Supplementary Information

$CuNiP_2O_7/CuFe_2O_4$ as Magnetically Separable Solid Acid Nanocatalyst for Synthesis of 1-Amidoalkyl-2-naphthols

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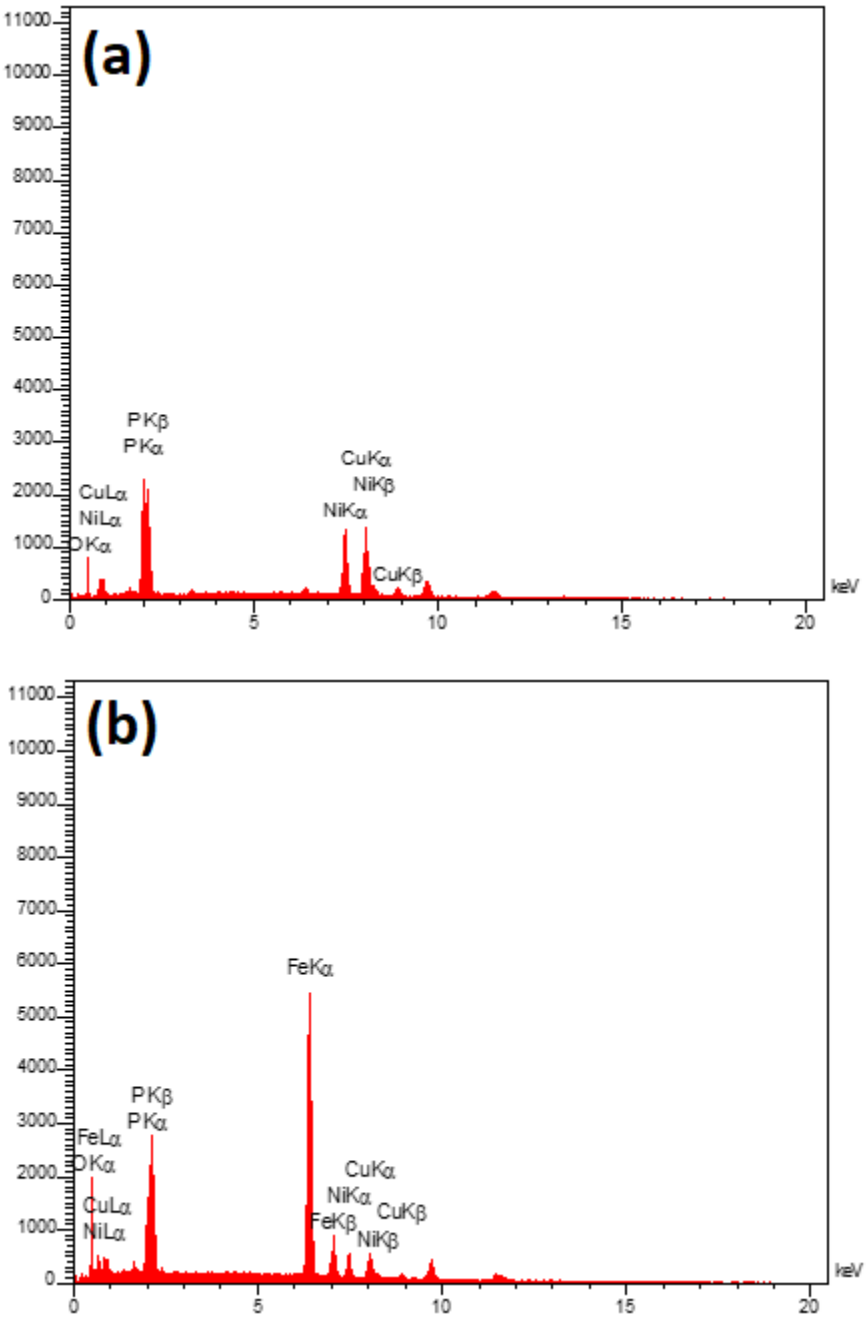


Fig. 1S. EDX spectra for the CNPO (a) and CNPO/CFO (b) samples.

Table S1a. Optimization of the temperature and reaction time.

Temperature (°C)	Time (min)	Yield (%)
70	60	40
80	45	62
90	**10**	**97**
100	10	97

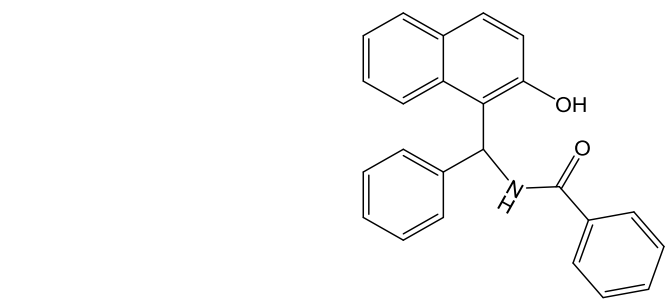
Table S1b. Optimization of the solvent under the constant temperature of 90 °C.

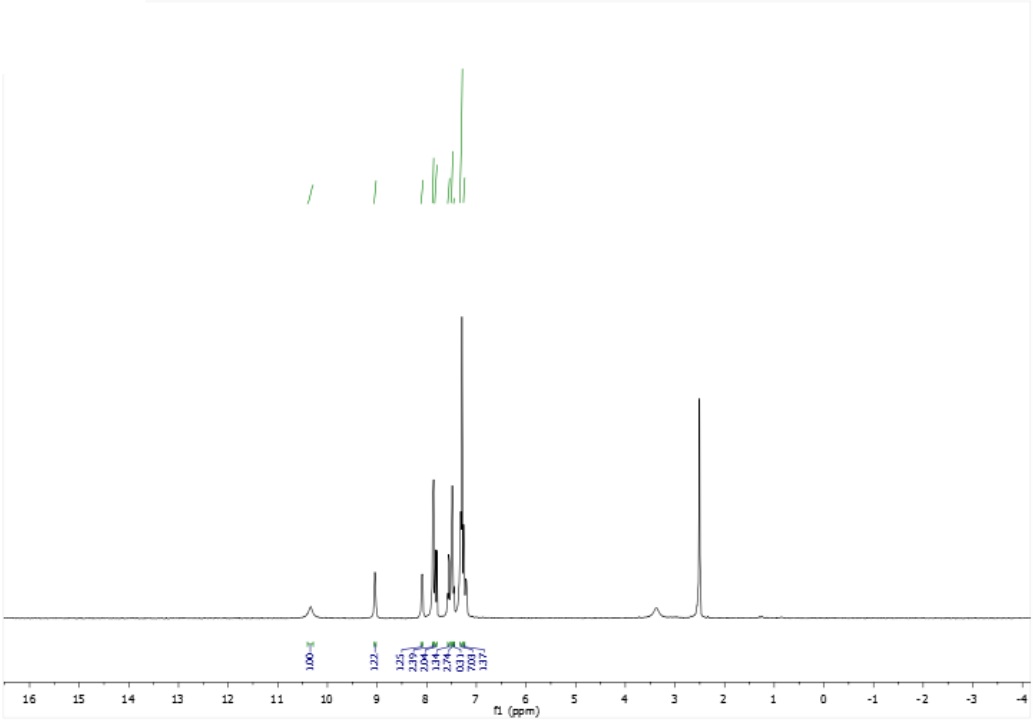
Solvent	Time (min)	Yield (%)
ethanol	160	64
Ethanol and water	160	-
methanol	160	25
Solvent free	**10**	**97**

Table S1c. Optimization of the catalyst amount under the constant temperature of 90 °C, reaction time of 10 min, and solvent free condition.

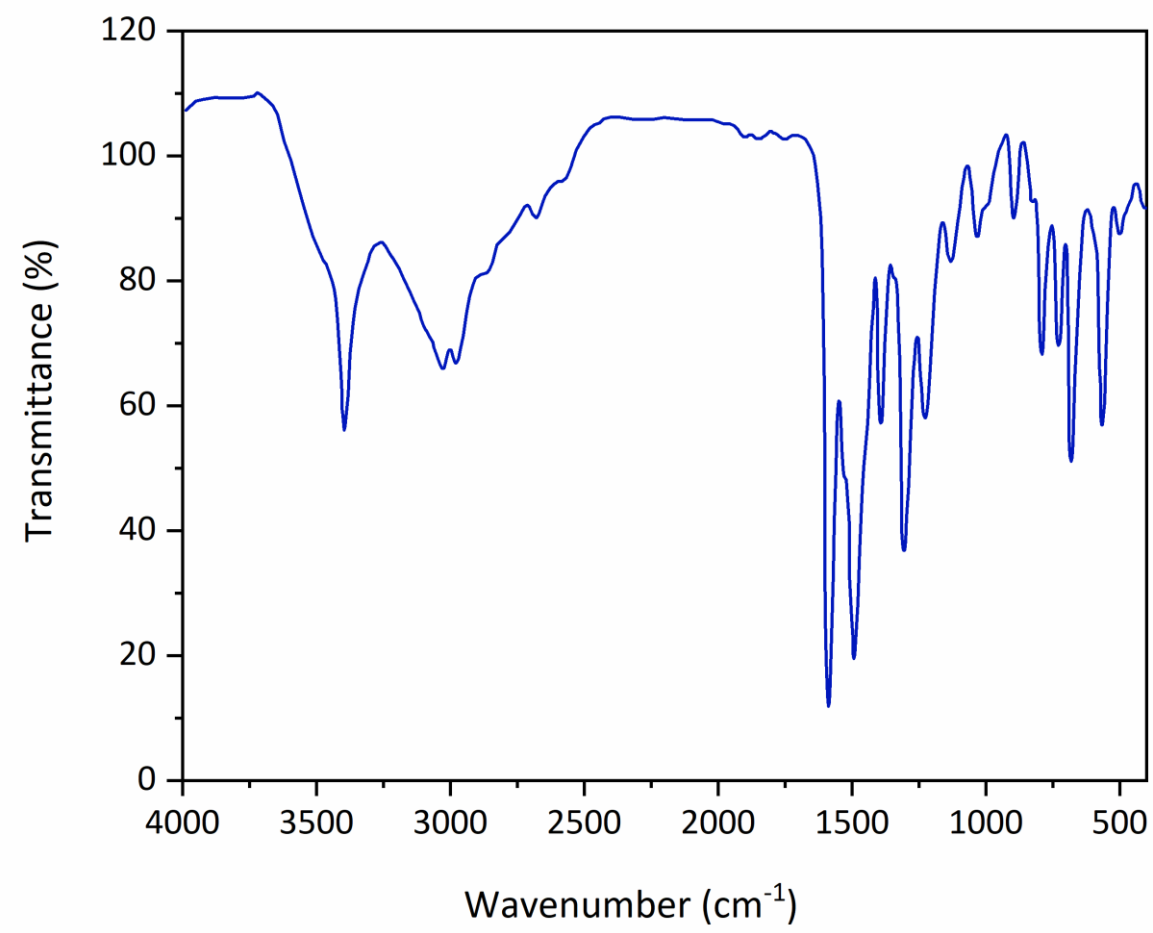
Catalyst amount (g)	Time (min)	Temperature (°C)	Yield (%)
0.01	10	90	53
0.02	10	90	55
0.03	10	99	67
0.04	**10**	**90**	**97**
0.05	10	90	97

Table 1 (entry 1): N-[(2-Hydroxynaphthalene-1-yl)-(phenyl)-methyl]-benzamide





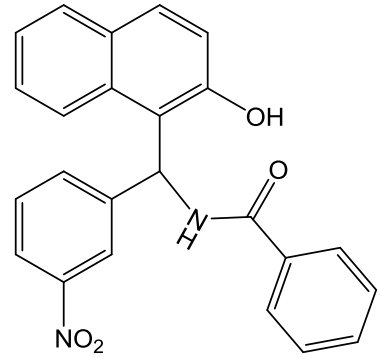
^{1}H NMR (400 MHz, DMSO-d_6) δ 10.34 (s, 1H), 9.03 (d, J = 8.4 Hz, 1H), 8.09 (d, J = 8.7 Hz, 1H), 7.87 (d, J = 7.7 Hz, 2H), 7.84 – 7.79 (m, 2H), 7.56 (t, J = 7.2 Hz, 1H), 7.49 (d, J = 1.6 Hz, 3H), 7.33 – 7.28 (m, 7H), 7.25 (d, J = 8.8 Hz, 1H).

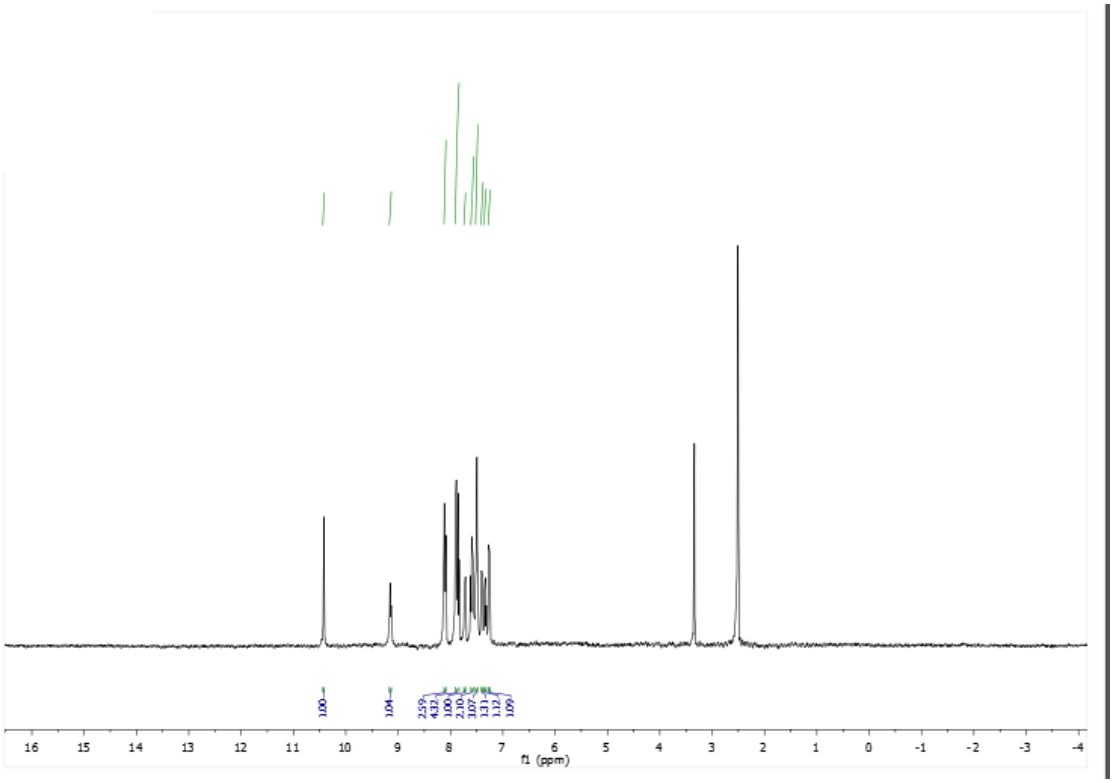


IR (KBr), ν cm^{-1}: 701, 753, 822, 1435, 1531, 1627, 3067, 3041.

