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Synergy of binary ionic liquids in a three component one-pot synthesis of 3,4,6-triarylpyridazine at ambient temperature

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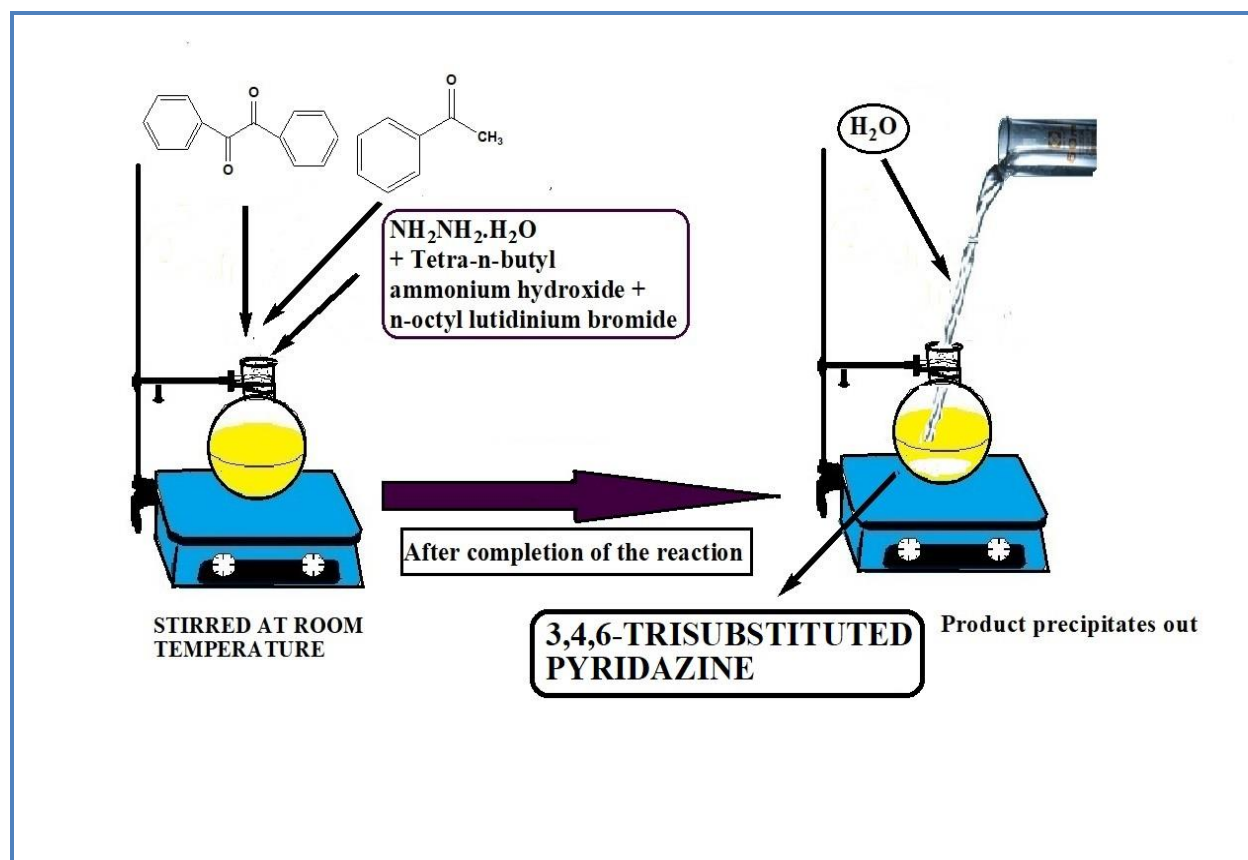
KEYWORDS

Pyridazine
Pyridinium based ionic liquid
Hydrazine
Tetra-n-butyl ammonium hydroxide
Acetophenones

ABSTRACT

A mixture of two ionic liquids have been used for a green synthesis of 3,4,6-triarylpyridazine in high yield and at room temperature. The two ionic liquids acts in tandem. The advantage of this method is a room temperature synthesis of the target compounds using green methodology. Product recovery is simple and the mixture of ionic liquids can be recovered and recycled.

