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Research Article

A Novel Four-Component Reaction between Secondary Amines and Hydroxybenzaldehydes with Isocyanides in Water: An Efficient One-Pot and Green Synthesis of Benzo[*b*]furan Derivatives

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The novel benzo[*b*]furan derivatives, **7a–i**, were synthesized and characterized by Ugi four-component reaction between 2-hydroxybenzaldehyde derivative **1**, a secondary amine **2**, and an isocyanide **3** in water. Those reactions were carried out at room temperature with moderate to good yields in one pot.

1. Introduction

The isocyanide-based MCRs have widely been applied in the versatile Ugi and Passerini reactions [1]. The great potential of the isocyanides for development of the multicomponent reactions lies in the diversity of bond-forming processes available, their functional group tolerance, and the high levels of chemo-, regio-, and stereoselectivity often observed [2–10]. The four-component condensation between the aldehydes, isocyanides, and ammonium formate affords *N*-substituted 2-formylaminocarboxamides [11]. The reaction between salicylaldehyde, isocyanides, and ammonium formate under Ugi four-component condensation conditions affords benzo[*b*]furan derivatives in low yields [12].

The furan derivatives, obtained from both synthetic and natural sources, have attracted much interest due to their wide pharmaceutical applications [13, 14]. Many furans from natural resources indicate interesting biological activities, such as the cytotoxic and antitumor properties [15] as well as antispasmodic [16], antimicrobial [17], and several other potentially useful activities [18].

As a continuation of our recent studies on isocyanide chemistry [19–22], we report the Ugi multicomponent reaction between 2-hydroxybenzaldehyde derivative **1**, a secondary amine **2**, and an isocyanide **3**.

2. Experimental

Starting materials and solvents were purchased from Merck (Germany) and Fluka (Switzerland) and were used without further purification. The reactions were monitored by TLC and NMR techniques, which indicated that there were no side products. IR spectra were measured on a Perkin-Elmer RXI, FT-IR spectrometer. ^{1}H and ^{13}C NMR spectra ($CDCl_3$) were recorded on a BrukerAvance spectrometer at 250.0 and 62.9 MHz, respectively. Elemental analyses were performed by using a Perkin-Elmer 2400(II) CHN/O analyzer. Mass spectra were recorded on a FINNIGAN-MATT 8430 mass spectrometer operating at an ionization potential of 20 eV. The TLC plates were prepared from Merck silica gel powder.

3. Synthesis of *N,N*-Dibenzyl-*N*-[5-nitro-2-(1,1,3,3-tetramethylbutylamino)-1-benzofuran-3-yl]amine (7a)

A mixture of dibenzylamine (0.19 mL, 1 mmol) and 2-hydroxy-5-nitrobenzaldehyde (0.167 g, 1 mmoL) (5 mL) was stirred in 5 mL water at room temperature for 60 minutes. To this mixture, 1,1,3,3-tetramethylbutyl isocyanide (0.17 mL, 1 mmoL) at 15°C was added rapidly, and the solution was allowed to stand for 24 h at room temperature. The solvent was removed under reduced pressure, and single-spot product (**7a**) was obtained. The mentioned product was purified through plate thin layer liquid chromatography PTLC method by using petroleum ether/diethyl ether (10 : 1) as eluent and red viscous oil was obtained. All other products (**7b–i**) were obtained by similar approach. The characterization data of the compounds are given below (**7a–i**).

