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LETTER ARTICLE

[bmim]BF_4-accelarated One-pot Synthesis of 2-amino Thiazole Derivatives

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ARTICLE HISTORY

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Abstract: An efficient and environmentally-friendly protocol for the synthesis of 2- (arylamino)thiazoles has been investigated. The two-step, three-component reaction of aryl ketones, *N*-bromosuccinimide, and thiourea derivatives was performed using [bmim][BF_4] ionic liquid as a solvent in one pot and gave products in good to excellent yields. Furthermore, the ionic liquid could be reused up to the 5th time without a considerable decrease in the reaction yield. Thirteen thiazoles were obtained in high yields (78-89%) under mild conditions (ambient temperature and relatively short reaction time) and their structures were confirmed by NMR data.

Keywords: Ionic liquid, thioureas, NBS, aryl ketones, heterocyclic compounds, thioamides.

1. INTRODUCTION

Thiazole derivatives are aromatic five-membered ring heterocyclic compounds that contain both sulfur and nitrogen at 1 and 3 positions. Thiazole-based compounds are found in a wide variety of bioactive molecules and natural products as well as synthetic sources. Several naturally occurring thiazoles play important roles in human life such as vitamin B1 (thiamin) and the penicillin antibiotic family. Many thiazoles possess a wide spectrum of bioactivities such as antibacterial, antiprotozoal, antimalarial, anticancer, anti-allergic, gene-modulating, anti-schizophrenia, antihypertension, anti-in*fl*ammation, and anti-HIV activities [1]. Some thiazole derivatives are well-known drugs in the markets for the treatment of various diseases such as cefixime, abafungin, dasatinib, cambendazole, meloxicam, and troglitazone (Fig. **1**). Furthermore, applications of thiazole derivatives in other fields such as building blocks for organic synthesis [2, 3], organic materials [4-6], and organic ligands [7-9] are also well documented.

With such broad applicability, increasing attention has been paid to the synthesis of thiazole-containing compounds and many synthetic protocols have been reported [1, 10]. The Hantzsch thiazole synthesis, one of the oldest and most straightforward methods to access thiazole derivatives, still receives great attention of chemists. Especially, one-pot synthesis of thiazoles from substituted ketones, brominated reagent, and thioamide or thiourea has attracted intensive research interest. However, these three-component reactions usually suffer from some drawbacks such as the long reaction time, high temperature, and low yields products. The use of ionic liquids for the Hantzsch thiazole synthesis can help chemists to avoid these drawbacks for which some studies can be found in the literature [10-13]. Herein, we have developed a mild and efficient method to prepare 2-amino thiazole derivatives from aryl ketones, *N*-bromosuccinimide (NBS), and thiourea derivatives using [bmim][BF_4] (1-Butyl-3-methylimidazolium tetrafluoroborate) ionic liquid as a green solvent. The synthesis features some advantages such as a readily available starting material, recyclability of ionic liquids, mild reaction conditions, and high efficiency.

2. RESULTS AND DISCUSSION

Recently, Muthyala and Kumar reported an efficient protocol for the preparation of thiazoles from aryl ketones, phenyl trimethyl ammonium tribromide (PTT), and thioamide using [bmim][BF_4] as a solvent and *p*-toluenesulfonic acid as a catalyst [14]. In another report, Stavber *et al*. performed the bromination reaction of aryl ketones using an ionic liquid without a catalyst [15]. We proposed that by using thioureas and NBS instead of thioamides and PTT, 2- (arylamino)thiazoles could be obtained using an ionic liquid solvent. Initially, we chose acetophenone and phenyl thiourea as model substrates along with NBS for the optimization of reaction conditions to synthesize 2- (phenylamino)thiazole. In the first step, a mixture of acetophenone (**1a**, 1 mmol) and NBS (1.25 mmol, 1.25 equiv.) in an ionic liquid (5 mL) was stirred vigorously at 45°C for a period of time. Phenyl thiou-

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rea (**2a**, 1.05 mmol, 1.05 equiv.) was then added to the reaction mixture and the resulting mixture was allowed to stir at room temperature for additional 30 minutes. Progress of the reaction was monitored by thin-layer chromatography. The results were summarized in Table **1**.