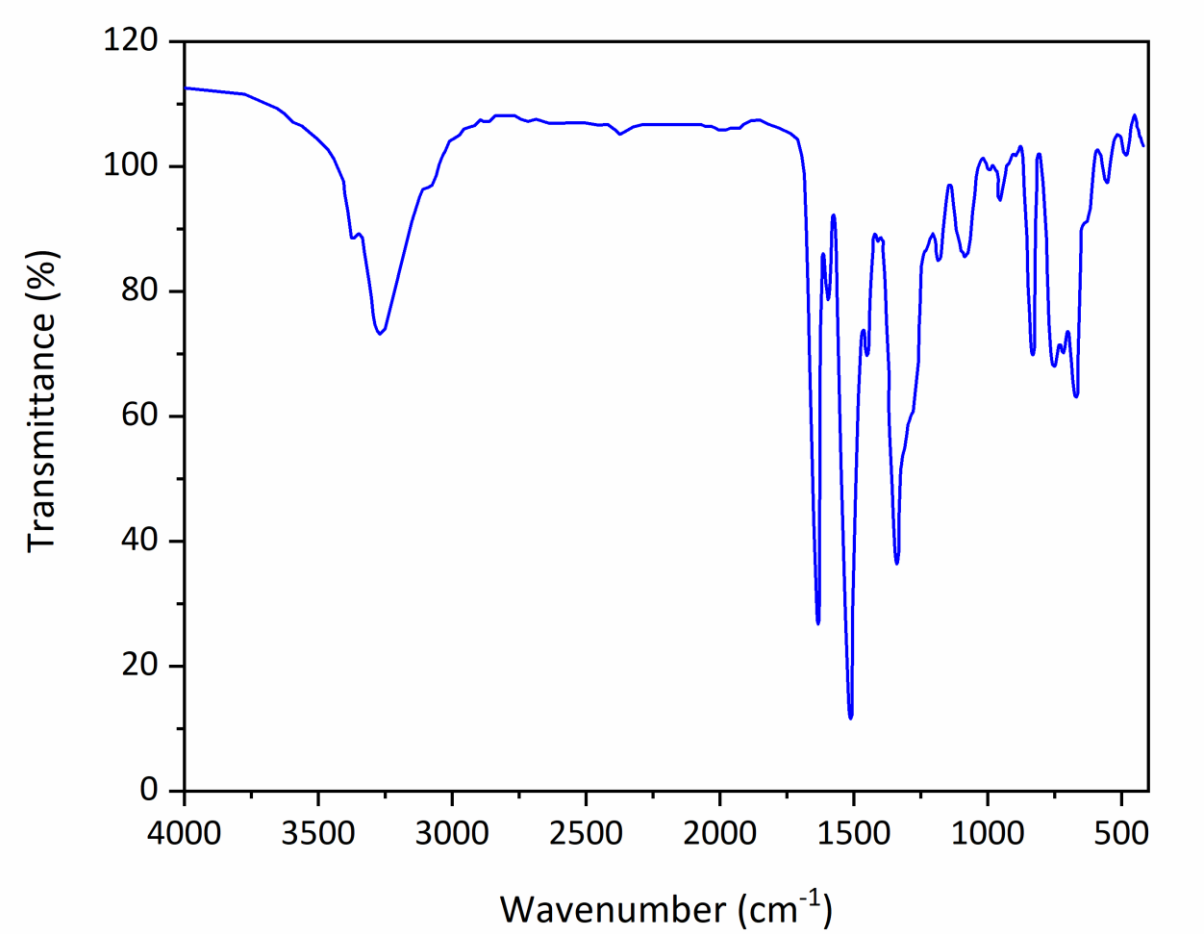
Wavenumber (cm^{-1})	Assignment
701	CH
753	CH
822	CH
1435	C=C
1627	C=O
3067	NH
3041	OH

Table 1 (entry 2): N-[(2-Hydroxynaphthalene-1-yl)- (3-nitrophenyl)-methyl]-benzamide





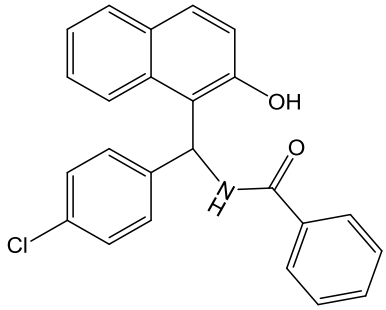
^{1}H NMR (400 MHz, DMSO-d_6) δ 10.42 (s, 1H), 9.15 (d, J = 8.0 Hz, 1H), 8.12 – 8.09 (m, 3H), 7.91 – 7.83 (m, 4H), 7.72 (d, J = 7.9 Hz, 1H), 7.57 (dd, J = 10.0, 6.9 Hz, 2H), 7.52 – 7.48 (m, 3H), 7.40 (d, J = 8.1 Hz, 1H), 7.34 (d, J = 7.4 Hz, 1H), 7.26 (d, J = 8.8 Hz, 1H).

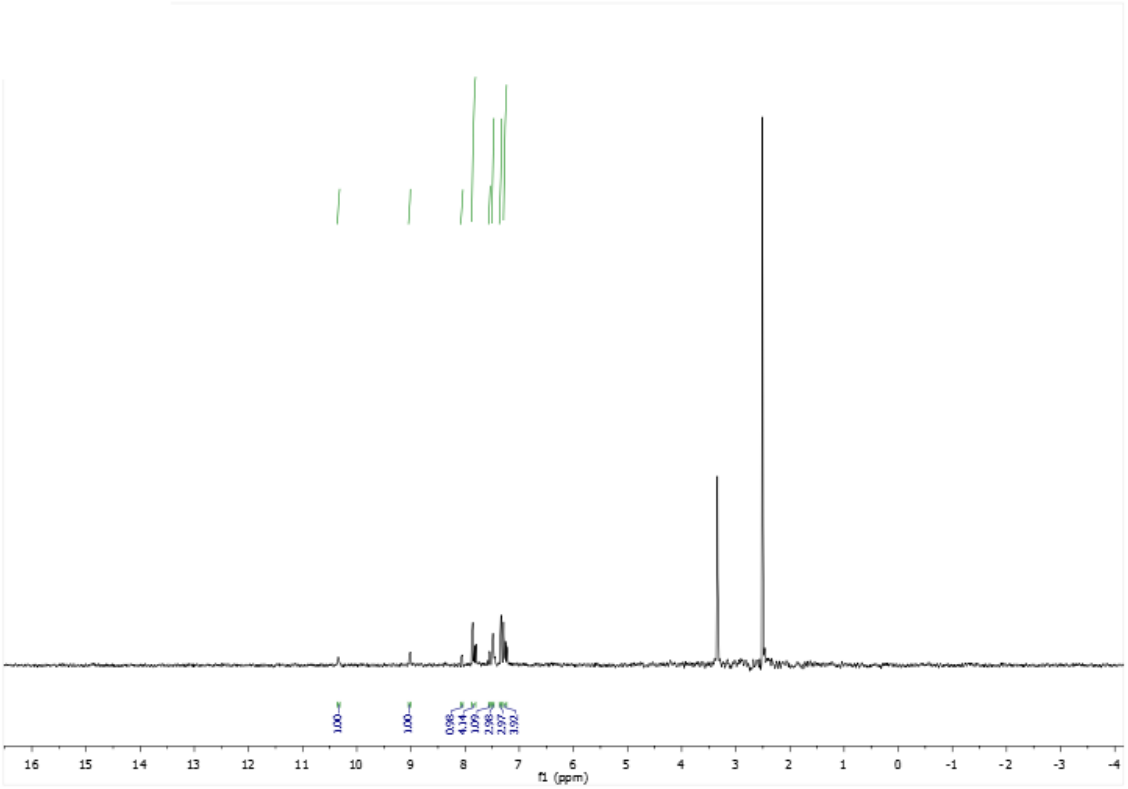


IR (KBr), ν cm^{-1}: 653, 732, 812, 1280, 1341, 1428, 1520, 1631, 3270, 3420.

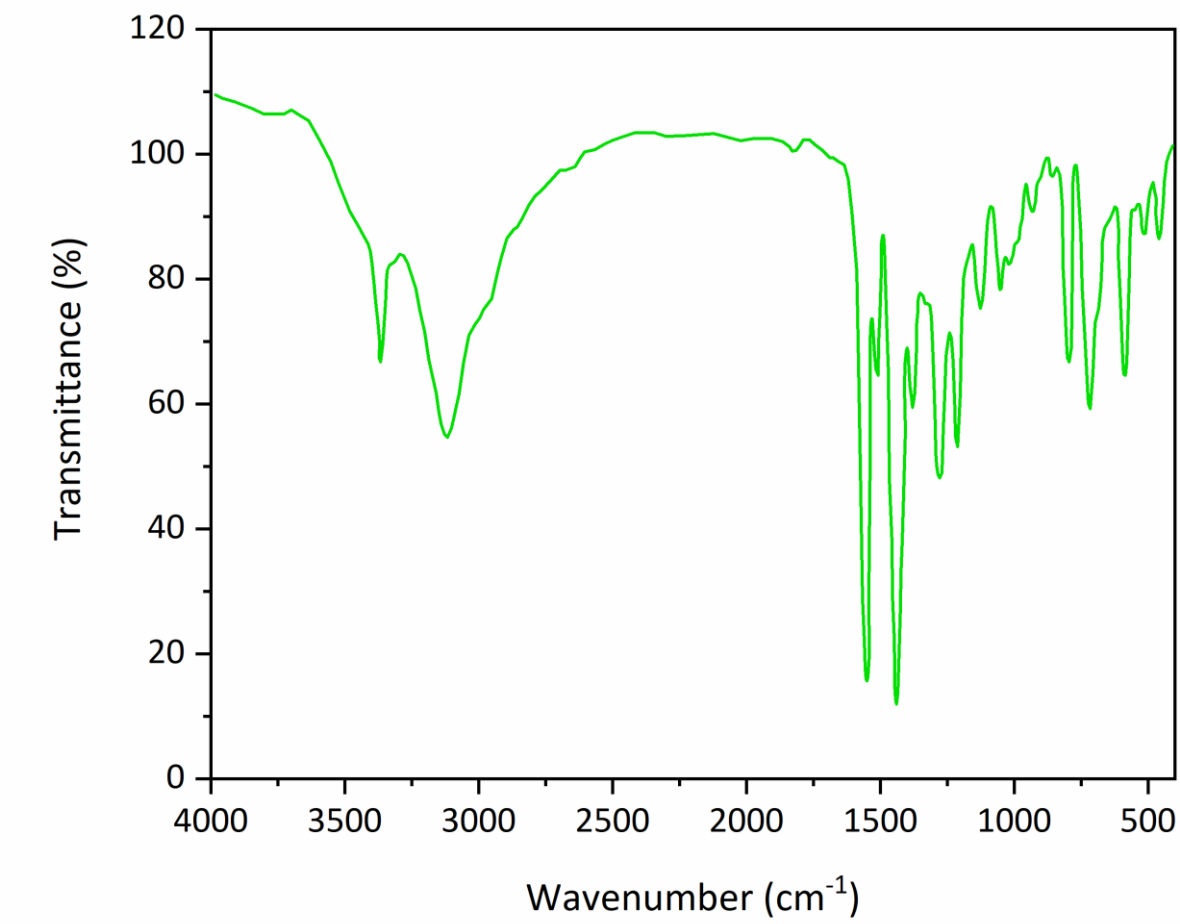
Wavenumber (cm^{-1})	Assignment
732	CH
812	CH
1280	C=C
1341	C=C
1428	C=C
1520	NO_2
1631	C=O
3270	OH
3420	NH

Table 1 (entry 3): N-[(2-Hydroxynaphthalene-1-yl)-(4-chlorophenyl)-methyl]-benzamide





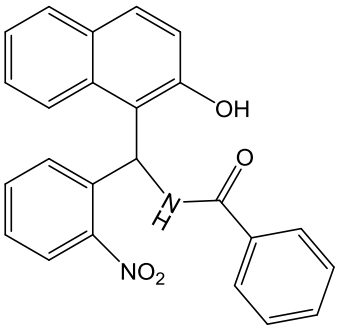
^{1}H NMR (400 MHz, DMSO-d_6) δ 10.34 (s, 1H), 9.02 (d, J = 8.5 Hz, 1H), 8.06 (d, J = 8.6 Hz, 1H), 7.84 (d, J = 13.9 Hz, 4H), 7.55 (d, J = 7.1 Hz, 1H), 7.49 (t, J = 7.2 Hz, 3H), 7.35 (d, J = 8.1 Hz, 3H), 7.29 – 7.23 (m, 4H).

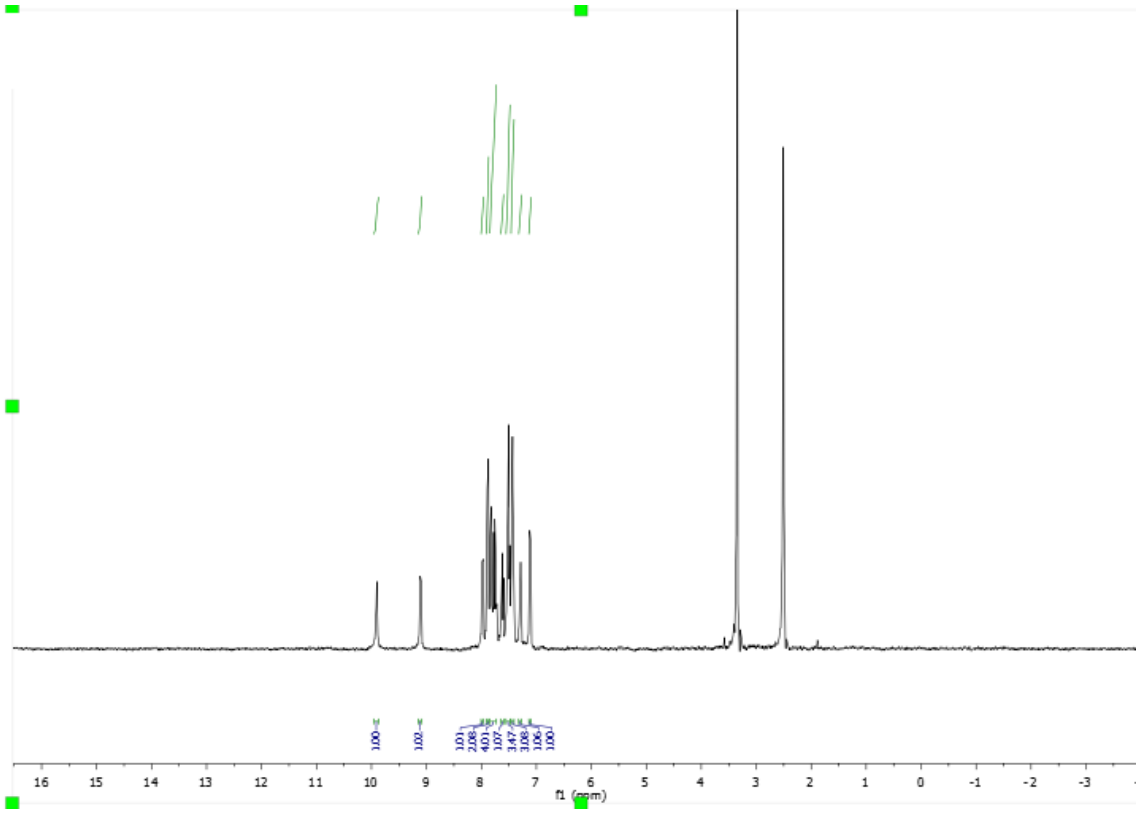


IR (KBr), ν cm^{-1}: 726, 812, 873, 1264, 1336, 1438, 1512, 1627, 3115, 3389.

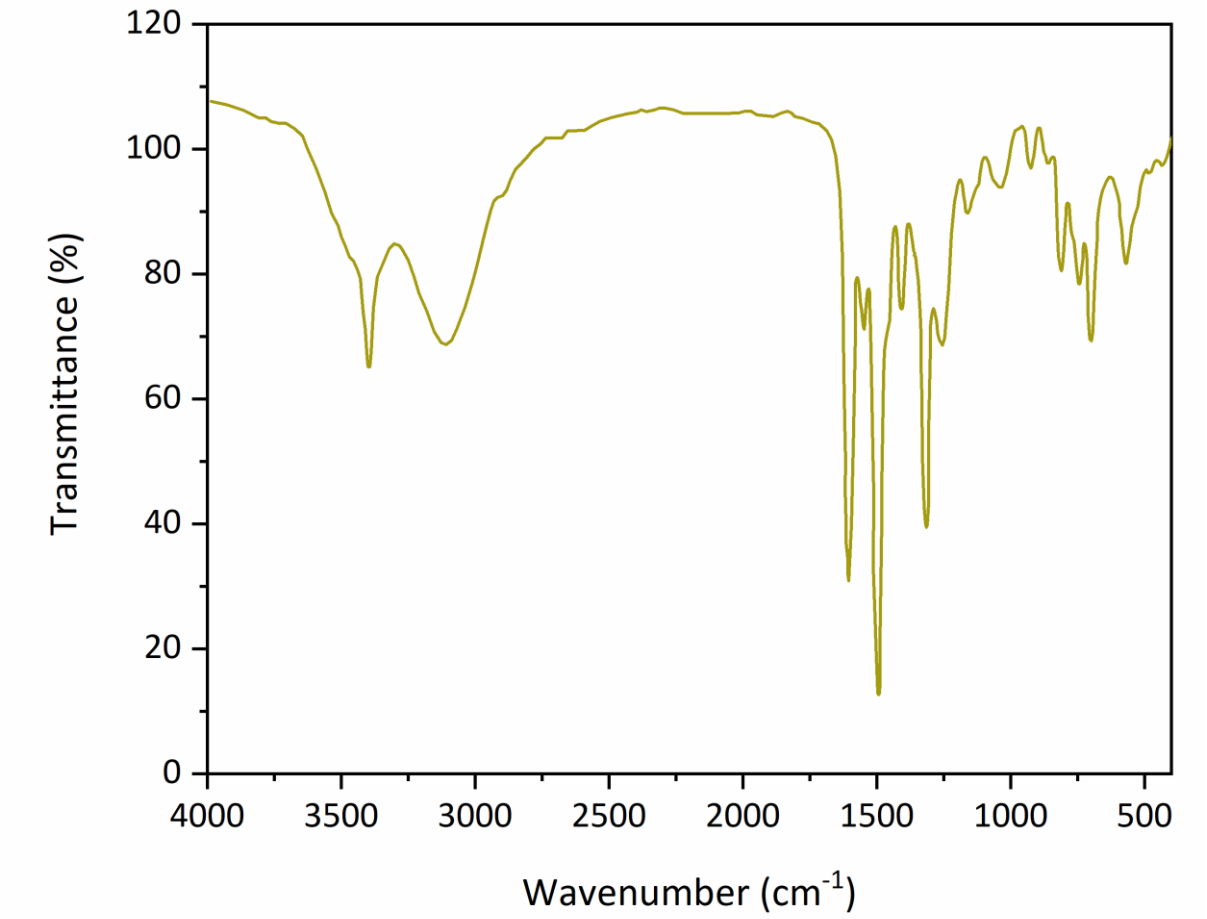
Wavenumber (cm^{-1})	Assignment
726	CH
812	CH
1627	C=O
3115	OH
3389	NH

Table 1 (entry 4): N-[(2-Hydroxynaphthalene-1-yl)-(2-nitrophenyl)-methyl]-benzamide





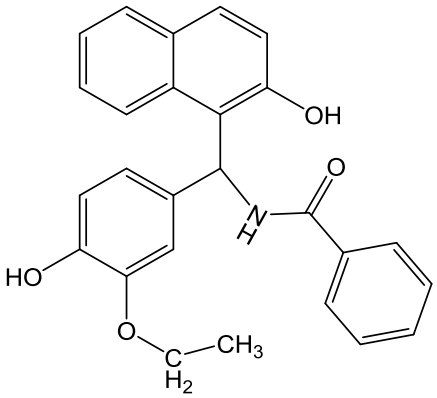
^{1}H NMR (400 MHz, DMSO-d_6) δ 9.90 (s, 1H), 9.10 (d, J = 7.7 Hz, 1H), 7.98 (d, J = 8.8 Hz, 1H), 7.91 – 7.87 (m, 2H), 7.84 – 7.73 (m, 4H), 7.62 (t, J = 7.7 Hz, 1H), 7.51 (dt, J = 9.9, 6.7 Hz, 3H), 7.46 – 7.40 (m, 3H), 7.29 (t, J = 7.5 Hz, 1H), 7.11 (d, J = 8.6 Hz, 1H).