Graphical Abstract



Introduction

Pyridazines are structurally important moiety in several biologically active and medicinally potent. Pyridazine and its derivatives continue to attract a great deal of attention due to their wide occurrence in natural products. Additionally, pyridazines interfere in various regulatory processes of enzymes and hence find wide applications in drug design. The pyridazine nucleus is an integral to several medicinally useful drugs such as analgesics, antibacterial, antidepressant, and anti-diabetic drugs [1]. Agricultural science takes advantage of their high biological activity in the preparation of potent insecticides [2], fungicides [3, 4], cardiotonics [5], and bacteriocides [6]. Functionalized pyridazines are also versatile building blocks for synthesis of the natural product. Their widespread use has attracted a great deal of attention from the synthetic organic chemists. Also, new methods of synthesis of various pyridazines with the unique structural features have been already reported and reviewed [7]. Classical methods of synthesis of pyridazines and their derivatives are including the Diazo-Wittig reaction starting from 1,3-diketones, 1,4-dicarbonyl compounds & hydrazine, reactions

involving 1,2 diketones, hydrazine derivatives in the presence of an ester containing an active methylene group [8].

Some of the recent methods include the application of Morita-Baylis-Hillman reaction using carbonates and the diazo compounds [9], *via* ring closure metathesis [10], retro-ene assisted palladium catalyzed synthesis using sonagashira condition [11], from 1,3-diketones and methyl ketones in a metal-free synthetic procedure [12], a one-pot procedure starting from an aromatic ketones and hydrazine, TMDA and butyl lithium [13] besides other major synthetic routes [14–16]. Most of the reported methods use expensive reagents that requires long reaction time and do not satisfy the requirements of the green chemical techniques. In a broad program of developing an efficient and eco-friendly method of synthesis of this versatile heterocycles, herein we report a general synthesis of trisubstituted pyridazine at room temperature and under mild condition using a binary mixture of ionic liquids.

ILs are recognized as environmentally harmless media because of their low vapor pressure, high thermal and chemical stability. Their unique properties and the possibility of easy recovery and reusability resulted in their wide acceptance as reaction media and catalyst for promoting various transformations. They are also known to influence the rate and selectivity of reactions [17–23]. Among the different types of ILs, the pyridinium based ionic liquids are novel in comparison with their imidazolium counterparts in stability, selectivity, and catalytic role. Exploration of their true potential of a variety of designer ILs are still being investigated [24].

Experimental

Matreials and methods

The used chemicals were purchased from Loba Chieme (India) and used as received. Melting points were recorded in open capillaries and are uncorrected. All new products were characterized by recording the IR spectra in KBr pellets in a Perkin Elmer FT-IR 1600 spectrophotometer and ^1H and ^{13}C NMR spectra were recorded in Bruker Bio Spin 300 MHz spectrometer using CDCl_3 as the solvent and TMS as the internal standard. Mass spectra of compounds were recorded in Micromass QTOF ESI-MS instrument (model HAB273) and conductivity measurement of the ionic liquids were obtained in Wayne Kerr Precision comp. Analyser 6440B.

Preparation of 1-n-octyl-3,5-dimethylpyridinium bromide

0.1 Mol of 3,5-dimethylpyridine and 0.1 mol of *n*-octylbromide were mixed and stirred for 20 hrs in the dark. A pale yellow coloured solid precipitated on completion of the reaction. The recovered

product was found to be pure and further purification was unnecessary. Being hygroscopic, the IL obtained was stored in a desiccator.

Preparation of 1-n-butyl-3,5-dimethylpyridinium bromide

0.1 Mol of 3,5-dimethyl- pyridine and 0.1 mol of *n*-butylbromide were mixed and stirred for 24 hrs in the dark. Precipitation of a pale yellow coloured solid product indicated the completion of the reaction. Complete conversion of the reactants was observed. The IL obtained was found to be almost pure and no further purification was necessary. The IL was stored in a dessiccator for further use.

Synthesis of pyridazines (General procedure)

In a 100 mL RB, a mixture of 2 mmol of benzil or substituted benzil, 2 mmol of acetophenones and 2 mmol of tetra-*n*-butyl ammoniumhydroxide were dissolved in 5 mL of ethanol. Under the constant stirring of 1 mL of hydrazine hydrate and 3 mol% of the IL added and stirring continued at room temperature for 90 min. The progress of the reaction was monitored by TLC for complete conversion in prepared silica gel plates using EtOAc–hexane (3:7). After completing the reaction, the mixture was quenched in ice water. A white precipitate was obtained which was recovered and further purified by column chromatography using EtOAc–hexane (3:7) as the eluent. Reduced pressure removal of the eluent mixture afforded the pure 2, 3, 5 triarylpyridazines.

