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Three-component synthesis of amidoalkyl naphthols catalyzed by bismuth(III) nitrate pentahydrate

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Abstract

Bismuth(III) nitrate pentahydrate catalyzed the three-component condensation of β-naphthol, aldehydes and amines/urea under solvent-free conditions to afford the corresponding amidoalkyl naphthols in excellent yields.
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Keywords: Amidoalkyl naphthols; Bismuth(III) nitrate; One-pot synthesis; Solvent-free conditions

Compounds bearing 1,3-amido oxygenated functional groups are ubiquitous to a variety of biologically important natural products and potent drugs including a number of nucleoside antibiotics and HIV protease inhibitors such as ritonavir, lipinavir, and the hypotensive [1]. In addition, the bradycardiac effects of these compounds have been evaluated [2]. The importance of amidoalkyl naphthols for their synthesis has attracted renewed attention and various improved procedures have been reported. These reported methods mainly include the one-pot three-component condensation of β-naphthol, aldehydes and amine/CH_3CN, which employs catalysts such as *p*-toluene sulfonic acid [3], H_2NSO_3H [4], $Al(H_2PO_4)_3$ [5], $Yb(OTf)_3$ [6], $Sr(OTf)_2$ [7], I_2 [8], Brønsted acidic ionic liquid [9], $K_5CoW_{12}O_{40}{\cdot}3H_2O$ [10], Indion-130 [11], Al_2O_3–$HClO_4$ [12], montmorillonite K10 [13], and silica sulfuric acid [14]. Most of these methods suffer from drawbacks including long reaction time, expensive reagent, toxic and corrosive solvent, high reaction temperature (>100 °C), high catalyst loading, strongly acidic conditions, and the use of additional microwave or ultrasonic irradiation. Moreover, aliphatic aldehydes did not give satisfactory yields in earlier reports. Therefore, the development of less expensive and high yielding catalytic method is desired.

Recently, the use of bismuth(III) nitrate as a catalyst or as a stoichiometric reagent in organic synthesis has increased considerably [15–18]. The main reasons for this are their low cost, nontoxicity, commercial availability, ease handling and resistant to air/moisture. That is why bismuth(III) compounds are termed as ''green'' reagents in organic synthesis [19–21]. In continuation of our work on the development of useful synthetic methodologies, we herein disclose the catalytic activity of bismuth(III) nitrate pentahydrate for the efficient three-component synthesis of amidoalkyl naphthols **4** under solvent-free conditions (Scheme 1).

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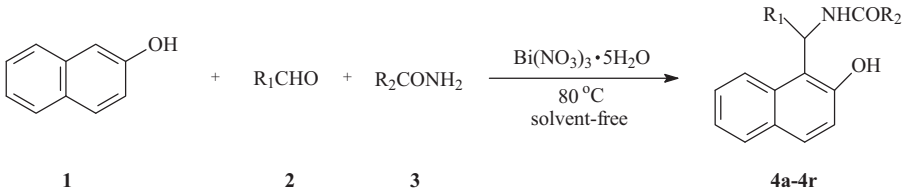

Scheme 1. One-pot three-component synthesis of amidoalkyl naphthols.

1. Experimental

General procedure for the synthesis of amidoalkyl naphthols (**4**): To a mixture of *β*-naphthol (10 mmol), an aldehyde (10 mmol), and an amide (11 mmol), $Bi(NO_3)_3{\cdot}5H_2O$ (0.2 mmol) was added. The reaction mixture was magnetically stirred on a preheated water bath at 80 °C. After completion of the reaction (monitored by TLC), the reaction mixture was cooled to r.t., washed with H_2O/EtOH (v/v = 1/1), and the residue was recrystallized from EtOH. The products were characterized by comparing their mp, IR, ^{1}H NMR, ^{13}C NMR and elemental analysis with those reported for the authentic samples. Spectral data for some representative compounds.

N-(1-(2-hydroxynaphthalen-1-yl)propyl)benzamide (**4j**). White solid. IR (KBr, cm^{-1}): 3405, 3184, 1635, 1532, 1514, 1344, 1073, 816, 747, 707. ^{1}H NMR (500 MHz, DMSO-d_6): *δ* 10.10 (s, 1H, OH), 8.62 (d, 1H, *J* = 8.0 Hz, NH), 8.23 (d, 1H, *J* = 8.8 Hz, ArH), 7.82 (t, 3H, *J* = 7.2 Hz, ArH), 7.70 (d, 1H, *J* = 8.8 Hz, ArH), 7.53–7.44 (m, 4H, ArH), 7.30 (t, 1H, *J* = 7.2 Hz, ArH), 7.20 (d, 1H, *J* = 8.8 Hz, ArH), 5.92 (q, 1H, *J* = 7.5 Hz, CH), 2.18–1.96 (m, 2H, CH_2), 0.93 (t, 3H, *J* = 7.3 Hz, CH_3). ^{13}C NMR (125 MHz, DMSO-d_6): *δ* 165.4, 152.9, 134.7, 132.1, 131.0, 128.5, 128.4, 128.3, 128.2, 126.9, 126.2, 122.5, 119.5, 118.6, 48.5, 26.9, 11.3. Anal. Calcd. for $C_{20}H_{19}NO_2$: C, 78.66; H, 6.27; N, 4.58. Found: C, 78.54; H, 6.33; N, 4.52%.