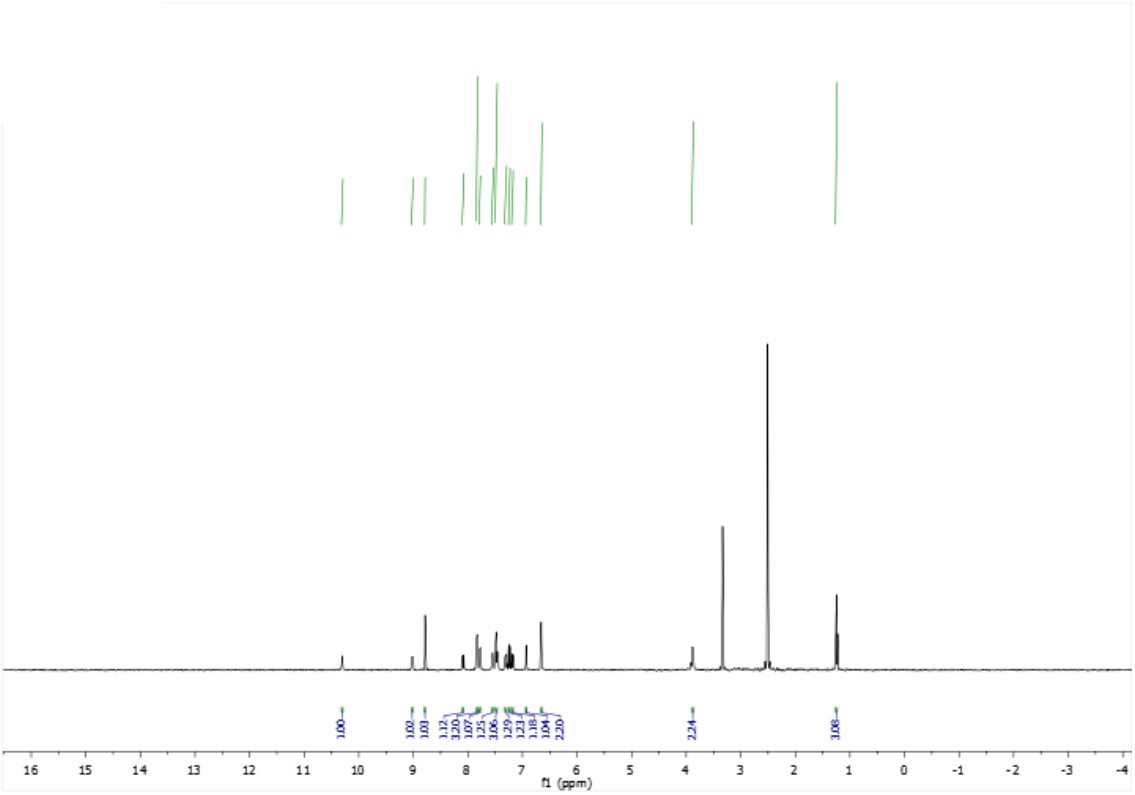


IR (KBr), ν cm^{-1}: 706, 751, 821, 1280, 1344, 1435, 1526, 1635, 3139, 3421.

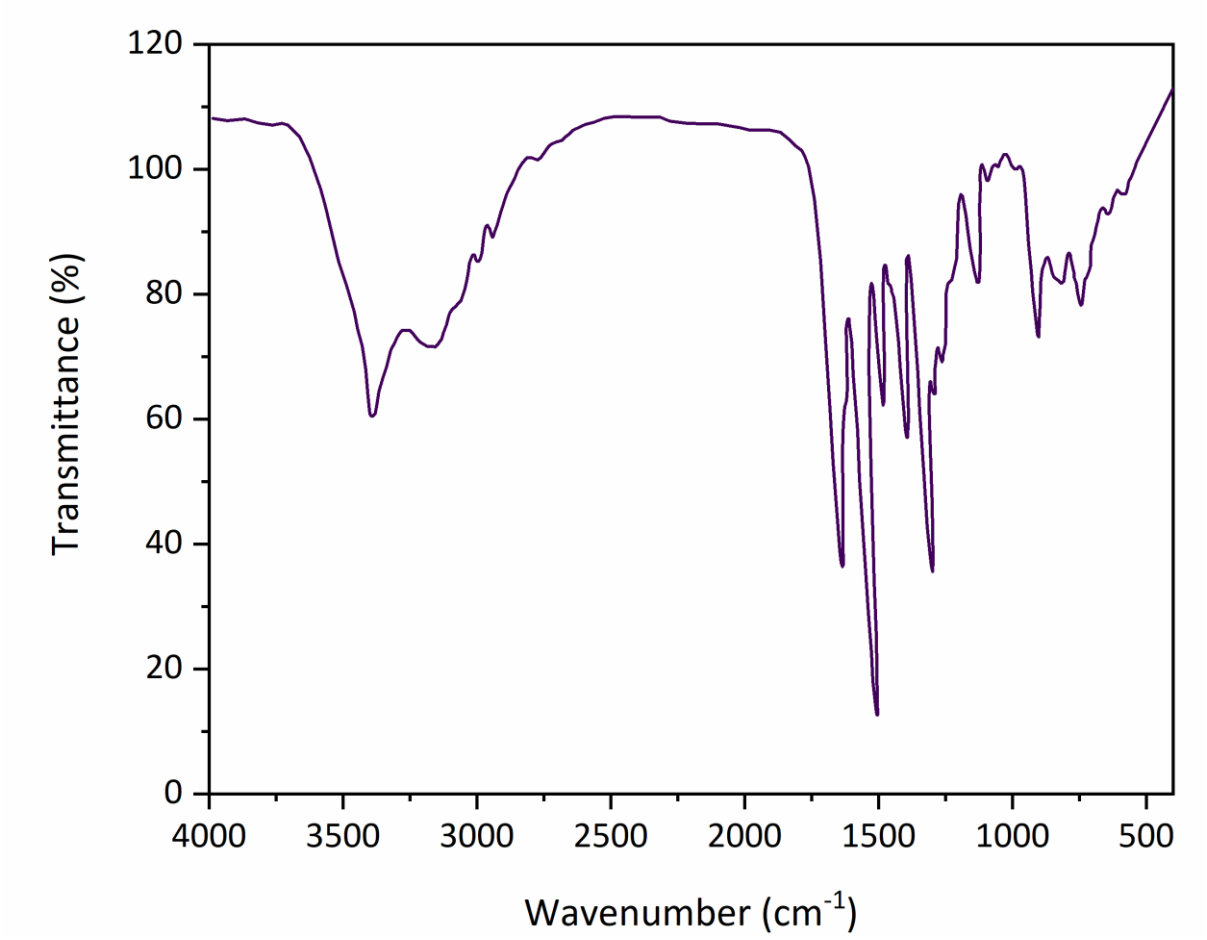
Wavenumber (cm^{-1})	Assignment
751	CH
821	CH
1344	NO_2
1435	NO_2
1526	NO_2
1635	C=O
3139	OH
3421	NH

Table 1 (entry 5): N-[(2-Hydroxynaphthalene-1-yl)-(3-ethoxy-4-hydroxyphenyl)-methyl]-benzamide





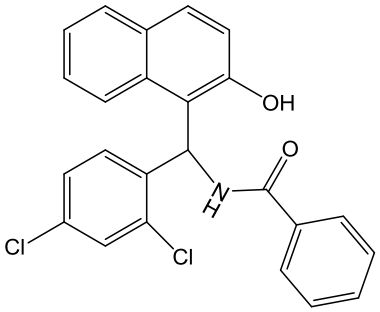
^{1}H NMR (400 MHz, DMSO-d_6) δ 10.30 (s, 1H), 9.02 (d, J = 8.6 Hz, 1H), 8.78 (s, 1H), 8.09 (d, J = 8.7 Hz, 1H), 7.85 – 7.82 (m, 3H), 7.78 (d, J = 8.9 Hz, 1H), 7.54 (d, J = 7.2 Hz, 1H), 7.48 (t, J = 7.5 Hz, 3H), 7.31 (t, J = 7.5 Hz, 1H), 7.24 (d, J = 8.9 Hz, 1H), 7.18 (d, J = 8.6 Hz, 1H), 6.93 (s, 1H), 6.65 (d, J = 2.8 Hz, 2H), 3.90 – 3.86 (m, 2H), 1.25 (d, J = 6.9 Hz, 3H).

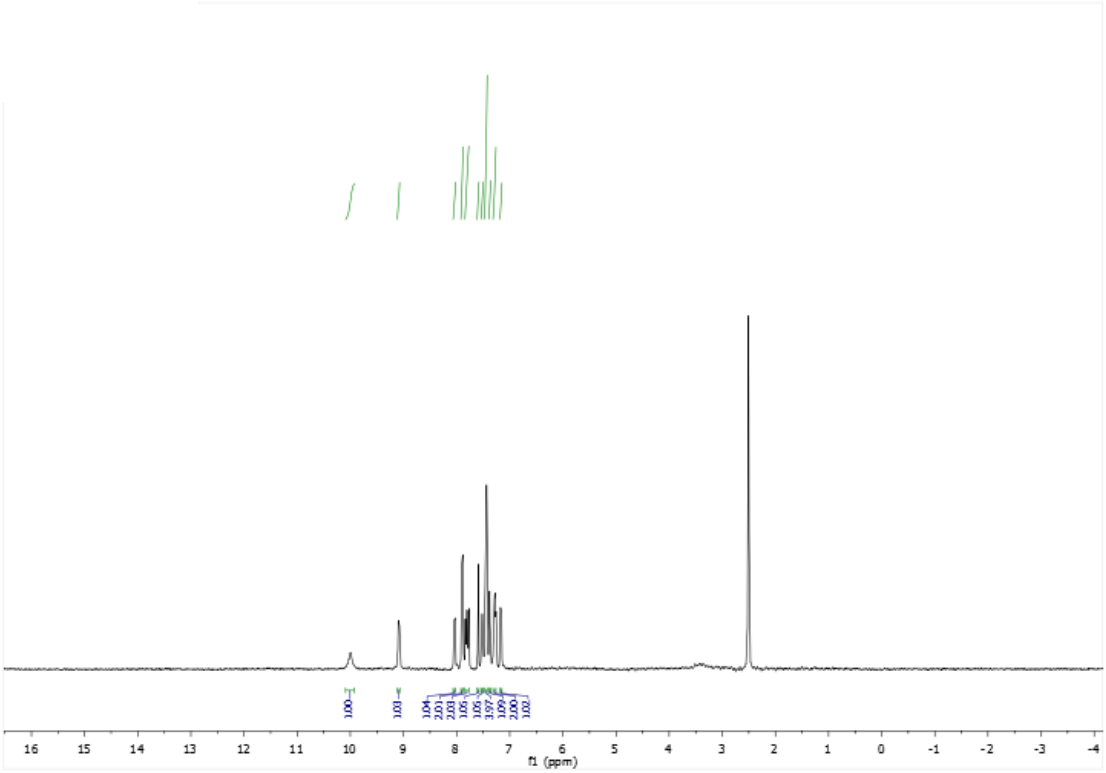


IR (KBr), ν cm^{-1}: 714, 814, 846, 1278, 1348, 1436, 1518, 1621, 3158, 3394.

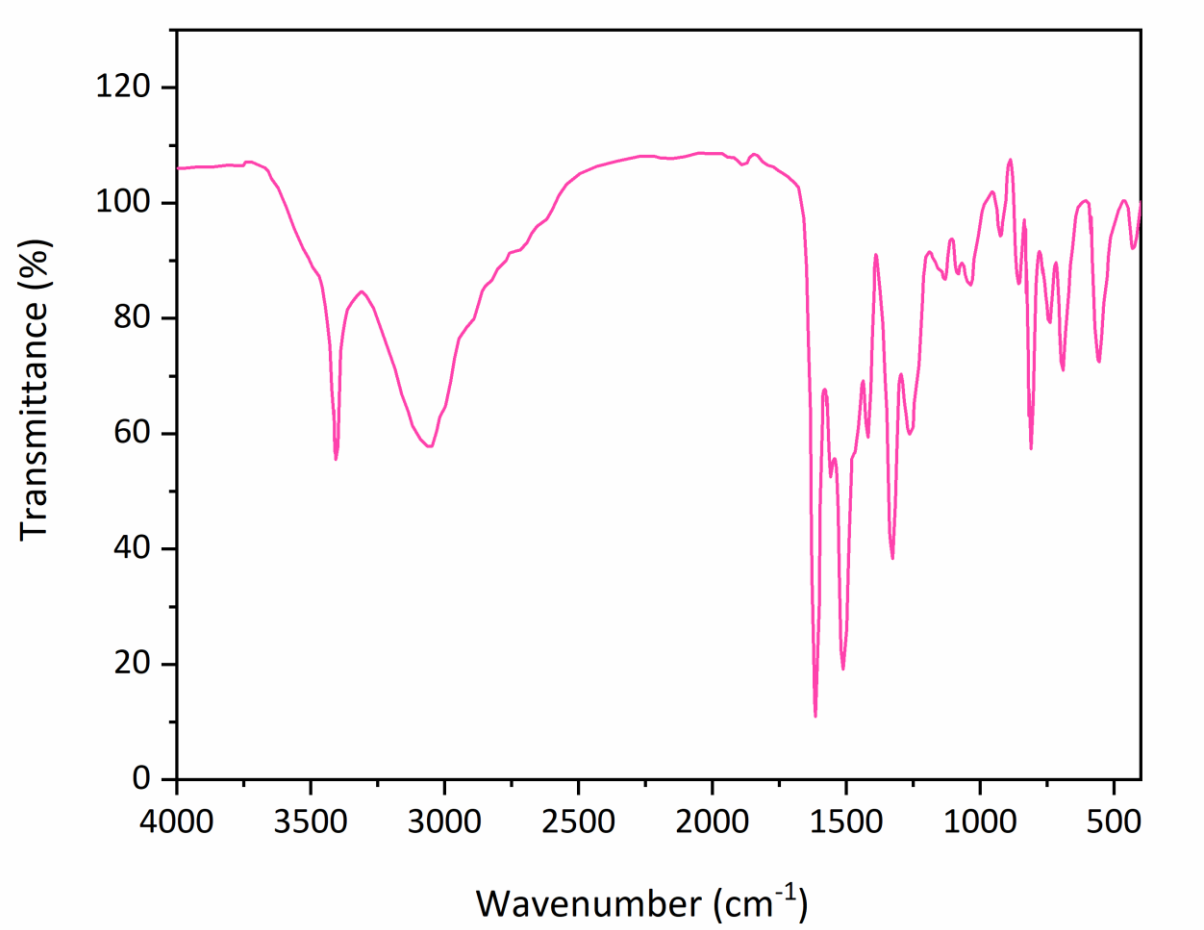
Wavenumber (cm^{-1})	Assignment
714	CH
814	CH
1348	C=C
1436	C=C
1518	C=O
1635	C=O
3158	OH
3394	NH

Table 1 (entry 6): N-[(2-Hydroxynaphthalene-1-yl)-(2,4-di-chlorophenyl)-methyl]-benzamide





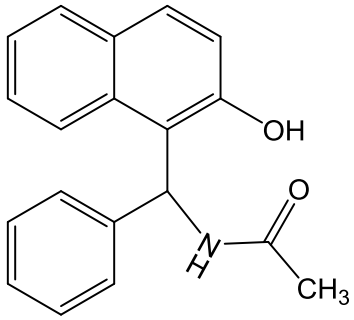
^{1}H NMR (400 MHz, DMSO-d_6) δ 10.30 (s, 1H), 9.02 (d, J = 8.6 Hz, 1H), 8.78 (s, 1H), 8.09 (d, J = 8.7 Hz, 1H), 7.85 – 7.82 (m, 3H), 7.78 (d, J = 8.9 Hz, 1H), 7.54 (d, J = 7.2 Hz, 1H), 7.48 (t, J = 7.5 Hz, 3H), 7.31 (t, J = 7.5 Hz, 1H), 7.24 (d, J = 8.9 Hz, 1H), 7.18 (d, J = 8.6 Hz, 1H), 6.93 (s, 1H), 6.65 (d, J = 2.8 Hz, 2H), 3.90 – 3.86 (m, 2H), 1.25 (d, J = 6.9 Hz, 3H).

