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A rapid synthesis of functionalized dimethylaminothiazoles from reaction of α-bromoketones with thioamide derivatives of tetramethyl guanidine in an ionic liquid

Ashraf Sadat Shahvelayati*

Department of Chemistry, Shahr-e Ray Branch, Islamic Azad University, PO Box 18155/144, Tehran, Iran

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Abstract: An efficient and rapid synthesis of dimethylaminothiazole *via* the reaction of ethyl bromopyruvate or phenacyl bromides with 2-(amidosulfanylenemethyl)-1,1,3,3-tetramethylguanidines is described in an ionic liquid at room temperature. The method is simple, rapid, and practical and the products are generated in good yields.

Keywords: Dimethylaminothiazole, Tetramethylguanidine, Ionic liquid, Ethyl bromopyruvate, Phenacyl bromide.

Introduction

Thiazoles occupy a prominent position among heterocycles. A large number of thiazoles obtained from microbial and marine origins exhibit important biological effects such as antitumor, antifungal, antibiotic, and antiviral activities [1,2]. Aminothiazoles are known to be ligands of estrogen receptors [3] as well as a novel class of adenosine receptor antagonists [4]. Other analogues are used as fungicides, inhibiting in vivo growth of Xanthomonas, as an ingredient of herbicides or as schistosomicidal and anthelmintic drugs. For example Fanetizole, a derivative of 2-aminothiazole, is an anti-inflammatory agent [5].

Synthetic thiazoles have also been shown to exhibit a wide variety of biological activities [6]. Several methods for the synthesis of thiazole derivatives have been developed. The classical method for the synthesis of thiazoles is the *Hantzsch* process, in which α-haloketone is condensed with a thioamide [7-9].

In the last few years there has been an increasing interest in applying ionic liquids (ILs) as the reaction media for a variety of chemical reactions [10-12].

Ionic liquids, combining an organic cation (e. g., 1-butyl-3-methylimidazolium cation) with a complex counter ion (e. g., Br^- or BF_4^-), are chemically inert, stable and non-volatile liquids.

Results and discussion

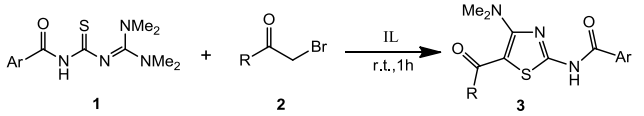
As part of our extensive program on the development of new routes in heterocyclic compounds synthesis [13-15], we report an efficient procedure for synthesis of dimethylaminothiazoles **3a-3j** from the reaction of 2-(amidosulfanylenemethyl)-1,1,3,3-tetramethyl guanidines with α-bromocarbonyl compounds in [bmim][Br] as a new solvent (Scheme **1**).

1-Butyl-3-methylimidazolium bromide abbreviated as [bmim][Br] is liquid at room temperature and is soluble in water, thus it is considered as a prospective environmentally friendly reaction medium.

The structures of compounds **3a-3j** were deduced from their IR, ^{1}H NMR, and ^{13}C NMR. The mass spectra of these compounds displayed molecular ion peaks at appropriate *m/z* values. The ^{1}H NMR spectrum of **3a** in $CDCl_3$ showed two singlets for methyl (δ = 3.16) and NH (δ = 9.90) protons, a triplet and a quartet (δ = 1.38, δ = 4.38) for ethyl group, along with characteristic signals phenyl group. The carbonyl

*Corresponding author. Tel: (+98) 2155229321, Fax: (+98) 2155229283, E-mail: avelayati@yahoo.com

