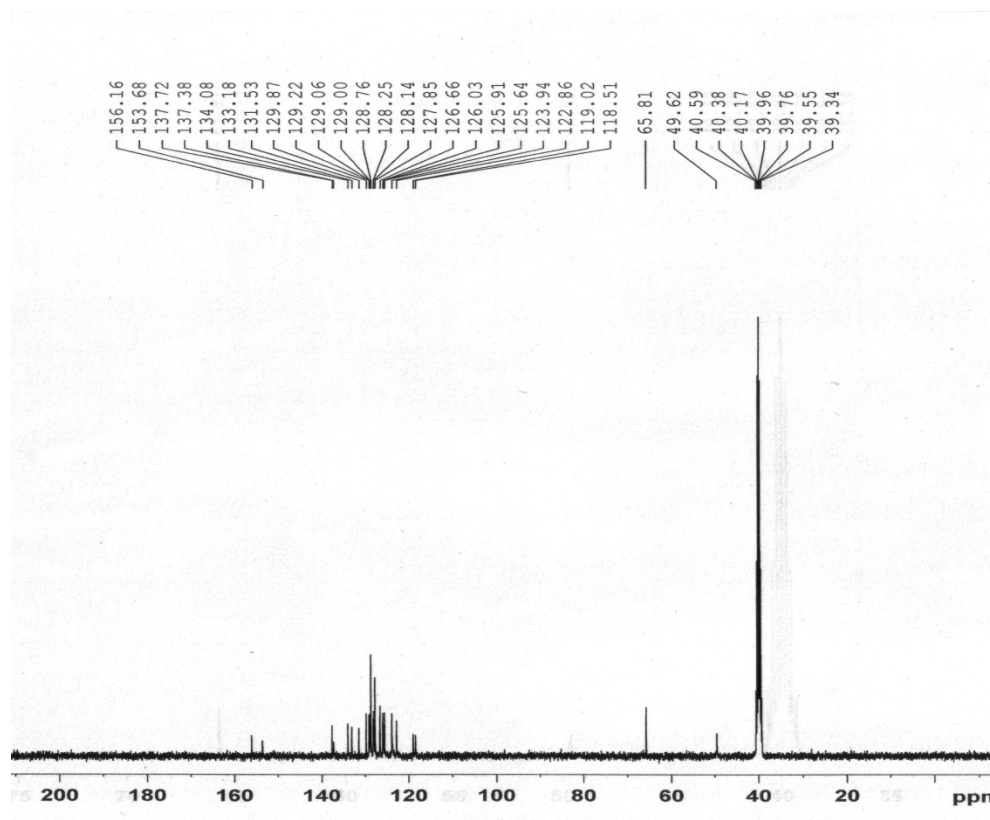
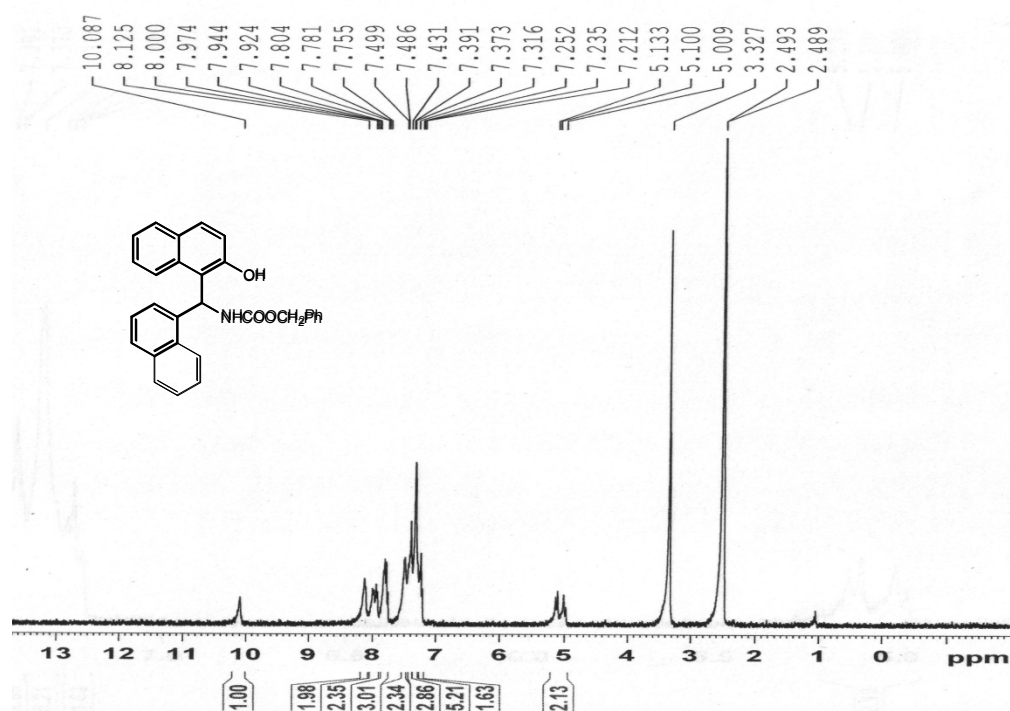


White solid; R_f (EtOAc/n-hexane: 4/6) = 0.35; m. p. 180-185 °C; ^1H NMR (400 MHz, DMSO- d_6): δ (ppm) 5.03 (d, J =12.4 Hz, 1H), 5.09 (d, J =12.4 Hz, 1H), 6.95–7.92 (17H), 9.91 (s, 1H, CHO), 10.15 (s, 1H, OH); ^{13}C NMR (100 MHz, DMSO- d_6): δ (ppm) 50.82, 66.28, 118.71, 118.84, 123.05, 123.51, 127.09, 