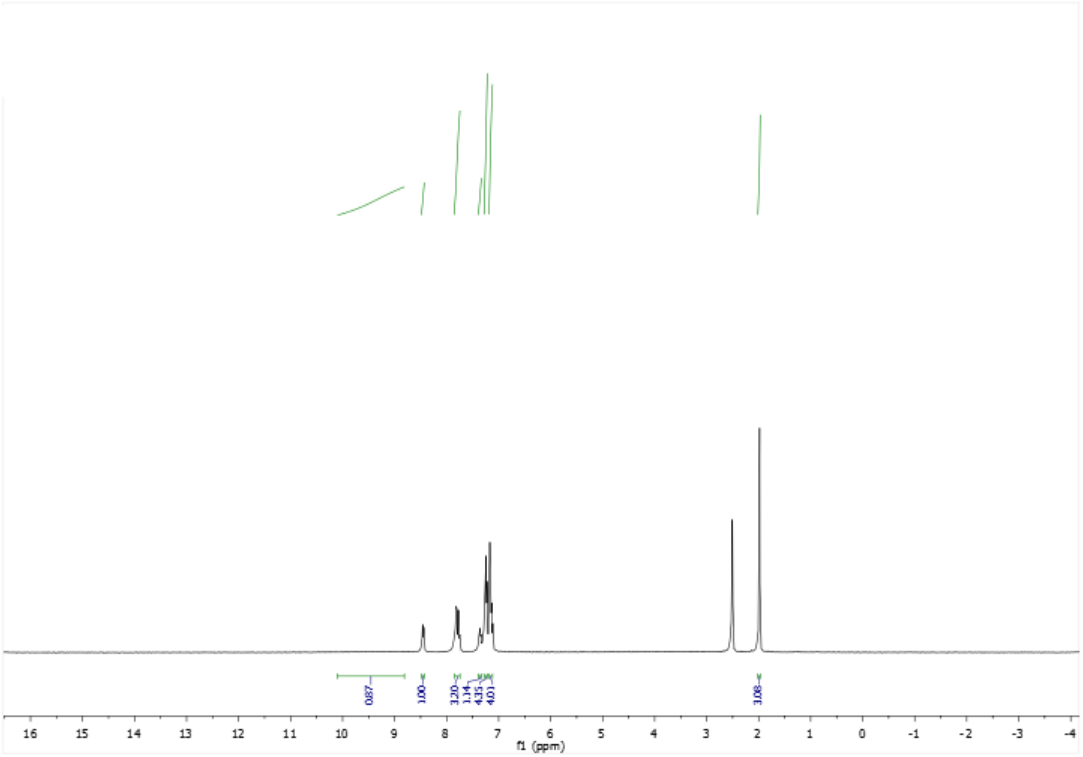


IR (KBr), ν cm^{-1}: 707, 754, 822, 1276, 1346, 1438, 1530, 1682, 3072, 3421.

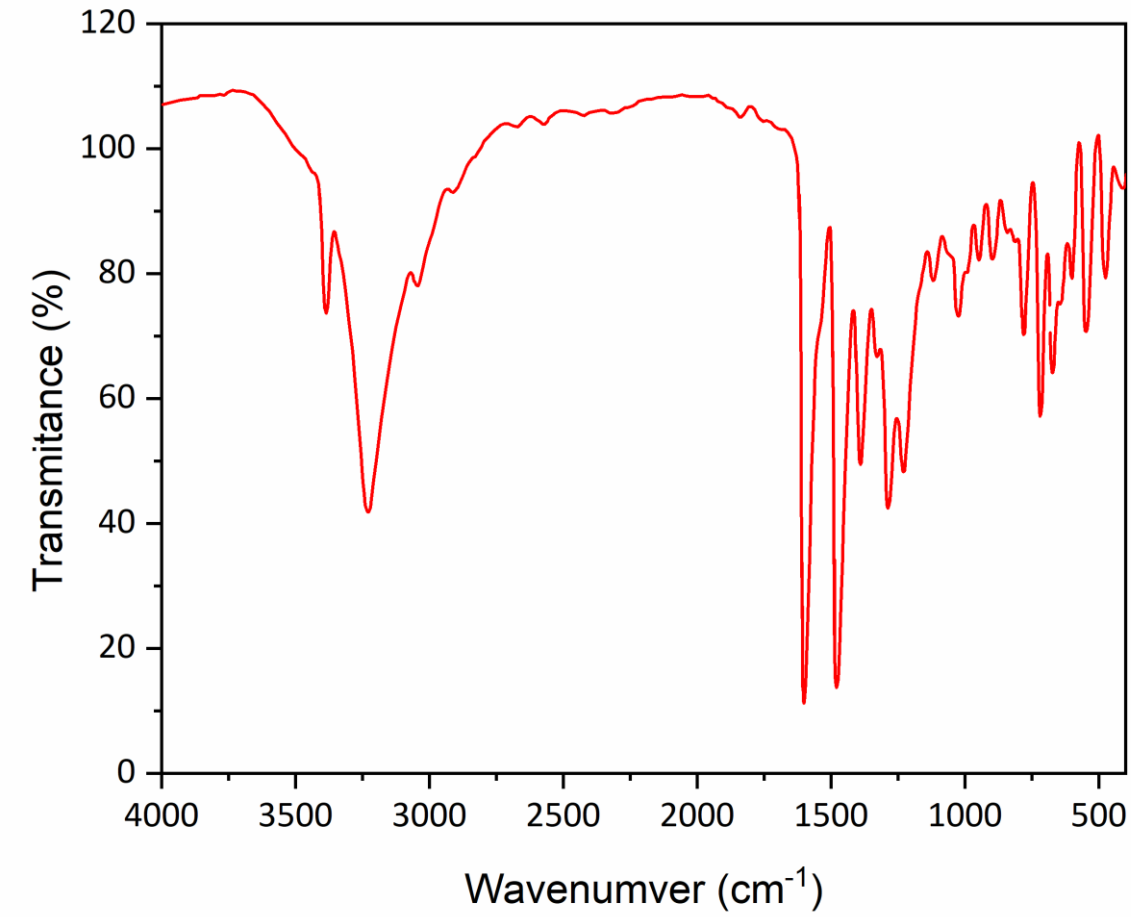
Wavenumber (cm^{-1})	Assignment
707	CH
754	CH
1438	C=C
1530	C=O
1682	C=O
3072	OH
3421	NH

Table 1 (entry 7): N-[(2-Hydroxynaphthalene-1-yl)-(phenyl)-methyl]-acetamide





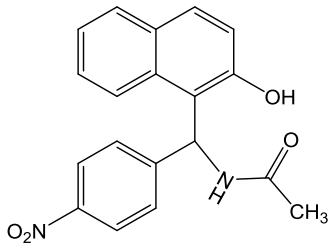
^{1}H NMR (400 MHz, DMSO-d_6) δ 9.4 (S ,1H),8.45 (d, J = 8.3 Hz, 1H), 7.78 (dd, J = 16.6, 8.5 Hz, 3H), 7.36 (t, J = 7.6 Hz, 1H), 7.25 (ddd, J = 11.2, 8.4, 4.7 Hz, 4H), 7.18 – 7.12 (m, 4H), 1.98 (s, 3H).

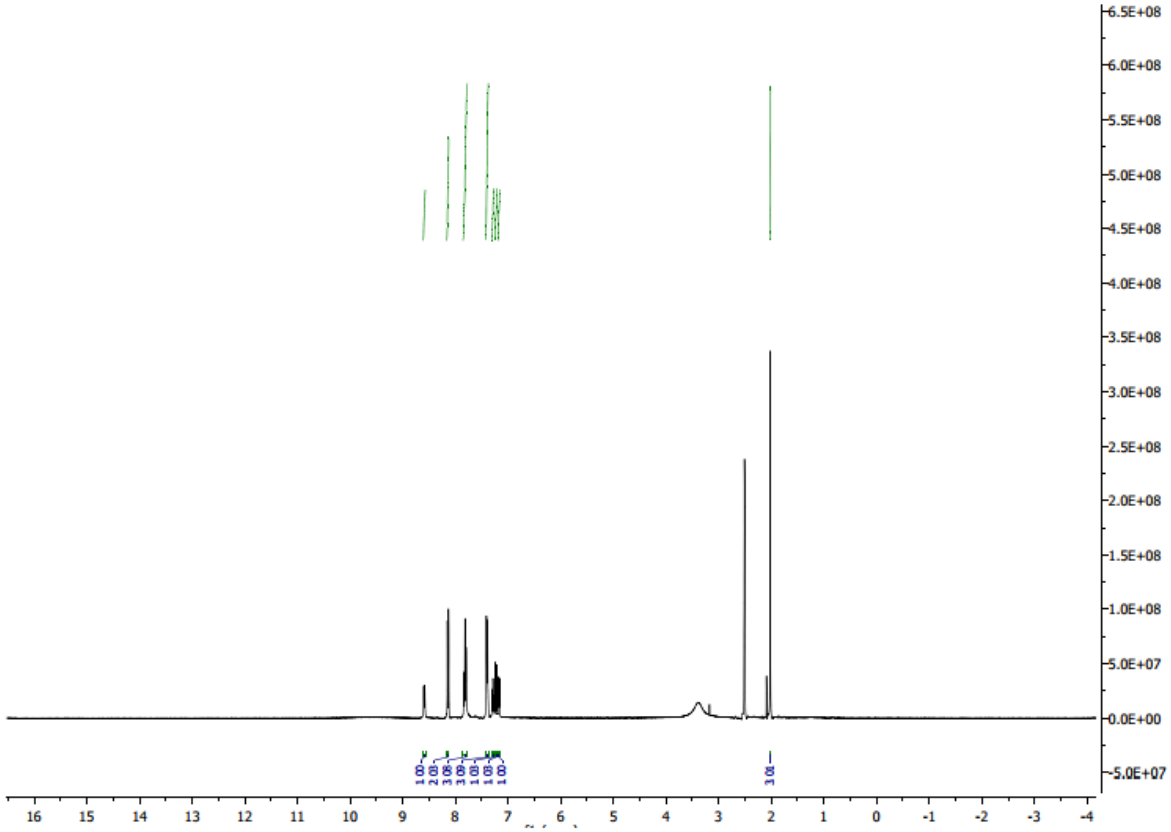


IR (KBr), ν cm^{-1}: 724, 808, 824, 1274, 1332, 1436, 1516, 1687, 3242, 3397.

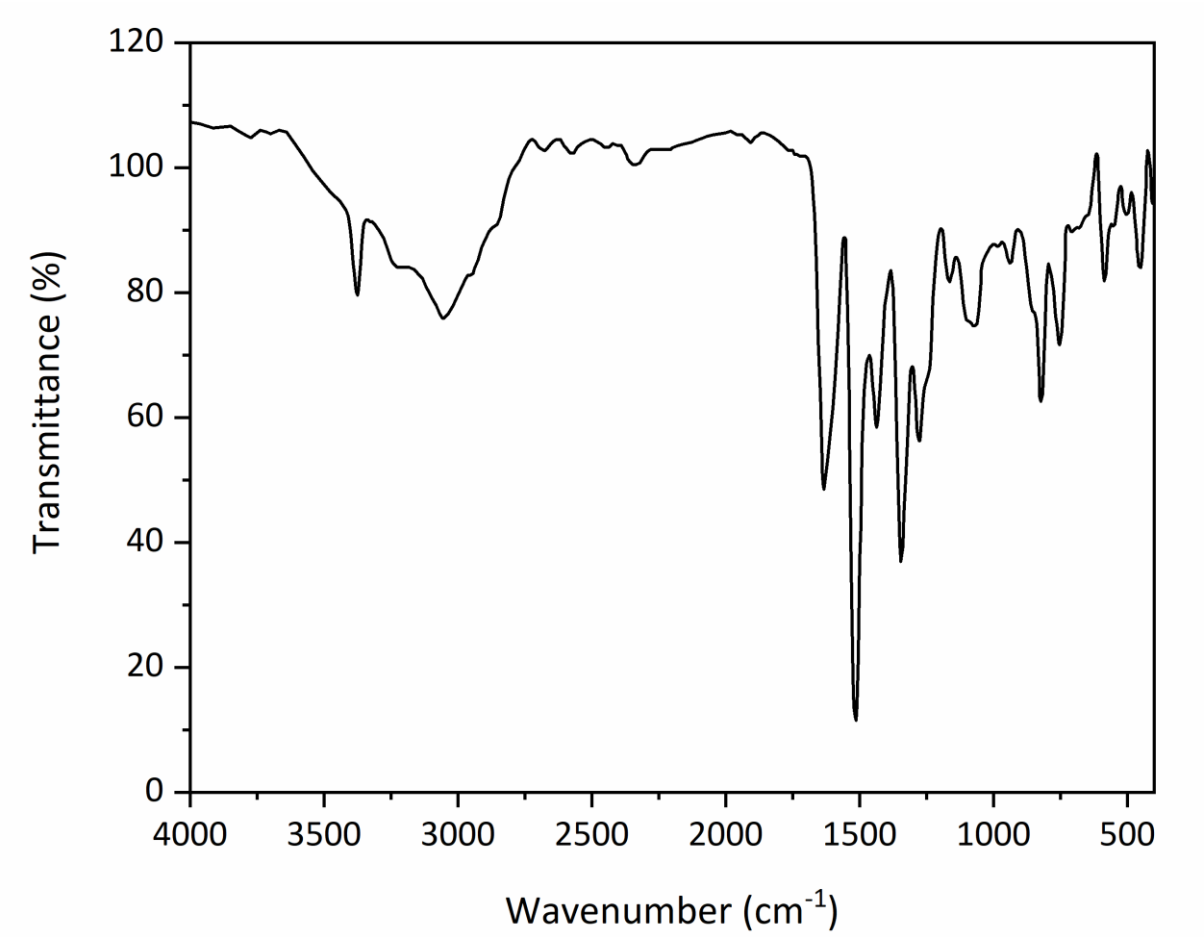
Wavenumber (cm^{-1})	Assignment
724	CH
808	CH
824	CH
1687	C=O
3242	OH
3397	NH

Table 1 (entry 8): N-[(2-Hydroxynaphthalene-1-yl)-(4-nitrophenyl)-methyl]-acetamide





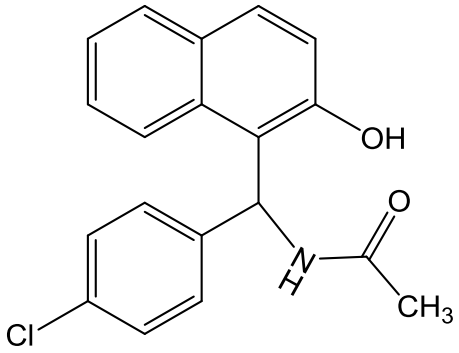
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.59 (d, J = 8.0 Hz, 1H), 8.14 (d, J = 8.6 Hz, 2H), 7.82 (t, J = 9.0 Hz, 3H), 7.40 (d, J = 8.4 Hz, 3H), 7.29 (t, J = 7.5 Hz, 3H), 7.23 (d, J = 8.9 Hz, 1H), 7.17 (d, J = 7.8 Hz, 1H), 2.02(S, 3H).