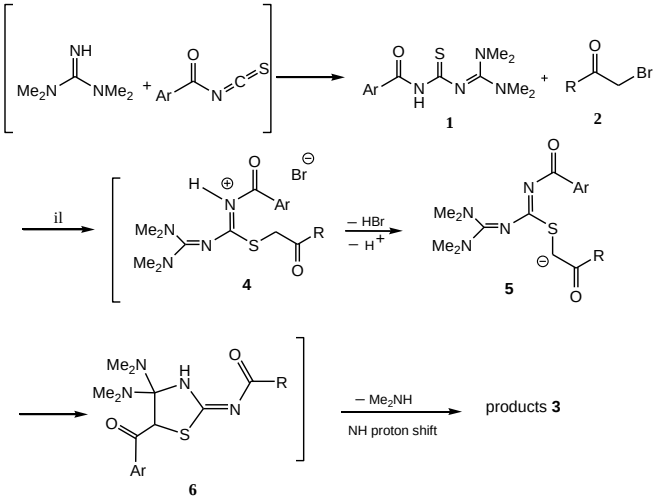
group resonances in the ^{13}C NMR spectra of **3a** appeared at 165.4, 166.0, and 170.5 ppm. The mass spectrum of **3a** displayed the molecular ion peak at *m/z* = 347. The ^{1}H NMR and ^{13}C NMR spectra of **3b**–**3j** were similar to those for **3a** except for the side chains, which exhibited characteristic resonances in the appropriate regions of the spectrum.



1,2	A	R'	Yiled (%) of 3
a	Ph	CO_2Et	90
b	Ph	4-BrC_6H_4	93
c	Ph	4-$MeOC_6H_4$	82
d	2-ClC_6H_4	CO_2Et	70
e	2-ClC_6H_4	4-BrC_6H_4	75
f	2-ClC_6H_4	4-$MeOC_6H_4$	72
g	4-$O_2NC_6H_4$	CO_2Et	70
h	4-MeC_6H_4	CO_2Et	92
i	4-MeC_6H_4	4-BrC_6H_4	90
j	4-MeC_6H_4	4-$MeOC_6H_4$	89

Scheme 1: Formation of compounds **3** in an ionic liquid

Mechanistically, 2-(amidosulfanylenemethyl)-1,1,3,3-tetramethylguanidine **1** , generated from the reaction of 1,1,3,3-tetramethylguanidine and aroylisothiocyanates, undergoes nucleophilic alkylation with *α*-bromoketones **2** to produce intermediate **4**. This intermediate undergoes HBr elimination and subsequent enolization to generate **5**, which undergoes intramolecular cyclization reaction to form the heterocyclic intermediate **6**. Subsequent NH proton shift and loss of dimethylamine affords functionalized dimethylaminothiazoles **3** (Scheme **2**).



Scheme 2: Proposed mechanism for the formation of compounds **3.**

Conclusion

In conclusion, we have described a convenient route to synthesis of functionalized dimethylaminothiazoles in [bmim][Br] as a new solvent in a short time. This catalyst-free synthetic method is facile and rapid; work up procedure is easy and gives pure target compounds.

Experimental

Chemicals and Apparatus:

2-(amido sulfanylene methyl)-1,1,3,3-tetramethyl guanidines were prepared from reaction of tetramethylguanidine and aroylisothiocyanates. bromopyruvate and phenacyl bromides were obtained from Fluka and were used without further purification, [bmim]Br was synthesized from the reaction of *N*-methylimidazole and *n*-butyl bromide [16]. Melting points were obtained uncorrected using an Electrothermal-9100 apparatus. IR spectra: Shimadzu IR-460 spectrometer, ^{1}H and ^{13}C NMR spectra: Bruker DRX-300AVANC instrument, in $CDCl_3$ at 300 MHz and 75 MHz, respectively, δ in ppm *J* in Hz, EI-MS (70 eV): Finningan-MAT-8430 mass spectrometer, in *m/z*. Elemental analyses (C, H, and N) were performed with a Heraeus CHN-O-Rapid analyzer.

*General procedure for the preparation of compounds **3:***

A mixture of substituted thioamide **1** (1mmol) and *α*-Bromo ketones **2** (1 mmol) was stirred in 1 ml of [bmim]Br for 1 h at r.t. Then 5 ml of water was added and the mixture was extracted with AcOEt (2 × 10 ml). The solvent was evaporated under reduced pressure to leave a residue that was purified by column chromatography (silica gel (230-400 mesh; *Merck*), hexane/AcOEt 3:1) to afford desired pure products **3**. Of course some products were precipitated directly after adding water and further purification was done by recrystallization from a mixture of *Et*OAc/*n*-hexane.

