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One-pot rapid synthesis of thiazole-substituted pyrazolyl-4-thiazolidinones mediated by diisopropylethylammonium acetate

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Abstract A convenient one-pot, rapid and scalable synthetic protocol has been developed for recently reported anti-inflammatory agents, thiazole-substituted pyrazolyl-4-thiazolidinones. Quantitative multicomponent cyclocondensation of 3-(4-methyl-2-substituted thiazol-5-yl)-1-phenyl-1*H*-pyrazole-4-carbaldehydes (**5a**, **b**), amines and mercaptoacetic acid has been carried out in safer medium, diisopropylethylammonium acetate, at room temperature. The optimisation details of the developed novel protocol are recorded.

Keywords Diisopropylethylammonium acetate (DIPEAc) · 4-Thiazolidinone · Multicomponent reaction

Introduction

Heterocyclic compounds have attracted a lot of attention because of their varied biological activities. Specifically, five-membered heterocycles with two heteroatoms have received particular attention, having proven utility in medicinal chemistry. Among them, 4-thiazolidinones represent an important class of five-membered heterocycles. Some of the thiazolidinones are found to possess interesting biological activities such as anticancer, antimalarial, tuberculostatic, antihistaminic, anticonvulsant, antibacterial and antiarrythmic activity [1]. Ralitoline, etozoline, pioglitazone and thiazolidomycin have this heteryl ring and are already in the market as medicaments [2]. This diversity in the biological response profile has attracted much attention from many researchers to explore this skeleton for its multiple potential activities. Motivated by the above recently reported

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observations, we reported new anti-inflammatory agents [3] having 4-thiazolydinoyl moiety.

Thiazolidinones are obtained by either one- or two-step synthesis processes. However, the most widely used approach to synthesise this ring system is through one-pot three-component tandem reaction between carbonyl compounds, primary amines, and mercaptoacetic acid or its derivatives. The reaction proceeds via initial formation of an imino intermediate, which undergoes attack by a sulphur nucleophile, i.e. mercaptoacetic acid, followed by intramolecular cyclisation with expulsion of water to yield the desired product. It is generally believed that the last step, i.e. removal of the water molecule, is rate determining and seems to be critical for obtaining 4-thiazolidinones in high yield [4]. Therefore, a number of strategies have been developed to remove the water molecules formed during the reaction in order to obtain high yield of the desired product. The media used for the condensation are volatile organic solvents such as toluene, benzene, 1,4-dioxane and tetrahydrofuran (THF). There are reports of acceleration of the above cyclocondensation using catalysts and desiccants such as *N*,*N*′-dicyclohexylcarbodiimide (DCC) [5], *O*-(benzotriazolyl)-*N*,*N*,*N*′,*N*′-tetramethyluronium hexafluorophosphate (HBTU) [6], ferrite [7], $ZnCl_2$ [8], sodium sulphate [9], [bmim][PF_6] [10, 11] and activated fly ash [12]. Use of microwave heating [13], and solid-phase [14] and polymer-supported [15] systems to run the cyclocondensation leading to 2,3-disubstituted 4-thiazolidinones has also been reported.

However, most of the methods described above suffer from certain limitations. To achieve the cyclocondensation, one must incorporate azeotropic distillation, microwave irradiation [16], use of stoichiometric amount of hazardous and costly catalysts, auxiliary reagents such as desiccants, inert and dry atmosphere or prolonged heating. Thus, a convenient, versatile, rapid and high-yielding synthetic method remains in demand to fulfil timely supply of a library of 4-thiazolidinones for biological evaluation and enrichment of the medicinal chemist's toolbox.