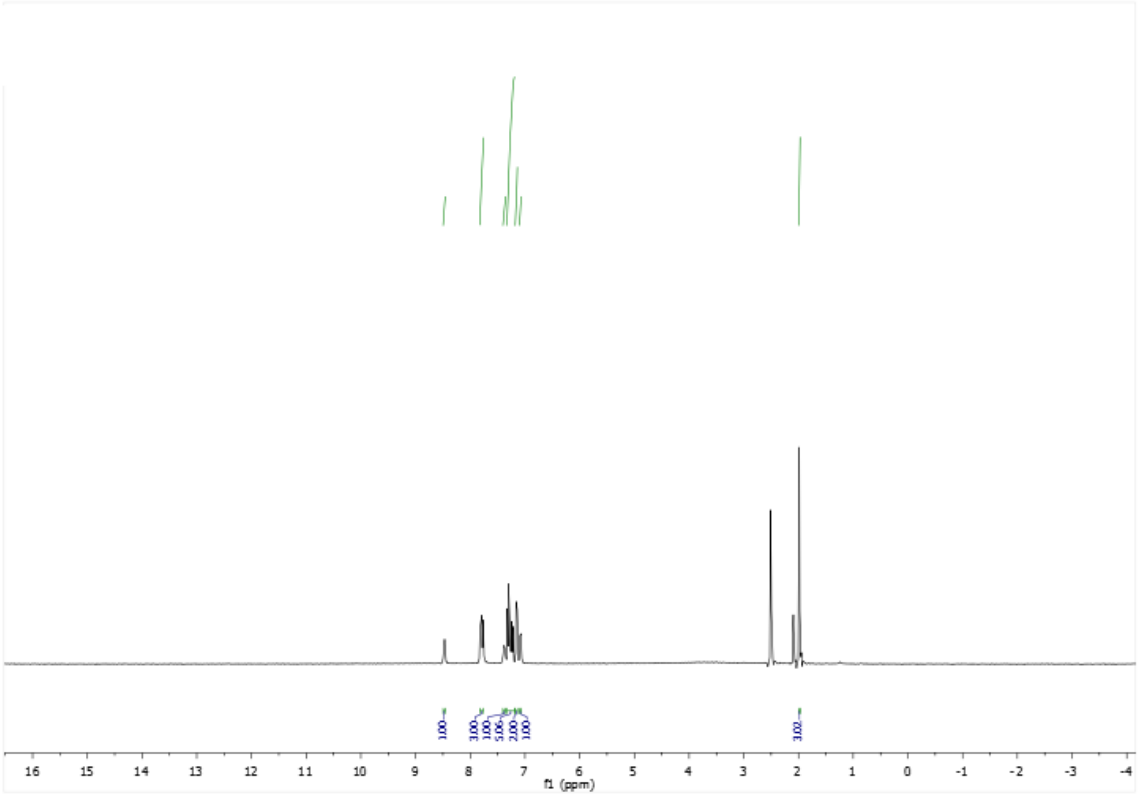


IR (KBr), ν cm^{-1}: 751, 821, 851, 1280, 1347, 1438, 1518, 1634, 3065, 3389.

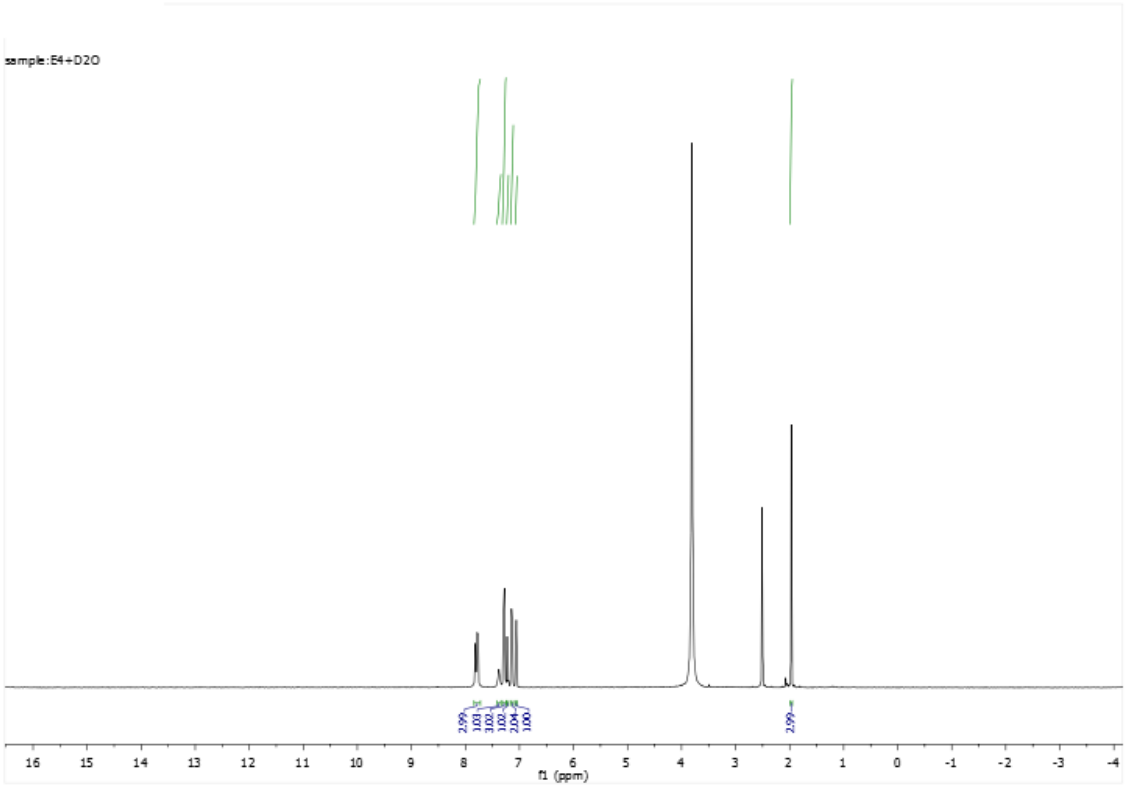
Wavenumber (cm^{-1})	Assignment
752	CH
821	CH
1518	NO_2
1634	C=O
3065	OH
3389	NH

Table 1 (entry 9): N-[(2-Hydroxynaphthalene-1-yl)-(4-chlorophenyl)-methyl]-acetamide

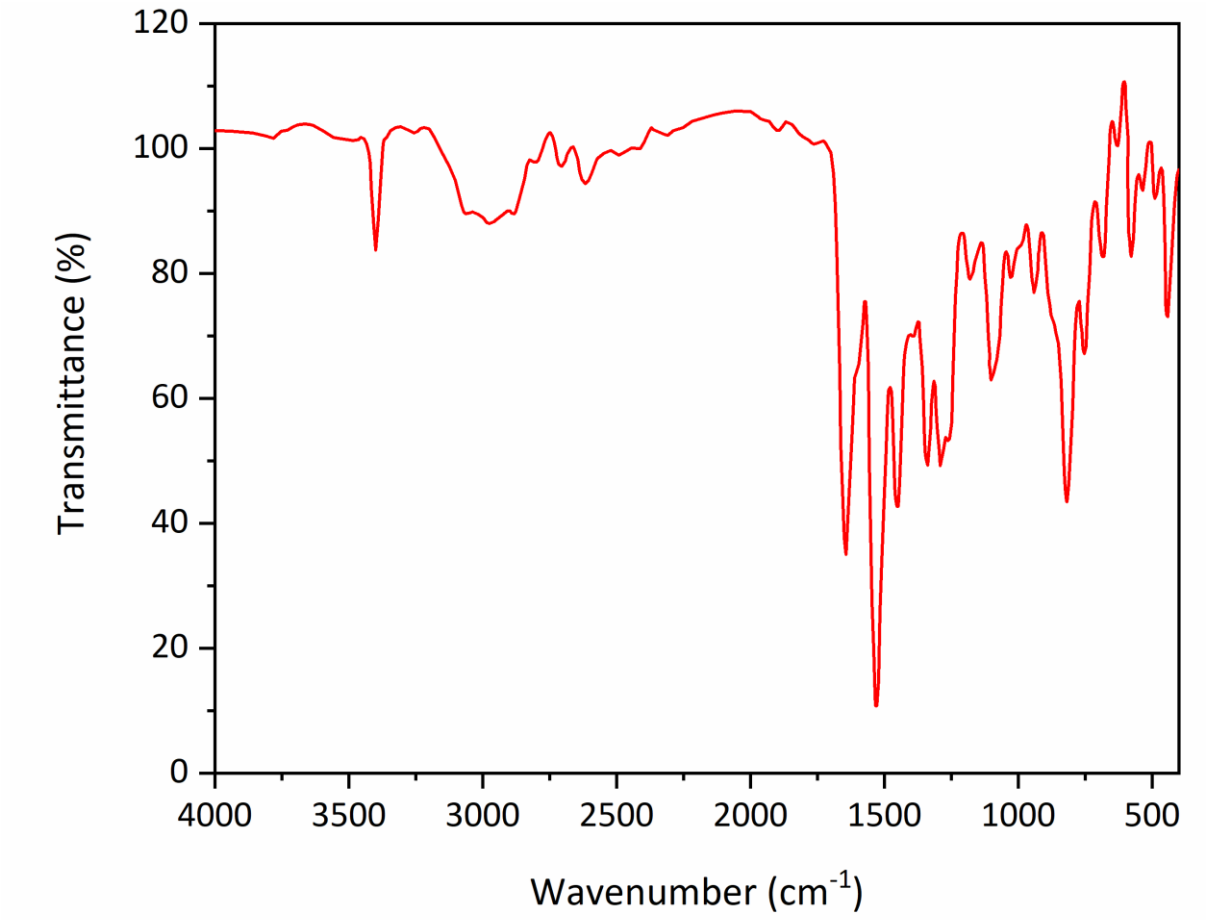




^{1}H NMR (400 MHz, DMSO-d_6) δ 8.48 (d, J = 8.2 Hz, 1H), 7.79 (dd, J = 14.7, 8.3 Hz, 3H), 7.38 (t, J = 7.6 Hz, 1H), 7.33 – 7.22 (m, 4H), 7.15 (d, J = 8.2 Hz, 2H), 7.08 (d, J = 8.2 Hz, 1H), 1.98 (s, 3H).



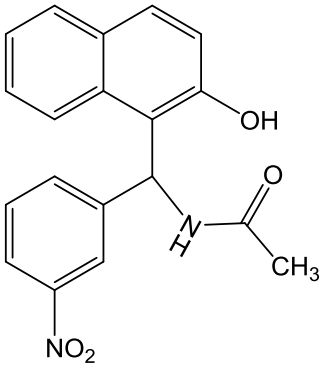
^{1}H NMR (400 MHz, DMSO-d_6 (D_2O changed)) δ 7.79 (dd, J = 13.1, 8.5 Hz, 3H), 7.38 (t, J = 7.4 Hz, 1H), 7.28 (dd, J = 7.6, 4.7 Hz, 3H), 7.22 (d, J = 8.9 Hz, 1H), 7.14 (d, J = 8.3 Hz, 2H), 7.06 (s, 1H), 1.97 (s, 3H).

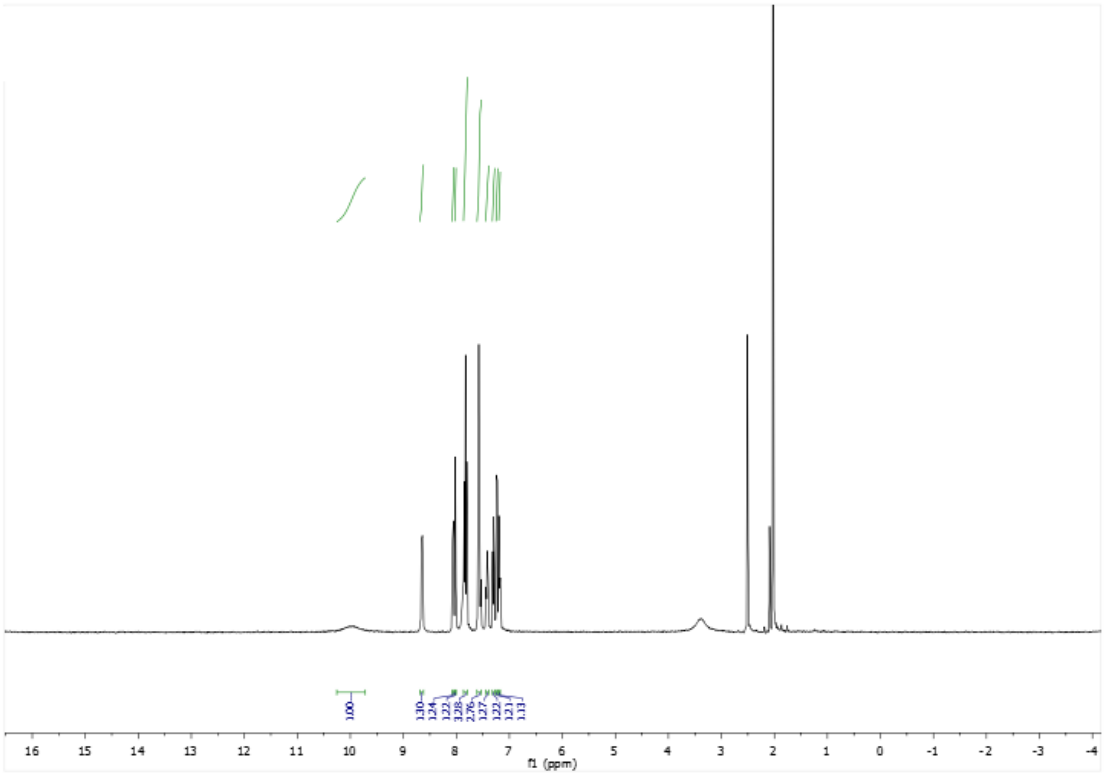


IR (KBr), ν cm^{-1}: 750, 814, 850, 1276, 1326, 1437, 1516, 1627, 3115, 3389.

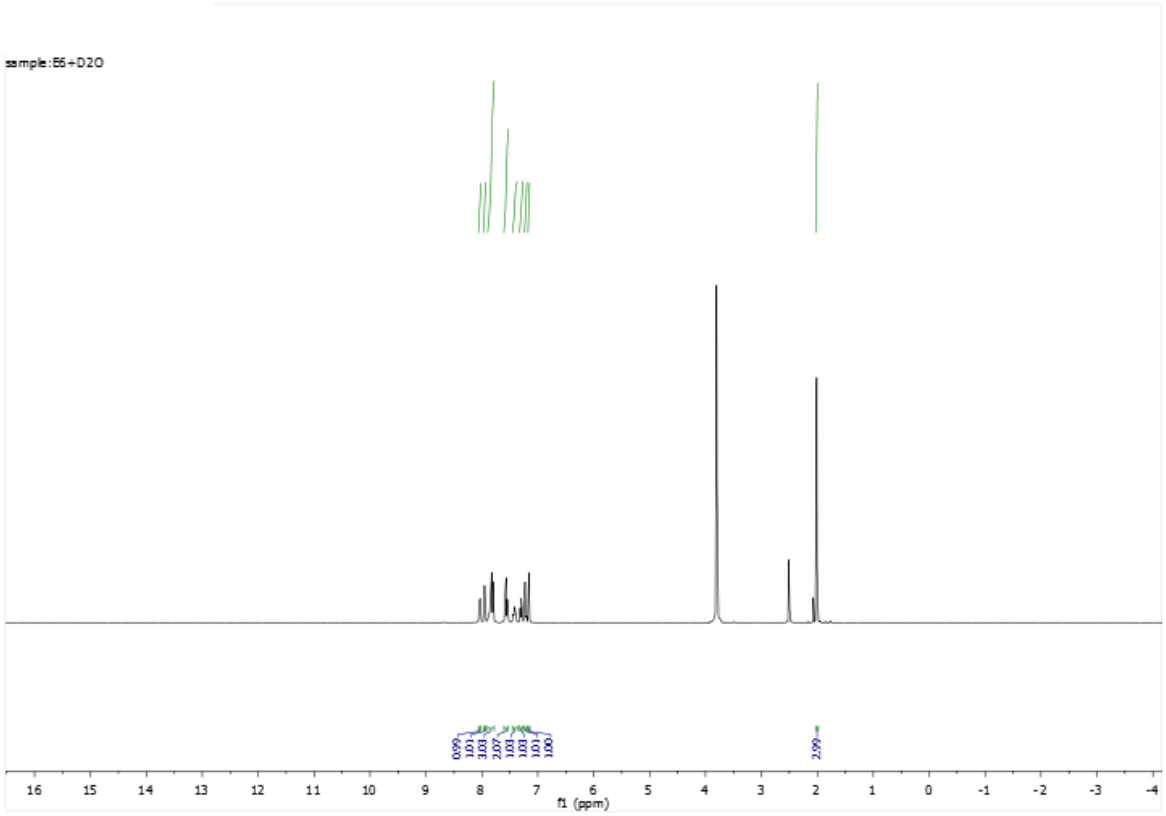
Wavenumber (cm^{-1})	Assignment
750	CH
814	CH
850	C-Cl
1276	C=O
1326	C=O
1627	C=O
3115	OH
3389	NH

Table 1 (entry 10): N-[(2-Hydroxynaphthalene-1-yl)-(3-nitrophenyl)-methyl]-acetamide

