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ORIGINAL PAPER

One-step three-component and solvent-free synthesis of thiazoles from tertiary thioamides

Hassan Zali-Boeini[1] · **Seyed Gholamhossein Mansouri**[1]

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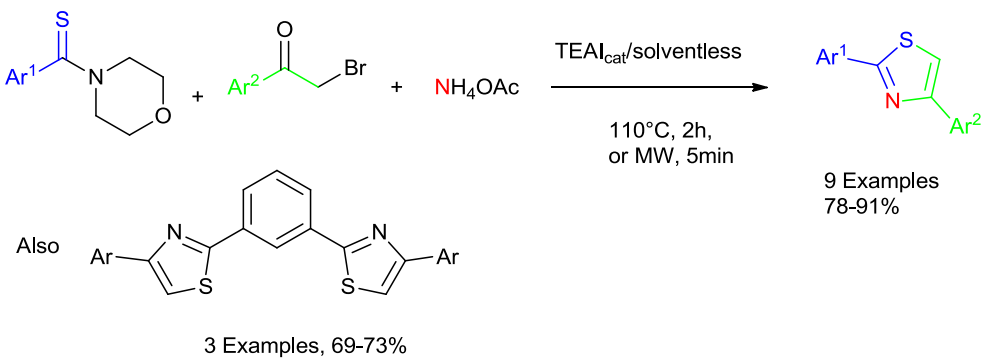
Abstract A novel one-pot three-component reaction has been developed for construction of thiazole derivatives under solvent-free conditions. Hence, tertiary thioamides, α-haloketones, and NH_4OAc were grinded together and allowed to react thermally at 110 °C and/or under microwave irradiation to produce the corresponding thiazole derivatives in very good yields.

Graphical Abstract



Keywords Synthetic methods · Sulfur heterocycles · Thiazole · Green chemistry

Introduction

It is well known that thiazoles are among the most important heterocycles that are frequently found in natural products [1, 2], pharmaceuticals [3–8], and agricultural chemistry [9]. They also have gained wide applications in the areas of organic electronics as electroluminescence and electron transport materials [10–12], electro-optic [13, 14] and photonic devices [15–18]. The thiazole ring as a latent formyl group has been frequently used as a useful building block in new synthetic transformations [19]. The most known approach to the synthesis of thiazole ring is the *Hantzsch* synthesis in which primary thioamides react with α-halocarbonyls to produce thiazole derivatives in a single step [20]. Also, α-halogenation of β-keto esters with *N*-bromosuccinimide, followed by cyclization with thiourea or selenourea produce 2-amino-4-alkyl- and 2-amino-4-arylthiazole-5-carboxylates and the corresponding selenazole analogues, respectively [21]. Thiazoles also could be easily accessed by the reaction of α-acylaminoketones and

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✉ Hassan Zali-Boeini
h.zali@chem.ui.ac.ir

[1] Department of Chemistry, Faculty of Science, University of Isfahan, 81746-73441 Isfahan, Iran

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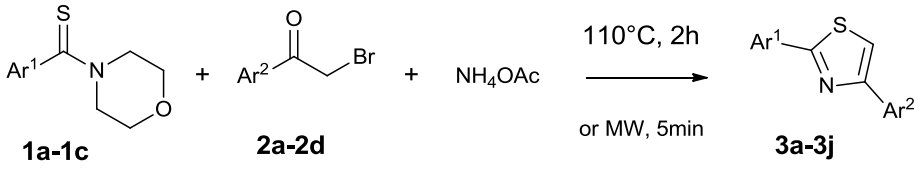
Scheme 1 Synthesis of thiazole derivatives by reaction of tertiary thioamides, α-haloketones, and NH_4OAc



Ar^1: **1a** (C_6H_5), **1b** (4-$MeOC_6H_4$), **1c** (4-FC_6H_4)
Ar^2: **2a** (C_6H_5), **2b** (4-MeC_6H_4), **2c** (Cl-FC_6H_4), **2d** (Br-FC_6H_4)

