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An efficient and recyclable bifunctional acid–base ionic liquid for synthesis of 1*H*-indazolo[1,2-*b*]phthalazinetriones

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Abstract A bifunctional ionic liquid, 1,4-dimethyl(4-sulfobutyl)piperazinium hydrogen sulfate ([DMSBP][HSO_4], **1**), containing both acid and basic sites was prepared. This ionic liquid was used as a homogeneous and reusable catalyst and efficiently promoted synthesis of 1*H*-indazolo[1,2-*b*]phthalazinetriones (**5a**–**m**) in a one-pot, four-component condensation reaction of phthalic anhydride, hydrazinium hydroxide, dimedone, and aromatic aldehydes under solvent-free conditions at 80 °C. Advantages of the method include short reaction time, high yields, and simple purification.

Keywords Bifunctional ionic liquid · Green chemistry · 1*H*-Indazolo[1,2-*b*]phthalazinetrione · Solvent-free

Introduction

The design and synthesis of catalysts with synchronic activation of acidic and basic sites is an emerging field in chemistry. Cooperation of the different active sites has been used to promote consecutive reactions with high rates and selectivity [1–7].

Modern synthesis emphasizes use of environmentally benign processes in which use of toxic and hazardous solvents is avoided [8, 9]. These objectives are perfectly supported by ionic liquids, which are known to promote organic reactions as benign dual solvent-catalysts with high performance [10, 11]. This is because ionic liquids have such notable advantages as extremely low volatility, high thermal stability, adjustable solubility, and versatile structures [12–16]. The term "task-specific ionic

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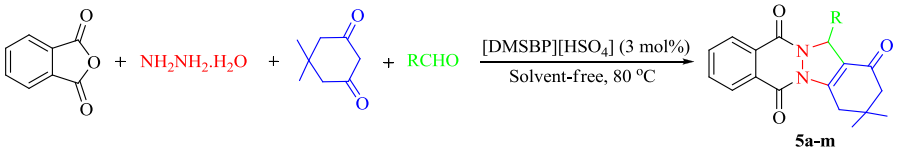


Scheme 1 Synthesis of 1*H*-indazolo[1,2-*b*]phthalazinetriones with an ionic liquid as catalyst

liquids" has been used to refer to ionic liquids which have particular properties or reactivities by virtue of their functional groups [17–19].

Ionic liquid catalysts with basic and/or acidic bifunctional groups have frequently been reported for Knoevenagel reactions [20–24]. To the best of our knowledge, however, bifunctional ionic liquids have not been widely used in multicomponent reactions.

Phthalazine derivatives are an important class of heterocyclic compounds which contain bridgehead hydrazine and have such properties as anticonvulsant activity [25], a potent vasorelaxant effect [26], and cardiovascular activity [27]. Several catalysts, for example *p*-toluenesulfonic acid [28], silica-supported polyphosphoric acid [29], *N*-halosulfonamides [30], and ionic liquids [31, 32], have recently been used for synthesis of these compounds.

In continuation of our study on ionic liquids and their applications [33], we have developed an efficient method for synthesis of 1*H*-indazolo[1,2-*b*]phthalazinetriones (**5a**–**m**) by one-pot, four-component condensation reaction of hydrazinium hydroxide, phthalic anhydride, dimedone, and aromatic aldehydes in the presence of an acid–base bifunctional reusable catalyst (**1**) at 80 °C under solvent free conditions (Scheme 1).

Experimental

General

All chemicals were high-grade quality purchased from Merck and Aldrich and used without further purification. Melting points were taken in open capillary tubes with a Buchi 510 melting point apparatus and are uncorrected. NMR spectra were recorded in DMSO-d_6 by use of Bruker 300 and 400-MHz and Jeol 90-MHz spectrometers. FT-IR spectra were recorded by use of a Perkin–Elmer 781 spectrophotometer. Mass spectra were obtained with Shimadzu GC–MS-QP 1100 EX.

