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New one-pot approach to regio-synthesis of substituted 2-aminothiazoles from the corresponding keto-aziridines

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Abstract Ring expansion of keto-aziridines to the corresponding 2-aminothiazoles (54–67 %) using ammonium thiocyanate in the presence of $RuCl_3$ under refluxing acetonitrile is described. A plausible mechanism for the synthesis of substituted 2-aminothiazoles has been proposed.

Keywords Aziridine · 2-Aminothiazoles · Ring expansion · $RuCl_3$

Introduction

Thiazole derivatives are the prominent players in the pharmaceutical research as they possess several biological properties [1–8].

Recently, thiazole analogues have been described as the possible core skeletons of A3 receptor antagonists with moderate affinity and selectivity [9–11]. In addition, thiazole derivatives are reported to be cyclin dependent kinase (CDK) inhibitors for the treatment of cancer and 11b-hydroxysteroid dehydrogenase type 1 (11b-HSD1) inhibitors for the treatment of metabolic disorders [12]. Particularly, 2-aminothiazoles have been reported to possess antitumor [13], antiviral [14, 15], antibacterial [16–18], anti-prion [19], psychotropic activities [20], anti-allergies [21], anti-hypertension [22], and anti-inflammation [23]. 2-Aminothiazole analogue MB06322 has also been used for the neuropeptide Y5 (NPY5) receptor for the treatment of eating disorders such as obesity and hyperphagia [24]. This class of compounds is well known as a prodrug for the treatment of type 2 diabetes [25] and aminothiazole-4-carboxylate derivatives as active compounds against *Mycobacterium tuberculosis* H37Rv and the α-ketoacyl-ACP synthase mtFabH [26]. Besides, the aromatic compounds containing sulfur are candidates for organic semiconductors [27]. 2-Aminothiazoles are generally prepared by the condensation of α-halo ketones/α-tosylketone with thioureas [28–34] or α-thiocyanato carbonyl compounds with aromatic or aliphatic amine hydrochlorides [35–38].

Despite work reported on the synthesis of 2-aminothiazole derivatives, novel and widely applicable methods for the synthesis of them are still in demand.

In continuation of our efforts in the development and synthesis of heterocycle molecules, especially from keto-aziridines [39–43], we have discovered a new and simple one-pot method for the synthesis of 2-aminothiazoles by the reaction of 2-aroyl-3-arylaziridines with ammonium thiocyanate in the presence of $RuCl_3$. This method describes a novel method which for the first time utilizes keto-aziridine for the synthesis of 2-aminothiazoles in a one-pot reaction. According to our knowledge, the synthesis of -5-arolyl-4-aryl-2-aminothiazole derivatives has not been reported in the literature, except, one report that shows synthesis of 4-phenyl-2-aminothiazole from α-halo ketones using ammonium thiocyanate in the presence of *N*-methylimidazole [43].

Results and discussion

Trans-2-aroyl-3-arylaziridines **(1)** were prepared via a Gabriel-Cromwell procedure, by bromination of the related α,β-unsaturated carbonyl compounds, followed by the

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reaction with ammonia solution in methanol at room temperature [44].

Our studies in this area began by choosing 2-benzoyl-3-phenylaziridine **1a** and NH_4SCN as the model substrates to test the possibility of reaction in acetonitrile but it failed. In order to evaluate the effect of catalyst in these reactions, we tried the reaction of 2-benzoyl-3-phenyl aziridine **1a** with NH_4SCN in the presence of H_2SO_4 and $AgNO_3$ or some Lewis acids such as I_2, $BF_3{\cdot}OEt_2$, $ZnCl_2$, $RuCl_3$, $Fe(NO_3)_3$, $AlCl_3$ or $NiCl_2$ in acetonitrile under refluxing conditions (Scheme 1; Table 1). The results of this study revealed that the ring expansion reaction in the presence of $AlCl_3$, $Fe(NO_3)_3$ and $NiCl_2$ (entry 1–3) gave no products but in the presence of H_2SO_4, $BF_3{\cdot}OEt_2$, $AgNO_3$ or $ZnCl_2$ (entry 4–7) gave a mixture of products and Chalcone with only a small amount (17–33 %) of the desired 2-amino-thiazole (**2a**), while with I_2 a moderate yield (45 %) of 2-aminothiazole (**2a**) was obtained (entry 8).

Surprisingly only $RuCl_3$ (20 % mol) was shown to be an effective catalyst for the ring expansion of 2-benzoyl-3-phenyl aziridine **1a** to 2-amino-4-benzoyl-5-phenyl thiazole **(2a)** (entry 9–10). No reaction occurred at lower temperatures even in the presence of $RuCl_3$ (20 % mol). In addition with KSCN in the presence of $RuCl_3$ (20 % mol) in refluxing acetonitrile, a small amount of the desired thiazole (**2a**) was obtained (33 %).