Spectral data of some representative products

3,4,6-triphenylpyridazine (Entry 1)

^1H NMR (300 MHz, CDCl_3): δ 7.28-7.41 (Ar-H, m), 7.45-7.5 (Ar-H, m), 7.51-7.78 (Ar-H, m), 7.79-8.09 (Pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 125.2, 125.3, 127.3, 127.7, 127.8, 128.6, 128.7, 128.7, 129.46, 129.5, 129.6, 129.7, 129.8, 129.9, 131.1, 133.0, 136.2, 138.8, 155.5, 158.2.

3,4-diphenyl-6-(4-chlorophenyl)pyridazine (Entry 3)

^1H NMR (300 MHz, CDCl_3): δ 7.26-7.39 (Ar-H, m), 7.41-7.50 (Ar-H, m), 7.75-7.92(Ar-H, m) , 8.02-8.04 (Pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 127.7, 128.9, 129.2, 129.7, 130.1, 131.0, 138.1, 145.6, 191.9.

3,4-diphenyl-6-(4-methoxyphenyl)pyridazine (Entry 4)

^1H NMR (300 MHz, CDCl_3): δ 4.28 (s, OCH_3) , 7.27-7.37 (Ar-H, m), 7.43-7.48 (Ar-H, m), 7.52-7.57 (Ar-H, m), 7.94-7.97 (Pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 55.2, 114.0, 124.4, 124.9, 125.0, 125.5, 126.8, 127.2, 131.7, 132.0, 135.2, 135.5, 139.1, 156.7, 157.1, 164.4, 164.8.

3,4-diphenyl-6-(4-bromophenyl)pyridazine (Entry 5)

^1H NMR (300 MHz, CDCl_3): δ 7.28-7.34 (Ar-H, m) , 7.40-7.55 (Ar-H, m) , 7.90-7.93 (Ar-H, m), 8.05-8.07 (pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 127.7, 128.9, 129.2, 129.4, 129.7, 130.1, 131.5, 138.1, 145.4.

3,4-diphenyl-6-(4-hydroxyphenyl)pyridazine (Entry 6)

^1H NMR (300 MHz, CDCl_3): δ 7.27-7.35 (Ar-H, m), 7.41-7.44 (Ar-H, m), 7.46-7.63 (Ar-H, m), 7.85-7.95 (pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 126.3, 127.7, 128.2, 128.4, 128.7, 128.9, 129.2, 129.4, 129.6, 130.1, 131.5, 138.1, 145.3.

3,4-diphenyl-6-(4-nitrophenyl)pyridazine (Entry 7)

^1H NMR (300 MHz, CDCl_3): δ 7.26-7.35 (Ar-H, m), 7.41-7.46 (Ar-H, m), 7.50-7.54 (Ar-H, m), 7.55-7.95 (pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 127.7, 127.8, 128.6, 128.7, 128.7, 129.4, 129.5, 129.7, 129.7, 129.8, 129.9, 131.1, 133.0, 136.3, 138.8, 148.8, 155.5, 158.2.

3,4-diphenyl-6-(2,4-dihydroxyphenyl)pyridazine (Entry 8)

^1H NMR (300 MHz, CDCl_3): δ 7.36-7.39 (Ar-H, m), 7.41-7.46 (Ar-H, m), 7.47-7.51 (Ar-H, m), 7.86-7.89 (pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 126.3, 127.7, 128.2, 128.4, 128.7, 128.9, 129.2, 129.2, 129.4, 129.6, 130.1, 131.5, 138.1, 145.4.

3,4-diphenyl-6-(3-bromophenyl)pyridazine (Entry 9)

^1H NMR (300 MHz, CDCl_3): δ 7.26-7.37 (Ar-H, m), 7.38-7.39 (Ar-H, m), 7.45-7.51 (Ar-H, m), 7.86-8.08 (pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 126.3, 127.2, 127.7, 128.2, 128.4, 128.6, 128.7, 128.8, 128.9, 129.1, 129.2, 129.4, 129.6, 128.7, 129.7, 131.5, 133.8, 145.3.

3,4-diphenyl-6-(2-hydroxyphenyl)pyridazine (Entry 10)

^1H NMR (300 MHz, CDCl_3): δ 7.26-7.35 (Ar-H, m), 7.37-7.44 (Ar-H, m), 7.46-7.60 (Ar-H, m), 7.95-7.96 (pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 126.3, 127.7, 128.2, 128.4, 128.7, 128.9, 129.2, 129.4, 129.6, 130.1, 131.5, 138.1, 145.3, 191.8.

