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Facile One-Pot Synthesis of 1-Amidoalkyl-2-Naphthols by $RuCl_2(PPh_3)_3$-Catalyzed Multi-Component Reactions

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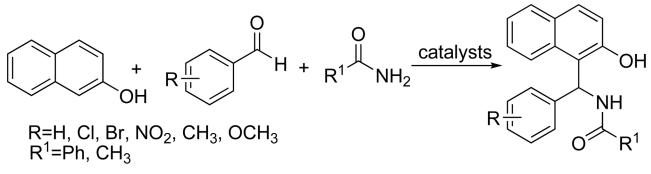
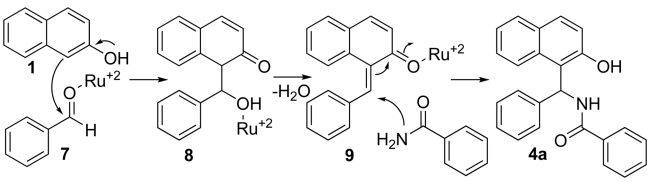
Molecules bearing 1,3-amino oxygenated functional groups have been reported to exhibit a variety of biological and pharmacological activities including nucleoside antibiotics and HIV protease inhibitors such as ritonavir and lipinavir.[1] Importantly, 1-amidoalkyl-2-naphthols can be easily converted to biologically active 1-aminomethyl-2-naphthols by amide hydrolysis. These compounds also exhibit potent antihypertensive, adrenoceptor-blocking, and Ca^{+2} channel-blocking activities.[2] Because of the importance of these compounds, numerous methods for the synthesis of 1-amidoalkyl-2-naphthols have been described. The reported methods mainly include one-pot three-component reactions of 2-naphthol, aromatic aldehydes, and amides (Scheme 1). These reactions have been extensively studied with the use of a variety of catalysts and reagents such as *p*-toluenesulfonic acid,[3] silica sulfuric acid,[4] NH_2SO_3H,[5] $Fe(HSO_4)_3$,[6] $Al(H_2PO_4)_3$,[7] $Ce(SO_4)_2$,[8] *N*-(4-sulfonic acid)-butyl triethyl ammonium hydrogen sulfate,[9] $HClO_4$-Al_2O_3,[10] $HClO_4$-SiO_2,[11] $FeCl_3$-SiO_2,[12] $ZnCl_2$-SiO_2,[13] $Yb(OTf)_3$,[14] $Sr(OTf)_2$,[15] $Bi(NO_3)_3$ $5H_2O$,[16] $Hf(NPf_2)_4$,[17] $K_5CoW_{12}O_{40}{\cdot}3H_2O$,[18] MoO_3-ZrO_2,[19] P_2O_5,[20] and montmorillonite K10 clay.[21]

Although numerous methods for the synthesis of 1-amidoalkyl-2-naphthols have been reported,[3-22] there is still demand for simpler, less toxic, more effective, and milder catalysts. Our interest in developing a mild and efficient synthetic method that provides a variety of 1-amidoalkyl-2-naphthols has led us to looking into more convenient and safely usable catalysts. Among these, we think tris(triphenylphosphine)ruthenium(II) dichloride is a viable alternative, and may be a promising catalyst for the synthesis of 1-amidoalkyl-2-naphthols due to its easy availability, sustainability, non-toxicity, and environmentally friendly properties.[23] We report herein an $RuCl_2(PPh_3)_3$-catalyzed one-pot three-component reaction for the synthesis of biologically interesting 1-amidoalkyl-2-naphthols.



Scheme 1



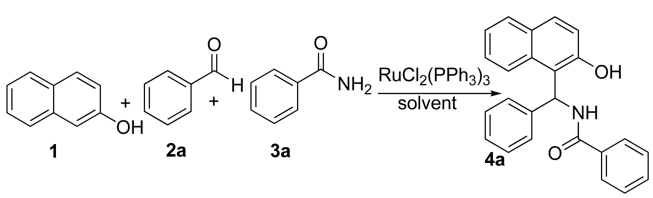
Scheme 2

Results and Discussion

The reaction of 2-naphthol (**1**, 1.0 mmol) with benzaldehyde (**2a**, 1.2 mmol) and benzamide (**3a**, 1.2 mmol) was first examined in the presence of 5 mol % $RuCl_2(PPh_3)_3$ in several solvents (Table 1). Across the range of solvents tested, the best yield was obtained in refluxing toluene in 88% yield. The desired product **4a** was determined by analysis of spectral data and compared directly with the reported data.[15]