Spectroscopic data for compounds 3a-j:

*Ethyl 2-benzoylamino-4-dimethylamino-thiazol-5-yl-oxo-acetate **(3a)**:*

Orange powder, mp 177-178 ºC, yield: 0.31 g (90%). IR (KBr) (ν_{max}/cm^{-1}): 3437, 1724, 1676, 1616, 1542, 1307, 1226, 1098. ^{1}H NMR (300 MHz, $CDCl_3$): σ = 1.38 (3 H, t, ^{3}J = 7.1, Me), 3.16 (6 H, s, Me_2N), 4.38 (2 H, q, ^{3}J = 7.1, CH_2O), 7.52 (2 H, t, ^{3}J = 7.4, CH), 7.64 (1 H, t, ^{3}J = 7.4, CH), 8.01 (2 H, d, ^{3}J = 7.4, 2 CH), 9.90 (1 H, s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.5 (Me), 43.0 (Me_2N), 62.9 (CH_2O), 99.5 (C), 128.0 (2 CH), 129.5 (2 CH), 131.7 (CH), 133.8 (C), 164.1

(C), 165.2 (C), 165.4 (C=O), 166.0 (C=O), 170.5 (C=O). MS (EI, 70 eV): *m/z* (%) = 347 (M^+, 13), 332 (38), 302 (66), 242 (21), 105 (100), 77 (39), 45 (22). Anal. Calcd for $C_{16}H_{17}N_3O_4S$ (347.39): C, 55.32; H, 4.93; N, 12.10. Found: C, 55.71; H, 5.03; N, 11.87.

N-(5-(4-Bromobenzoyl)-4-dimethylamino-thiazol-2-yl)benzamide ***(3b):***

Yellow powder, mp 233-235 ºC, yield: 0.40 g (93%). IR (KBr) (ν_{max}/cm^{-1}): 3450, 1674, 1589, 1534, 1387, 1251, 1211, 1170. ^{1}H NMR (300 MHz, $CDCl_3$): σ = 3.15 (6 H, s, Me_2N), 7.56 (2 H, t, ^3J = 7.3, 2 CH), 7.60 (2 H, d, ^3J = 8.4, 2 CH), 7.65 (1 H, t, ^3J = 7.3, CH), 7.73 (2 H, d, ^3J = 8.4, 2 CH), 7.95 (2 H, d, ^3J = 7.3, 2 CH), 9.90 (1 H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 42.7 (Me_2N), 100.1 (C), 126.7 (C), 127.9 (2 CH), 129.5 (2 CH), 130.5 (2 CH), 131.5 (C), 132.0 (2 CH), 133.8 (C), 133.9 (C), 140.5 (C), 160.9 (C), 165.1 (C=O), 184.2 (C=O). MS (EI, 70 eV): *m/z* (%) = 431 (M^++2, 14), 429 (M^+, 13), 414 (33), 412 (30), 185 (11), 183 (11), 105 (100), 77 (43), 45 (9). Anal. Calcd for $C_{19}H_{16}BrN_3O_2S$ (430.32): C, 53.03; H, 3.75; N, 9.76. Found: C, 52.86; H, 3.83; N, 10.01.

