RESEARCH LETTER

Ionic liquid [omim][NO3], a green medium for room-temperature synthesis of benzothiazinone derivatives in one pot

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An efficient one-pot procedure is developed at room temperature for the reaction of 2-aminothiophenols with 2-bromoalkanoates in ionic liquid (IL) [omim][NO3], providing the corresponding benzothiazin-3-one derivatives in high yields without using any other base or additive. Products are easily obtained by simple extraction of the reaction mixtures with Et2O. In most cases, evaporation of the volatile contents leads to precipitation of the products and therefore the use of chromatographic separations is avoided. The IL is then recovered and reused in next reactions without loosing its activity. The procedure is successfully extended to the synthesis of the respective 2-carboxylate derivatives via the reaction of 2-aminothiophenols with 2-bromomalonates.

Keywords: green synthesis; 1,4-benzothiazin-3-ones; ionic liquids

Introduction

In line with global enforcements for more severe environmental regulations, use of ionic liquids (ILs) as green and reusable media in various fields of chemistry has dramatically increased in the new millennium (1–3). Particularly, tailoring ILs with appropriate physical and chemical properties is of great prominence in today’s synthetic organic chemistry to enhance the selectivity and reactivity of various transformations (4–7). As a result, ILs are now considered as very promising benign surrogates to classic organic solvents since they possess very low vapor pressure, are often recoverable, show high thermal stability, dissolve a wide range of organic reactants, and can be stored for relatively long periods of time (8–10).

The 2H-benzo[b][1,4]thiazin-3(4H)-one (e.g. 3) derivatives are considered as an important group of heterocycles since they exhibit key medicinal (11–13), biological (14, 15), industrial (16–18), and agricultural (19) activities. Consequently, extensive investigations have been devoted by synthetic chemists to offer general methods for the synthesis of these structural motifs. One of the existing procedures goes through a multi-step substitution-reduction pathway which involves the addition of a thiol acetate to halonitrobenzenes followed by the reduction of the nitro group and a final annulation step (20–22). Another common method is substitution of the chlorine atom in 2-chloroanilines with sodium sulfide followed by annulation of the aminothiophenol intermediate with derivatives of chloroacetic acid (23). Other procedures include the annulation of o-aminothiophenols or their corresponding disulfides with α-substituted carbonyl compounds (24–29) and Smiles rearrangement. The latter involves either the one-pot combination of 2-chlorothiophenols with chloroacetyl chlorides and amines (30, 31) or ring expansion of smaller heterocycles into the benzothiazinone skeleton (32–34). However, many of these procedures require more than one-step reactions, demand high temperature treatment of the starting materials, give low or moderate yields of products, or need reactants or reagents which are not commercially available. In continuation of our investigations on heterocyclic systems, and on the basis of our previous studies on the development of environmentally clean procedures (35–38), we recently reported an IL mediated procedure for one-pot chemoselective synthesis of benzoxazin-3-one derivatives from their corresponding 2-aminophenols using DBU or K2CO3 (39,40), where the IL could be recovered and reused over several recycles without loosing its performance. On this track, we now report the base-free application of the results to [omim][NO3] mediated cyclization of 2-aminothiophenols 1 with 2-bromoalkanoates 2 in the presence of no extra additive (Scheme 1). The procedure takes place under mild conditions in one pot, reactions complete at room temperature, a broad...
range of starting materials can take part in the procedure, and the IL is recycled and reused efficiently in next reactions.