*N,N-Dibenzyl-N-[5-nitro-2-(1,1,3,3-tetramethylbutylamino)-1-benzofuran-3-yl]amine, **7a***. Red viscous oil; yield: 98%. IR (KBr) (υ_{max}, cm^{-1}): 3400 (NH), 1646, 1530, 1476, 1346, 1223. ^{1}H NMR (250 MHz, $CDCl_3$) δ_H: 0.96 (9H, s, CMe_3), 1.06 (6H, s, CMe_2NH), 1.47 (2H, s, CH_2CMe_3), 4.20 (4H, s, $2CH_2$, benzyl), 4.10 (1H, s, NH, exchanged by D_2O addition), 7.20–7.39 (11H, m, H–Ar), 7.89 (1H, dd, $^3J_{HH}$ = 6.5 Hz, $^4J_{HH}$ = 2.3 Hz, H-4, benzofuran), 8.15 (1H, d, $^4J_{HH}$ = 2.3 Hz, H-6, benzofuran). ^{13}C NMR (62.9 MHz, $CDCl_3$) dC: 30.03 ($2CH_3$ of CMe_2NH), 31.55 ($3CH_3$, CMe_3), 31.64 (C, CMe_3), 54.21 (CH_2, CH_2CMe_3), 56.01 (C, CMe_2NH), 58.91 ($2CH_2$, benzyl), 109.87, 111.50, 128.74 (3CH, benzofuran), 103.64, 114.93, 151.95, 158.32 (4C, benzofuran), 143.91 ($C(NO_2)$), 127.34, 128.60, 129.18 (10CH), 138.96 ($2C_{ipso}$).

*N,N-Dibenzyl-N-[2-(cyclohexylamino)-5-nitro-1-benzofuran-3-yl]amine, **7b***. Red viscous oil; yield: 94%. IR (KBr) (υ_{max}, cm^{-1}): 3415 (NH), 2853, 1646, 1530, 1461, 1346, 1261. ^{1}H NMR (250 MHz, $CDCl_3$) δ_H: 1.10–1.81 (10H, 2m, $5CH_2$, cyclohexyl), 3.22 (1H, m, CH–N, cyclohexyl), 4.24 (4H, s, $2CH_2$, benzyl), 3.85 (1H, br s, NH, exchanged by D_2O addition), 7.20–7.38 (11H, m, H–Ar), 7.86 (1H, dd, $^3J_{HH}$ = 6.5 Hz, $^4J_{HH}$ = 2.3 Hz, H-4, benzofuran), 8.09 (1H, d, $^4J_{HH}$ = 2.3 Hz, H-6, benzofuran). ^{13}C NMR (62.9 MHz, $CDCl_3$) δ_C: 24.70 ($2CH_{2,\beta}$, cyclohexyl), 25.44 ($1CH_{2,\gamma}$, cyclohexyl), 33.87 ($2CH_{2,\alpha}$, cyclohexyl), 48.07 (CH–N, cyclohexyl), 58.70 ($2CH_2$, benzyl), 109.76, 111.68, 129.07 (3CH, benzofuran), 102.56, 115.05, 151.76, 157.68 (4C, benzofuran), 143.89 ($C(NO_2)$), 127.27, 128.25, 129.15 (10CH), 138.96 ($2C_{ipso}$).

*N-Benzyl-N-ethyl–N-[5-nitro-2-(1,1,3,3-tetramethylbutylamino)-1-benzofuran-3-yl]amine, **7c***. Red viscous oil; yield: 70%. IR(KBr) (υ_{max}, cm^{-1}): 3479 (NH), 2775, 1638, 1551, 1504, 1374, 1289. ^{1}H NMR (250 MHz, $CDCl_3$) δ_H: 0.98 (9H, s, CMe_3), 1.24 (6H, s, CMe_2NH), 1.54 (2H, s, CH_2CMe_3), 1.59 (3H, CH_3, NCH_2CH_3), 3.22 (CH_2, NCH_2), 4.17 (2H, s, CH_2), 4.76 (1H, s, NH, exchanged by D_2O addition), 7.22–7.27 (6H, m, H–Ar), 7.87 (1H, m, benzofuran), 8.12 (1H, s, H-4, benzofuran). ^{13}C NMR (62.9 MHz, $CDCl_3$) δ_C: 29.86 (CH_3, NCH_2CH_3), 30.24 ($2CH_3$, CMe_2NH), 31.56 ($3CH_3$, CMe_3), 31.71 (C, CMe_3), 49.02 (CH_2, CH_2CMe_3), 54.35 (C, CMe_2NH), 56.43 (CH_2, benzyl), 59.73 (NCH_2), 109.95, 115.12, 144.5, 158.05 (4C, benzofuran), 111.46, 114.23, 127.40 (3CH, benzofuran), 152.3 ($C(NO_2)$), 126.95, 128.26, 128.71 (5CH), 128.24 (C_{ipso}).