P_2S_5 (*Gabriel* synthesis) [22]. They can be also obtained in good yields via the reaction of 1*H*-1-(1′-alkynyl)-5-methyl-1,2,3-benziodoxathiole 3,3-dioxides with primary thioamides [23]. Reaction of dithiooxamide (rubeanic acid) with aromatic aldehydes has been utilized for the synthesis of thiazolothiazole derivatives [24]. Reaction of mercaptoacetaldehyde dimer, ammonia, and aldehydes is leading to 2,5-dihydro-1,3-thiazoles [25]. It has been shown that, when *S*-alkylated thioamides or thioureas are reacted with certain acid derivatives, various types of substituted thiazoles are prepared in low to moderate yields [26]. Direct one-step reaction of thiocarboxylic acids, α-haloketones, and NH_4OAc in refluxing AcOH has been used for the preparation of thiazole derivatives [27]. 2-Aminothiazole derivatives have been prepared by the reaction of 2-*N*-acylglycinamides with a thionating reagent followed by subsequent cyclization mediated by triflouroacetic anhydride (TFAA) [28]. Recently 5-aminothiazoles have been prepared based on cyclization of their diamide adducts, prepared by the *Ugi* reaction, in the presence of Lawesson's reagent [29]. More recently a new approach for the construction of thiazole ring has been developed using the cyclocondensation reaction of primary thioamides and alkynyl(aryl)iodonium reagent [30]. Direct conversion of alkenes to the corresponding 2-aminothiazoles is possible via reaction with thiourea in the presence of 2-iodoxybenzoic acid (IBX) as oxidant [31, 32]. Dimethyl cyanodithioimidocarbonates as starting material have been successfully employed for the synthesis of 1,3-thiazoles and 1,3-selenazoles [33]. Another method benefits from base-induced cyclization reaction of isocyanides bearing an active methylene with methyl arenecarbodithioates for the synthesis of 4,5-disubstituted thiazoles [34].

Following our studies on exploration of new synthetic methods for the construction of sulfur containing rings [35–40], herein we wish to report for the first time an efficient one-step method for the synthesis of disubstituted thiazoles via the three-component reaction of tertiary thioamides, α-haloketones, and NH_4OAc under solvent-free condition (Scheme 1).

Experimental

The chemicals used in this work were purchased from Fluka and Merck chemical companies. The progress of the reaction was monitored by TLC using 0.25-μm pre-coated silica gel plates. Melting points were determined using Stuart Scientific SMP2 apparatus and are uncorrected. ^1H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on Bruker-Avance 400.

General procedure for the one-pot three-component preparation of thiazoles 3a–3i

In a 25-mL round-bottom flask, finely grinded morpholino(aryl)methanethione (1 mmol), α-bromoketone (1.15 mmol), ammonium acetate (1.6 mmol, 125 mg), and tetraethylammonium iodide (TEAI, 0.1 mmol, 26 mg) were heated at melting point (110 °C) and (or subjected to MW heating in a domestic microwave oven at medium power for 5 min) heating was continued for 2 h. After cooling, the reaction mixture was triturated with hexane–EtOAc (4:1, 10 mL). Then, the crude precipitated product was filtered and recrystallized from a suitable solvent (EtOH). In some cases (**3d** and **3h**) after work-up of the reaction a crude oily product was obtained. In these cases, the crude product was purified by column chromatography on silica gel using a mixture of hexane–EtOAc (4:1) as eluent.

General procedure for the one-pot three-component preparation of thiazoles 3j–3 l

In a 25-mL round-bottom flask, 1,3-phenylenebis(morph olinomethanethione) (1 mmol, 336 mg), α-bromoketone (2.3 mmol), ammonium acetate (3.2 mmol, 250 mg), and TEAI (0.1 mmol, 26 mg) were heated at 110 °C to make a melted reaction mixture and heating was continued for a further 2 h. Thereafter, the reaction mixture was boiled with MeOH (10 ml) and cooled. Then, the crude precipitated product was filtered and recrystallized from EtOH to

obtain 1,3-bis(4-arylthiazol-2-yl)benzene derivatives as off-white or light yellow powders.