Recently, room-temperature ionic liquids (RTILs) have been found to be useful as green media for value-added transformations [17]. RTILs act as 'neoteric solvents' for a broad range of chemical and industrial processes. ILs are emerging as more promising catalysts in various fields such as organic synthesis, materials science, electrochemistry and separation technology [18–22]. The ability to dissolve many organic and inorganic substances makes RTILs eco-friendly reaction media/catalysts. ILs completely consist of weakly coordinating ions, i.e. organic cation with inorganic/organic anion, possessing desirable properties, and are liquids at or close to room temperature. An interesting aspect is that ILs not only act as media for organic substrates but also display catalytic behaviour [17].

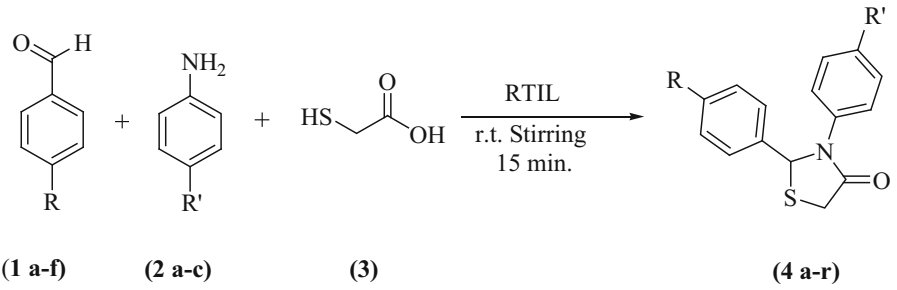
Owing to the significance of 4-thiazolidinones and considering the urgent need for cost-effective and environmentally benign novel synthetic protocols for therapeutically interesting 4-thiazolidinones, our group has developed and reported various synthetic methods having one or another type of advantage. While conducting multicomponent cyclocondensation leading to 4-thiazolidinones and to accelerate the rate of the cyclocondensation, in our reports we used alternative

media such as PEG-400 [23] and *N*-methylpyridinium tosylate (ionic liquid) [24]. We even reported the catalytic role of heterogeneous catalysts such as silica chloride [25] and biocatalyst, baker's yeast [26] in accelerating the cyclocondensation. Synthesis of the title compounds has been carried out by traditional/classical protocols and recently even by our reported alternative synthetic method [3]. It has been noticed that the cyclocondensations carried out in these reports were under prolonged heating and finally gave moderate yields. Therefore, we felt that there was still scope to develop a rapid, benign, cost-effective multicomponent one-pot method to obtain the title products.

Results and discussion

Considering the significance of RTILs, here in the search for the best experimental reaction conditions, screening of different room-temperature ionic liquids was carried out for cyclocondensation of benzaldehyde **1a**, aniline **2a** and mercaptoacetic acid **3** (Scheme 1) as a standard model reaction. For evaluation of the effect of solvents on the one-pot model reaction, initially various solvents such as toluene, 1,4-dioxane, acetonitrile, methanol, ethanol and various freshly prepared room-temperature ionic liquids [27] were screened at room temperature for the model reaction (Table 1, entries 1–10). Amongst them, diisopropylethylammonium acetate (DIPEAc) and RTIL were found to be an excellent medium and catalyst, furnishing the product in excellent yield of 88 % (Table 1, entry 10). In the absence of solvent, the neat one-pot multicomponent cyclocondensation at room temperature did not exhibit similar reaction condensations.

To confirm the role of DIPEAc as a catalyst, one-pot cyclocondensation of benzaldehyde, aniline and mercaptoacetic acid was separately attempted in media, viz. acetonitrile, ethanol and dichloromethane, in presence of 20, 50 and 100 mol% of DIPEAc. It was noted that, at R.T. in absence of DIPEAc, condensation could not occur in these media. However, the condensation was found to take place at moderate rate. It was also observed that, when equimolar quantity of DIPEAc was incorporated in these media, the rate of condensation was noticeable (Table 2). This



Scheme 1 Synthesis of 4-thiazolidinones from aromatic carbonyl compounds, aromatic amines and mercaptoacetic acid in RTIL