*N-Benzyl-N-ethyl–N-[2-(cyclohexylamino)-5-nitro-1-benzofuran-3-yl]amine, **7d***. Red viscous oil; yield: 32%. IR(neat) (υ_{max}, cm^{-1}): 3418 (NH), 1633, 1505, 1455, 1393, 1299. ^{1}H NMR (250 MHz, $CDCl_3$) δ_H: 1.14–1.76 (10H, 2m, $5CH_2$, cyclohexyl), 2.64 (2H, s, NCH_2), 2.16 (3H, s, NCH_3), 3.24 (1H, m, CH–N, cyclohexyl), 4.71 (2H, s, CH_2, benzyl), 4.26 (1H, br s, NH, exchanged by D_2O addition), 7.24–7.34 (6H, m, H–Ar), 7.89 (1H, m, H-4, benzofuran), 8.04 (1H, H-6, benzofuran). ^{13}C NMR (62.9 MHz, $CDCl_3$) δ_C: 24.74–25.39 ($5CH_2$, cyclohexyl), 25.39 (CH_3, NCH_2CH_3), 33.00 (CH_2, NCH_2CH_3), 33.79 (CH–N, cyclohexyl), 52.38 (CH_2, benzyl), 110.01–129.09 (8CH, 6C).

*N-Benzyl-N-methyl–N-[5-nitro-2-(1,1,3,3-tetramethylbutylamino)-1-benzofuran-3-yl]amine, **7e***. Red viscous oil; yield: 95%. IR(KBr) (υ_{max}, cm^{-1}): 3479 (NH), 1626, 1551, 1478, 1358, 1292. ^{1}H NMR (250 MHz, $CDCl_3$) δ_H: 1.00 (9H, s, CMe_3), 1.301 (6H, s, CMe_2NH), 1.618 (2H, s, CH_2CMe_3), 2.86 (CH_3, NCH_3), 4.11 (2H, s, CH_2), 4.31 (1H, s, NH, exchanged by D_2O addition), 7.11–7.31 (6H, m, H–Ar), 7.89 (1H, dd, $^3J_{HH}$ = 6.8 Hz, $^4J_{HH}$ = 2.3 Hz, H-4, benzofuran), 8.17 (1H, d, $^4J_{HH}$ = 2.3 Hz, H-4, benzofuran). ^{13}C NMR (62.9 MHz, $CDCl_3$) δ_C: 30.35 ($2CH_3$, CMe_2NH), 31.58 ($3CH_3$, CMe_3), 31.58 (C, CMe_3), 54.30 (CH_2, CH_2CMe_3), 55.45 (C, CMe_2NH), 56.53 (CH_2, benzyl), 60.97 (NCH_3), 106.89, 115.14, 152.01, 157.25 (4C, benzofuran), 109.99, 111.90, 127.34 (3CH, benzofuran), 143.80 ($C(NO_2)$), 127.34, 128.29, 129.03 (5CH), 138.78 (C_{ipso}).