N-(4-Dimethylamino-5-(4-methoxy-benzoyl)thiazol-2-yl)benzamide ***(3c)****:*

Yellow powder, mp 217-218ºC, yield: 0.31 g (82%). IR (KBr) (ν_{max}/cm^{-1}): 3445, 1679, 1591, 1520, 1367, 1221, 1157. ^{1}H NMR (300 MHz, $CDCl_3$): σ = 3.11 (6 H, s, Me_2N), 3.89 (3 H, s, OMe), 6.95 (2 H, d, ^{3}J = 8.7, 2 CH), 7.53 (2 H, d, ^{3}J = 7.8, 2 CH), 7.63 (1 H, d, ^{3}J = 7.8, CH), 7.86 (2 H, d, ^{3}J = 8.7, 2 CH), 7.94 (2 H, d, ^{3}J = 7.8, 2 CH), 9.10 (1 H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): 42.6 (Me_2N), 55.9 (OMe), 100.4 (C), 114.0 (2 CH), 127.9 (2 CH), 129.5 (2 CH), 131.6 (2 CH), 131.7 (C), 133.7 (C), 134.4 (C), 160.3 (C), 160.9 (C), 162.8 (C), 165.0 (C=O), 184.9 (C=O). MS (EI, 70 eV): *m/z* (%) = 381 (M^+, 12), 366 (41), 276 (35), 246 (12), 135 (22), 105 (100), 77 (37), 45 (8). Anal. Calcd for $C_{20}H_{19}N_3O_3S$ (381.45): C, 62.97; H, 5.02; N, 11.02. Found: C, 63.48; H, 5.61; N, 11.73.

Ethyl (2-(2-chloro-benzoylamino)-4-dimethylamino-thiazol-5-yl)oxoacetate ***(3d)***:

Orange powder, mp 153-155 ºC, yield: 0.27 g (70%). IR (KBr) (ν_{max}/cm^{-1}): 3439, 1723, 1624, 1545, 1394, 1307, 1232, 1116. ^{1}H NMR (300 MHz, $CDCl_3$): σ = 1.43 (3 H, t, ^{3}J = 7.1, Me), 3.18 (6 H, s, Me_2N), 4.40 (2 H, q, ^{3}J = 7.1, OCH_2), 7.41-7.92 (4 H, m, 4 CH), 9.85 (1 H, s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): 14.5 (Me), 43.0 (Me_2N), 63.0 (OCH_2), 99.4 (C), 128.0 (CH), 131.2 (CH), 131.5 (C), 131.7 (C), 131.9 (CH), 133.7 (CH), 164.0 (C), 164.2 (C), 164.3 (C=O), 165.7 (C=O), 170.5 (C=O). MS (EI, 70 eV): *m/z* (%) = 381 (M^+, 13), 366 (45), 336 (65), 242 (14), 139 (100), 111 (18), 45 (21). Anal. Calcd for $C_{16}H_{16}ClN_3O_4S$ (381.83): C, 50.33; H, 4.22; N, 11.01. Found: C, 51.04; H, 5.20; N, 11.72.

N-(5-(4-Bromo-benzoyl)-4-dimethylamino-thiazol-2-yl)-2-Chlorobenzamide ***(3e)***:

Yellow powder, mp 206-207 ºC, yield: 0.35 g (75%). IR (KBr): 3385, 1688, 1610, 1539, 1386, 1263, 1129. ^{1}H NMR (75 MHz, $CDCl_3$): 3.17 (6 H, s, Me_2N), 7.41-7.91 (4 H, m, 4 CH), 7.61 (2 H, d, ^{3}J = 8.4, 2 CH), 7.74 (2 H, d, ^{3}J = 8.4, 2 CH), 9.78 (1 H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): 42.3 (Me_2N), 100.5 (C), 126.5 (C), 127.7 (CH), 128.0 (C), 130.4 (2 CH), 131.5 (CH), 132.0 (2 CH), 132.8 (CH), 132.9 (C), 133.5 (C), 140.7 (C), 162.5 (C), 164.1 (C), 166.4 (C=O), 184.2 (C=O). MS (EI, 70 eV): *m/z* (%) = 465 (M^++2, 16), 463 (M^+, 12), 448 (45), 450 (34), 185 (21), 183 (21), 139 (100), 43 (9). Anal. Calcd for $C_{19}H_{15}BrClN_3O_2S$ (464.76): C, 49.04; H, 3.25; N, 9.04. Found: C, 49.82; H, 4.02; N, 9.62.