These observations encouraged us to develop a direct method for the conversion of keto-aziridines **1a–g** to the corresponding 2-aminothiazole **2a–g** (Scheme 2). The results of these ring expansion reactions are summarized in Table 2.

The structure of 2-aminothiazoles was deuced from IR, ^1H, ^{13}C NMR, as well as elemental analyses. The ^1H NMR spectrum showed a broad singlet at δ 5.2–5.7 ppm corresponding to the two hydrogens of amino group attached to C-2. Whereas in the ^{13}C NMR spectrum, the signal at δ 165.2–166.3 ppm corresponds to C-2 of thiazole ring. The FT-IR spectra of the compounds exhibited the broad peak at 3,400–3,450 and 1,650–1,660 cm^{-1} which corresponds to the primary amino and carbonyl group, respectively. Based on elemental analyses of products and the above spectral data, the structure of the compounds was assigned as 5-aroyl-4-aryl-2-aminothiazoles. These observations reveal that the 5-benzoyl-4-phenyl-2-aminothiazole can be obtained by the reaction of 2-benzoyl-3-phenylaziridine with ammonium thiocyanate in the presence of $RuCl_3$ in refluxing acetonitrile.

Table 1 Reaction of *trans*-2-benzoyl-3-phenyl aziridines **(1a)** with ammonium thiocyanate in the presence of some different catalysts

Entry	Catalyst[b]	Time (h)	Yield (%)[a]	
			2a	**Chalcone**
1	$AlCl_3$	2	0	0
2	$Fe(NO_3)_3$	2	0	50
3	$NiCl_2$	2	0	0
4	H_2SO_4	7	19	21
5	$BF_3{\cdot}OEt_2$	2	28	0
6	$AgNO_3$	2	33	30
7	$ZnCl_2$	7	17	24
8	I_2	2	45	35
9	**$RuCl_3$**	**2.5**	**56**	**10**
10	$RuCl_3$[c]	2.5	54	28
11	$RuCl_3$[d]	8	29	23
12	–	8	0	12

[a] Isolated yield after purification

[b] Catalyst (20 % mol)

[c] $RuCl_3$ (60 % mol)

[d] In room temperature and in the presence of $RuCl_3$ (20 % mol)

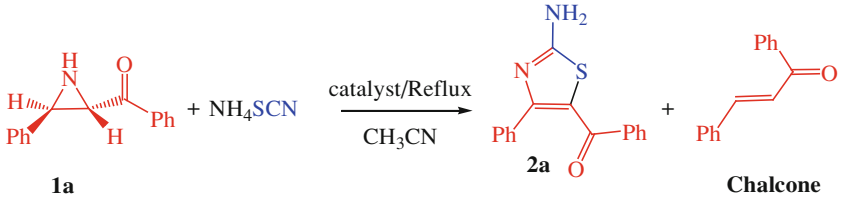
Bold values indicate the best conditions for transformation of keto-aziridine to the corresponding 2-aminothiazole

We previously reported the mechanism of the iodide ion catalyzed isomerization of 1-acylaziridines by attack of the nucleophile on the C-2 1-acylaziridines, which subsequently cyclizes to the oxazoline [39]. Now, the action of thiocyanate ion like iodide on 2-aroyl-3-aryl aziridine would involve a ring opening processes. No trace of another regio-isomer of these aminothiazoles were detected.

Although the exact mechanism of the reaction is not very clear, one possible sequence of steps to explain the conversion of keto-aziridines to corresponding 2-amino-thiazoles involves activation of aziridine ring with $RuCl_3$ by coordinating with the nitrogen and carbonyl group of the aziridine, then the reaction of thiocyanate with aziridine to give intermediate **A**. After that, amino group attacks the thiocyanate group of the intermediate **A**, which leads to the intermediate **B** and which is oxidized to 2-aminothiazole **2** in the presence of air (Scheme 3).

In conclusion, this work describes an efficient and novel one-pot method for the synthesis of 2-aminothiazole derivatives from the corresponding keto-aziridines with ammonium thiocyanate in the presence of $RuCl_3$ under refluxing conditions. The synthesis of 2-aminothiazoles

Scheme 1 The reaction of *trans*-2-benzoyl-3-phenyl aziridines with ammonium thiocyanate in the presence of different catalysts



1a **Chalcone**

Scheme 2 Synthesis of *trans*-2-aminothiazole from *trans*-2-aroyl-3-arylaziridines and ammonium thiocyanate in the presence of $RuCl_3$