3,4-di(4-methoxyphenyl)-6-(4-bromophenyl)pyridazine (Entry 12)

^1H NMR (300 MHz, CDCl_3): δ 3.89-3.91 (OCH₃, 1H), 4.06-4.08 (OCH₃, 1H), 7.15-7.26 (Ar-H, m), 7.43-7.68 (Ar-H, m), 7.68-7.89 (Ar-H, m), 8.12-8.13 (pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 52.1, 55.2, 56.7, 56.8, 113.4, 113.5, 113.8, 114.0, 124.4, 124.9, 125.0, 125.5, 126.8, 127.2, 131.7, 132.0, 135.5, 139.3, 156.7, 157.1, 164.4, 164.8.

3,4-di(4-methoxyphenyl)-6-(4-chlorophenyl)pyridazine (Entry 13)

^1H NMR (300 MHz, CDCl_3): δ 3.89-3.91 (d, OCH₃) 4.06-4.09 (d, OCH₃), 7.18-7.22 (Ar-H, m), 7.25-7.26 (Ar-H, m), 7.93-7.98 (Ar-H, m), 8.18-8.21 (pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 55.2, 55.2, 56.7, 56.8, 113.4, 113.5, 113.8, 114.0, 124.4, 124.9, 125.0, 125.5, 126.8, 127.2, 131.7, 132.0, 135.2, 135.5, 139.1, 156.7, 157.1, 164.4. 164.8.

3,4-di(4-methoxyphenyl)-6-phenylpyridazine (Entry 15)

^1H NMR (300 MHz, CDCl_3): δ 3.71 (s, OCH₃), 6.67-6.75 (m, Ar-H), 7.24-7.43 (m, Ar-H), 7.46-7.56 (Ar-H, m), 7.94-7.98 (m, pyridazine Ar-H). ^{13}C NMR (75 MHz, CDCl_3): δ 55.1, 55.2, 56.8, 56.8, 113.8, 113.9, 114.0, 124.3, 124.8, 125.3, 126.4, 126.8, 131.6, 135.2, 135.5, 139.0, 156.6, 156.9, 164.3, 164.74.

Results and discussion

In our continuing effort on utilization of the *N*-alkylpyridinium based ionic liquids for promoting several transformations including the synthesis of heterocycles, two different ionic liquids were prepared namely 1-butyl-3,5-dimethylpyridiniumbromide [1-*n*-B-3,5-diMpyrBr] and 1-octyl-3, 5-dimethylpyridiniumbromide [1-*n*-O-3,5-diMpyrBr] starting from 3,5-dialkylpyridine. The preparation involved stirring at room temperature, a mixture of the 3,5-dimethylpyridine and the corresponding alkyl halide for 20–24 hrs. The yield of the ILs was found to be quantitative.

The pyridinium ionic liquids were characterized by spectroscopic methods. The thermogravimetric analysis (TGA) curves of the two pyridinium based ILs are illustrated shown in [Figure 1](#) and [Figure 2](#) respectively. In case of pyridinium based ILs, the thermal stability varied with the nature of the cation and found to be higher in case of 1-butyl-3, 5-dimethyl pyridinium bromide compared to 1-octyl-3,5-dimethyl pyridinium bromide. TGA experiments were performed by using a TGA-DSC1, Mettler Toledo instrument. The samples were weighed and placed in a platinum crucible. They were then heated in a stream of nitrogen atmosphere, from room temperature to 700 °C with a heating rate usually of 10 °C /min. The TGA curves indicated the decomposition temperature of both the ILs indicating thermal stability up to 250 °C (Supplementary information).

Conductivity measurements of aqueous solutions of the ILs were determined and the results summarized in the [Table 1](#). It is reported that conductivity of aqueous solutions of 0.0001 molar concentration of typical ILs falls within the range of 1.0 mS/cm to 10.mS/cm and increases with dilution with water. The results were in agreement with those reported in literature [25].

Figure 1. TGA curve of [1-*n*-O-3,5-diMPyrBr]