The use of [bbim]BF_4 (benzyl-3-butylimidazolium tetrafluoroborate) as a solvent resulted in product **3a** in 63% yield in 4 hours (Table **1**, entry 1). In case of [bbim]ClO_4, the reaction yield dropped to 27% (Table **1**, entry 2). The yield was not considerably improved when the reaction time was increased to 6 h (Table **1**, entry 3). The use of [bbim]Br and [bbim]CF_3SO_3 did not produce adequate result, producing product **3a** in 41 and 51% yields, respectively (Table **1**, entries 4 and 5). To our delight, when [bmim]BF_4 was employed, a very good yield of product **3a** was obtained (Table **1**, entry 6). In next attempts, the use of three other ionic liquids, [bmim]PF_6, [bmim]HSO_4, [bmim]OAc, was not fully successful. Moderate yields of products were observed in these cases, although the reaction time was lengthened to 6 h. When NIS was employed as an iodinating reagent, a very low yield of **3a** was obtained (Table **1**, entry 10). Presumably, NIS favored the substitution to the aromatic ring. We concluded that [bmim]BF_4 is the most efficient solvent for our synthesis.

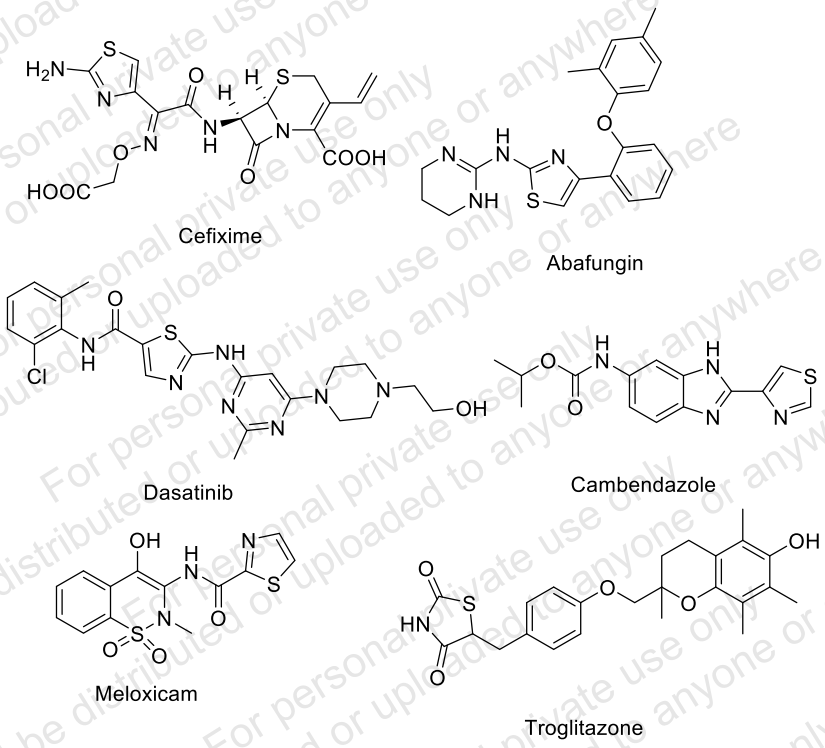
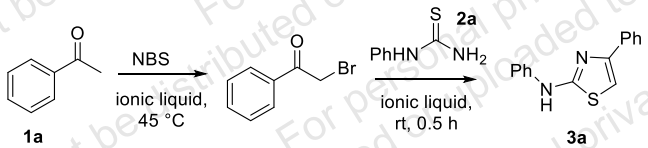


Fig. (1). Some drugs containg thiazole skeleton.

Table 1. Optimization reaction conditions for thiazole synthesis.