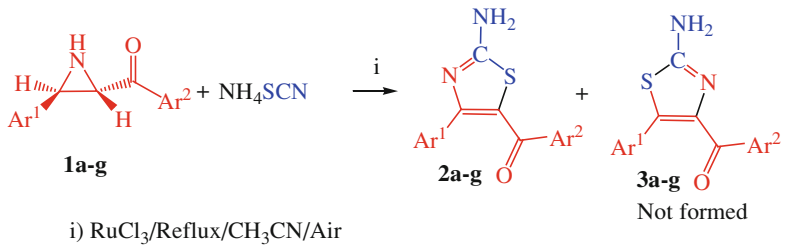
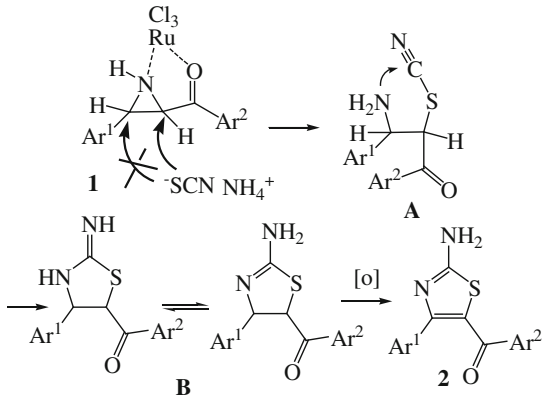


Table 2 Synthesis of 5-aroyl-4-aryl-2-aminothiazoles **(2)** from *trans*-2-aroyl-3-arylaziridines **(1)** with ammonium thiocyanate in the presence of $RuCl_3$ (20 % mol)

Entry	Ar_1	Ar_2	Time (h)	Yield (%)[a]
2a	C_6H_5	C_6H_5	7	56
2b	3-$NO_2C_6H_4$	C_6H_5	7	55
2c	4-ClC_6H_4	C_6H_5	7	60
2d	2,4-$Cl_2C_6H_3$	4-ClC_6H_4	7	62
2e	2,4-Cl_2C_6H3	C_6H_5	7	54
2f	3-$NO_2C_6H_4$	4-ClC_6H_4	2.5	55
2g	C_6H_5	4-BrC_6H_4	7	67

[a] Isolated yield after purification



Scheme 3 Proposed mechanism for synthesis of 5-aroyl-4-aryl-2-aminothiazoles **(2)** from *trans*-2-aroyl-3-arylaziridines **(1)** and ammonium thiocyanate in the presence of $RuCl_3$

from the corresponding keto-aziridines has not been reported in the literature.

Experimental

All yields refer to isolated products after purification by column chromatography or distillation in vacuum. Products were characterized with IR and ^{1}HNMR spectra, TLC, melting, and boiling points. NMR spectra were recorded on a Bruker AMX-400 spectrometer (^{1}H at 400 MHz and ^{13}C at 100 MHz) in $CDCl_3$ with chemical shift values in ppm downfield from TMS and IR spectra were recorded on FT Infrared spectroscope JASCO, FT/IR-6300. All solvents used were dried and distilled according to standard procedures.

Typical procedure for the synthesis of 2-aminothiazoles **(2)**

$RuCl_3$ (0.2 mmol) was added to a solution of aziridines (**1**) (1.0 mmol) and ammonium thiocyanate (1.0 mmol) in CH_3CN (10 mL). The mixture was refluxed for the stipulated time (Table 2). After the desired product formation indicated by TLC/or GC–MS, the solvent was evaporated in rotary vacuum evaporator to obtain the crude products. The crude product was purified by silica gel column chromatography (EtOAc/Hexane: 2/5) to provide the desired 2-aminothiazole (54–67 %).

5-Benzoyl-4-phenyl-2-aminothiazole **(2a)**

Yellow solid, Mp 94–98 °C; IR (KBr): 3,422, 3,081, 1,635, 1,597, 1,527, 1,448, 758, 686 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.70–7.68 (m, 2H), 7.40–7.11 (m, 8H), δ 5.7 (br s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm): 195.5, 165.6, 151.9, 136.4, 129.9, 129.8, 129.6, 129.3, 129.0, 128.8, 128.3, 123.9. Anal. Calcd for $C_{16}H_{12}N_2OS$: C, 68.55; H, 4.31; N, 9.99 %. Found: C, 68.43; H, 4.35; N, 9.97 %.

5-Benzoyl-4-(3-nitrophenyl)-2-aminothiazole **(2b)**

Yellow solid, Mp 122–125 °C; IR (KBr): 3,428, 3,062, 1,638, 1,590, 1,527, 1,355, 1,440 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.02 (d, J = 2.0 Hz, 1H), 7.8 (d, J = 7.2 Hz, 2H), 7.6 (d, J = 7.6 Hz, 1H), 7.5 (d, J = 3.2 Hz, 1H), 7.41–7.25 (m, 2H), 7.0 (t, J = 7.9 Hz, 1H), 5.5 (br s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm): 189.9, 165.3, 152.0, 138.2, 136.1, 133.1, 132.2, 132.1, 132.0, 130.6, 129.8, 129.2, 128.7, 124.0 ppm. Anal. Calcd for $C_{16}H_{11}N_3O_3S$: C, 59.07; H, 3.41; N, 12.92 %. Found: C, 58.98; H, 3.45; N, 12.87 %.