Spectral data of compounds 3a–3l

Compound **3a**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.42 (m, 4H), 8.07 (m, 4H), 7.52 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.9 (C-2 thiazole), 162.9 (C-4 thiazole), 134.4, 130.2, 128.8, 128.5, 128.2, 126.5, 116.1, 115.9, 112.5 (C-5 thiazole); Anal. Calcd. for $C_{15}H_{11}NS$: C, 75.91; H, 4.67; N, 5.90; S, 13.51 Found: C, 75.84; H, 4.71; N, 5.79; S, 13.57.

Compound **3b**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.07–8.05 (m, 2H), 7.91 (d, j = 8.0 Hz, 2H), 7.50–7.44 (m, 3H), 7.43 (s, 1H), 7.27 (d, j = 8.0 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.7 (C-2 thiazole), 162.3 (C-4 thiazole), 156.7, 138.3, 131.6, 130.8, 129.3, 128.6, 128.2, 126.2, 116.7, 111.8 (C-5 thiazole), 20.7; Anal. Calcd. for $C_{16}H_{13}NS$: C, 76.46; H, 5.21; N, 5.57; S, 12.76 Found: C, 76.34; H, 5.31; N, 5.70; S, 12.59.

Compound **3c**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.06–8.03 (m, 2H), 7.96–7.93 (m, 2H), 7.50–7.40 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.1 (C-2 thiazole), 155.1 (C-4 thiazole), 134.0, 133.6, 133.0, 130.2, 128.94, 128.89, 127.7, 126.6, 112.8 (C-5 thiazole); Anal. Calcd. for $C_{15}H_{10}ClNS$: C, 66.29; H, 3.71; Cl, 13.05; N, 5.15; S, 11.80 Found: C, 66.34; H, 3.61; Cl, 13.13; N, 5.10; S, 11.69.

Compound **3d**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.01–7.98 (m, 4H), 7.47–7.43 (m, 2H), 7.41 (s, 1H), 7.38–7.34 (m, 1H), 7.01–6.97 (m, 2H), 3.88 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.8 (C-2 thiazole), 161.3 (C-4 thiazole), 154.7, 133.5, 131.3, 128.3, 127.2, 126.5, 122.2, 114.6, 112.1 (C-5 thiazole), 54.4; Anal. Calcd. for $C_{16}H_{13}NOS$: C, 71.88; H, 4.90; N, 5.24; S, 11.99 Found: C, 71.74; H, 4.97; N, 5.18; S, 12.04.

Compound **3e**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.07–8.02 (m, 2H), 8.01–7.98 (m, 2H), 7.47 (s, 1H), 7.45 (d, j = 7.8 Hz, 2H), 7.37 (t, j = 7.4 Hz, 1H), 7.19–7.13 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.9 (C-2 thiazole), 162.9 (C-4 thiazole), 156.4, 134.4, 130.2, 128.7, 128.6, 128.5, 128.2, 126.5, 116.1, 115.9, 112.5 (C-5 thiazole); Anal. Calcd. for $C_{15}H_{10}FNS$: C, 70.57; H, 3.95; F, 7.44; N, 5.49; S, 12.56 Found: C, 70.49; H, 3.90; F, 7.49; N, 5.42; S, 12.49.

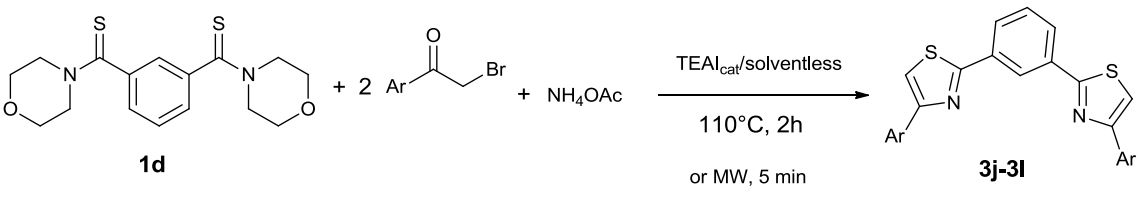
Compound **3f**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04–7.99 (m, 2H), 7.95–7.91 (m, 2H), 7.45 (s, 1H), 7.44–7.40 (m, 2H), 7.19–7.13 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.9 (C-2 thiazole), 165.0 (C-4 thiazole), 163.0, 155.2, 134.1, 132.9, 130.0, 128.9, 128.6, 128.5, 127.7, 116.1, 115.9, 112.8 (C-5 thiazole); Anal. Calcd. for $C_{15}H_9ClFNS$: C, 62.18; H, 3.13; Cl, 12.24; F, 6.56; N, 4.83; S, 11.07 Found: C, 62.24; H, 3.17; Cl, 12.18; F, 6.51; N, 4.78; S, 11.11.