*Procedure for synthesis of 1,4-dimethyl(4-sulfobutyl)piperazinium hydrogen sulfate [DMSBP][HSO$_4$] (**1**)*

1,4-Dimethylpiperazine (**4**, 10.0 mmol, 1.4 mL) was added to 1,4-butane sultone (10.0 mmol, 1.0 mL) and stirred under solvent-free conditions at room temperature. A white solid zwitterion was formed early in the procedure and the reaction mixture

was stirred further (10 h) to complete the reaction. The zwitterion was washed with diethyl ether (3 × 5 mL), isolated by filtration to remove impurities, and dried under vacuum at 110 °C (yield 95 %). In the next step, sulfuric acid (10 mmol, 0.5 mL) was added dropwise to this zwitterion (10 mmol, 2.5 g) and the mixture was stirred at 80 °C for 6 h, to convert the solid zwitterion into a liquid. The ionic liquid obtained was then washed with ether (3 × 5 mL) to remove the non-ionic residues and dried under vacuum at 110 °C (yield 94 %).

Spectroscopic data for (**1**): IR (Nujol): ν_{max} = 2950–3540 (br, acidic O–H), 1182 (C–N), 1040 and 901 cm^{-1} (SO_3); ^1H NMR (400 MHz, D_2O): δ (ppm) 1.79–1.87 (m, 2H, CH_2), 1.93–2.02 (m, 2H, CH_2), 2.38 (s, 3H, NCH_3), 2.78–2.94 (m, 4H, H-piperazinium), 2.99 (t, J = 7.6, 2H, NCH_2), 3.13 (s, 3H, NCH_3) and 3.44–3.49 (m, 6H, SCH_2 and H-piperazinium); ^{13}C NMR (100 MHz, D_2O): δ (ppm) 19.99, 21.05, 43.70, 47.33, 49.68, 49.85, 53.84, 59.68. MS, *m/z* (%): 348 (M^+), 251 (M^+ – HSO_4), 170 (M^+ – HSO_4SO_3) 157 (M^+ – $HSO_4CH_2SO_3$), 129 (M^+ – HSO_4 $(CH_2)_3$ – SO_3), 114 (M^+ – $HSO_4(CH_2)_4SO_3$).

*Procedure for synthesis of 1,4-dimethylpiperazine-diium hydrogen sulfate (**2**)*

A solution of 1,4-dimethylpiperazine (**4**, 4.0 mmol, 0.56 mL) was added dropwise to sulfuric acid (8.0 mmol, 98 wt%), at 0 °C over a period of 15 min. in three-necked flask with magnetic stirrer. The reaction mixture was then stirred for 4 h. The mixture was washed with diethyl ether (3 × 10 mL) to remove any non-ionic residues and dried under vacuum at 90 °C (m.p: 92 °C, yield 90 %).

Spectroscopic data for (**2**): IR (KBr): ν_{max} = 3250–3550 (br, acidic OH), 1182 (C–N), 1120 cm^{-1} (–SO_3); ^1H NMR (DMSO): δ (ppm) 2.8 (s, 6H, $2CH_3$), 3.34 (s, 8H, $4CH_2$) and 5.40 (s, 2H, N–H) ppm; ^{13}C NMR (100 MHz, D_2O): δ (ppm): 41.80 and 49.96.

*Procedure for synthesis of 1,4-dimethyl-disulfopiperazine-diium chloride (**3**)*

A solution of 1,4-dimethylpiperazine (**4**, 4.0 mmol, 0.56 mL) in CH_2Cl_2 (20 mL) was added dropwise to a stirred solution of chlorosulfonic acid (8.0 mmol, 0.53 mL) in dry CH_2Cl_2 (20 mL) over a period of 10 min at 0 °C, then stirred for a further 4 h. The solvent was evaporated, and the solid residue was washed with diethyl ether (3 × 10 mL) to remove any non-ionic residues and dried under vacuum at 90 °C to give a white solid with double acid sites as acid catalyst (mp: 165 °C, yield 95 %).

Spectroscopic data for (**3**): IR (KBr): ν_{max} = 3150–3552 (br, acidic OH), 1242 (C–N), 1039 and 920 cm^{-1} (–SO_3); ^1H NMR (DMSO): δ (ppm) 2.48 (s, 6H, $2CH_3$), 3.13 (s, 8H, $4CH_2$) and 8.99 (s, 2H, N–H) ppm; ^{13}C NMR (100 MHz, D_2O): δ (ppm): 41.68 and 48.96.