*N-Benzyl-N-methyl–N-[2-(cyclohexylamino)-5-nitro-1-benzofuran-3-yl]amine, **7f***. Red viscous oil; yield: 48%. IR(neat) (υ_{max}, cm^{-1}): 3444 (NH), 1632, 1556, 1483, 1341, 1298. ^{1}H NMR (250 MHz, $CDCl_3$) δ_H: 1.106–2.913 (10H, 2m, $5CH_2$, cyclohexyl), 3.113 (3H, s, NCH_3), 3.372 (1H, m, CH–N, cyclohexyl), 4.048 (1H, d, CH_2, benzyl), 4.584 (1H, d, CH_2, benzyl), 4.474 (1H, s, NH, exchanged by D_2O addition), 6.898–7.449 (5H, m, H–Ar), 7.897 (1H, dd, $^4J_{HH}$ = 2.3 Hz, $^5J_{HH}$ = 1 Hz, H-4, benzofuran), 8.176 (1H, dd, $^3J_{HH}$ = 6.8 Hz, $^4J_{HH}$ = 2.3 Hz, H-4, benzofuran), 8.645 (1H, dd, $^3J_{HH}$ = 6.8 Hz, $^4J_{HH}$ = 2.3Hz, H-4, benzofuran). ^{13}C NMR (62.9 MHz, $CDCl_3$) δ_C: 24.58 ($2CH_{2,\beta}$, cyclohexyl), 25.44 ($1CH_{2,\gamma}$, cyclohexyl), 33.87 ($2CH_{2,\alpha}$, cyclohexyl), 48.07 (CH–N, cyclohexyl), 53.77 (CH_3, NCH_3), 59.51 (CH_2, benzyl), 128.23–129.08 (5C, benzofuran), 129.9–129.34 (3CH, benzofuran), 129.97 ($C(NO_2)$), 127.14–128.11 (5CH).

*N,N-Dibenzyl-N-[5-bromo-2-(1,1,3,3-tetramethylbutylamino)-1-benzofuran-3-yl]amine, **7g***. Yellow viscous oil; yield: 95%. IR(neat) (υ_{max}, cm^{-1}): 3423 (NH), 2980, 1642, 1462, 1365, 1225. ^{1}H NMR (250 MHz, $CDCl_3$) δ_H: 0.96 (9H, s, CMe_3), 1.05 (6H, s, CMe_2NH), 1.44 (2H, s, CH_2CMe_3), 3.96 (1H, s, NH, exchanged by D_2O addition), 4.14 (4H, s, CH_2, benzyl),

Table 1: Condition and yield of reactions for synthesis of benzo[*b*]furan derivatives, **7a–i**, in water.

Entry	Products	R′	R	X	Yield (%)
1	**7a**	1,1,3,3-Tetramethylbutyl	Benzyl	NO_2	90
2	**7b**	Cyclohexyl	Benzyl	NO_2	98
3	**7c**	1,1,3,3-Tetramethylbutyl	Ethyl	NO_2	81
4	**7d**	Cyclohexyl	Ethyl	NO_2	89
5	**7e**	1,1,3,3-Tetramethylbutyl	Methyl	NO_2	42
6	**7f**	Cyclohexyl	Methyl	NO_2	90
7	**7g**	1,1,3,3-Tetramethylbutyl	Benzyl	Br	48
8	**7h**	Cyclohexyl	Benzyl	Br	91
9	**7i**	1,1,3,3-Tetramethylbutyl	Ethyl	Br	80

7.05–7.49 (13H, m, H–Ar). ^{13}C NMR (62.9 MHz, $CDCl_3$) δ_C: 30.00 ($2CH_3$, CMe_2NH), 31.58 ($3CH_3$, CMe_3), 31.64 (C, CMe_3), 54.32 (CH_2, CH_2CMe_3), 55.83 (C, CMe_2NH), 58.79 ($2CH_2$, benzyl), 118.70, 121.11, 130.01 (3CH, benzofuran), 103.74, 115.48, 147.74, 156.78 (4 C, benzofuran), 111.44 (C(Br)), 127.18, 128.23, 129.21 (10 CH), 139.31 ($2C_{ipso}$).

N,N-Dibenzyl-N-[5-bromo-2-(cyclohexylamino)-1-benzofuran-3-yl]amine, ***7h***. Yellow viscous oil; yield: 90%. IR(neat) (υ_{max}, cm^{-1}): 3412 (NH), 2936, 1631, 1522, 1455, 1364, 1267. ^{1}H NMR (250 MHz, $CDCl_3$) δ_H: 0.8–1.81 (10H, 2m, $5CH_2$, cyclohexyl), 3.15 (1H, m, CH–N, cyclohexyl), 4.14 (4H, s, $2CH_2$, benzyl), 3.73 (1H, br s, NH, exchanged by D_2O addition), 7.03–7.50 (13H, m, H–Ar). ^{13}C NMR (62.9 MHz, $CDCl_3$) δ_C: 24.79 ($2CH_{2,\beta}$, cyclohexyl), 25.54 ($1CH_{2,\gamma}$, cyclohexyl), 33.90 ($2CH_{2,\alpha}$ of cyclohexyl), 50.37 (CHNH, cyclohexyl), 58.61 ($2CH_2$, benzyl), 118.90, 121.27, 128.79 (3CH, benzofuran), 102.67, 115.51, 147.54, 156.22 (4C, benzofuran), 111.35 (C(Br)), 127.13, 128.20, 129.13 (10CH), 139.31 ($2C_{ipso}$).