In order to extend the utility of this methodology for the synthesis of a variety of 1-amidoalkyl-2-naphthols, further reactions of 2-naphthol with different several aryl aldehydes and amides were examined. These reactions were carried out in the presence of 5 mol % of $RuCl_2(PPh_3)_3$ in refluxing toluene for 10-12 h using the optimized reaction conditions described above. The results are summarized in Table 2. The reactions worked well with substituted aromatic aldehydes

Table 1. Reaction of 2-naphtol (**1**) with benzaldehyde (**2a**) and benzamide (**3a**) in the presence of 5 mol % of $RuCl_2(PPh_3)_3$ in several solvents



Entry	Solvent	Condition	Yield (%)
1	CH_2Cl_2	reflux, 12 h	45
2	acetone	reflux, 12 h	35
3	THF	reflux, 12 h	52
4	toluene	reflux, 10 h	88
5	DMF	150 °C, 12 h	60

bearing either electron-donating or -withdrawing groups on the benzene ring. For example, reaction of 2-naphthol with 4-methylbenzaldehyde and benzamide provided **4b** in 85% yield (entry 1, Table 2), whereas treatment with 4-methoxybenzaldehyde and benzamide afforded **4c** in 81% yield (entry 2). Similarly, reactions of **2b** and **2c** with acetamide afforded **4d** and **4e** in 70 and 75% yield (entries 3-4). Other aromatic aldehydes of 2-chlorobenzaldehyde, 4-chlorobenzaldehyde, and 4-bromobenzaldehyde with substituents of electron-withdrawing groups on the benzene ring were reacted successfully with benzamide or acetamide to afford products **4f-4i** in 80, 75, 70, and 75% yield, respectively (entries 5-8). Similarly, treatment of 4-nitrobenzaldehyde with benzamide or acetamide provided the desired products **4j** and **4k** in 62 and 70% yield, respectively (entries 9-10). These reactions provided rapid access to various 1-amidoalkyl-2-naphthols **4b-4k** in good yields.

The formation of **4a** can be explained by the proposed mechanism as shown in Scheme 2. Benzaldehyde (**2a**) forms an oxygen-bonded complex in the presence of $RuCl_2(PPh_3)_3$ catalyst to give **7**, which is attacked by 2-naphthol (**1**) to produce the intermediate **8** through aldol-type reaction. Elimination of water gives another *ortho*-quinone methide **9**, which undergoes 1,4-addition to yield final product **4a**.

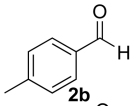
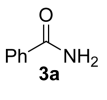
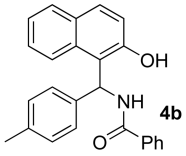
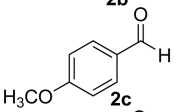
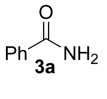
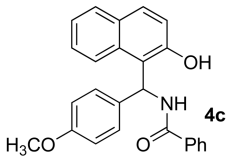
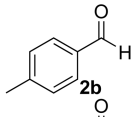
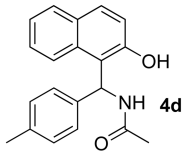
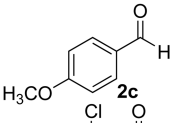
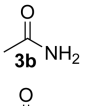
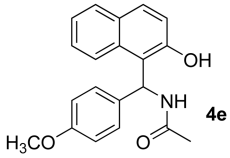
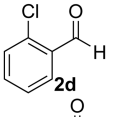
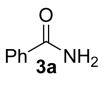
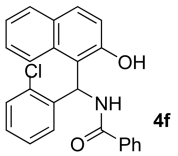
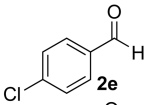
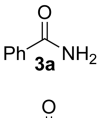
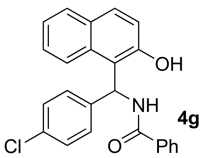
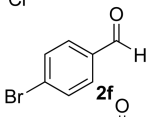
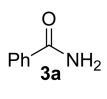
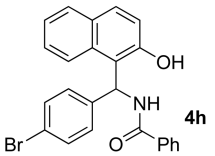
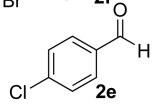
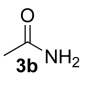
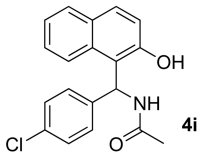
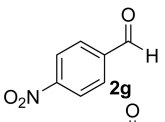
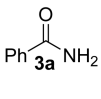
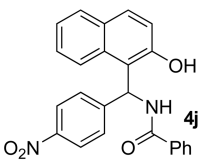
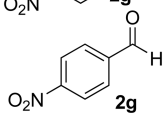
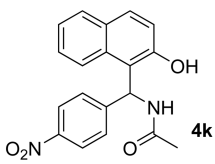
In summary, we have developed an efficient and general synthesis of 1-amidoalkyl-2-naphthols by $RuCl_2(PPh_3)_3$-catalyzed one-pot multi-component reaction of 2-naphthol with aromatic aldehydes and amides. The advantages of these methodologies are easy handling, mild reaction conditions, and use of an effective and non-toxic catalyst.