N-(1-(2-hydroxynaphthalen-1-yl)propyl)acetamide (**4p**). White solid. IR (KBr, cm^{-1}): 3430, 3236, 2963, 1644, 1583, 1517, 1333, 1079, 814, 746, 709. ^{1}H NMR (500 MHz, DMSO-d_6): *δ* 9.94 (s, 1H, OH), 8.08 (d, 1H, *J* = 8.6 Hz, NH), 8.03 (s, 1H, ArH), 7.72 (d, 1H, *J* = 8.0 Hz, ArH), 7.64 (d, 1H, *J* = 8.8 Hz, ArH), 7.39 (t, 1H, *J* = 7.4 Hz, ArH), 7.21 (t, 1H, *J* = 7.4 Hz, ArH), 7.13 (d, 1H, *J* = 9.1 Hz, ArH), 5.63 (q, 1H, *J* = 7.8 Hz, CH), 1.89–1.84 (m, 2H, CH_2), 1.81 (s, 3H, CH_3), 0.77 (t, 3H, *J* = 7.4 Hz, CH_3). ^{13}C NMR (125 MHz, DMSO-d_6): *δ* 169.1, 153.5, 132.9, 129.0, 128.9, 128.7, 126.6, 123.1, 122.7, 120.0, 119.1, 48.0, 27.2, 23.2, 11.9. Anal. Calcd. for $C_{15}H_{17}NO_2$: C, 74.05; H, 7.04; N, 5.75. Found: C, 74.17; H, 6.98; N, 5.68%.

N-(1-(2-hydroxynaphthalen-1-yl)butyl)acetamide (**4q**). White solid. IR (KBr, cm^{-1}): 3409, 3220, 2956, 1642, 1583, 1531, 1515, 1336, 1076, 816, 749, 705. ^{1}H NMR (500 MHz, DMSO-d_6): *δ* 9.86 (s, 1H, OH), 8.13 (d, 1H, *J* = 8.6 Hz, NH), 8.02 (s, 1H, ArH), 7.78 (d, 1H, *J* = 7.7 Hz, ArH), 7.68 (d, 1H, *J* = 8.8 Hz, ArH), 7.46 (t, 1H, *J* = 7.2 Hz, ArH), 7.28 (t, 1H, *J* = 7.4 Hz, ArH), 7.18 (d, 1H, *J* = 8.8 Hz, ArH), 5.83 (q, 1H, *J* = 7.6 Hz, CH), 2.05–1.97 (m, 1H, CH_2), 1.88–1.80 (m, 4H, CH_2 and CH_3), 1.40–1.30 (m, 1H, CH_2), 1.22–1.13 (m, 1H, CH_2), 0.88 (t, 3H, *J* = 7.4 Hz, CH_3). ^{13}C NMR (125 MHz, DMSO-d_6): *δ* 168.4, 152.9, 132.2, 128.4, 128.2, 128.1, 126.0, 122.1, 119.8, 118.5, 45.5, 35.9, 22.7, 19.5, 13.7. Anal. Calcd. for $C_{16}H_{19}NO_2$: C, 74.68; H, 7.44; N, 5.44. Found: C, 74.79; H, 7.37; N, 5.36%.

1-((2-Hydroxynaphthalen-1-yl)(phenyl)methyl)urea (**4r**). White solid. IR (KBr, cm^{-1}): 3447, 3212, 2932, 1651, 1535, 1438, 1354, 1063, 814, 751, 698. ^{1}H NMR (500 MHz, DMSO-d_6): *δ* 9.97 (s, 1H, OH), 7.83–7.75 (m, 3H, ArH), 7.40 (s, 1H, NH), 7.29–7.11 (m, 7H, ArH), 6.94 (s, 2H), 5.86 (s, 2H, NH_2). ^{13}C NMR (125 MHz, DMSO-d_6): *δ* 158.5, 152.9, 144.2, 128.9, 128.6, 127.8, 126.4, 125.8, 122.4, 120.2, 118.5, 48.1. Anal. Calcd. for $C_{18}H_{16}N_2O_2$: C, 73.95; H, 5.51; N, 9.58. Found: C, 74.08; H, 5.59; N, 9.46%.

2. Results and discussion

First, in order to optimize the reaction conditions, various reaction media were screened using the model reaction of *β*-naphthol, benzaldehyde and benzamide in Table 1. It was found that the best results were obtained with 2 mol% $Bi(NO_3)_3{\cdot}5H_2O$ under solvent-free conditions (Table 1, entry 9). The reaction was completed within 12 min and the expected product was obtained in a 96% yield, while diminish the amount of catalyst would decrease the product yield (Table 1, entry 10).

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