N-Benzyl-N-ethyl–N-[5-bromo-2-(1,1,3,3-tetramethylbutylamino)-1-benzofuran-3-yl]amine, ***7i***. Yellow viscous oil; yield: 68%. IR(neat) (υ_{max}, cm^{-1}): 3501 (NH), 2999, 1668, 1565, 1465, 1374, 1238. ^{1}H NMR (250 MHz, $CDCl_3$) δ_H: 1.03 (9H, s, CMe_3), 1.21 (6H, s, CMe_2NH), 1.45 (2H, s, CH_2CMe_3), 2.17 (3H, s, NCH_2CH_3), 3.95 (1H, s, NH, exchanged by D_2O addition), 4.16 (2H, CH_2, NCH_2), 5.29 (2H, s, CH_2, benzyl), 7.04–7.6 (5H, m, H–Ar), 7.77, 7.8, 7.86 (3H, d, benzofuran). ^{13}C NMR (62.9 MHz, $CDCl_3$) δ_C: 30.96 ($2CH_3$, CMe_2NH), 31.43 ($3CH_3$, CMe_3), 31.52 (C, CMe_3), 41.96 (CH_3, NCH_2CH_3), 50.67 (CH_2, CH_2CMe_3), 53.38 (CH_2, NCH_2), 55.26 (C, CMe_2NH), 60.39 (CH_2, benzyl), 111.22, 115.00, 116.91, 118.87, 121.12 (5CH), 123.12(C), 127.39, 127.64, 128.16 (3CH, benzofuran), 130.15 (C(Br)), 131.21, 135.42 (2C, benzofuran), 140.41, 141.18 ($2C_{ipso}$).

4. Results and Discussion

Here, we report a simple, one-pot, four-component reaction between electron-poor 2-hydroxybenzaldehyde derivative **1**, secondary amines **2**, and isocyanides **4**, in water at room temperature, leading to benzo[*b*]furan derivatives **7** (Figure 1 and Table 1). The reaction proceeds smoothly and cleanly under mild conditions in water and is therefore considered to be a green chemistry method. The structures of the products were deduced from elemental analyses, IR, ^{1}H NMR, and ^{13}C NMR spectra.

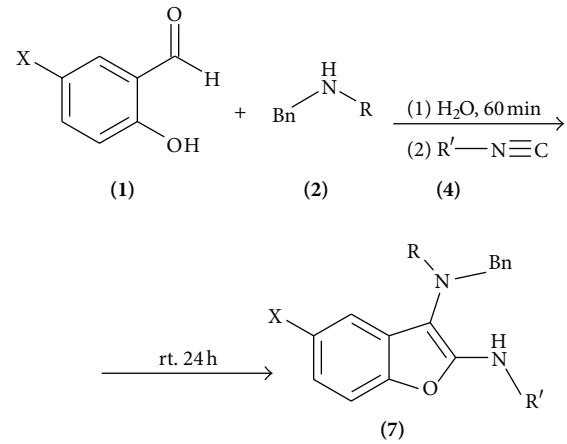

Figure 1: Four-component synthesis of benzo[*b*]furan derivatives, **7a–i**, in water.

We also used *N*-benzyl-*tert*-butylamine in above reaction, but the yields of the corresponding products **7** were very low, and several by-products were observed. As indicated in Table 1, the reactions proceeded efficiently with electron-withdrawing 2-hydroxybenzaldehyde derivatives **1**, while electron-releasing 2-hydroxybenzaldehyde derivatives are not suitable starting materials in these reactions. The high yields of **7a–i** can be explained by the greater electrophilicity of carbonyl groups of electron-withdrawing 2-hydroxybenzaldehyde derivatives relative to the carbonyl groups of electron-releasing 2-hydroxybenzaldehyde derivatives. 1,1,3,3-Tetramethylbutyl isocyanide leads to decreasing the yield of **7** due to steric effects.