Table 1 Screening of solvents

Entry	Solvent	Time (min)	Yield (%)[a]
1	Toluene	15	No condensation
2	1,4-Dioxane	15	No condensation
3	Acetonitrile	15	No condensation
4	Methanol	15	No condensation
5	Ethanol	15	No condensation
6	Dichloromethane	15	No condensation
7	Piperidine ammonium acetate	15	17
8	Pyrrolidine ammonium acetate	15	22
9	Triethylethylammonium acetate	15	41
10	Diisopropylethylammonium acetate	15	88

Reaction conditions: **1a** (0.009 mol), **2a** (0.009 mol), **3** (0.028 mol), in solvent (5 ml), stirred at room temperature

[a] Isolated yields

Table 2 Effect of catalyst (DIPEAc) concentration in presence of solvent

Entry	Solvent	Time (min)	Yield (%)[a] in presence of DIPEAc		
			20 mol%	50 mol%	100 mol%
1	Acetonitrile	15	36	38	51
2	Ethanol	15	28	34	43
3	Dichloromethane	15	21	28	33

Reaction conditions: **1a** (0.009 mol), **2a** (0.009 mol), **3** (0.028 mol), in solvent (5 ml), stirred at room temperature

[a] Isolated yields

Table 3 Recovery and recycling of DIPEAc

Entry	Run	Yield (%)[a]
1	I	88
2	II	86
3	III	80
4	IV	76

Reaction conditions: **1a** (0.009 mol), **2a** (0.009 mol), **3** (0.028 mol), DIPEAc (5 ml) stirred at room temperature for 15 min

[a] Isolated yields

observation is in agreement with the idea that DIPEAc plays a role not only as a medium but also as a catalyst in this cyclocondensation. Therefore, DIPEAc was chosen as the medium and catalyst of choice for further optimisation studies.

We studied the reusability of room-temperature ionic liquid (DIPEAc). It was observed that the recovered IL worked with good efficiency up to the second run (Table 3, entries 1, 2), while in the third and fourth runs (Table 3, entries 3, 4) the product yield decreased slightly.

Inspired by these observations and to generalise the synthetic procedure, a variety of electronically divergent aromatic aldehydes and aromatic amines were treated with mercaptoacetic acid at room temperature in presence of DIPEAc. Aromatic aldehydes with several functionalities such as Cl, OH, CH_3, OCH_3 and NO_2 were found to be compatible under the optimised reaction condition. Aldehydes having no substituent on phenyl ring reacted smoothly, and the products were obtained in excellent yields ranging from 88 to 93 % (Table 4, entries 1–3). Electron-donating group such as OCH_3, CH_3 and OH present in the aldehyde afforded moderate yields of the products (Table 4, entries 4–12). Presence of Cl group in the aldehydes (Table 4, entries 13–15) showed good effect on yield. Aldehydes possessing electron-withdrawing group gave moderate yields (Table 4, entries 16–18). The products, 4-thiazolidinones, isolated in this study were characterised and their M.P.s

Table 4 Synthesis of 4-thiazolidinones (**4a–r**)

Entry	Compound	R	R′	Yield (%)[a]	Melting point (°C)[b]
1	**4a**	H	H	88	105–107
2	**4b**	H	CH_3	73	112–115
3	**4c**	H	Cl	93	111–113
4	**4d**	OCH_3	H	80	110–111
5	**4e**	OCH_3	CH_3	78	148–150
6	**4f**	OCH_3	Cl	84	157–160
7	**4g**	CH_3	H	74	115–117
8	**4h**	CH_3	CH_3	79	120–122
9	**4i**	CH_3	Cl	82	155–157
10	**4j**	OH	H	76	182–184
11	**4k**	OH	CH_3	81	210–211
12	**4l**	OH	Cl	75	158–160
13	**4m**	Cl	H	75	130–132
14	**4n**	Cl	CH_3	79	162–165
15	**4o**	Cl	Cl	78	124–126
16	**4p**	NO_2	H	76	105–107
17	**4q**	NO_2	CH_3	81	157–159
18	**4r**	NO_2	Cl	70	139–141