128.30, 128.82, 129.09, 129.96, 130.19, 132.43, 135.09, 137.37, 150.11, 153.53, 156.66, 193.05 ; IR (KBr, cm^{-1}): 3407, 3299, 3063, 1697, 1682, 1628, 1606, 1578, 1515, 1438, 1323, 1274, 1214, 1169, 1137, 1047, 944, 815, 745, 696; MS (EI, 70 eV): m/z (%) =411(M^+).

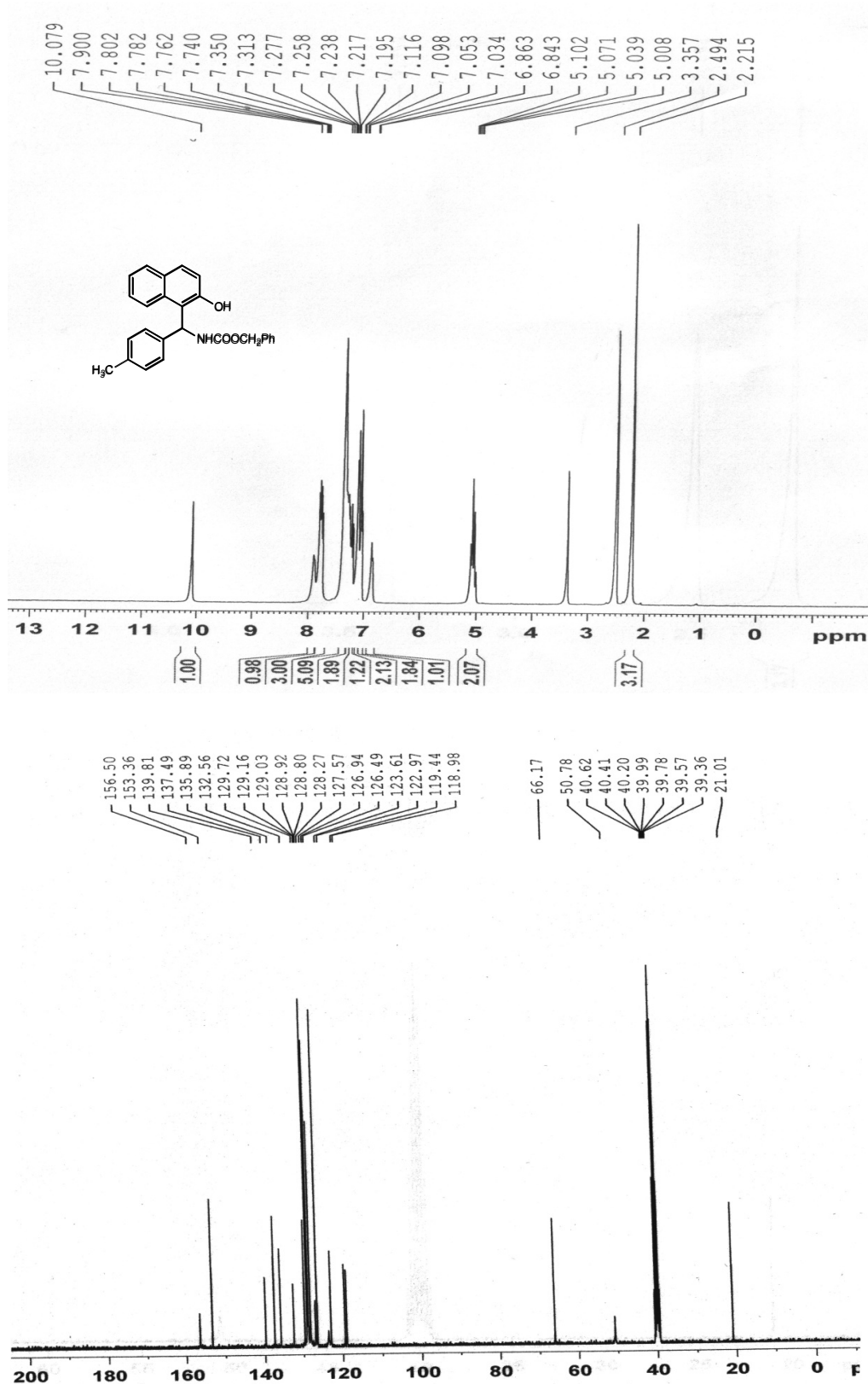
Benzyl (4-bromophenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate 10a.