Results and discussion

We first optimized the conditions for the reaction between 2-aminothiophenol 1a with ethyl 2-bromoacetate 2a under different conditions. On the basis of our previous experience (40), we used carbonate bases to initiate the reactions. As a result, K₂CO₃ had the best performance causing about 35% conversion of the reactants to product 3a in [omim][BF₄], while Na₂CO₃ and NaHCO₃ led to yields lower than 10%. In the absence of any IL, no product was obtained after a long reaction time proving the effect of the IL in catalyzing the reaction. Addition of water to the reaction mixture enhanced the outcome and increased the yield of 3a to 98% within 24 hr. Surprisingly, in the absence of water or any base, [omim][NO₃] led to the same yield after only 2 hr (Table 1, entry 1). Therefore, we applied the optimum result to other reactants to evaluate the generality of the reaction. As a result, when different 2-aminothiophenols bearing electron withdrawing- (entries 2-9) and electron-releasing substituents (entries 10-13) were employed to annulate with various 2-bromoalkanoates, high yields of the respective products were obtained.

With these results in hand, we decided to further evaluate the synthetic scope of the procedure by applying the conditions to the reactions of 2-aminothiophenols with different 2-bromo-2-alkylmalonates 4a-c, as summarized in Table 2. Under the same conditions ([omim][NO₃] only), 2-aminothiophenol 1a reacted with 4a to rapidly produce ethyl 3-oxobenzothiazine-2-carboxylate 5a in high yield (Entry 1). Similarly, reactions of 1a with 4b and 4c gave high yields of 5b and 5c, respectively (entries 2–3). This was also the case for the reactions of 1b, 1c, and 1e (X = Cl, Y = H) leading to efficient formation of the respective products (entries 4–8). Due to higher reactivity of the malonate species, reactions proceeded rapidly in all cases and completed within 1–2 hr.

In all the reactions discussed above, annulation of the reactants took place in one pot and at ambient temperature. Reactions were fast and products were obtained by extraction by ether. In most cases, concentration of the extract under reduced pressure led to precipitation of the products which were purified by recrystallization from ether. Moreover, this allowed the recovery of [omim][NO₃] and its reuse in next reactions without noticeable loss of activity as shown in five consecutive reactions of 1a with 4a (Figure 1).

Experimental

Reactions were monitored by TLC using silica gel coated plates and EtOAc/hexane solutions as the mobile phase. Melting points are uncorrected. FT-IR spectra were recorded using KBr disks on a Bruker Vector-22 infrared spectrometer and absorptions are reported as wave numbers (cm⁻¹). ¹H NMR (500 MHz) and ¹³C NMR (125 MHz) spectra were obtained on a FT-NMR Bruker Ultra Shield instrument as CDCl₃ solutions and the chemical shifts are expressed as δ units with Me₄Si as the internal standard. Mass spectra were obtained on a Finnigan Mat 8430 apparatus at ionization potential of 70 eV. Elemental analyses were performed using a Thermo Finnigan Flash EA 1112 instrument. ILs were prepared using available literature (41–43). The IL was recovered by removing its volatile contents (under reduced pressure at 50°C for 1 hr). Reagents were purchased from commercial sources and were freshly used after being purified by standard procedures. The identity of the known products was confirmed by the comparison of their melting points and their NMR data with those available in the literature. New products were characterized by their ¹H NMR, ¹³C NMR, IR and mass spectra and their purity was confirmed by elemental analyses.

A typical procedure

A mixture of 1a (1 mmol) and 4a (1 mmol) in [omim][NO₃] (218 μL, 1 mmol) was stirred in a flask...
Table 1. [Omm][NO₃] catalyzed synthesis of products 3.

| Entry | Reactants | Product | Time (hr) | Yield (%)a |
|-------|-----------|---------|-----------|------------|
| 1     | 1a + 2a   | 3a      | 2         | 98         |
| 2     | 1b + 2a   | 3b      | 4         | 93         |
| 3     | 1b + 2b   | 3c      | 4         | 87         |
| 4     | 1b + 2c   | 3d      | 4         | 85         |
| 5     | 1b + 2d   | 3e      | 6         | 85         |
| 6     | 1b + 2e   | 3f      | 48        | 83         |
| 7     | 1c + 2a   | 3g      | 24        | 95         |
| 8     | 1c + 2b   | 3h      | 30        | 92         |
| 9     | 1c + 2e   | 3i      | 48        | 88         |
| 10    | 1d + 2c   | 3k      | 3         | 88         |
| 11    | 1d + 2d   | 3l      | 3         | 88         |
| 12    | 1e + 2a   | 3m      | 0.25      | 90         |
| 13    | 1e + 2e   | 3n      | 4         | 80         |