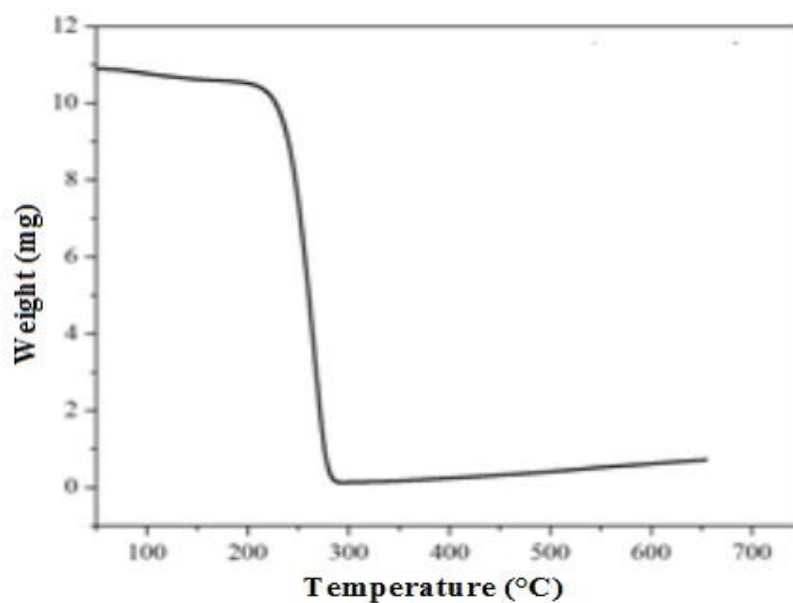


Figure 2. TGA curve of [1-*n*-B-3,5-diMPyrBr]

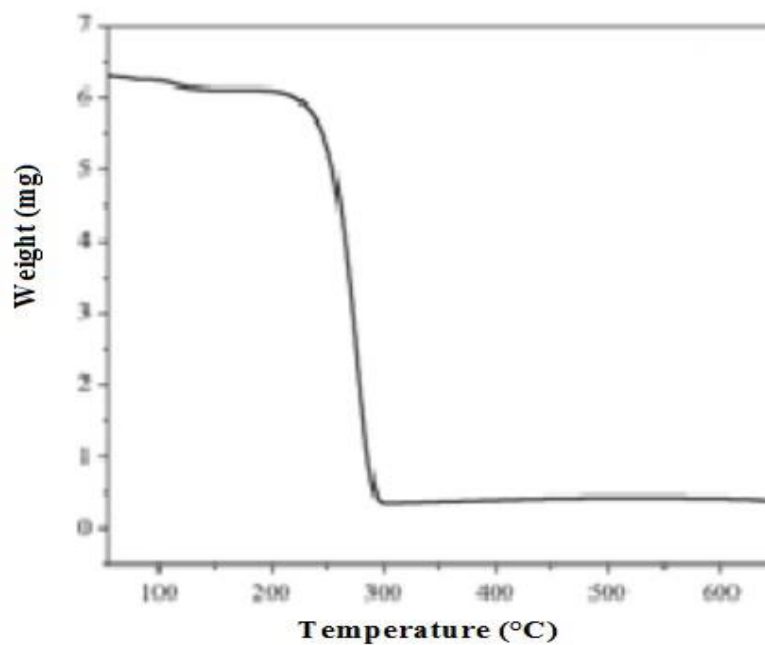


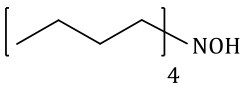
Table 1. Variation of conductivity in aqueous solution of pyridinium ionic liquids

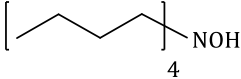
Entry	Ionic liquid	Initial concentration	Conductivity on dilution			
			1 mL	2 mL	3 mL	4 mL
1	1-octyl-3,5-dimethylpyr-bromide	0.0001 M	1.756	1.95	2.206	2.532
2	1-butyl-3,5-dimethylpyr-bromide	0.0001 M	1.109	1.23	1.39	1.421

The ILs were explored for their utility in the synthesis of pyridazines. Initially, a representative three component reaction involving acetophenone, benzyl, and hydrazine hydrate was examined by using both the ionic liquids in ethanol solution. No perceptible change was observed. It was reported that the presence of a base is essential for initiating the reaction. Consequently, the reactions were repeated using four different bases namely K_2CO_3 , CH_3ONa , KOH , and a solid base Al_2O_3 -OK successively and their suitability for initiating the reaction was explored. The solid base was prepared following a reported procedure [26]. However, none of these bases gave satisfactory yield of the pyridazines. In order to drive the reaction we then explored the possibility of using a mixture of two ILs, one basic and the other neutral. Tetra-*N*-butylammonium hydroxide was used as the basic IL for the purpose.