Compound **3g**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.05–8.00 (m, 2H), 7.87 (d, j = 8.5 Hz, 2H), 7.58 (d, j = 8.5 Hz, 2H), 7.49 (s, 1H), 7.19–7.14 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.9 (C-2 thiazole), 165.0 (C-4 thiazole), 163.0, 155.2, 133.3, 131.9, 130.0, 128.6, 128.5, 128.0, 122.2, 116.1, 115.9, 112.9 (C-5 thiazole); Anal. Calcd. for $C_{15}H_9BrFNS$: C, 53.91; H, 2.71; Br, 23.91; F, 5.68; N, 4.19; S, 9.59 Found: C, 53.85; H, 2.66; Br, 23.97; F, 5.74; N, 4.14; S, 9.52.

Compound **3h**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95 (d, j = 8.7 Hz, 2H), 7.87 (d, j = 8.4 Hz, 2H), 7.57 (d, j = 8.4 Hz, 2H), 7.40 (s, 1H), 6.98 (d, j = 8.7 Hz, 2H), 3.88 (s, 3H);^{13}C NMR (125 MHz, $CDCl_3$): δ 168.0 (C-2 thiazole), 161.3 (C-4 thiazole), 154.9, 133.6, 131.8, 128.1, 128.0, 126.6, 122.0, 114.3, 112.1 (C-5 thiazole), 55.4; Anal. Calcd. for $C_{16}H_{12}BrNOS$: C, 55.50; H, 3.49; Br, 23.08; N, 4.05; S, 9.26 Found: C, 55.56; H, 3.45; Br, 23.13; N, 3.99; S, 9.21.