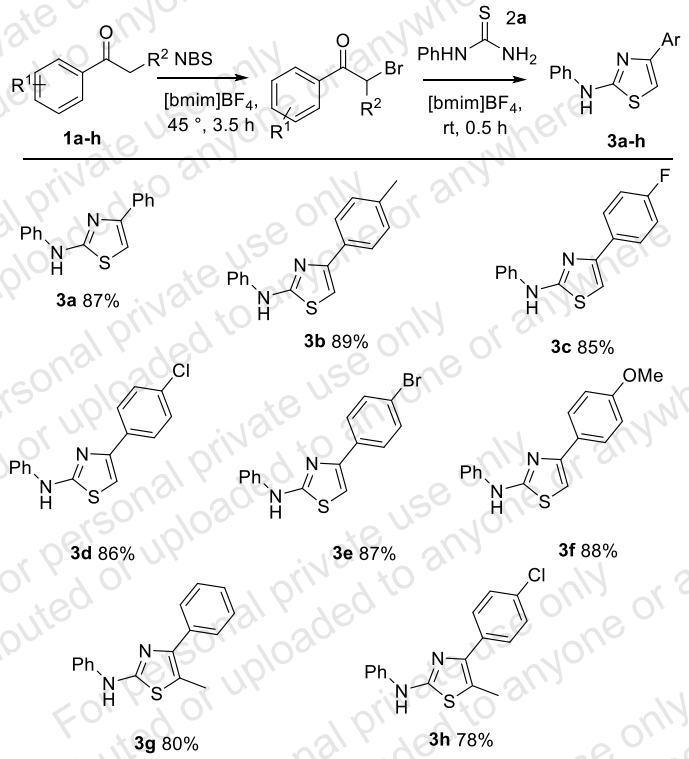


Entry	Solvent	Time (h, for Two Steps)	Yeild of 3a (%)
1	[bbim]BF_4	4	63
2	[bbim]ClO_4	4	27
3	[bbim]ClO_4	6	35
4	[bbim]Br	6	41
5	[bbim]CF_3SO_3	6	51
6	[bmim]BF_4	4	87

Table 1 Contd…

Entry	Solvent	Time (h, for Two Steps)	Yeild of 3a (%)
7	$[bmim]PF_6$	6	57
8	$[bmim]HSO_4$	6	66
9	[bmim]OAc	6	61
10	$[bmim]BF_4$	4	9[a]

Note: [a]NIS was used.



Scheme 1. Scope of arylketones[a, b]
Note: [a]Reaction conditions: ketone (1 mmol), NBS (1.25 mol), [bmim][BF_4] (5 mL) 3.5 h, then phenyl thiourea (1.05 mmol), 0.5 h; [b] Yield: isolated yield.

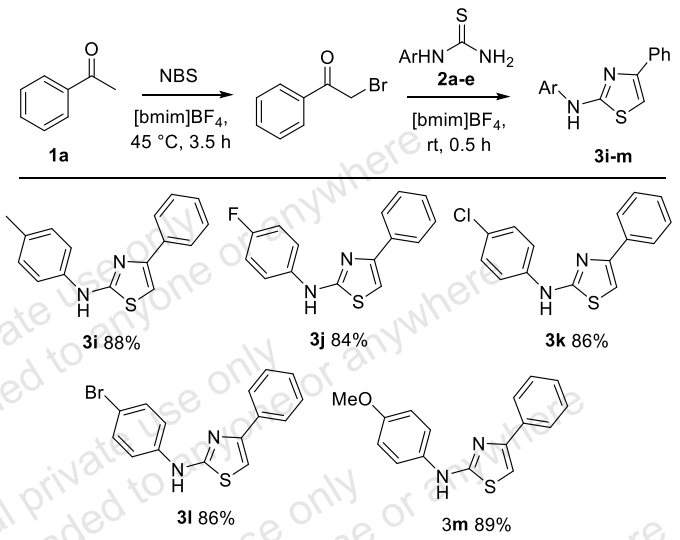
With the optimal conditions in hand, we next explored the scope of the reaction. At first, the scope of aryl ketones was examined and results are given in Scheme **1**. Generally, various thiazole derivatives were provided in high yields (78-89%). It can be observed that acetophenones containing both electron-withdrawing and electron-releasing substituents at the para position were tolerated as good substrates for this reaction under these conditions and products were obtained in good to excellent yield (**3a**-**f**). The electronic effect of substituents at the phenyl ring might not play important roles in reaction yields. However, lower yields were afforded for two arylethyl ketones (**3g** and **3h**). A decrease in reaction yields in these reactions might be due to the steric effect and electronic effect of the methyl group at its ketone counterpart.

Subsequently, various aryl thioureas were examined under optimized reaction conditions and results are given in Scheme **2**. A series of para-substituted aryl thioureas, including electron-donating groups and electron-withdrawing groups, was converted into the corresponding thiazoles (**3i-3m**) in high yields.

The advantage of the use of ionic liquids as novel reaction media for this transformation is that the ionic liquid can be easily recovered and recycled in subsequent reactions. The ionic liquid reaction medium could be reused for the same reaction, maintaining the good yields of thiazole up to the 5th time, as shown in Table **2**. The one-pot procedure described by Muthyala and Kumar [14] required longer time (more than 13 h for two steps) and needed other chemicals (p-TsOH.H_2O for the first step and K_2CO_3 for the second step). In another report, PEG-400 was chosen as the solvent for this one-pot reaction [16] but the synthesis also needed longer time and the solvent was not recycled.