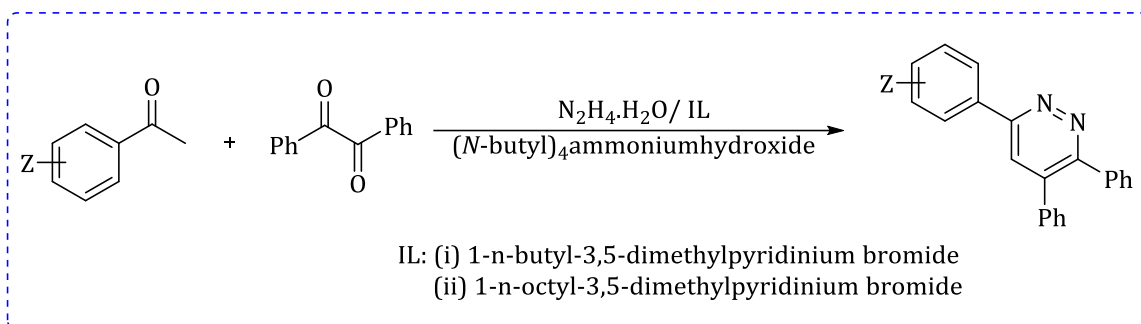
A comparison of the reaction conditions and the yield of the pyridazine obtained is revealed in Table 2. The results indicated that the use of tetra-*N*-butylammonium hydroxide as the basic IL along with the pyridinium based ionic liquids gave excellent yield of the product in a short reaction time and at room temperature. The two ionic liquids, one basic and the other neutral have acted in tandem

Table 2. Effect of bases on the formation of 3,4,6-triarylpyridazine at ambient temperature

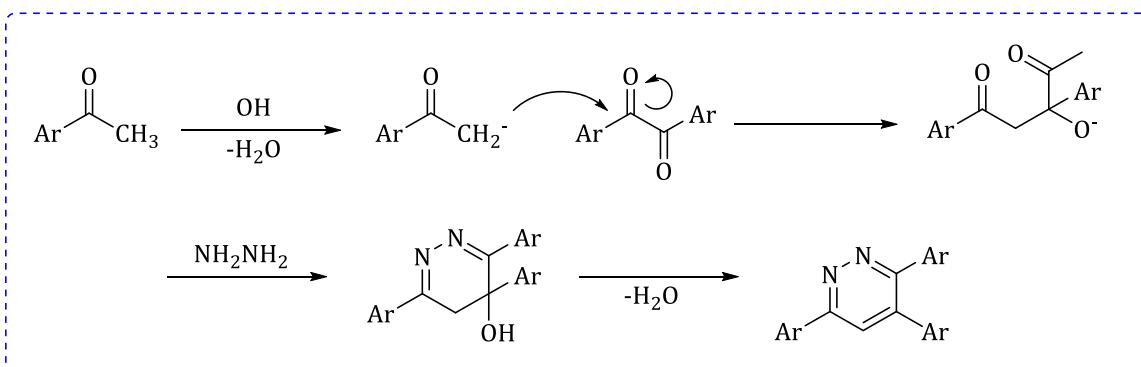
Entry	Solvent	Base	Time (hrs)	Yield (%)
1	Ethanol	K_2CO_3	3.5	Trace
2	Ethanol	$NaOMe$	3	30
3	Ethanol	KOH	3	40
4	Ethanol	Al_2O_3 -OK	2.5	62
5	1- <i>n</i> -butyl-3,5-dimethylpyridiniumbromide	base free	3	—
6	1- <i>n</i> -octyl-3,5-dimethylpyridiniumbromide	base free	3	—
7	1- <i>n</i> -octyl-3,5-dimethylpyridiniumbromide		1.5	87

8	1- <i>n</i> -butyl-3,5-dimethylpyridiniumbromide		1.5	70
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The reaction was standardized by using a variety of acetophenones and substituted benzil and the results are summarized in [Table 2](#)



Scheme 1. Synthesis of 3,4,6-triarylpyridazine mediated by binary ionic liquid mixture