Experimental

General Procedure for the Synthesis of 4a-4k. To a mixture of 2-naphthol (144 mg, 1.0 mmol), aldehyde (1.2 mmol), and amide (1.2 mmol) in toluene (10 mL) was added $RuCl_2(PPh_3)_3$ (48 mg, 0.05 mmol) under N_2. The mixture was heated under reflux for 10-12 h. After completion of reaction as indicated by TLC, the solvent was removed under reduced pressure. The residue was purified by column chromatography on silica gel to give product.

***N*-((2-Hydroxynaphthalen-1-yl)(4-methylphenyl)meth-**

Table 2. $RuCl_2(PPh_3)_3$-catalyzed synthesis of a variety of 1-amidoalkyl-2-naphthols

Entry	Aldehydes	Amides	Time (h)	Product	Yield (%)
1	 **2b**	 **3a**	12	 **4b**	85
2	 **2c**	 **3a**	12	 **4c**	81
3	 **2b**	 **3b**	12	 **4d**	70
4	 **2c**	 **3b**	12	 **4e**	75
5	 **2d**	 **3a**	10	 **4f**	80
6	 **2e**	 **3a**	10	 **4g**	75
7	 **2f**	 **3a**	10	 **4h**	70
8	 **2e**	 **3b**	10	 **4i**	75
9	 **2g**	 **3a**	10	 **4j**	62
10	 **2g**	 **3b**	10	 **4k**	70

yl)benzamide (4a):[15] Yield 88%; mp 230-232 °C; ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.29 (1H, s), 8.99 (1H, d, J = 8.4 Hz), 8.11 (1H, d, J = 8.7 Hz), 7.94-7.79 (4H, m), 7.55-7.45 (4H, m), 7.35-7.20 (8H, m); ^{13}C NMR (DMSO-d_6, 75 MHz) δ 165.6, 161.8, 153.1, 142.0, 134.3, 132.2, 131.3, 129.3, 128.5, 128.4, 128.3, 128.1, 127.0, 126.7, 126.4, 126.3, 122.6, 118.6, 118.3, 49.2; IR (KBr) 3377, 1638, 1533, 1344, 1264, 1091, 806 cm^{-1}.

***N*-((2-Hydroxynaphthalen-1-yl)(4-methylphenyl)methyl)benzamide (4b):**[15] Yield 85%; mp 194-198 °C; ^{1}H NMR ($CDCl_3$+DMSO-d_6, 300 MHz) δ 9.56 (1H, s), 8.78 (1H, br), 8.08 (1H, d, J = 8.4 Hz), 7.75 (2H, d, J = 7.8 Hz), 7.66 (1H, d, J = 8.1 Hz), 7.58 (1H, d, J = 9.0 Hz), 7.36-7.12 (9H, m), 6.92 (2H, d, J = 7.8 Hz), 2.15 (3H, s); ^{13}C NMR (DMSO-d_6, 75 MHz) δ 163.7, 153.2, 139.0, 135.7, 134.4, 132.3, 131.4, 129.3, 128.8, 128.6, 128.5, 128.4, 127.1, 126.8, 126.5, 122.7, 118.7, 118.5, 49.1, 20.6; IR (KBr) 3752, 3425, 2371, 1628, 1572, 1517, 1481, 1438, 1343, 1274, 1179, 943, 814 cm^{-1}.

***N*-((2-Hydroxynaphth-1-yl)(4-methoxyphenyl)methyl)-benzamide (4c):**[15] Yield 81%; mp 200-204 °C; ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.40 (1H, br), 9.07 (1H, d, J = 8.7 Hz), 8.09 (1H, d, J = 8.7 Hz), 7.86 (2H, d, J = 8.4 Hz), 7.80 (2H, d, J = 9.3 Hz), 7.56-7.44 (2H, m), 7.33-7.21 (7H, m), 6.85 (2H, d, J = 8.7 Hz), 3.68 (3H, s); ^{13}C NMR ($CDCl_3$ + DMSO-d_6, 75 MHz) δ 166.1, 158.0, 152.7, 134.5, 134.0, 132.3, 131.0, 129.1, 128.4, 128.2, 128.2, 127.6, 126.7, 126.7, 122.7, 122.2, 118.6, 118.5, 113.2, 54.9, 49.2; IR (KBr) 3751, 3425, 2369, 1630, 1573, 1516, 1436, 1346, 1260, 1173, 1028, 939, 822 cm^{-1}.