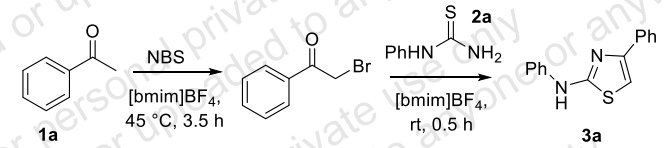
3. MATERIALS AND METHODS

All chemicals were purchased from Sigma-Aldrich and used without further purification due to their high purity. ^{1}H and ^{13}C NMR spectra were recorded on a Varian Inova NMR Spectrometer (^{1}H NMR running at 500 MHz and ^{13}C NMR running at 125 MHz) instrument using $CDCl_3$ as a solvent and Me_4Si as the internal standard. ^{1}H NMR chemical shifts



Scheme 2. Scope of arylthioureas[a, b]
Note: [a]Reaction conditions: acetophenone (1 mmol), NBS (1.25 mol), [bmim][BF_4] (5 mL) 3.5 h, then phenyl thiourea (1.05 mmol), 0.5 h; [b] Yield: isolated yield.

Table 2. Recyclability of [bmim][BF_4].



Reuse	1	2	3	4	5
Yields (%)	86	84	84	83	81

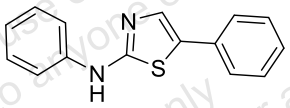
are quoted in δ values in ppm and are referenced relative to the chemical shift of $CDCl_3$ (7.26 ppm). Coupling constants (*J*) are reported in Hz, with signal multiplicities designated at singlet (s), doublet (d), doublet of doublet (dd), triplet (t), quartet (q), and multiplet (m). ^{13}C NMR chemical shifts are quoted in δ values in ppm and are referenced relative to the chemical shift of $CDCl_3$. Column chromatography was performed using Meck silica gel (40-63 μm) packed by the slurry method, under a positive pressure of air.

4. EXPERIMENTAL

4.1. General Procedure for Thiazole Synthesis

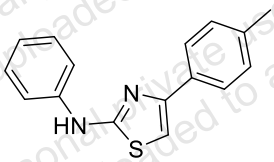
Acetophenone (**1a**, 120 mg, 1 mmol), and NBS (221 mg, 1.25 mmol, 1.25 equiv.) were added to [bmim]BF_4 (5 mL). The mixture was stirred at 45°C for 3.5 hours. Phenyl thiourea (167 mg, **2a**, 1.1 mmol, 1.1 equiv) was added and the resulting mixture was then allowed to stir at room temperature for 0.5 hour. After completion, the reaction mixture was extracted with diethyl ether (7 mL × 4). The organic extracts were combined and dried over Na_2SO_4. After removal of the solvent under reduced pressure, the residue was purified by column chromatography on silica gel to give the desired product **3a** as a red solid in 87 % yield. The ionic liquid was washed with water (5 mL), dried with a vacuum pump for 2 h at 80°C, and reused for the next run without further purification.

4.1.1. N,4-Diphenylthiazol-2-amine *(3a)*



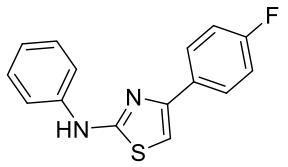
Red solid (219 mg, 87%). ^1H NMR (500 MHz, $CDCl_3$) δ 7.87 (d, J = 8.0 Hz, 2H,), 7.42 - 7.28 (m, 7H), 7.07 (t, J = 7.0 Hz, 1H), 6.83 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.6, 151.4, 140.4, 134.6, 129.5, 128.6, 127.8, 126.3, 123.0, 118.3, 101.8. NMR data are consistent with the literature report [17].

4.1.2. N-Phenyl-4-(p-tolyl)thiazol-2-amine (3b)



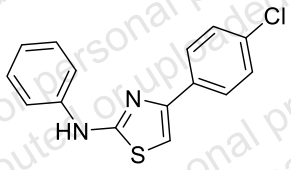
Red solid (237 mg, 89%). ^1H NMR (500 MHz, $CDCl_3$) 7.75 (d, *J* = 8.0 Hz, 2H), 7.39 - 7.32 (m, 4H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.05 (t, *J* = 6.5 Hz, 1H), 6.77 (s, 1H), 2.38 (s, 3H, Me). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.6, 151.4, 140.4, 137.7, 131.9, 129.4, 129.2, 126.1, 122.9, 118.2, 101.0, 21.3 (Me). NMR data are consistent with the literature report [17].