at room temperature for 1 hr. After completion of the reaction, based on TLC monitoring, the reaction mixture was extracted with diethyl ether. The organic phase was washed with brine, dried over anhydrous Na₂SO₄ and concentrated under reduced pressure to obtain the solid product 5a which was further purified by recrystallization from ether. The IL was recovered by evaporating its ether content and reused in the following reaction.

**Spectral data of new compounds**

7-Fluoro-2-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (3c)

White solid; mp 170–173°C; ¹H NMR (500 MHz, CDCl₃) δ 9.88 (br s, NH), 6.92 (dd, J = 8.0, 2.5 Hz, 1H), 6.86 (dd, J = 9.0, 5.0 Hz, 1H), 6.77 (ddd, J = 9.0, 8.0, 2.5 Hz, 1H), 3.41 (q, J = 7.0 Hz, 1H), 1.39 (d, J = 7.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 168.4, 159.5, 157.6, 133.4, 133.1, 121.1, 121.1, 118.5, 118.5, 114.8, 114.6, 114.3, 114.1, 37.0, 15.7; IR (KBr) 3190, 3076, 2953, 1853, 1670, 1490, 831, 601 cm⁻¹; MS 197, 154, 138, 114, 59; Elemental analyses calcld for C₉H₈FNOS: C%, 54.81; H%, 4.09; N%, 7.10. Found: C%, 54.67; H%, 4.15; N%, 7.31%.

2-Ethyl-7-fluoro-2H-benzo[b][1,4]thiazin-3(4H)-one (3d)

White solid; mp 127–128°C; ¹H NMR (500 MHz, CDCl₃) δ 9.98 (br s, NH), 7.07 (dd, J = 8.3, 2.5 Hz, 1H), 6.94 (dd, J = 8.7, 5.0 Hz, 1H), 6.90 (ddd, J = 8.7, 7.9, 2.7 Hz, 1H), 3.35 (dd, J = 8.8, 5.9 Hz, 1H), 2.02–1.93 (m, 1H), 1.71–1.62 (m, 1H), 1.10 (t, J = 7.3 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 168.9, 160.0, 158.1, 132.8, 132.8, 120.7, 120.6, 118.5, 115.8, 115.4, 115.2, 114.5, 44.7, 23.7, 11.7; IR (KBr) 3304, 3199, 3084, 2970, 1859, 1856, 1490, 848, 798 cm⁻¹; MS 211, 182, 154, 138, 114, 83, 69, 27; Elemental analyses calcld for C₁₂H₁₀FNOS: C%, 56.85; H%, 4.77; N%, 6.63. Found: C%, 56.57; H%, 4.92; N%, 6.88%.

7-Fluoro-2-propyl-2H-benzo[b][1,4]thiazin-3(4H)-one (3e)

White solid; mp 143–145°C; ¹H NMR (500 MHz, CDCl₃) δ 9.41 (br s, NH), 7.07 (dd, J = 8.3, 2.2 Hz, 1H), 6.93 (dd, J = 8.7, 5.0 Hz, 1H), 6.90 (ddd, J = 8.7, 7.9, 2.7 Hz, 1H), 3.34 (dd, J = 6.0, 2.5 Hz, 1H), 1.93–1.88 (m, 1H), 1.65–1.58 (m, 2H), 1.49–1.46 (m, 1H), 0.96 (t, J = 7.2 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 168.7, 160.0, 158.1, 132.7, 132.1, 120.9, 120.8, 118.3, 118.3, 115.5, 115.3, 114.5, 42.7, 32.2, 20.3, 13.8; IR (KBr) 3282, 3068, 2860, 1865, 1666, 1668, 1487, 798, 603 cm⁻¹; MS 225, 183, 154, 150, 127, 83, 55, 27.4.