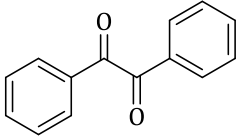
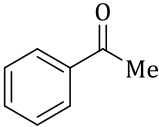
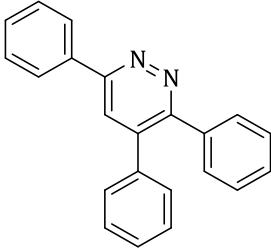
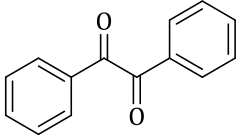
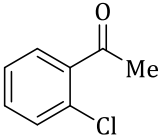
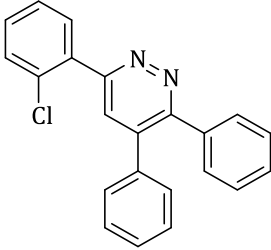
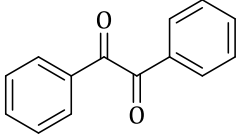
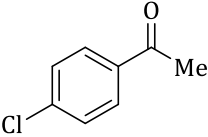
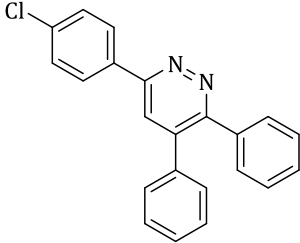
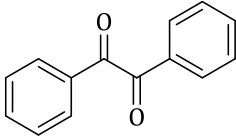
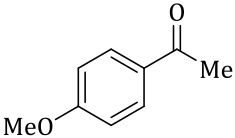
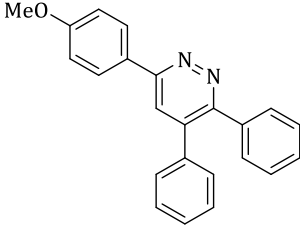
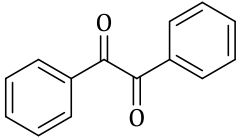
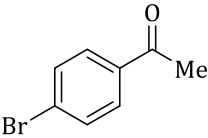
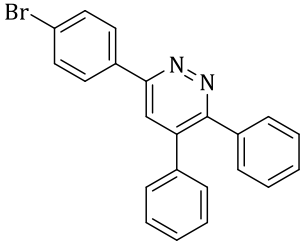
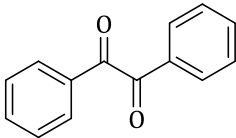
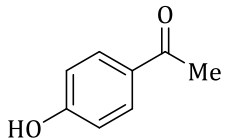
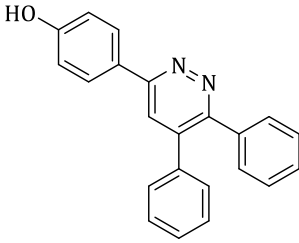


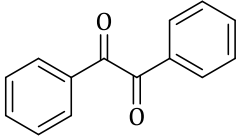
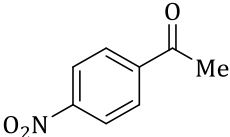
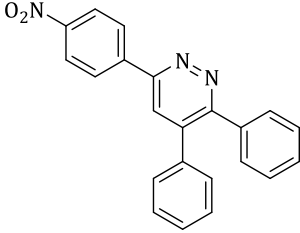
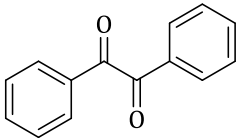
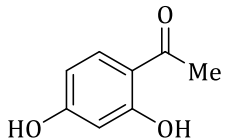
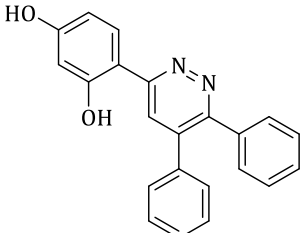
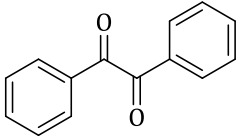
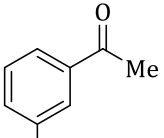
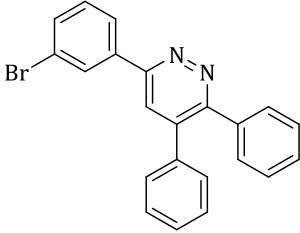
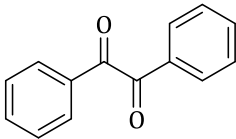
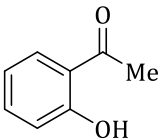
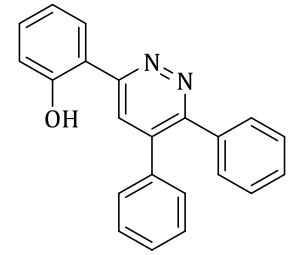
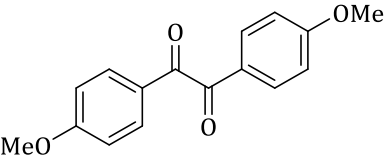
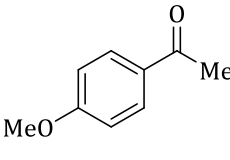
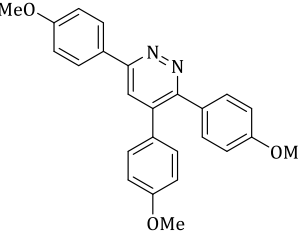
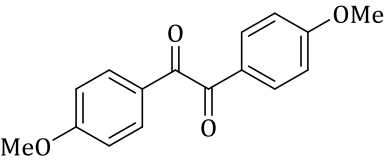
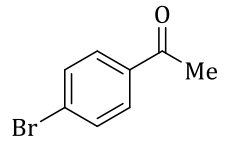
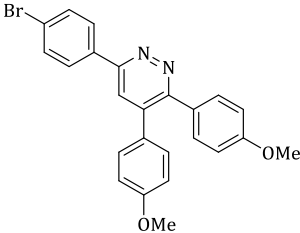
Scheme 2. Proposed mechanism of synthesis of 3, 4, 6-triarylpyridazine