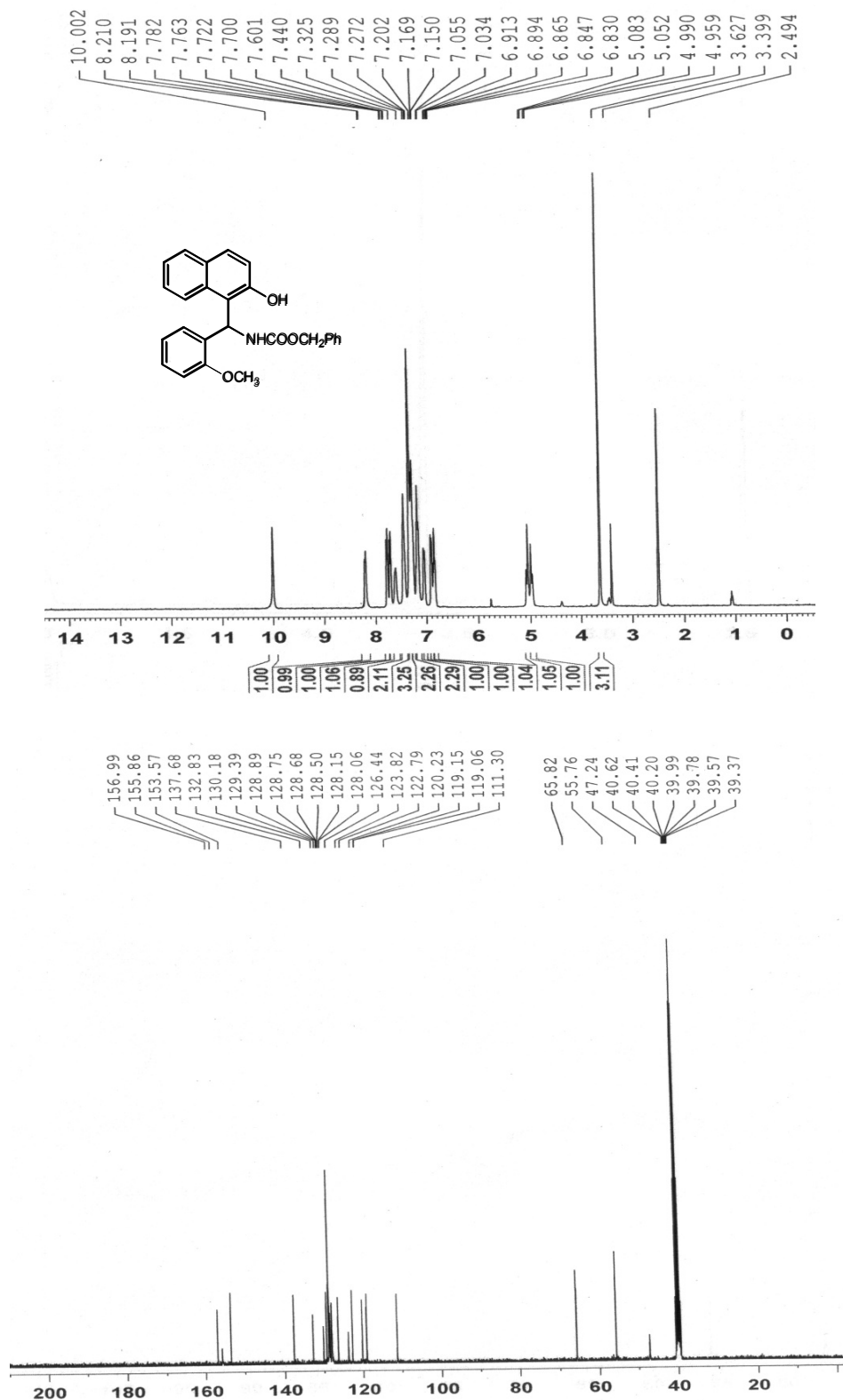
Benzyl (2-hydroxynaphthalen-1-yl)(naphthalen-1-yl)methylcarbamate 11a.