N-[4-Dimethylamino-5-(4-methoxybenzoyl)thiazol-2-yl]-2-Chloro-benzamide ***(3f)****:*

Yellow powder, mp 203-205 ºC, yield: 0.30 g (72%). IR (KBr) (ν_{max}/cm^{-1}): 3410, 1679, 1608, 1541, 1391, 1255, 1117. ^{1}H NMR (300 MHz, $CDCl_3$): σ = 3.13 (6 H, s, Me_2N), 3.89 (3 H, s, MeO), 6.96 (2 H, d, ^3J = 8.7, 2 CH), 7.41-7.86 (4 H, m, 4 CH), 7.87 (2 H, d, ^3J = 8.7, 2 CH), 9.79 (1 H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 42.5 (Me_2N), 55.8 (MeO), 100.5 (C), 113.9 (2 CH), 127.9 (CH), 131.2 (2 CH), 131.5 (CH), 132.0 (CH), 132.6 (C), 133.4 (CH), 133.5 (CH), 134.4 (C), 159.6 (C), 161.8 (C), 162.8 (C), 164.1 (C=O), 184.9 (C=O). MS (EI, 70 eV): *m/z* (%) = 417 (M^++2, 8), 415 (M^+, 20), 400 (48), 304 (33), 276 (24), 139 (100), 135 (19), 43 (10). Anal. Calcd for $C_{20}H_{18}ClN_3O_3S$ (415.89): C, 57.76; H, 4.36; N, 10.10. Found: C, 58.05; H, 4.43; N, 10.29.

Ethyl (4-dimethylamino-2-(4-nitro-benzoylamino)-thiazol-5-yl)oxoacetate ***(3g)****:*

Viscous oil, yield: 0.27 g (70%). IR (KBr) (ν_{max}/cm^{-1}): 3460, 1729, 1682, 1621, 1562, 1317, 1216, 1078. ^{1}H NMR (300 MHz, $CDCl_3$): σ = 1.40 (3 H, t, ^{3}J = 7.2, Me), 3.19 (6 H, s, Me_2N), 4.40 (2 H, q, ^{3}J = 7.2, OCH_2), 8.23 (2 H, d, ^{3}J = 8.7, 2 CH), 8.41 (2 H, d, ^{3}J = 8.7, 2 CH), 10.21 (1 H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): 14.5 (Me), 43.1 (Me_2N), 63.1 (OCH_2), 100.1 (C), 124.6 (2 CH), 129.4 (2 CH), 132.1 (C), 137.0 (C),

151.1 (C), 163.5 (C), 164.0 (C=O), 164.7 (C=O), 170.6 (C=O). MS (EI, 70 eV): *m/z* (%) = 392 (M^+, 21), 377 (39), 347 (59), 242 (24), 150 (100), 122 (16), 45 (23). Anal. Calcd for $C_{16}H_{16}N_4O_6S$ (392.39): C, 48.97; H, 4.11; N, 14.28. Found: C, 49.63; H, 4.78 ; N, 13.85.

Ethyl (4-dimethylamino-2-(4-methyl-benzoylamino)-thiazol-5-yl)oxoacetate ***(3h)****:*

Orange powder, mp 168-169 ºC, yield: 0.33 g (92%). IR (KBr) (ν_{max}/cm^{-1}): 3426, 1689, 1604, 1556, 1377, 1273, 1120. ^{1}H NMR (300 MHz, $CDCl_3$): σ = 1.42 (3 H, t, ^{3}J = 7.1, Me), 2.46 (3 H, s, Me), 3.18 (6 H, s, Me_2N), 4.39 (2 H, q, ^{3}J = 7.1, OCH_2), 7.34 (2 H, d, ^{3}J = 8.2, 2 CH), 7.88 (2 H, d, ^{3}J = 8.2, 2 CH), 9.72 (1 H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): 14.5 (Me), 22.1 (Me), 43.0 (NMe_2), 62.9 (OCH_2), 99.9 (C), 128.0 (2 CH), 128.8 (C), 130.2 (2 CH), 144.8 (C), 164.1 (C), 165.1 (C), 165.2(C=O), 166.1 (C=O), 170.5 (C=O). MS (EI, 70 eV): *m/z* (%) = 361 (M^+, 17), 346 (40), 315 (73), 242 (18), 119 (100), 91 (31), 45 (19). Anal. Calcd for $C_{17}H_{19}N_3O_4S$ (361.42): C, 56.50; H, 5.30; N, 11.63. Found: C, 57.32; H, 5.77; N, 10.89.