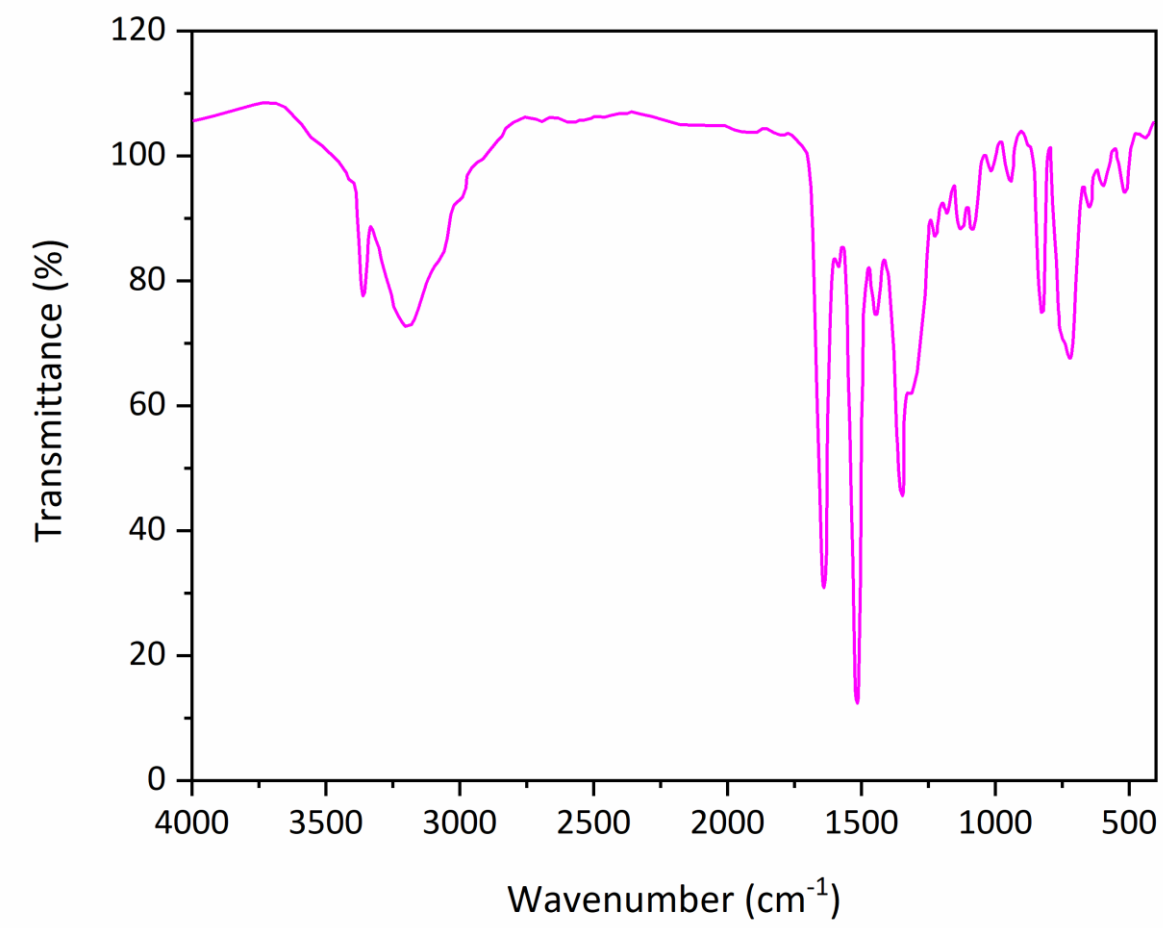




^{1}H NMR (400 MHz, DMSO-d_6) δ 9.97 (s, 1H), 8.65 (d, J = 8.0 Hz, 1H), 8.06 (dt, J = 7.1, 2.2 Hz, 1H), 8.02 (s, 1H), 7.83 (t, J = 8.7 Hz, 3H), 7.61 – 7.53 (m, 3H), 7.42 (t, J = 7.8 Hz, 1H), 7.30 (t, J = 7.4 Hz, 1H), 7.24 (d, J = 8.8 Hz, 1H), 7.19 (d, J = 7.9 Hz, 1H), 2.03 (s, 3H).



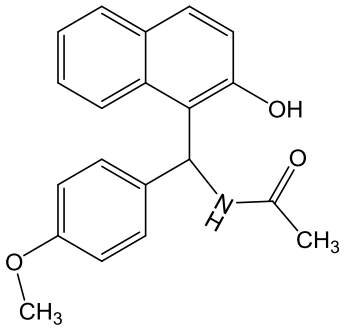
^{1}H NMR (400 MHz, DMSO-d_6 (D_2O changed)) δ 8.04 (dt, J = 7.6, 2.1 Hz, 1H), 7.95 (d, J = 2.3 Hz, 1H), 7.90 – 7.78 (m, 3H), 7.61 – 7.52 (m, 2H), 7.42 (t, J = 7.8 Hz, 1H), 7.30 (t, J = 7.4 Hz, 1H), 7.22 (d, J = 8.9 Hz, 1H), 7.16 (s, 1H), 2.01 (s, 3H).

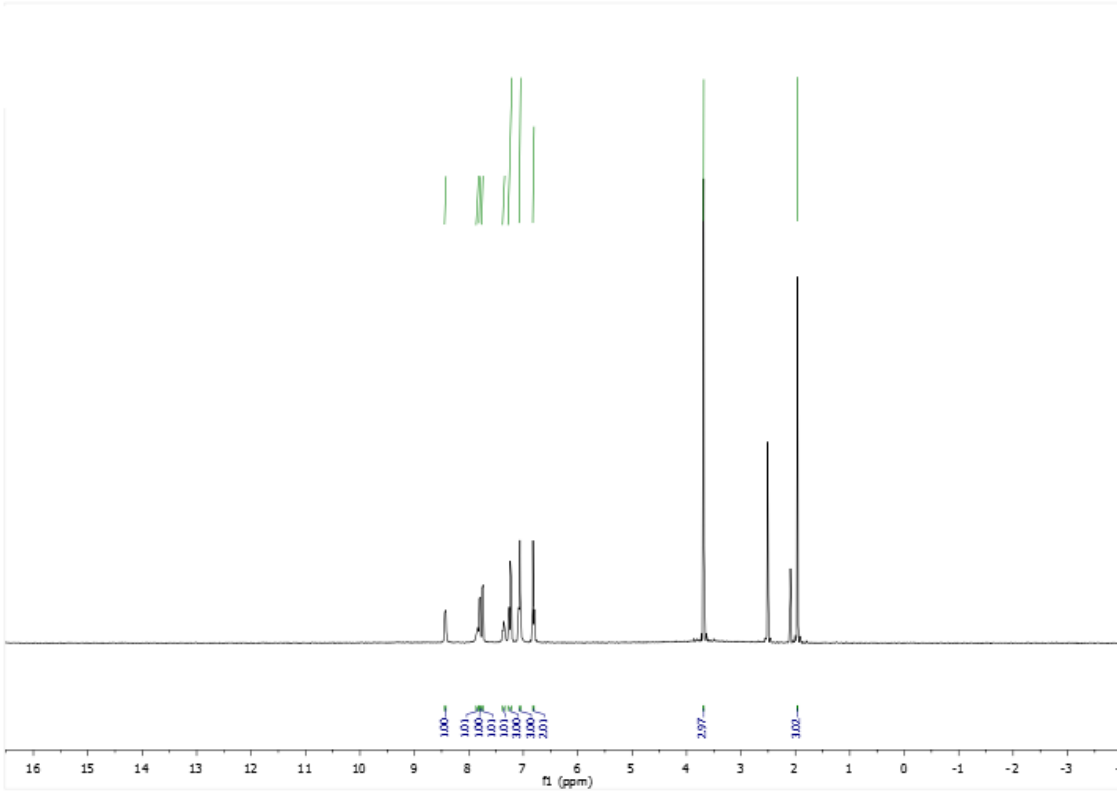


IR (KBr), ν cm^{-1}: 707, 808, 1206, 1436, 1347, 1520, 1644, 3206, 3372.

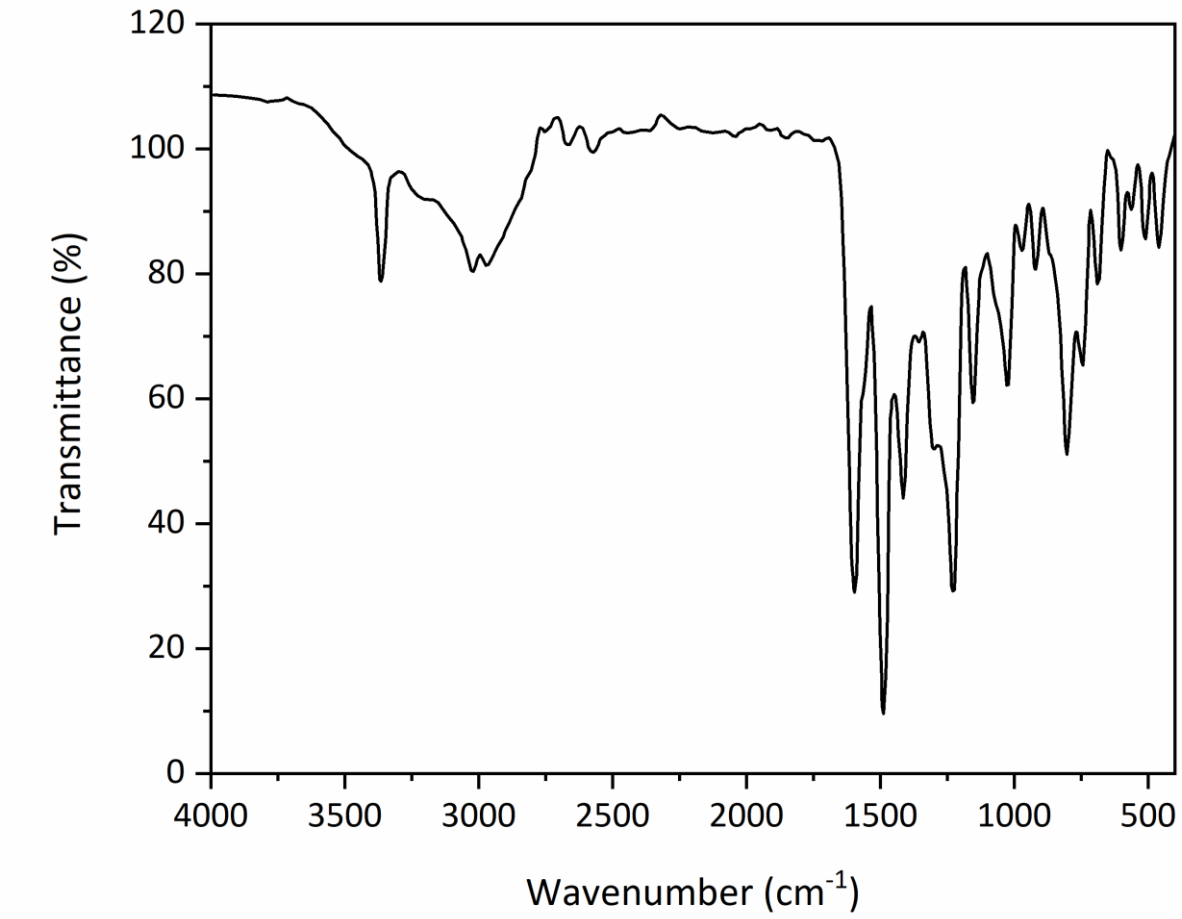
Wavenumber (cm^{-1})	Assignment
707	CH
808	CH
1520	NO_2
1644	C=O
3206	OH
3372	NH

Table 1 (entry 11): N-[(2-Hydroxynaphthalene-1-yl)-(4-methoxyphenyl)-methyl]-acetamide





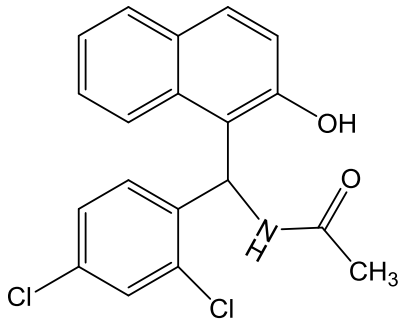
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.43 (d, J = 8.5 Hz, 1H), 7.84 (d, J = 7.6 Hz, 1H), 7.80 (d, J = 6.9 Hz, 1H), 7.76 (s, 1H), 7.36 (s, 1H), 7.25 (dd, J = 15.7, 8.2 Hz, 3H), 7.06 (t, J = 4.2 Hz, 3H), 6.82 – 6.80 (m, 2H), 3.69 (s, 3H), 1.96 (s, 3H).

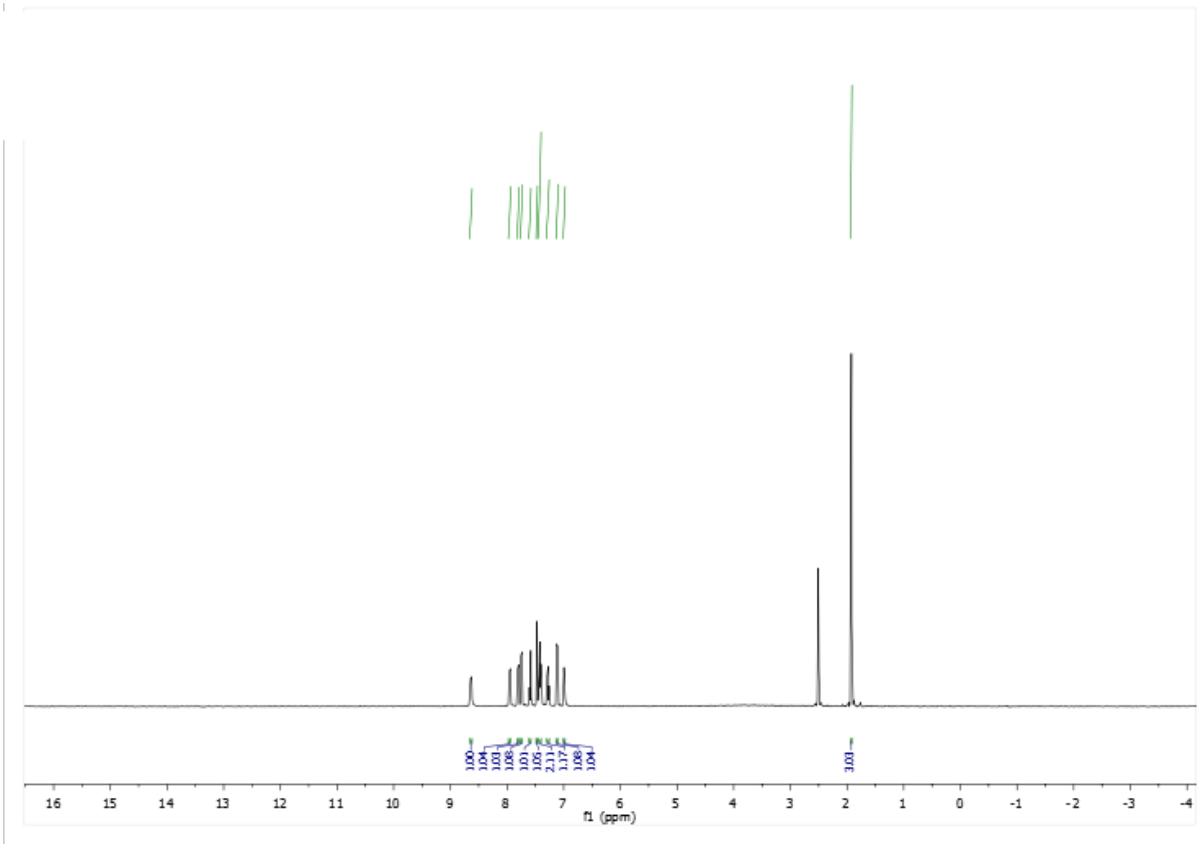


IR (KBr), ν cm^{-1}: 750, 811, 822, 1117, 1250, 1438, 1516, 1626, 3056, 3394.