Reaction conditions: aryl carbaldehyde (0.009 mol), **2a** (0.009 mol), **3** (0.028 mol), DIPEAc (5 ml) stirred at room temperature for 15 min

[a] Isolated yields

[b] The melting points of these products prepared in this work are in good agreement with those reported in literature [24, 25, 28]

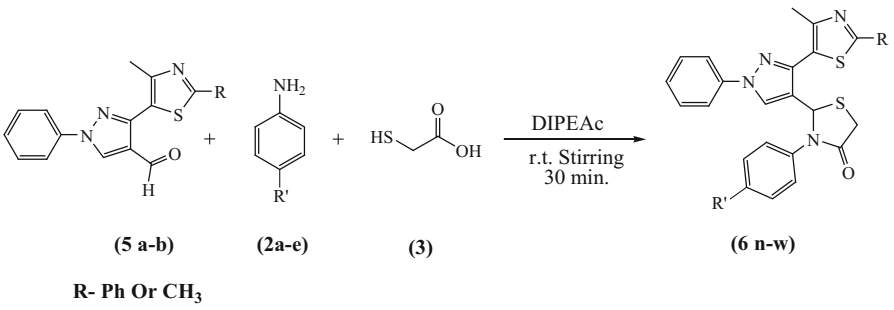
and other physical data found to be in good agreement with those reported in literature [24, 26, 28].

Encouraged by this success and in continuation of our endeavour towards synthesis of bioactive heterocycles, here we applied the above same strategy for one-pot cyclocondensation of heteryl aldehydes to obtain the thiazolidinones (Scheme 2). Hence, we attempted one-pot cyclocondensation of 3-(4-methyl-2-substituted thiazol-5-yl)-1-phenyl-1*H*-pyrazole-4-carbaldehydes (**5a**, **b**), aromatic amines (**2a**–**e**) and mercaptoacetic acid (**3**) under the above optimised condition to obtain recently reported anti-inflammatory agents, 2-(3-(4-methyl-2-substituted thiazol-5-yl)-2-phenyl-1*H*-pyrazol-4-yl)-3-phenylthiazolidin-4-ones [3].

It was observed that 3-(4-methyl-2-substituted thiazol-5-yl)-1-phenyl-1*H*-pyrazole-4-carbaldehydes reacted cleanly under the above reaction condition (Scheme 2) and the products were obtained in 73 to 88 % yields without any difficulty within 30 min at room temperature. Aromatic amines having halide groups reacted smoothly, and the products were obtained in excellent yields ranging from 79 to 88 % (Table 5, entries 3–5, 8–10). Slightly moderate yields of the product were obtained when heterocyclic aldehydes were allowed to react with aromatic amines having electron-donating group such as CH_3 (Table 5, entries 2, 7). Aniline gave moderate yield with the heterocyclic aldehyde (Table 5, entries 1, 6). All the products (**6n**–**w**) were characterised by infrared (IR), ^{1}H nuclear magnetic resonance (NMR), ^{13}C NMR and mass analysis, and the analytical data were in agreement with our earlier reports [3].

A plausible mechanism for the rapid formation of 4-thiazolidinones using RTIL, diisopropylethylammonium acetate (DIPEAc) would be as follows:

1. The rate acceleration for the formation of 4-thiazolidinone has been found to be enhanced due to the dual nature of the ionic liquid, i.e. as a catalyst and medium. RTIL might be helping to create a high initial concentration of the reactants in solvation at room temperature.
2. The electrophilic character of carbonyl carbons of the aldehyde would have been enhanced because of their non-covalent interaction with ammonium cation of the RTIL, diisopropylethylammonium acetate.