N-(5-(4-Bromobenzoyl)-4-(dimethylamino)thiazol-2-yl)-4-methylbenzamide ***(3i)****:*

Yellow powder, mp 185-187 ºC, yield: 0.40 g (90%). IR (KBr) (ν_{max}/cm^{-1}): 3443, 1678, 1598, 1543, 1390, 1246, 1136. ^{1}H NMR (300 MHz, $CDCl_3$): σ = 2.45 (3 H, s, Me), 3.16 (6 H, s, Me_2N), 7.34 (2 H, d, ^{3}J = 8.1, 2 CH), 7.59 (2 H, d, ^{3}J = 8.4, 2 CH), 7.73 (2 H, d, ^{3}J = 8.4, 2 CH), 7.84 (2 H, d, ^{3}J = 8.1, 2 CH), 9.62 (1 H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): 22.1 (Me), 42.7 (NMe_2), 100.1 (C), 126.6 (C), 127.9 (2 CH), 128.7 (C), 130.2 (2 CH), 130.5 (2 CH), 132.0 (2 CH), 140.6 (C), 144.8 (C), 161.1 (C), 161.1 (C), 165.1 (C=O), 184.2 (C=O). MS (EI, 70 eV): *m/z* (%) = 445 (M^++2, 18), 443 (M^+, 18), 428 (32), 430 (31), 185 (13), 183 (13), 119 (100), 91 (45), 43 (8). Anal. Calcd for $C_{20}H_{18}BrN_3O_2S$ (444.34): C, 54.06; H, 4.08; N, 9.46. Found: C, 54.74; H, 4.52; N, 9.89.

N-(4-(Dimethylamino)-5-(4-methoxybenzoyl)thiazol-2-yl)-4-methylbenzamide ***(3j)****:*

Yellow powder, mp 211-212 ºC, Yield: 0.35 g (89%). IR (KBr) (ν_{max}/cm^{-1}): 3434, 1681, 1595, 1539, 1389, 1258, 1150. ^{1}H NMR (300 MHz, $CDCl_3$): σ = 2.44 (3 H, s, Me), 3.11 (6 H, s, Me_2N), 3.89 (3 H, s, OMe), 6.94 (2 H, d, ^{3}J = 8.2, 2 CH), 7.32 (2 H, d, ^{3}J = 8.2, 2 CH), 7.84 (2H, d, ^{3}J = 7.1, 2 CH), 7.87 (2 H, d, ^{3}J = 7.1, 2 CH), 9.68 (1 H, br s, NH). ^{13}C NMR (75 MHz, $CDCl_3$): 22.0 (Me), 42.5 (Me_2N), 55.8 (OMe), 100.3 (C), 114.0 (2 CH), 127.9 (2 CH), 128.9 (C), 130.2 (2 CH), 131.1 (2 CH), 134.4 (C), 144.6 (C), 160.4 (C), 161.7 (C), 162.8 (C), 164.9 (C=O), 184.9 (C=O). MS (EI, 70 eV): *m/z* (%) = 395 (M^+, 15), 380 (38), 276 (25), 260 (18), 135 (27), 119 (100), 91 (57), 43 (10). Anal. Calcd for $C_{21}H_{21}N_3O_3S$ (395.47): C, 63.78; H; 5.35, N; 10.63. Found: C, 64.11, H 5.78, N 11.23.

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