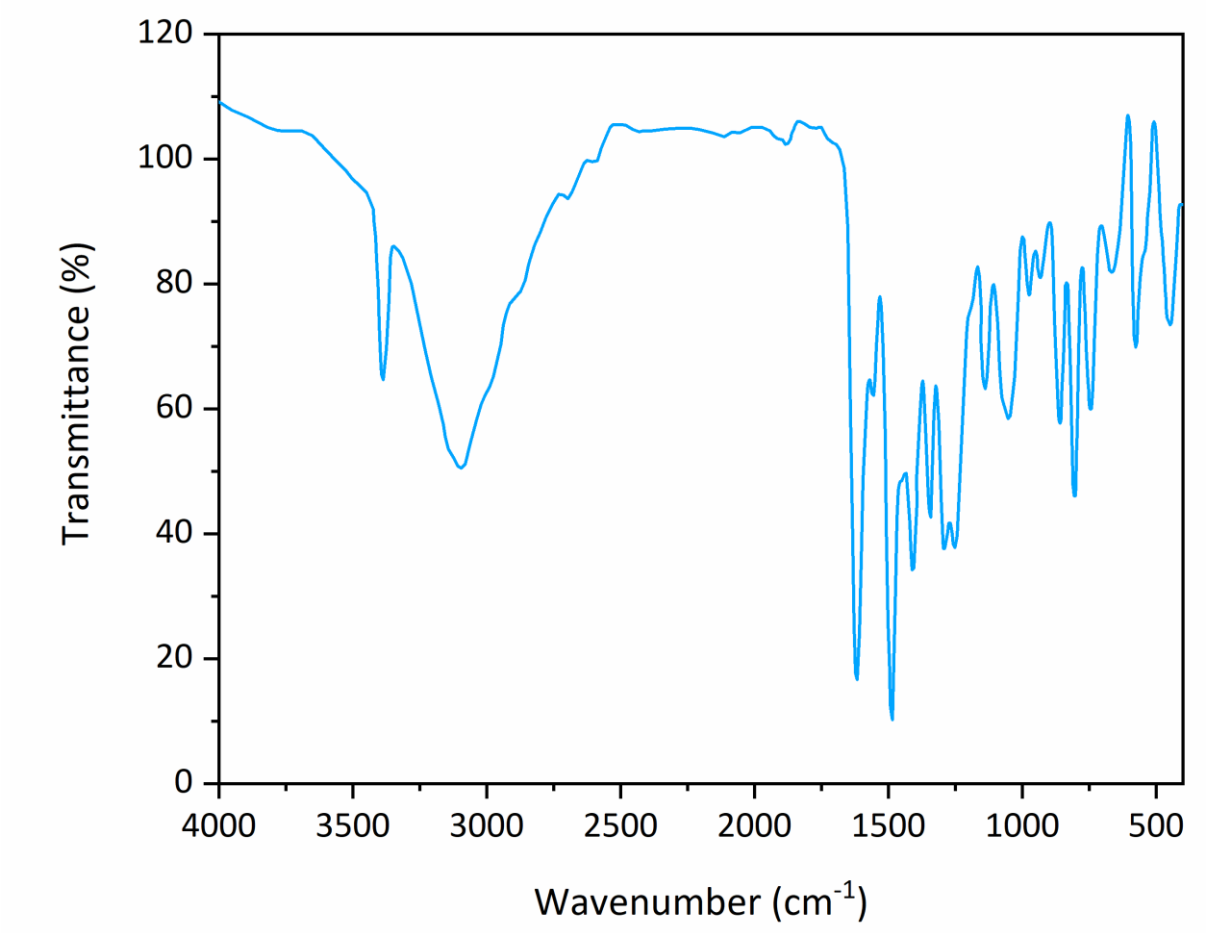
Wavenumber (cm^{-1})	Assignment
750	CH
811	CH
1626	C=O
3056	OH
3394	NH

Table 1(entry 12): N-[(2-Hydroxynaphthalene-1-yl)-(2,4-di-chlorophenyl)-methyl]-acetamide





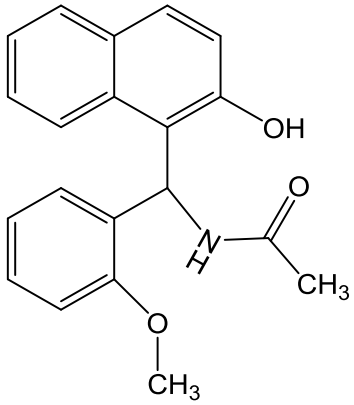
^{1}H NMR (400 MHz, DMSO-d_6) δ 8.63 (d, J = 7.9 Hz, 1H), 7.95 (d, J = 8.6 Hz, 1H), 7.80 (dd, J = 8.2, 1.4 Hz, 1H), 7.74 (d, J = 8.8 Hz, 1H), 7.59 (d, J = 8.5 Hz, 1H), 7.47 (d, J = 2.3 Hz, 1H), 7.46 – 7.40 (m, 2H), 7.28 (t, J = 7.5 Hz, 1H), 7.11 (d, J = 8.8 Hz, 1H), 6.99 (d, J = 7.7 Hz, 1H), 1.92 (s, 3H).

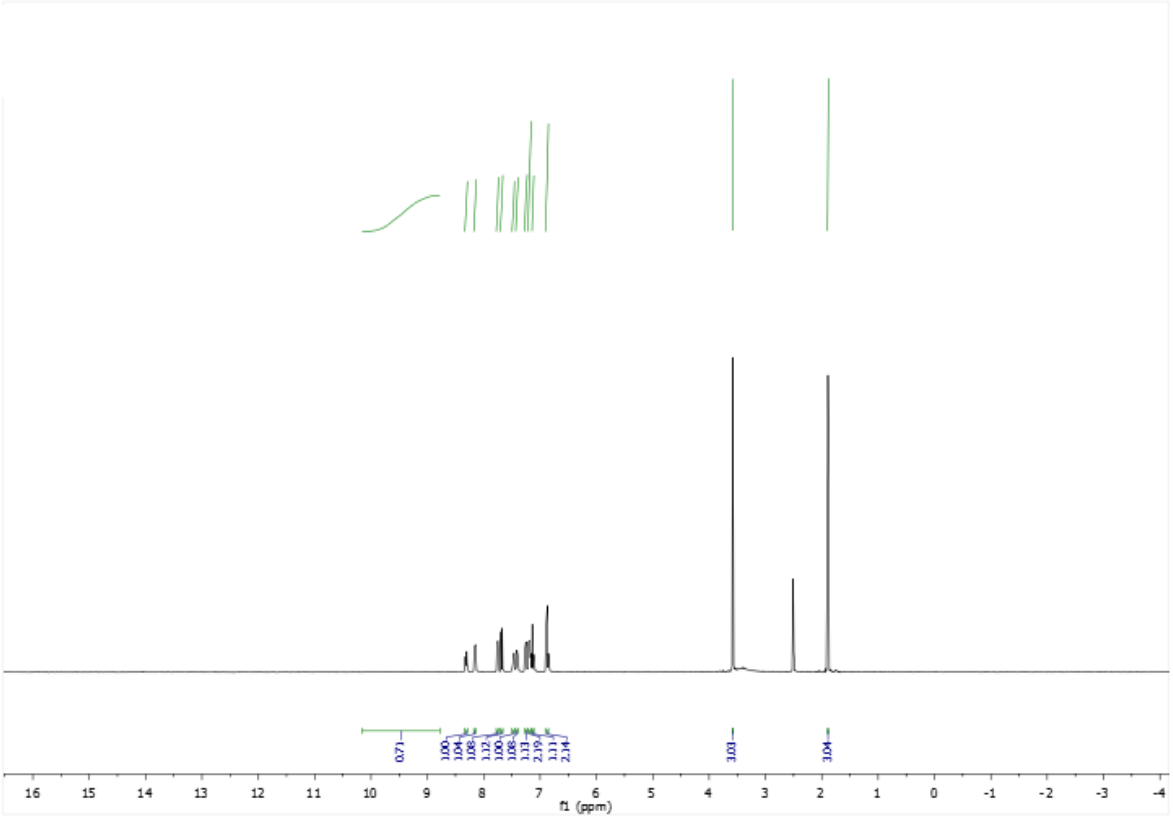


IR (KBr), ν cm^{-1}: 751, 813, 869, 1312, 1367, 1434, 1515, 1646, 3118, 3402.

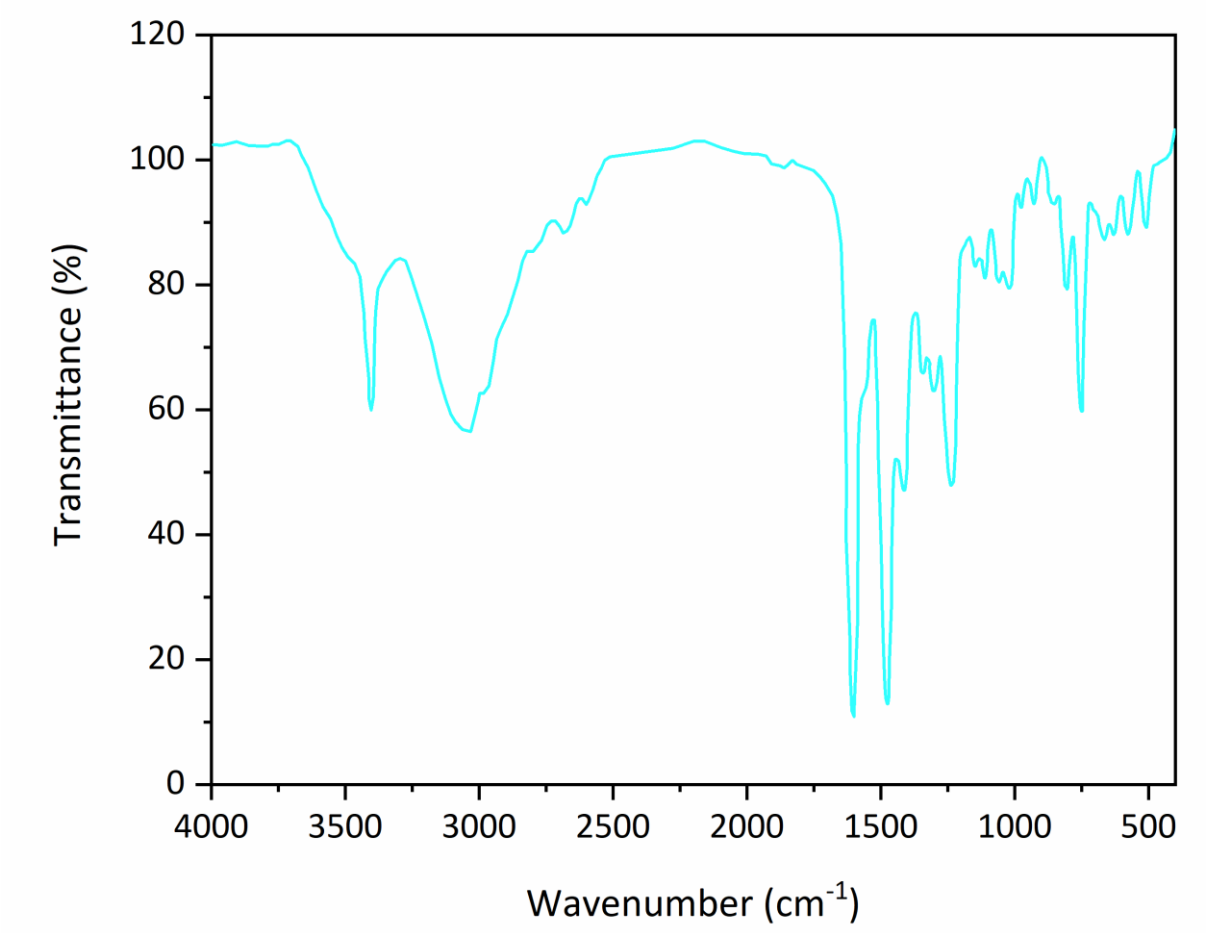
Wavenumber (cm^{-1})	Assignment
751	CH
813	CH
1646	C=O
3118	OH
3402	NH

Table 1 (entry 13): N-[(2-Hydroxynaphthalene-1-yl)-(2-methoxyphenyl)-methyl]-acetamide





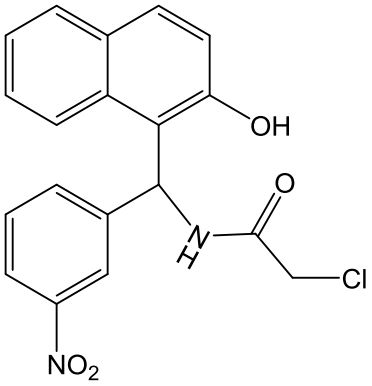
^{1}H NMR (400 MHz, DMSO-d_6) δ 9.5 (S,1H), 8.31 (d, J = 8.5 Hz, 1H), 8.16 (d, J = 8.7 Hz, 1H), 7.76 (dd, J = 8.1, 1.4 Hz, 1H), 7.68 (d, J = 8.8 Hz, 1H), 7.47 (d, J = 7.2 Hz, 1H), 7.41 (ddd, J = 8.5, 6.8, 1.4 Hz, 1H), 7.25 (dd, J = 7.9, 6.8 Hz, 1H), 7.18 (ddd, J = 8.2, 6.3, 4.8 Hz, 2H), 7.13 (d, J = 8.8 Hz, 1H), 6.91 – 6.85 (m, 2H), 3.58 (s, 3H), 1.89 (s, 3H).

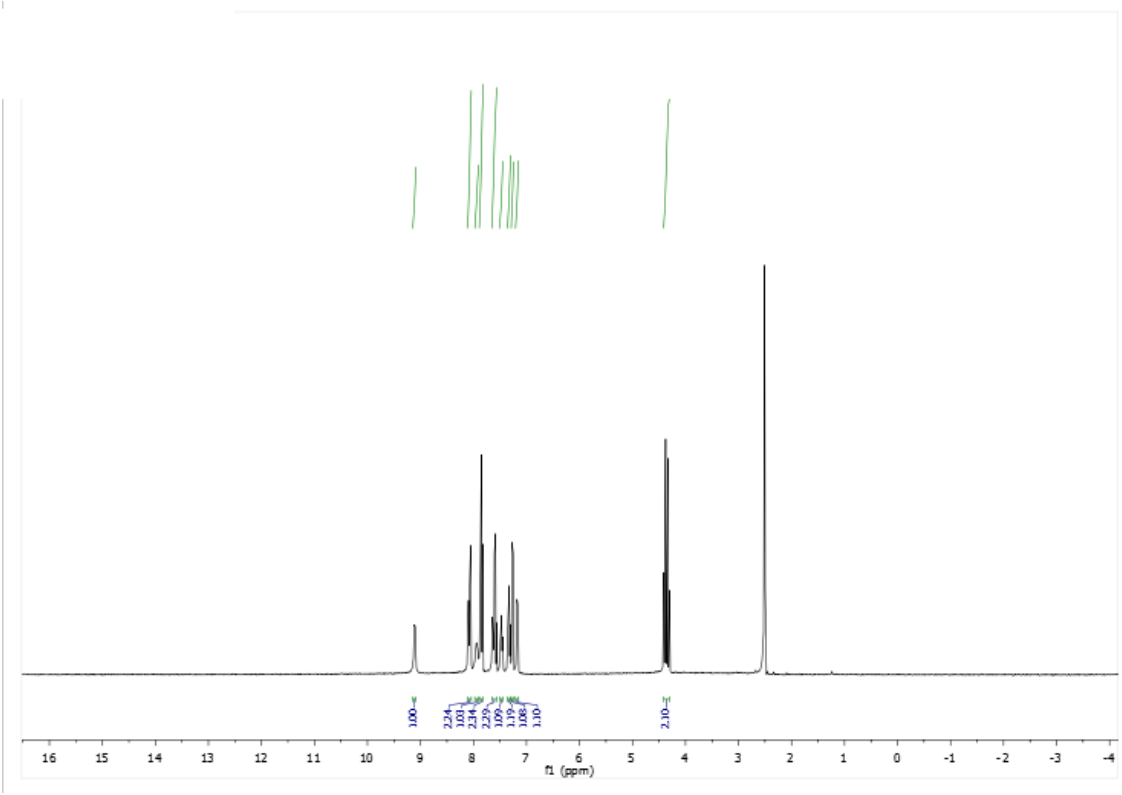


IR (KBr), ν cm^{-1}: 753, 809, 850, 1263, 1327, 1443, 1513, 1624, 3067, 3429.

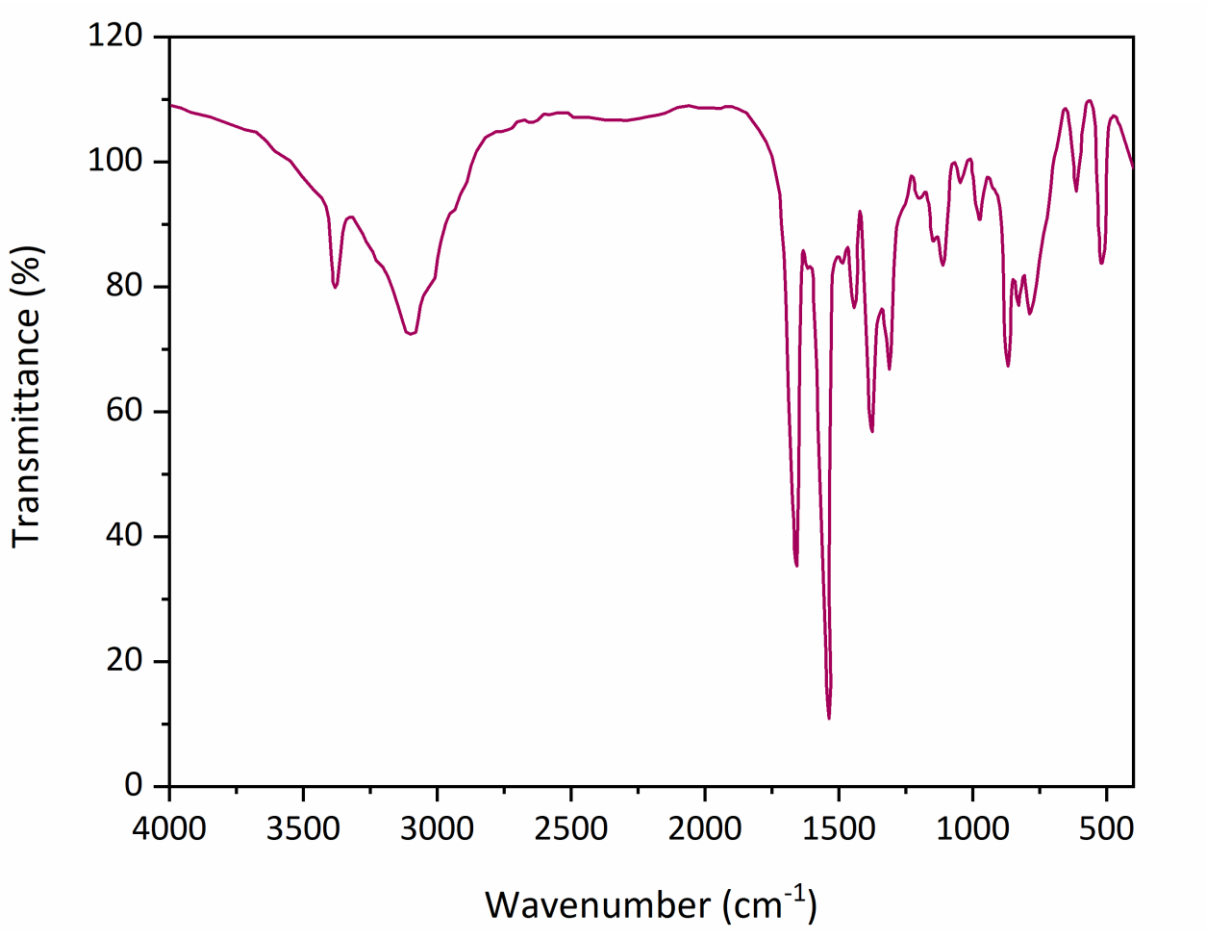
Wavenumber (cm^{-1})	Assignment
753	CH
809	CH
1624	C=O
3067	OH
3429	NH