***N*-((2-Hydroxynaphthalen-1-yl)(4-methylphenyl)methyl)acetamide (4d):**[6] Yield 70%; mp 225-227°C; ^{1}H NMR (DMSO-d_6, 300 MHz) δ 9.99 (1H, s), 8.43(1H, d, J = 8.4 Hz), 7.86-7.74 (3H, m), 7.35 (1H, dd, J = 6.9, 7.8 Hz), 7.26 (1H, d, J = 7.5 Hz), 7.22 (1H, d, J = 8.7 Hz), 7.09 (1H, d, J = 8.4 Hz), 7.04 (4H, s), 2.22 (3H, s), 1.97 (3H, s); ^{13}C NMR (DMSO-d_6, 75 MHz) δ 169.2, 153.2, 139.6, 135.0, 132.3, 129.1, 128.6, 128.2, 126.3, 126.0, 123.4, 122.3, 118.9, 118.5, 47.6, 22.7, 20.6; IR (KBr) 3396, 1625, 1519, 1438, 1330, 1269, 1177, 1064, 809 cm^{-1}.

***N*-((2-Hydroxynaphthalen-1-yl)(4-methoxyphenyl)methyl)acetamide (4e):**[15] Yield 75%; mp 182-184 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 10.01 (1H, br), 8.46 (1H, d, J = 7.8 Hz), 8.06 (1H, d, J = 7.5 Hz), 7.78 (1H, d, J = 7.2 Hz), 7.65 (1H, d, J = 9.0 Hz), 7.34-7.27 (6H, m), 6.76 (2H, d, J = 9.0 Hz), 3.70 (3H, s), 2.01(3H, s); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.7, 158.3, 153.0, 133.3, 132.3, 129.6, 128.7, 128.5, 127.8, 126.8, 122.9, 122.2, 118.7, 117.8, 113.5, 55.0, 49.6, 23.2; IR (KBr) 3753, 3452, 3371, 2925, 2857, 1626, 1515, 1444, 1376, 1301, 1248, 1179, 1034, 816, 749 cm^{-1}.

***N*-((2-Hydroxynaphthalen-1-yl)(2-chlorophenyl)methyl)benzamide (4f):**[24] Yield 80%; mp 260-262 °C ; ^{1}H NMR ((DMSO-d_6, 300 MHz) δ 9.96 (1H, s), 8.98 (1H, s), 8.07 (1H, d, J = 8.1 Hz), 7.88 (2H, d, J = 6.0 Hz), 7.79 (2H, dd, J = 8.1, 12.0 Hz), 7.45-7.34 (7H, m), 7.27-7.12 (4H, m); ^{13}C NMR (DMSO-d_6, 75 MHz) δ 165.3, 153.6, 138.7, 134.2, 132.8, 132.7, 131.0, 130.1, 129.3, 128.5, 128.3, 128.1, 127.3, 126.5, 126.2, 122.7, 122.3, 118.6, 48.6; IR (KBr) 3423, 3072, 1956, 1817, 1631, 1531, 1439, 1344, 1282, 1182, 1048, 939, 818, 755, 704 cm^{-1}.

***N*-((2-Hydroxynaphthalen-1-yl)(4-chlorophenyl)methyl)benzamide (4g):**[15] Yield 75%; mp 227-229 °C; ^{1}H NMR ($CDCl_3$+DMSO-d_6, 300 MHz) δ 8.58 (1H, d, J = 9.0 Hz), 8.04 (1H, d, J = 8.4 Hz), 7.75-7.72 (2H, m), 7.65 (1H, d, J = 7.8 Hz), 7.59 (1H, d, J = 9.0 Hz), 7.39-7.04 (12H, m); ^{13}C NMR (Acetone-d_6, 75 MHz) δ 167.3, 163.4, 154.2, 142.5, 135.8, 133.8, 133.0, 132.4, 130.9, 130.3, 129.8, 129.5, 129.4, 129.2, 128.1, 124.2, 123.6, 119.8, 119.7, 50.3; IR (KBr) 3422, 2369, 2340, 1639, 1575, 1525, 1486, 1437, 1342, 1273, 811, 695 cm^{-1}.