5-Benzoyl-4-(4-chlorophenyl)-2-aminothiazole **(2c)**

Yellow solid, Mp 129-132 °C; IR (KBr): 3,417, 3,077, 1,639, 1,597, 1,501, 755, 679 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.8 (d, J = 8.2 Hz, 2H), 7.40–7.26 (m, 7H), 5.4 (br s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm):

192.4, 165.2, 131.6, 130.5, 129.6, 129.3, 128.9, 128.7, 128.6, 128.5, 128.4, 128.2 ppm. Anal. Calcd for $C_{16}H_{11}ClN_2OS$: C, 61.05; H, 3.52; N, 8.90 %. Found: C, 61.13; H, 3.55; N, 8.92 %.

5-(4-Chlorobenzoyl)-4-(2,4-dichlorophenyl)-2-aminothiazole **(2d)**

Yellow solid, Mp 132–135 °C; IR (KBr): 3,441, 3,075, 1,640, 1,588, 1,503, 1,448, 751, 685 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) (δ, ppm):7.9 (d, J = 8.6 Hz, 2H), 7.41–7.37 (m, 3H), 7.28 (d, J = 2.4 Hz, 1H), 7.20 (dd, J = 8.4, 2.0 Hz, 1H), 5.5 (br s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm): 189.2, 165.3, 149.8, 145.5, 139.3, 137.2, 135.5, 132.8, 131.6, 129.7, 129.1, 128.4, 123.2, 122.0 ppm. Anal. Calcd for $C_{16}H_9Cl_3N_2OS$: C, 50.09; H, 2.36; N, 7.30 %. Found: C, 50.12; H, 2.55; N, 7.32 %.

5-Benzoyl-4-(2,4-dichlorophenyl)-2-aminothiazole **(2e)**

Yellow solid, Mp 120-122 °C; IR (KBr): 3,445, 3081, 1,625, 1,585, 1,495, 1,441, 748, 686 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) (δ, ppm): 7.9 (d, J = 7.9 Hz, 2H), 7.2–7.4 (m, 5H), 7.0 (dd, J = 8.2, 2 Hz, 1H), 5.2 (br s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm): 183.4, 166.1, 143.1, 136.6, 133.3, 132.8, 131.5, 131.0, 129.6, 128.9, 128.0, 126.9, 124.3, 123.1 ppm. Anal. Calcd for $C_{16}H_{10}Cl_2N_2OS$: C, 55.03; H, 2.89; N, 8.02 %. Found: C, 54.98; H, 2.91; N, 8.09 %.

5-(4-Chlorobenzoyl)-4-(3-nitrophenyl)-2-aminothiazole **(2f)**

Yellow solid, Mp 108–111 °C; IR (KBr): 3,422, 3,081, 1,635, 1,597, 1,527, 1,355, 1,451, 751, 681 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.2 (d, J = 2.1 Hz, 1H), 8.1 (dd, J = 8.2, 2.1 Hz, 1H), 7.8 (d, J = 7.9, 2.0 Hz, 2H), 7.7 (d, J = 7.6 Hz, 1H), 7.5 (t, J = 8.2 Hz, 1H), 7.4 (dd, J = 8.2, 2.0 Hz, 2H), 5.7 (br s, 2H).^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm): 188.2, 165.7, 148.6, 146.0, 144.6, 139.7, 135.3, 132.3, 131.6, 129.4, 129.1, 128.6, 124.2, 123.2 ppm. Anal. Calcd for $C_{16}H_{10}ClN_3O_3S$: C, 53.41; H, 2.80; N, 11.68 %. Found: C, 53.48; H, 2.91; N, 11.69 %.

5-(4-Bromobenzoyl)-4-phenyl-2-aminothiazole **(2g)**

Yellow solid, Mp 135–138 °C; IR (KBr): 3,439, 3,086, 1,643, 1,591, 1,511, 1,451, 738, 666 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) (δ, ppm): 8.2 (d, J = 8.1 Hz, 2H), 7.41–7.37 (m, 3H), 7.36–7.22 (m, 4H), 5.5 (br s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) (δ, ppm): 189.4, 166.2, 131.9, 130.4, 129.5, 129.0, 128.8, 128.7, 128.6, 128.5, 128.4, 128.1 ppm. Anal. Calcd for $C_{16}H_{11}BrN_2OS$: C, 53.49; H, 3.09; N, 7.80 %. Found: C, 53.45; H, 2.99; N, 7.69 %.

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