Although we have not established the mechanism of the reaction in an experimental manner, a plausible reaction sequence that accounts for the formation of **7** was shown in Figure 2. Thus, the condensation of 2-hydroxybenzaldehyde derivative **1** and secondary amine **2** gives an iminium ion intermediate **3**, which is then attacked by the alkyl isocyanide **4** to afford intermediate **5**. The cyclization of the ionic

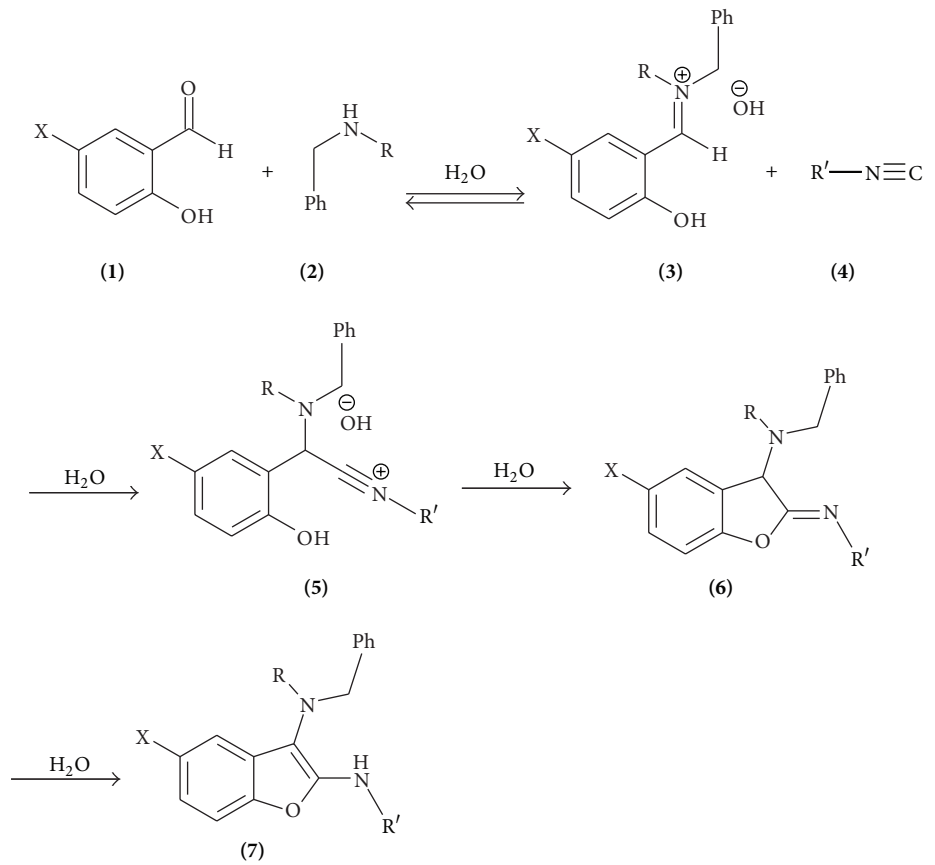

Figure 2: Proposed mechanism for the formation of benzo[*b*]furan derivatives, **7a-i**, in water.

intermediate **5** leads to the benzofuran **6**. Tautomerization of **6** could then lead to formation of the benzo[*b*]furan derivatives **7**.

5. Conclusion

In this work, we have developed a mild and efficient protocol for the preparation of benzo[*b*]furan derivatives. Our method offers several advantages over existing methods, including good yields, cleaner reactions, and simple workup which make it a useful and environmentally attractive strategy for the synthesis of benzo[*b*]furan derivatives, with promising bioactivity.

Conflict of Interests

The authors have no conflict of interests with any trademark mentioned in the paper.

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