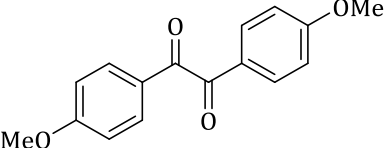
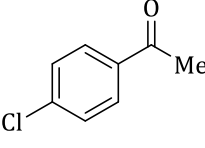
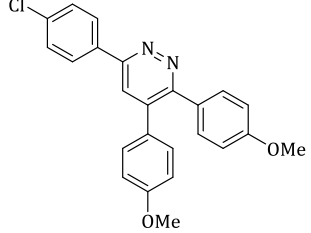
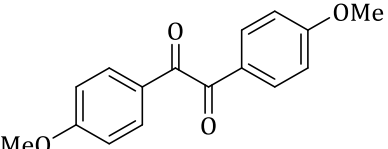
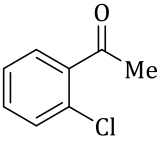
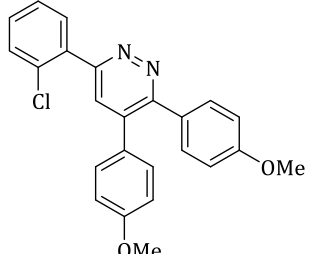
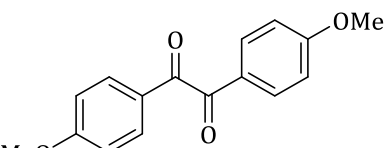
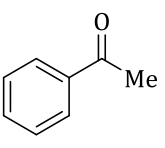
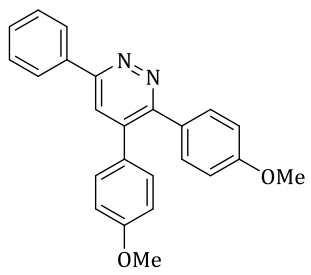
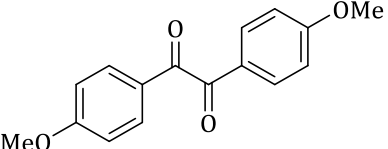
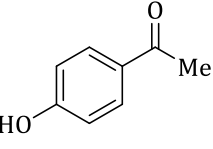
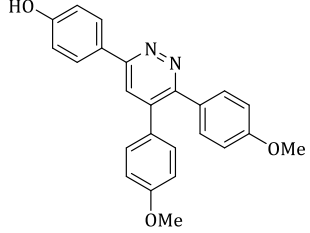
to give the desired product further, the results indicated the superiority of 1-*N*-octyl-3,5-pyridiniumbromide over the corresponding butyl derivative and consequently the reaction were standardized using a 1:1 mixture of the ILs namely 1-*N*-butyl-3,5-dimethylpyridiniumbromide and tetra-*N*-butylammoniumhydroxide. The latter acting as the base necessary. The ionic liquid mixture could be separated by aqueous extraction. Dropwise addition of diethylether, precipitated the ionic liquid mixture which could be reused however with reduced efficiency. The reaction carried out is demonstrated in [Scheme 2](#), and experimental results are summarized in [Table 3](#).

Table 3. Synthesis of 3,4,6-triarylpyridazine using binary ionic liquid mixtures

Entry	1,2 dione	Acetophenone	product	M.p. (°C)	Yield (%)
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1				168	87
2				172	94
3				169	90
4				162	85
5				137	92
6				131	97

7				147	97
8				145	96
9				128	90
10				142	97
11				152	91
12				162	90

13				158	90
14				162	95
15				165	93
16				167	97

Conclusion

The procedure for the synthesis of pyridazine derivatives consists of two ionic liquids, one basic and other neutral, provides a simple procedure to the best of our knowledge, the synergy of two ionic liquid in the synthesis of pyridazine derivatives have not been reported earlier. Improvements over other methods include mild reaction conditions, easy recovery of products, and high yields, without using any toxic chemicals. The reaction was applicable for a wide variety of substrates and some of the products have not been reported earlier.

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Disclosure statement

No potential conflict of interest was reported by the authors.

Supporting Information

Additional supporting information related to this article can be found, in the online version, at http://www.ajgreenchem.com/article_51780.html.

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