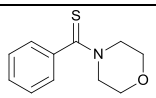
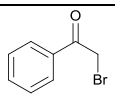
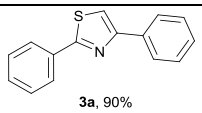
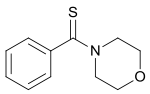
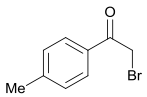
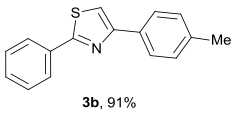
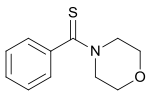
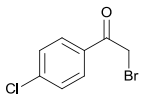
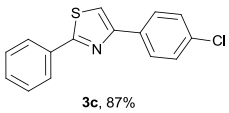
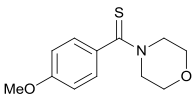
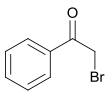
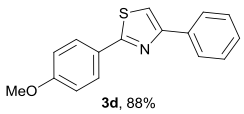
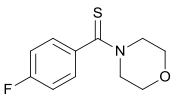
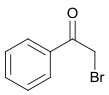
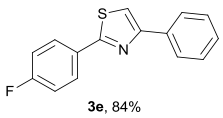
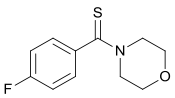
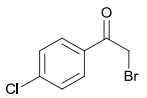
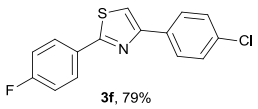
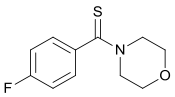
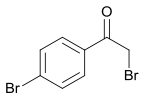
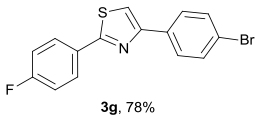
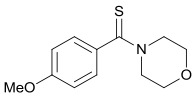
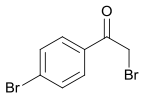
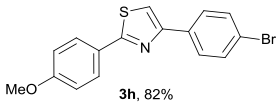
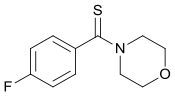
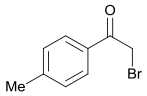
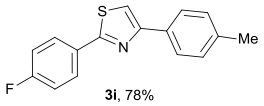
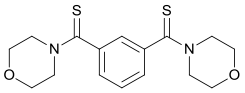
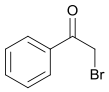
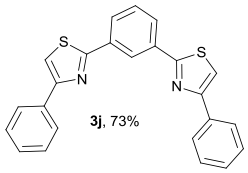
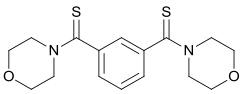
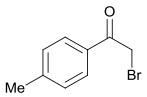
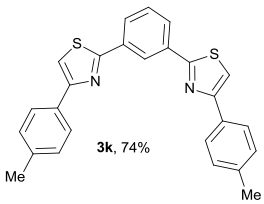
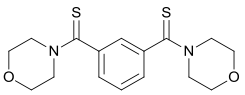
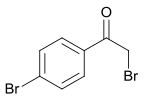
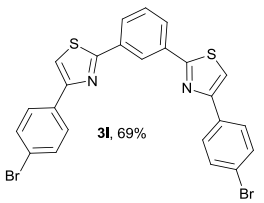
Compound **3i**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.06–8.02 (m, 2H), 7.89 (d, j = 8.1 Hz, 2H), 7.41 (s, 1H), 7.26 (d, j = 7.9 Hz, 2H), 7.19–7.13 (m, 2H), 2.41 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.5 (C-2 thiazole), 164.9 (C-4 thiazole), 162.9, 156.5, 138.1, 131.8, 130.2, 129.4, 128.53, 128.47, 126.4, 116.0, 115.8, 111.7 (C-5 thiazole), 21.3; Anal. Calcd. for $C_{16}H_{12}FNS$: C, 71.35; H, 4.49; F, 7.05; N, 5.20; S, 11.91 Found: C, 71.28; H, 4.46; F, 7.11; N, 5.16; S, 11.94.

Compound **3j**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.68 (s, 1H), 8.14 (d, j = 7.8 Hz, 1H), 8.13 (d, j = 7.8 Hz, 1H), 8.05 (d, j = 7.7 Hz, 4H), 7.58 (t, j = 7.7 Hz, 1H), 7.54 (s, 2H), 7.49 (t, j = 7.6 Hz, 4H), 7.39 (t, j = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.0 (C-2 thiazole), 156.6 (C-4 thiazole), 134.6, 134.5, 129.5, 128.8, 128.3, 128.1, 126.5,



Scheme 2 Application of bisthioamides for the synthesis of 1,3-bis(4-arylthiazol-2-yl)benzene derivatives

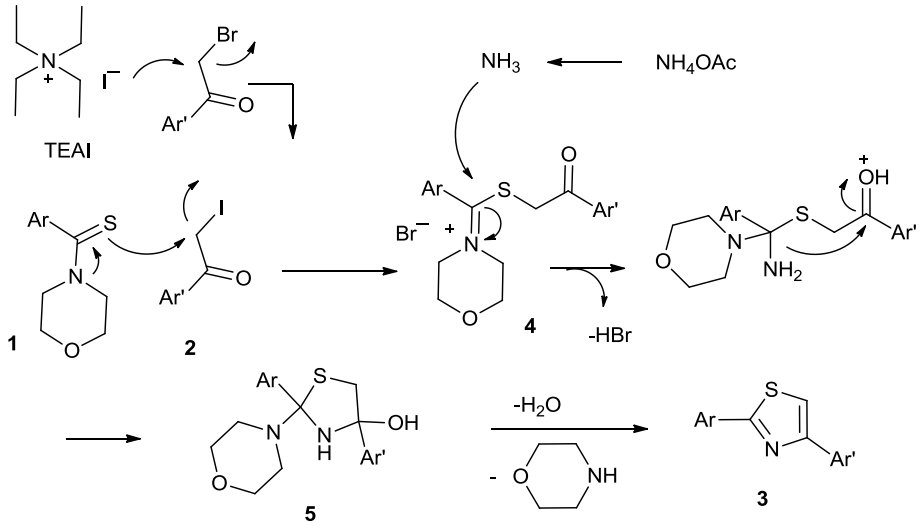
Table 1 A novel solvent-free approach for the synthesis of thiazole derivatives[a, b]