4.1.3. 4-(4-Fluorophenyl)-N-phenylthiazol-2-amine (3c)



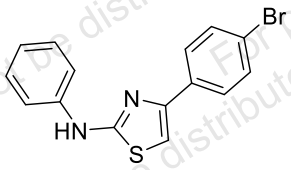
Red solid (230 mg, 85%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (dd, *J* = 8.0, 6.0 Hz, 2H), 7.35 (d, *J* = 6.0 Hz, 4H), 7.07 (t, *J* = 8.5 Hz, 3H), 6.74 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.8, 162.6 (d, *J* = 246 Hz, CF), 150.3, 140.3, 130.9, 129.5, 127.8 (d, *J* = 8 Hz), 123.1, 118.3, 115.5 (d, *J* = 22 Hz), 101.3. NMR data are consistent with the literature report [18].

4.1.4. 4-(4-Chlorophenyl)-N-phenylthiazol-2-amine (3d)



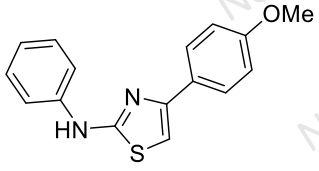
Yellow solid (246 mg, 86%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.5 Hz, 2H), 7.36 - 7.28 (m, 6H), 7.10 - 7.05 (m, 1H), 6.78 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.2, 150.1, 140.2, 133.1, 133.1, 129.5, 128.7, 127.5, 123.3, 118.5, 102.1. NMR data are consistent with the literature report [19].

4.1.5. 4-(4-Bromophenyl)-N-phenylthiazol-2-amine (3e)



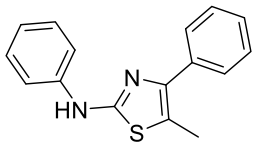
Red solid (287 mg, 87%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.5 Hz, 2H), 7.52 (d, *J* = 8.5 Hz, 2H), 7.36 (dd, *J* = 8.0, 6.0 Hz, 4H), 7.09 (td, *J* = 5.5, 3.0 Hz, 1H), 6.82 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.8, 150.3, 140.2, 133.5, 131.7, 129.5, 127.7, 123.2, 121.8, 118.4, 102.3. NMR data are consistent with the literature report [19].

4.1.6. 4-(4-Methoxyphenyl)-N-phenylthiazol-2-amine (3f)



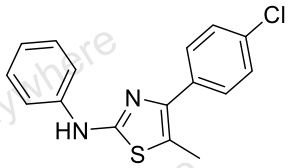
Yellow solid (121 mg, 88%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.5 Hz, 2H), 7.39 - 7.30 (m, 4H), 7.05 (t, *J* = 6.5 Hz, 1H), 6.93 (d, *J* = 8.5 Hz, 2H), 6.69 (s, 1H), 3.83 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.6, 159.4, 151.1, 140.4, 129.4, 127.7, 127.4, 122.9, 118.2, 114.0, 100.0, 55.3 (OMe). NMR data are consistent with the literature report [20].

4.1.7. 5-Methyl-N,4-diphenylthiazol-2-amine (3g)



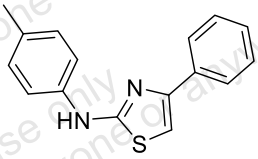
Yellow solid (213 mg, 80%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.0 Hz, 2H), 7.41 (t, *J* = 7.0 Hz, 2H), 7.32-7.21 (m, 5H), 7.01 (d, *J* = 6.5 Hz, 1H), 2.44 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.1, 146.1, 140.6, 135.1, 129.5, 128.6, 128.3, 127.4, 122.6, 118.1, 116.8, 12.3 (Me). NMR data are consistent with the literature report [21].

4.1.8. 4-(4-Chlorophenyl)-5-methyl-N-phenylthiazol-2-amine (3h)



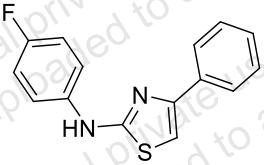
Yellow solid (234 mg, 78%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.34 - 7.24 (m, 4H), 7.06 (t, *J* = 7.0 Hz, 1H), 2.45 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.3, 144.8, 140.4, 133.5, 133.3, 129.7, 129.5, 128.5, 123.0, 118.3, 117.0, 12.3 (Me). NMR data are consistent with the literature report [21].