Table 1 (entry 14): N-[(2-Hydroxynaphthalene-1-yl)-(3-nitrophenyl)-methyl]-chloroacetamide



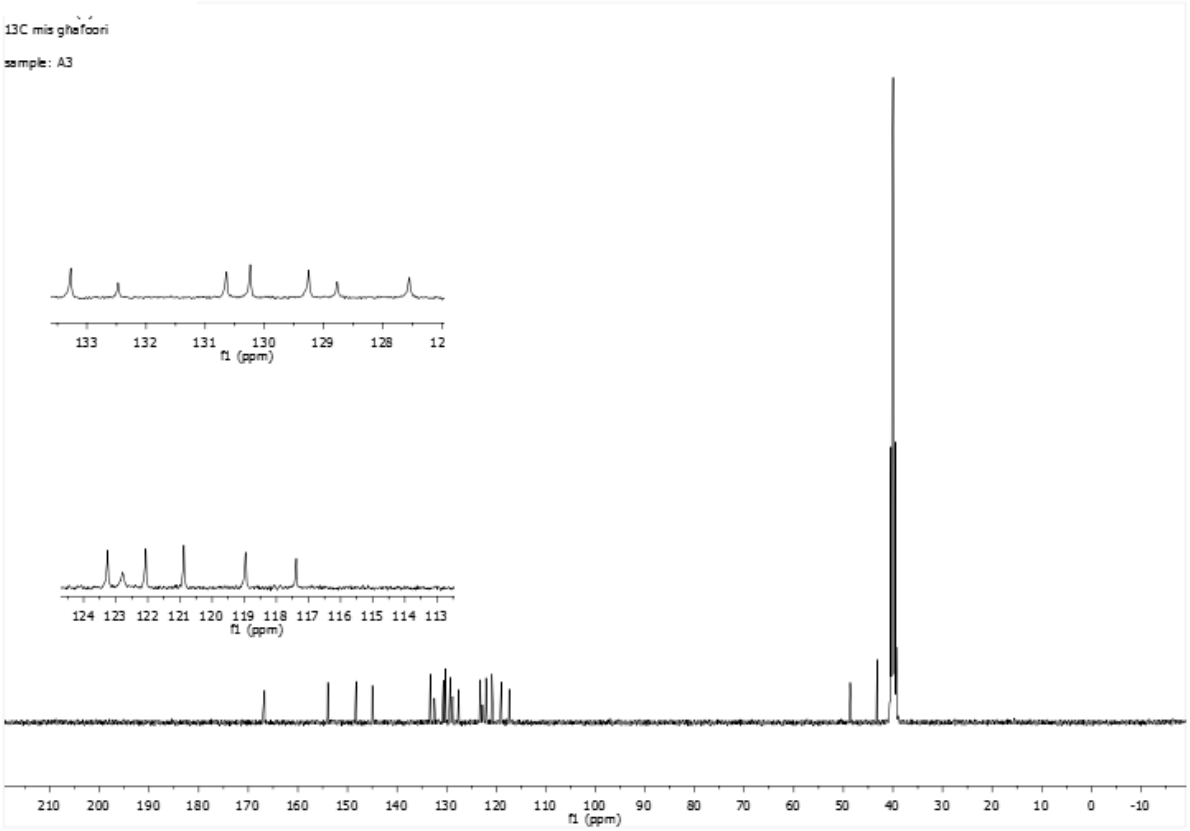


^{1}H NMR (400 MHz, DMSO-d_6) δ 9.11 (d, J = 8.2 Hz, 1H), 8.11 – 8.04 (m, 2H), 7.94 (s, 1H), 7.89 – 7.82 (m, 2H), 7.60 (dt, J = 15.6, 7.8 Hz, 2H), 7.47 (t, J = 7.7 Hz, 1H), 7.32 (t, J = 7.5 Hz, 1H), 7.26 (d, J = 8.9 Hz, 1H), 7.17 (d, J = 7.7 Hz, 1H), 4.41 – 4.30 (m, 2H).



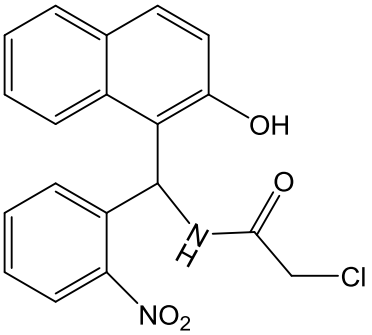
IR (KBr), ν cm^{-1}: 724, 779, 821, 1274, 1346, 1402, 1525, 1635, 3090, 3382.

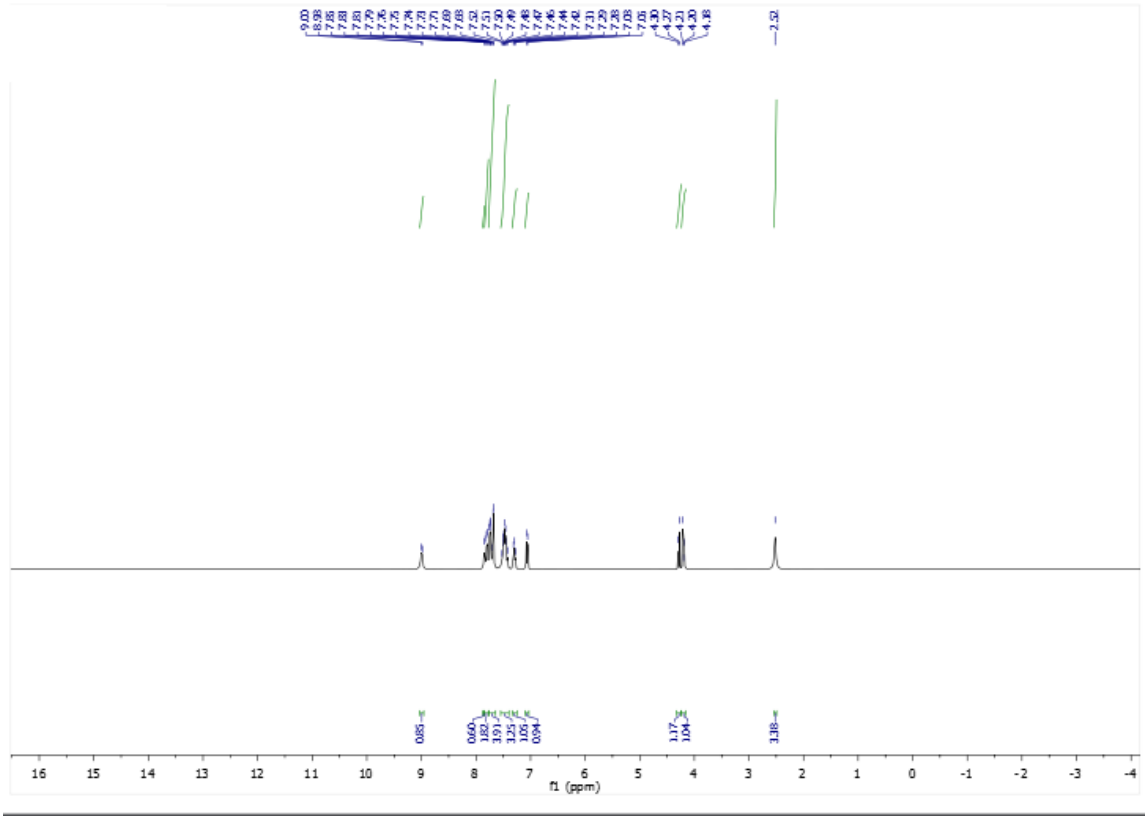
Wavenumber (cm^{-1})	Assignment
724	CH
779	CH
1346	C=C
1525	NO_2
1635	C=O
3090	OH
3382	NH



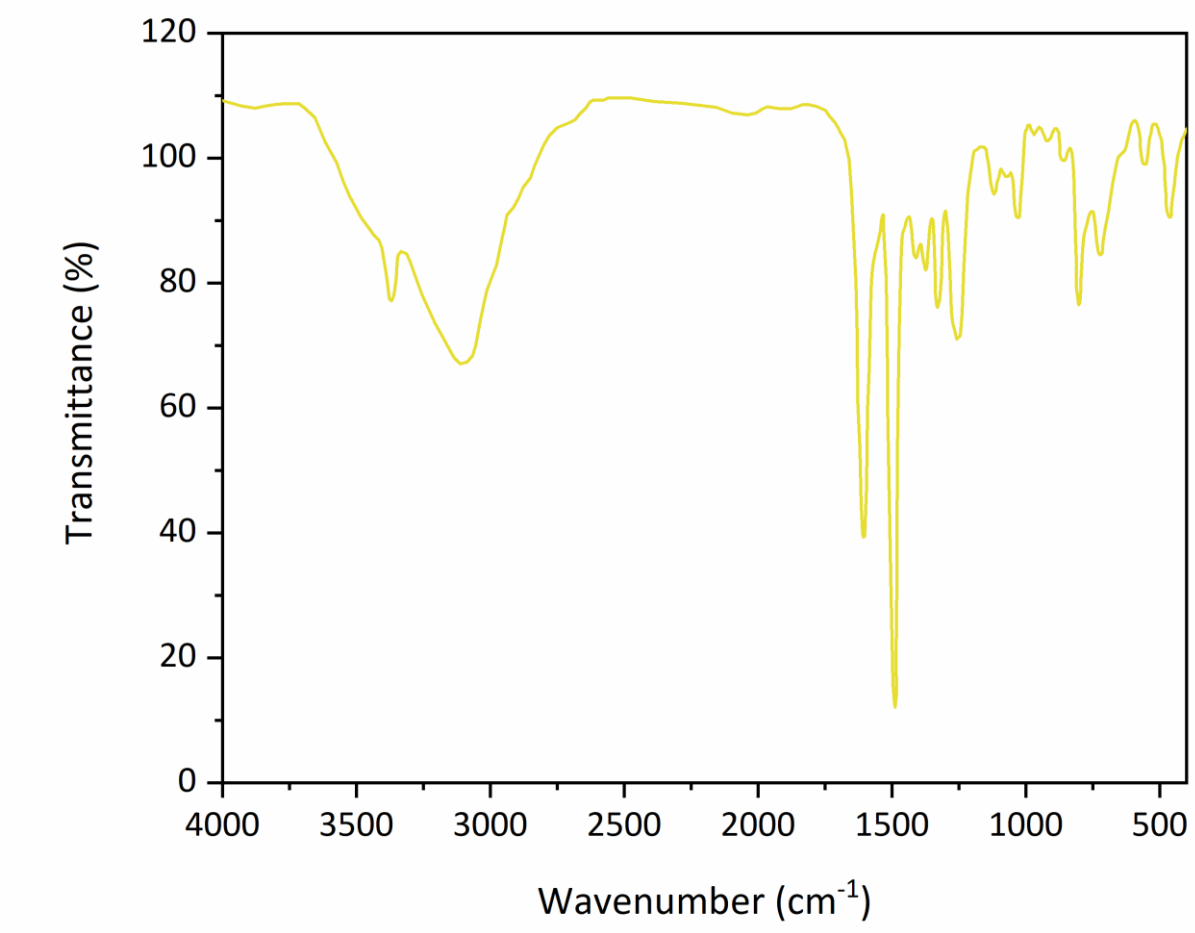
^{13}C NMR (101 MHz, DMSO-d_6) δ 166.72, 153.96, 148.26, 144.99, 133.28, 132.47, 130.64, 130.24, 129.25, 128.77, 127.56, 123.26, 122.78, 122.07, 120.88, 118.96, 117.39, 48.64, 43.18.

Table 1 (entry 15): N-[(2-Hydroxynaphthalene-1-yl)-(2-nitrophenyl)-methyl]-chloroacetamide



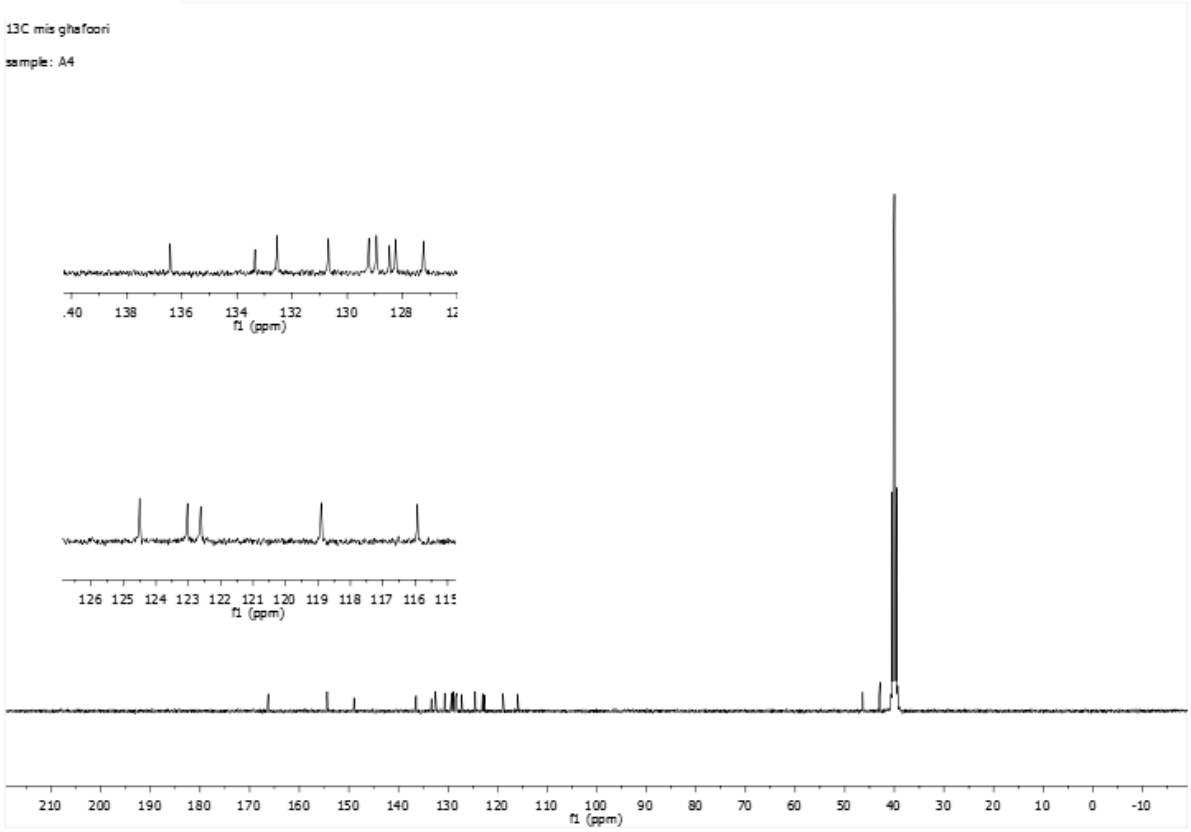


^{1}H NMR (400 MHz, DMSO-d_6) δ 8.99 (d, J = 8.3 Hz, 1H), 7.85 (s, 1H), 7.84 – 7.77 (m, 2H), 7.74 (dd, J = 8.5, 6.3 Hz, 2H), 7.68 (d, J = 4.4 Hz, 1H), 7.48 (ddd, J = 14.7, 9.4, 5.4 Hz, 3H), 7.29 (t, J = 7.4 Hz, 1H), 7.07 (d, J = 8.9 Hz, 1H), 4.28 (d, J = 12.9 Hz, 1H), 4.20 (d, J = 12.9 Hz, 1H).



IR (KBr), ν cm^{-1}: 732, 810, 852, 1277, 1353, 1401, 1526, 1640, 2123, 3381, 3500.

Wavenumber (cm^{-1})	Assignment
732	CH
810	CH
1401	C=C
1526	NO_2
1640	C=O
3381	OH
3500	NH



^{13}C NMR (101 MHz, DMSO-d_6) δ 166.26, 154.37, 148.97, 136.42, 133.35, 132.55, 130.68, 129.22, 128.94, 128.47, 128.24, 127.23, 124.50, 123.03, 122.62, 118.90, 115.93, 46.38, 42.90.