Entry	Thioamide	α-haloketones	Product
1			 **3a**, 90%
2			 **3b**, 91%
3			 **3c**, 87%
4			 **3d**, 88%
5			 **3e**, 84%
6			 **3f**, 79%
7			 **3g**, 78%
8			 **3h**, 82%
9			 **3i**, 78%
10			 **3j**, 73%
11			 **3k**, 74%
12			 **3l**, 69%

[a] Reaction conditions: morpholino(aryl)methanethione (1 mmol), α-bromoketone (1.2 mmol), NH_4OAc (2 mmol, 154 mg), and TEAI (0.1 mmol, 26 mg) and heated at 110 °C for 2 h

[b] Yields refer to pure isolated products

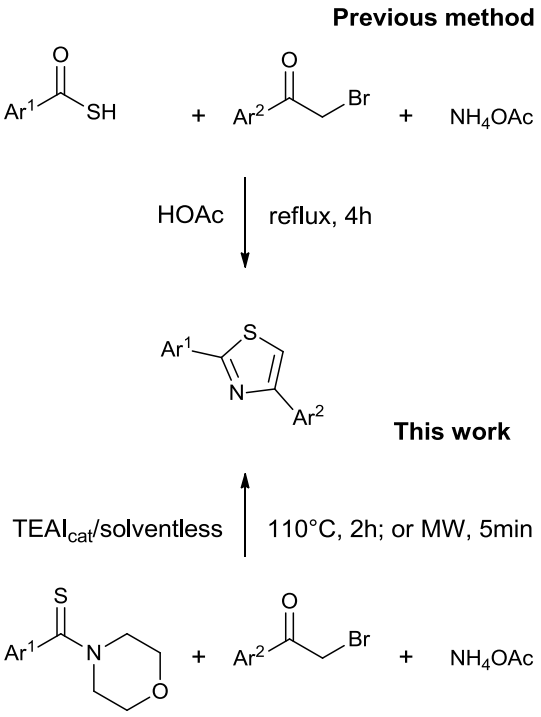
Scheme 3 A proposed mechanism for the construction of thiazole ring



124.6, 113.0 (C-5 thiazole); Anal. Calcd. for $C_{24}H_{16}N_2S_2$: C, 72.70; H, 4.07; N, 7.06; S, 16.17 Found: C, 72.76; H, 4.11; N, 7.01; S, 16.21.

Compound **3k**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.66 (s, 1H), 8.13 (d, j = 7.7 Hz, 1H), 8.12 (d, j = 7.7 Hz, 1H), 7.93 (d, j = 8.0 Hz, 4H), 7.57 (t, j = 7.7 Hz, 1H), 7.48 (s, 2H), 7.29 (d, j = 8.0 Hz, 4H), 2.42 (s, 6H);^{13}C NMR (125 MHz, $CDCl_3$): δ 166.9 (C-2 thiazole), 156.7 (C-4 thiazole), 138.1, 134.6, 131.8, 129.5, 129.4, 128.0, 126.4, 124.6, 112.2 (C-5 thiazole), 21.3; Anal. Calcd. for $C_{26}H_{20}N_2S_2$: C, 73.55; H, 4.75; N, 6.60; S, 15.10 Found: C, 73.49; H, 4.71; N, 6.66; S, 15.04.