Scheme 2 Synthesis of 2-(3-(4-methyl-2-substituted thiazol-5-yl)-2-phenyl-1*H*-pyrazol-4-yl)-3-phenylthiazolidin-4-ones

Table 5 Synthesis of 2-(3-(4-methyl-2-substituted thiazol-5-yl)-2-phenyl-1*H*-pyrazol-4-yl)-3-phenylthiazolidin-4-ones

Entry	Compound	R	R′	Yield (%)[a]	Melting point (°C)[b]
1	**6n**	Ph	H	73	208–210
2	**6o**	Ph	CH_3	75	169–171
3	**6p**	Ph	F	82	178–180
4	**6q**	Ph	Cl	85	180–182
5	**6r**	Ph	Br	81	165–167
6	**6s**	CH_3	H	74	137–139
7	**6t**	CH_3	CH_3	77	155–157
8	**6u**	CH_3	F	88	163–165
9	**6v**	CH_3	Cl	79	175–177
10	**6w**	CH_3	Br	80	171–173

Reaction conditions: heterocyclic carbaldehyde **5a** (0.0028 mol), **2a** (0.0028 mol), **3** (0.0086 mol), DIPEAc (5 ml) stirred at room temperature for 30 min

[a] Isolated yields

[b] The melting points of these products prepared in this work are in good agreement with those reported in literature [3]

3. Acetate anion may be responsible for enhancing nucleophilicity of mercapto group of mercaptoacetic acid, causing its facile addition on the imino intermediate generated in situ.

These factors might be responsible for accelerating the rate of cyclocondensation and hence enhancing yields of the desired 4-thiazolidinones.

Experimental

The chemicals used were of laboratory grade. Melting points of all synthesised compounds were determined in open capillary tubes and are uncorrected. ^{1}H NMR spectra were recorded on a Bruker DRX-300 and 400 MHz NMR spectrometer and ^{13}C NMR spectra were recorded on a Bruker DRX-75 and 100 MHz NMR in $CDCl_3$/dimethyl sulphoxide (DMSO)-d_6 using tetramethylsilane (TMS) as internal standard, and chemical shifts are in δ (ppm). Direct analysis in real time (DART) mass spectra were recorded on a Vokudelna ES + 2000. High-resolution mass spectra (HRMS) were recorded on an Agilent 6520 quadrupole time-of-flight (QTOF) electrospray ionisation (ESI) HRMS instrument. The purity of each compound was checked by thin-layer chromatography (TLC) using silica gel, 60F254 aluminium sheets as adsorbent, with visualisation accomplished by iodine/ultraviolet light.

General procedure for synthesis of 2,3-diaryl-4-thiazolidinones (**4a**–**r**)

A mixture of aromatic carbaldehydes (**1a**–**f**) (0.009 mol), aromatic amines (**2a**–**c**) (0.009 mol) and mercaptoacetic acid (**3**) (0.028 mol) was dissolved in DIPEAc (5 ml), then the solution was stirred at room temperature for 15 min. Reaction progress was monitored by TLC using ethyl acetate:hexane (3:7) as solvents. Then, after 15 min, reaction mass was poured on cold water and washed with $NaHCO_3$, and the obtained crude products were filtered and crystallised from ethanol. The reaction is depicted in Scheme 1.

General procedure for 2-(3-(4-methyl-2-substituted thiazol-5-yl)-2-phenyl-1*H*-pyrazol-4-yl)-3-phenylthiazolidin-4-ones (**6n**–**w**)

A mixture of 3-(4-methyl-2-substituted thiazol-5-yl)-1-phenyl-1*H*-pyrazole-4-carbaldehydes (**5a**, **b**) (0.0028 mol), aromatic amines **(2a-e)** (0.0028 mol) and mercaptoacetic acid (**3**) (0.0086 mol) was dissolved in DIPEAc (5 ml), then the solution was stirred at room temperature for 30 min. Reaction progress was monitored by TLC using ethyl acetate:hexane (3:7) as solvents. Then, after 30 min, reaction mass was poured on cold water and washed with $NaHCO_3$, and the obtained crude products were filtered and crystallised from ethanol. The reaction is depicted in Scheme 2.