4.1.9. 4-Phenyl-N-(p-tolyl)thiazol-2-amine (3i)



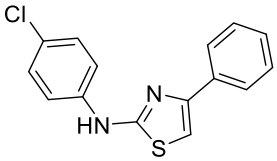
Red solid (234 mg, 88%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.0 Hz, 2H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.321 (d, *J* = 7.5 Hz, 1H), 7.29 (d, *J* = 3.5 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.78 (s, 1H), 2.33 (s, 3H). ^{13} C NMR (125 MHz, $CDCl_3$) δ 165.5, 151.2, 137.8, 134.6, 133.1, 130.0, 128.6, 127.9, 126.1, 119.0, 101.5, 20.8 (Me). NMR data are consistent with the literature report [22].

4.1.10. N-(4-Fluorophenyl)-4-phenylthiazol-2-amine (3j)



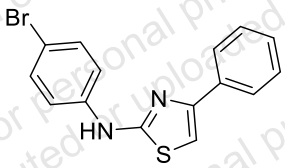
Brown solid (230 mg, 84%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.0 Hz, 2H), 7.44 - 7.36 (m, 4H), 7.33 - 7.28 (m, 1H), 7.05 (t, *J* = 8.5 Hz, 2H), 6.79 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.5, 157.5 (d, *J* = 246 Hz, CF), 151.6, 136.7, 134.6, 128.6, 128.1, 126.1, 120.7 (d, *J* = 8 Hz), 116.1 (d, *J* = 23 Hz), 101.7. NMR data are consistent with the literature report [23].

4.1.11. N-(4-Chlorophenyl)-4-phenylthiazol-2-amine (3k)



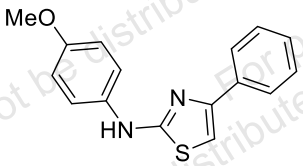
Yellow solid (246 mg, 86%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (d, *J* = 7.5 Hz, 2H), 7.39 (t, *J* = 7.5 Hz, 2H), 7.37 - 7.28 (m, 3H), 7.28 - 7.22 (m, 2H), 6.82 (s, 1H). 13C NMR (125 MHz, $CDCl_3$) δ 164.4, 151.3, 138.9, 134.3, 129.5, 128.6, 128.0, 127.7, 126.2, 119.5, 102.0. NMR data are consistent with the literature report [23].

4.1.12. N-(4-Bromophenyl)-4-phenylthiazol-2-amine (3l)



Red solid (284 mg, 86%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 (d, *J* = 7.5 Hz, 2H), 7.39 (t, *J* = 8.0 Hz, 4H), 7.32 (t, *J* = 7.0 Hz, 1H), 7.20 (d, *J* = 8.0 Hz, 2H), 6.83 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.4, 151.4, 139.4, 134.4, 132.2, 128.8, 128.1, 126.2, 119.8, 115.1, 102.1. NMR data are consistent with the literature report [11].

4.1.13. N-(4-Methoxyphenyl)-4-phenylthiazol-2-amine (3m)



White solid (251 mg, 89%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 2H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.27 - 7.20 (m, 3H), 6.83 (d, *J* = 9.0 Hz, 2H), 6.69 (s, 1H), 3.76 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.3, 156.4, 151.4, 134.7, 133.8, 128.5, 127.7, 126.1, 122.2, 114.7, 101.2, 55.6 (OMe). NMR data are consistent with the literature report [14].

CONCLUSION

In summary, a mild and efficient method for the synthesis of 2- (arylamino)thiazoles *via* three-component reaction of aryl ketones, aryl thioureas, and NBS using [bmim][BF_4] ionic liquid as a solvent has been developed. Advantageous features of the synthesis include mild reaction conditions, readily available starting materials, recyclability of ionic liquid, and high yield of products. Thirteen thiazoles were prepared in good to excellent yields. Structures of products were confirmed by ^{1}H and ^{13}C NMR. The reaction mechanism is being investigated in our lab and will be reported in the due course.

LIST OF ABBREVIATIONS

NBS = *N*-bromosuccinimide

PTT = Phenyl Trimethyl Ammonium Tribromide

CONSENT FOR PUBLICATION

Not applicable.

FUNDING

None.

CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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SUPPLEMENTARY MATERIAL

Supplementary Material is available on the publisher's website along with the published article, containing spectroscopic data of all synthesized compounds.

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