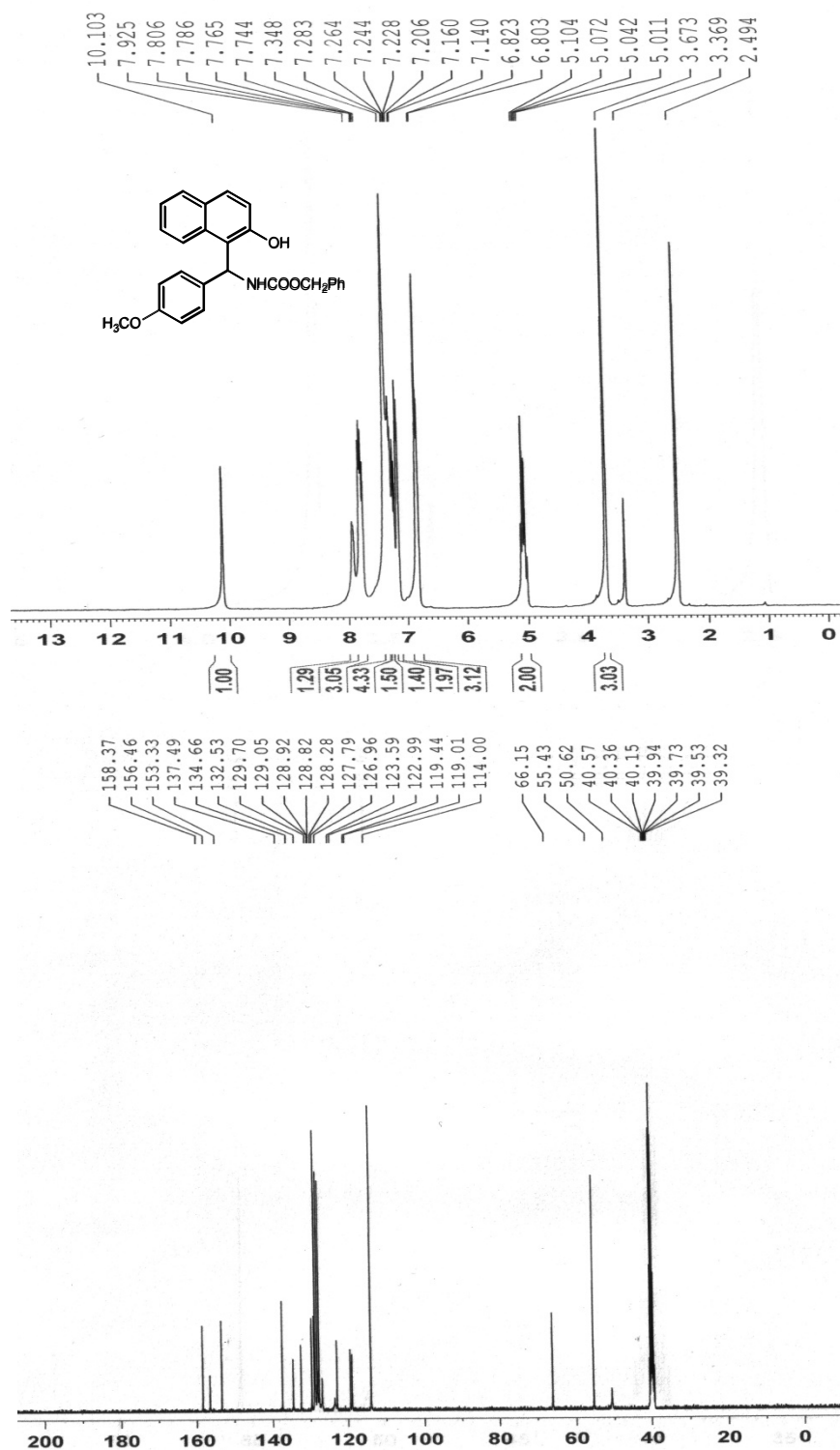
Benzyl (2-hydroxynaphthalen-1-yl)(p-tolyl)methylcarbamate 12a.