*Isolated yields.*
Elemental analyses calcd for C$_{11}$H$_{12}$FNOS:
\%C, 58.65; \%H, 5.37; \%N, 6.22.
Found: \%C, 58.97; \%H, 5.58; \%N, 6.31%.

7-Fluoro-2,2-dimethyl-2H-benzo[b][1,4]thiazin-3(4H)-one (3f)
White solid: mp 143–145°C; \(^1\)H NMR (500 MHz, CDCl$_3$) \(\delta\) 8.55 (br s, NH), 7.06 (dd, \(J\) = 8.2, 2.7 Hz, 1H), 6.92 (ddd, \(J\) = 8.7, 8.2, 2.7 Hz, 1H), 6.86 (ddd, \(J\) = 8.7, 4.8 Hz, 1H), 1.51 (s, 6H); \(^13\)C NMR (125 MHz, CDCl$_3$) \(\delta\) 171.1, 159.9, 157.9, 132.9, 132.3, 122.0, 121.9, 117.9, 117.9, 115.1, 114.9, 114.5, 114.3, 43.0, 24.6; IR (KBr) 3194, 2974, 1664, 1492, 1251, 854, 601 cm$^{-1}$; MS 211, 196, 168, 127, 114, 83, 69, 39; Elemental analyses calcd for C$_{10}$H$_{10}$FNOS: \%C, 56.85; \%H, 4.77; \%N, 6.63. Found: \%C, 56.52; \%H, 4.49; \%N, 6.74%.

2-Ethyl-7-methyl-2H-benzo[b][1,4]thiazin-3(4H)-one (3k)
White solid: mp 160–162°C; \(^1\)H NMR (500 MHz, CDCl$_3$) \(\delta\) 8.55 (br s, NH), 7.15 (s, 1H), 6.99 (dd, \(J\) = 8.1, 1.1 Hz, 1H), 6.78 (d, \(J\) = 8.0 Hz, 1H), 3.34 (dd, \(J\) = 8.8, 5.9 Hz, 1H), 2.32 (s, 3H), 1.96 (ddq, 8.0 Hz, 1H), 1.49 (s, 6H); \(^13\)C NMR (125 MHz, CDCl$_3$) \(\delta\) 172.1, 159.9, 157.9, 132.9, 132.3, 122.0, 121.9, 115.1, 114.9, 114.5, 114.3, 103.0, 24.6; IR (KBr) 3194, 2974, 1664, 1492, 1251, 854, 601 cm$^{-1}$; MS 211, 196, 168, 127, 114, 83, 69, 39, 21.

Table 2. [Omim][NO$_3$] catalyzed synthesis of products 5.

| Entry | Reactant 1 | Reactant 4 | Product | Yield (%)$^a$ |
|-------|------------|------------|---------|--------------|
| 1     | 1a         | 4a         | 5a      | 90           |
| 2     | 1a         | 4b         | 5b      | 93           |
| 3     | 1a         | 4c         | 5c      | 88           |
| 4     | 1b         | 4a         | 5d      | 96           |
| 5     | 1b         | 4b         | 5e      | 95           |
| 6     | 1b         | 4c         | 5f      | 95           |
| 7     | 1c         | 4b         | 5g      | 95           |
| 8     | 1e         | 4b         | 5h      | 93           |

$^a$Isolated yields.