***N*-((2-Hydroxynaphthalen-1-yl)(4-bromophenyl)methyl)benzamide (4h):**[9] Yield 70%; mp 206-208 °C; ^{1}H NMR (DMSO-d_6, 300 MHz) δ 10.39 (1H, br), 9.05 (1H, d, J = 8.4 Hz), 8.07 (1H, d, J = 8.7 Hz), 7.88 (2H, d, J = 7.5 Hz), 7.83 (2H, dd, J = 7.8, 8.4 Hz), 7.57-7.45 (6H, m), 7.32 (1H, d, J = 8.1 Hz), 7.25 (4H, dd, J = 8.7, 8.1 Hz); ^{13}C NMR (DMSO-d_6, 75 MHz) δ 166.0, 161.9, 153.3, 141.6, 134.2, 132.3, 131.5, 131.1, 129.7, 128.8, 128.7, 128.5, 128.4, 127.3, 126.9, 122.8, 119.7, 118.7, 117.9, 48.8; IR (KBr) 3411, 3197, 2363, 1632, 1512, 1336, 1266, 1067, 811, 723 cm^{-1}.

***N*-((2-Hydroxynaphthalen-1-yl)(4-chlorophenyl)methyl)-acetamide (4i):**[7] Yield 75%; mp 224-226 °C; ^{1}H NMR ($CDCl_3$+DMSO-d_6, 300 MHz) δ 9.41 (1H, s), 7.74 (2H, d, J = 8.7 Hz), 7.47 (1H, d, J = 7.8 Hz), 7.40 (1H, d , J = 9.0 Hz), 7.11 (1H, d, J = 7.8, 7.5 Hz), 7.00 (1H, dd, J = 7.5, 7.2 Hz), 6.92-6.85 (6H, m), 1.77 (3H, s); ^{13}C NMR ($CDCl_3$+DMSO-d_6, 75 MHz) δ 168.7, 152.4, 140.4, 134.0, 131.6, 130.9, 128.7, 127.8, 127.7, 127.1, 126.0, 122.0, 121.6, 118.0, 117.6, 47.6, 22.4; IR (KBr) 3751, 3395, 2371, 1631, 1520, 1438, 1372, 1332, 1278, 1245, 1175, 1093, 933, 817 cm^{-1}.

***N*-((2-Hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)-benzamide (4j):**[25] Yield 62%; mp 235-237 °C; ^{1}H NMR ($CDCl_3$+DMSO-d_6, 300 MHz) δ 9.90 (1H, br), 8.75 (1H, d, J = 8.7 Hz), 8.13 (1H, d, J = 8.7 Hz), 8.02 (2H, d, J = 8.4 Hz), 7.83 (2H, d, J = 7.5 Hz), 7.76 (1H, d, J = 7.8 Hz), 7.71 (1H, d, J = 9.0 Hz), 7.50-7.40 (7H, m), 7.30 (1H, d, J = 7.8, 7.2 Hz), 7.23 (1H, d, J = 9.0 Hz); ^{13}C NMR ($CDCl_3$+DMSO-d_6, 75 MHz) δ 166.2, 152.7, 149.7, 146.0, 133.5, 131.8, 131.2, 129.6, 128.2, 128.0, 126.8, 126.5, 122.7, 122.6, 121.4, 118.1, 117.0, 48.8; IR (KBr) 3421, 2384, 2278, 1737, 1642, 1519, 1345, 1057, 851, 815, 743, 709 cm^{-1}.

***N*-((2-Hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)-acetamide (4k):**[7] Yield 70%; mp 230-234 °C; ^{1}H NMR ($CDCl_3$+DMSO-d_6, 300 MHz) δ 9.73 (1H, br), 8.01-7.92 (4H, m), 7.71 (1H, d, J = 8.1 Hz), 7.65 (1H, d, J = 8.7 Hz), 7.36 (3H, dd, J = 10.8, 8.7 Hz), 7.26-7.13 (3H, m), 2.03 (3H, s); ^{13}C NMR ($CDCl_3$+DMSO-d_6, 75 MHz) δ 169.2, 156.7, 152.6, 149.8, 131.6, 129.2, 127.9, 127.9, 126.4, 126.3, 122.3, 122.2, 121.5, 117.8, 116.9, 47.8, 22.4; IR (KBr) 3394, 1634, 1522, 1442, 1279, 819 cm^{-1}.

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