General procedure for synthesis of diisopropylethylammonium acetate (DIPEAc)

A mixture of acetic acid (0.02 mol) and *N*-ethyl-*N*-isopropylpropan-2-amine (0.02 mol) was stirred at 0–10 °C for 30 min. The viscous liquid, diisopropylethylammonium acetate, was obtained [26].

Recovery of ionic liquid

An attempt was made to recover the room-temperature ionic liquid (DIPEAc). After completion of the reaction, the reaction mixture was poured on ice water, and the product was separated by filtration. The filtrate was subjected to evaporation of water to get viscous liquid, which on cooling gave the ionic liquid. The recovered ionic liquid was reused for three more cycles of the same cyclocondensation and found to act satisfactorily.

Spectral data for representative compounds

2-(4-Chlorophenyl)-3-p-tolylthiazolidin-4-one (***4n***) ^{1}H NMR ($CDCl_3$, 300 MHz) $\delta = 2.26$ (s, 3H, CH_3), 3.83–3.86 (d, methylene 1H), 3.96–4.02 (d, methylene 1H), 5.99 (s, 1H, CH), 7.01–7.29 (m, overlapping 8H, Ar–H). ^{13}C NMR ($CDCl_3$, 75 MHz) $\delta = 21.2$, 33.5, 65.1, 125.3, 125.7, 127.3, 129.2, 130.1, 130.3, 134.6, 134.8, 137.5, 142.0, 171.0. DART-MS (ESI+): (*m*/*z*, % intensity): 304 (M+, 100) $C_{16}H_{14}ClNOS$.

2-(3-(4-Methyl-2-phenylthiazol-5-yl)-2-phenyl-1H-pyrazol-4-yl)-3-phenylthiazolidin-4-one (***6n***) IR (KBr) V_{max}: 3,045, 2,910, 1,680, 1,585, 765 cm^{-1}. ^1H NMR (DMSO-d_6, 400 MHz) $d = 2.48$ (3H, s, CH_3), 3.85–3.89 (1H, d, $J = 16$ Hz), 3.97–4.01 (1H, d, $J = 16$ Hz), 6.41 (1H, s), 7.08–7.91 (14H, m, Ar–H), 8.85 (1H, s, pyrazolyl). ^{13}C NMR (DMSO-d_6, 100 MHz) $\delta = 16.2$, 32.8, 55.7, 115.4, 115.8, 118.2, 121.1, 122.0, 126.1, 127.0, 128.2, 128.3, 129.2, 129.8, 130.7, 132.8, 133.6, 138.5, 141.9, 152.2, 165.8, 170.1. HRESIMS *m/z* (pos): 529.0923 $[M + H]^+$ for $C_{28}H_{21}ClN_4OS_2$.

Conclusions

We have developed an exceedingly simple, scalable and novel synthetic protocol for synthesis of 4-thiazolidinone derivatives. To the best our knowledge, this is the first report of application of diisopropylethylammonium acetate (DIPEAc) for synthesis of 4-thiazolidinones at room temperature. The new catalytic and green medium procedure offers the following distinct advantages: (1) one-pot multicomponent synthesis of thiazolidinones with broad substrate scope, (2) no requirement for additional reagents, (3) high yields, (4) ease of product isolation/purification, (5) ease of preparation/handling of RTIL and (6) catalyst reuse that fulfils the triple bottom-line philosophy of green chemistry. Therefore, this one-pot multicomponent procedure clearly represents an appealing methodology for synthesis of 4-thiazolidinones in both academia and pharmaceutical industry, and is even better than our earlier efforts towards synthesis of 2-(3-(4-methyl-2-substituted thiazol-5-yl)-2-phenyl-1*H*-pyrazol-4-yl)-3-phenylthiazolidin-4-ones [3].

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