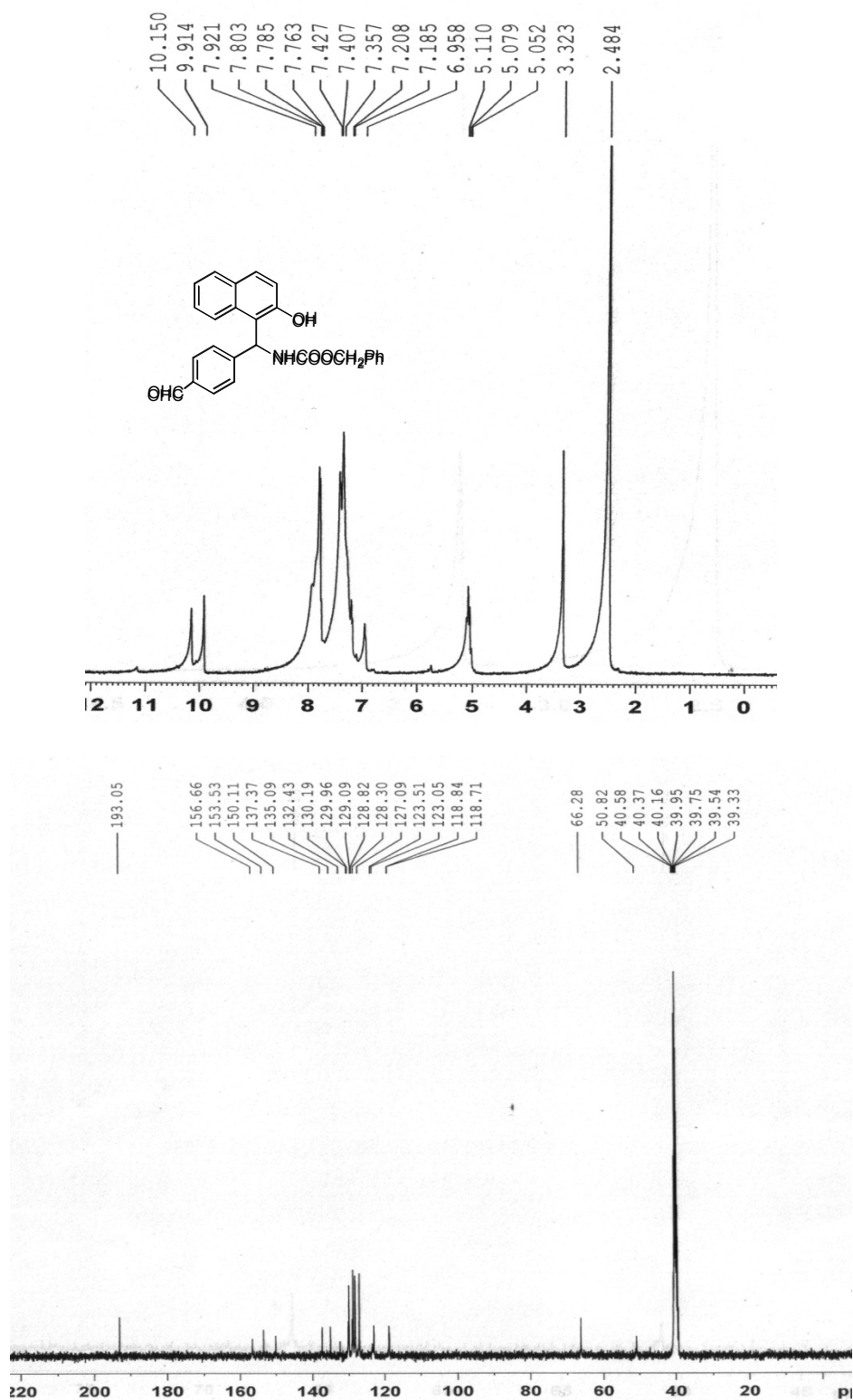
Benzyl (2-hydroxynaphthalen-1-yl)(2-methoxyphenyl)methylcarbamate 13a.



Benzyl (2-hydroxynaphthalen-1-yl)(4-methoxyphenyl)methylcarbamate 14a.



Benzyl (4-formylphenyl)(2-hydroxynaphthalen-1-yl)methylcarbamate 1b.



Benzyl (1,4-phenylenebis((2-hydroxynaphthalen-1-yl)methylene)dicarbamate 2b.