Compound **3l**: ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.64 (s, 1H), 8.12–8.10 (m, 2H), 7.91 (d, j = 8.3 Hz, 4H), 7.62–7.53 (m, 7H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 167.2 (C-2 thiazole), 155.4 (C-4 thiazole), 134.4, 133.3, 131.9, 129.6, 128.2, 128.1, 124.6, 122.3, 113.4 (C-5 thiazole); Anal. Calcd. for $C_{24}H_{14}Br_2N_2S_2$: C, 52.00; H, 2.55; Br, 28.83; N, 5.05; S, 11.57 Found: C, 51.96; H, 2.52; Br, 28.88; N, 5.09; S, 11.53.



Scheme 4 Simplicity and immediacy of our approach in comparison with a previously reported method

Results and discussion

At the beginning of our investigation, thioamide **1a** (1 mmol) as a test substrate was mixed with α-bromoacetophenone **2a** (1.2 mmol) and NH_4OAc (1.2 mmol). Then, the mixture was grinded and heated at melting point (100–110 °C) for 2 h. The reaction mixture was proceeded smoothly, and the product **3a** was obtained in moderate yield (56 %). We found that with increasing the amount of NH_4OAc to 2 molar equivalents the product yield of the reaction was meaningfully raised up to 77 %. Surprisingly, it was found that the product yield of the reaction was significantly improved with addition of small amounts of tetraethylammonium iodide (TEAI, 0.1 mmol) to the reaction media. We assume that, this is due to catalytic role of iodide ion in activating α-bromoketone **2** and partial increasing of polarity of the reaction mixture. Therefore, when the reaction was carried out using thioamide **1a** (1 mmol), α-bromoacetophenone **2a** (1.2 mmol) and NH_4OAc (2 mmol) in the presence of TEAI (0.1 mmol) at 110 °C, the product **3a** was produced in a very good yield (90 %). Unfortunately, further increase in the reaction temperature resulted in diminishing the yield of **3a** accompanied with the formation of some dark tarry

materials. The necessary thioamides **1a**–**1c** are readily prepared by Willgerodt–Kindler reaction of the corresponding aldehydes with morpholine and sulfur and/or with thionation of the corresponding amides [41–44]. In order to show the generality of the reaction, other types of tertiary thioamides such as bisthioamide **1d** were chosen and reacted at the same reaction conditions to produce compounds **3j**–**3l** as 1,3-bis(4-arylthiazol-2-yl)benzene derivatives (Scheme 2) in good yields (69–74 %) and the results are given in Table 1.

A plausible mechanism is also proposed for the course of reaction and shown in scheme 3. First, α-bromoacetophenone **2** is activated by the aid of TEAI and undergone a nucleophilic attack by thioamide **1** to produce *S*-alkylated thioamide intermediate **4**. Then NH_4OAc as a source of NH_3 makes a nucleophilic attack on this intermediate and cyclizes to intermediate **5**. Eventually, this intermediate transforms to thiazole **3** thru elimination of a morpholine and water molecules.

The simplicity and the immediacy of the presented approach are demonstrated in scheme 4 by comparison with the method with one of the previously reported methods. In one of the reported methods, thiazole derivatives have been prepared by the reaction of thiocarboxylic acids and α-haloketones with NH_4OAc in refluxing AcOH [27]. Although this method is rather effective, but investigation of a narrow substituent's scope (only five examples), exploiting thiocarboxylic acids which are unpleasant and noxious materials and performing the reaction in refluxing acetic acid which is a corrosive and irritating solvent are major drawbacks of the method. We found that replacing thiocarboxylic acids with tertiary thioamides which are safe and odorless materials and performing the reaction course under solvent-free conditions avoids the environmental issues associated with using of a solvent media.

Conclusion

To sum up, a new one-step three-component and solvent-free approach for the synthesis of diverse thiazole derivatives has been developed. Construction of thiazoles using cheap and readily available starting materials, solvent-free synthesis, microwave acceleration of the reaction, using a nontoxic and odorless sulfur source, simple work-up of the reaction mixture, and easy isolation and purification of final product are some advantages of the presented method. These benefits promote the method as a favorable alternative to the synthesis of thiazole derivatives, which are important chemicals in drug development and industrial chemistry.

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