Elemental analyses calcd for C$_{10}$H$_{10}$FNOS: \%C, 56.85; \%H, 4.77; \%N, 6.63. Found: \%C, 56.52; \%H, 4.49; \%N, 6.74%.
7-Methyl-2-propyl-2H-benzo[b][1,4]thiazin-3(4H)-one (3f)

White solid: mp 127–130°C; 1H NMR (500 MHz, CDCl3) δ 8.92 (br s, NH), 7.14 (s, 1H), 6.99 (dd, J = 7.9, 1.0 Hz, 1H), 6.80 (d, J = 8.0 Hz, 1H), 3.43 (dd, J = 8.6, 6.0 Hz, 1H), 2.32 (s, 3H), 2.00–1.93 (m, 1H), 1.70–1.64 (m, 2H), 1.50–1.45 (m, 1H), 0.95 (t, J = 7.2 Hz, 3H); 13C NMR (125 MHz, CDCl3) δ 168.7, 134.0, 133.9, 129.0, 128.1, 118.8, 117.1, 66.2, 42.9, 32.2, 20.3, 13.9; IR (KBr) 3196, 3074, 2960, 1867, 1662, 1496, 804 cm⁻¹; MS 221, 178, 150, 134, 109, 77, 69, 41, 27; Elemental analyses calcld for C13H15NOS: %C, 65.12; %H, 6.83; %N, 6.33. Found: %C, 65.23; %H, 6.74; %N, 6.55.

Ethyl 2-ethyl-3-oxo-3,4-dihydro-2H-benzo[b][1,4]thiazin-2-carboxylate (5e)

White solid: mp 110–115°C; 1H NMR (500 MHz, CDCl3) δ 9.19 (br s, NH), 7.10 (dd, J = 8.2, 2.4 Hz, 1H), 6.94 (dd, J = 8.7, 2.4 Hz, 1H), 6.91 (dd, J = 8.7, 8.6, 1.4 Hz, 1H), 4.19–4.09 (m, 2H), 2.30–2.21 (m, 2H), 1.16 (t, J = 7.3 Hz, 3H), 1.11 (t, J = 6.3 Hz, 3H); 13C NMR (125 MHz, CDCl3) δ 168.9, 166.2, 136.8, 128.1, 127.6, 124.3, 120.2, 117.9, 62.9, 56.4, 26.1, 14.2, 10.8; IR (KBr) cm⁻¹ 3446, 3199, 2983, 1739, 1668, 1236, 746. El-MS (m/z) 265 [M⁺]; 185, 131, 96; Elemental analyses calcld for C13H15NO3S: %C, 58.85; %H, 5.70; %N, 5.28. Found: %C, 58.66; %H, 5.67; %N, 5.52.

Ethyl 7-fluoro-3-oxo-3,4-dihydro-2H-benzo[b][1,4]thiazin-2-carboxylate (5d)

White solid: mp 154–156°C; 1H NMR (500 MHz, CDCl3) δ 9.40 (br s, NH), 7.25 (d, J = 8.2 Hz, 1H), 7.03 (d, J = 2.0 Hz, 1H), 7.00 (dd, J = 10.0, 2.0 Hz, 1H), 4.19–4.14 (m, 1H), 4.11–4.06 (m, 1H), 1.83 (s, 3H), 1.09 (t, J = 7.1 Hz, 3H); 13C NMR (125 MHz, CDCl3) δ 169.6, 167.3, 137.7, 133.6, 128.8, 124.2, 117.5, 117.2, 62.9, 50.1, 19.6, 14.2; IR (KBr) 3199, 3116, 2981, 1720, 1674, 1469, 1251, 854, 742 cm⁻¹; MS 285, 212, 184, 143, 108, 95, 69, 43, 29; Elemental analyses calcld for C13H13FNO3S: %C, 50.44; %H, 4.23; %N, 4.90. Found: %C, 50.37; %H, 4.35; %N, 4.92.

Conclusions

We believe that the present work provides a very efficient pathway for the synthesis of various 1,4-benzothiazin-3-one derivatives since it incorpo-
rates one-pot cyclization of 2-aminothiophenols with 2-bromoalkanotes of different steric and electronic nature. In addition, the process is chemoselective and occurs at room temperature, reactions are fast, products are obtained in high yields, and the medium can be recycled efficiently.

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