*General procedure for synthesis of 1H-indazolo[1,2-b]phthalazine-1,6,11(13H)-trione derivatives (**5a–n**)*

Dimedone (1.0 mmol) and an aromatic aldehyde (1.0 mmol) were added to a mixture of hydrazinium hydroxide (1.2 mmol), phthalic anhydride (1.0 mmol), and

1 (3 mol %, 0.01 g) at 80 °C and stirred under solvent-free conditions. Reaction was complete within 5–20 min (confirmed by thin-layer chromatography). The reaction was cooled to room temperature and H_2O (5 mL) added. The solid residue was isolated by filtration. Crude products were purified by recrystallization from 70:30 ethanol–water and characterized by comparison of their physical data with those of known 1*H*-indazolo[1,2-*b*]phthalazine-1,6,11(13*H*)-triones.

Selected spectral data for indazolophthalazinetrione derivatives

5c: (3,3-dimethyl-13-(3-nitrophenyl)-3,4-dihydro-1*H*-indazolo[1,2-*b*]phthalazine-1,6,11(2*H*,13*H*)-trione): IR (KBr): ν_{max} 2973, 1684, 1661, 1625 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 1.23 (6H, s, 2Me), 2.35 (2H, s, CH_2C), 3.24–3.47 (2H, AB system, $J = 18.9$ Hz, CH_aH_bCO), 6.53 (1H, s, CHN), 7.53–8.4 (8H, m, ArH); ^{13}C NMR (300 MHz, $CDCl_3$); 28.5, 28.8, 34.7,38.2, 50.9, 64.0, 117.2, 121.5, 123.7, 127.7, 128.3, 128.5, 128.9, 129.5; MS (ESI) *m/z* 417 (M + 1).

5f: (13-(2,4-dichlorophenyl)-3,3-dimethyl-3,4-dihydro-1*H*-indazolo[1,2-*b*]phthalazine-1,6,11(2*H*,13*H*)-trione: IR (KBr): ν_{max} 2967, 1664, 1627 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) 1.23 (6H, s, Me), 2.33 (2H, s, CH_2C), 3.27–3.43 (2H, AB system, $J = 19.4$ Hz, CH_aH_bCO), 6.64 (1H, s, CHN), 7.26–8.4 (7H, m, ArH); ^{13}C NMR (300 MHz, $CDCl_3$); 28.4, 28.7, 34.6, 38.0, 50.8, 63.5, 118.1, 127.6, 127.7, 128.1, 128.6, 129.0, 131.1, 131.7, 133.2, 133.7, 134.6, 135.1, 152.0, 154.3, 156.1, 192.0; MS (ESI) *m/z* 441 (M + 1).

5h: 3,3-dimethyl-13-(naphthalen-2-yl)-3,4-dihydro-1*H*-indazolo[1,2-*b*]phthalazine-1,6,11(2*H*,13*H*)-trione: IR (KBr): ν_{max} 2956, 1665, 1619 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): 1.22 (6H, s, 2Me), 2.33 (2H, s, CH_2C), 3.24–3.50 (2H, AB system, $J = 18.6$ Hz, CH_aH_bCO), 6.62 (1H, s, CHN), 7.45–8.38 (11H, m, ArH); ^{13}C NMR (300 MHz, $CDCl_3$): 28.4, 28.7, 34.6, 38.1, 50.9, 65.1, 118.6, 124.2, 126.2, 126.3, 126.8, 127.6, 127.7, 127.9, 128.2; MS (ESI) *m/z* 422 (M + 1).

Results and discussion

The structure of [DMSBP][HSO_4] was determined by use of IR, ^1H, ^{13}C NMR, and mass spectroscopy. The IR spectrum of this ionic liquid contained a broad peak at 2950–3540 cm^{-1} which is ascribed to O–H stretching of the SO_3H group. The peak observed at 1040 cm^{-1} is ascribed to vibrational modes of the O–SO_2 bond. The ^1H NMR spectrum contained two single peaks at 2.29 and 3.04 ppm which correspond to the two methyl groups. The different chemical shifts of the methyl groups adequately explains the asymmetric structure of this ionic liquid.

Thermal gravimetric analysis (TGA) and differential thermal gravimetric (DTG) analysis of **1** were also conducted. The results are depicted in Fig. 1, which shows the high thermal stability of [DMSBP][HSO_4], with decomposition at temperatures above 270 °C.

To determine the optimum conditions for synthesis of 1*H*-indazolo[1,2-*b*]phthalazinetriones we investigated the reaction of 4-chlorobenzaldehyde, dimedone, phthalic anhydride, and hydrazinium hydroxide as model reaction in the presence of different catalysts. To investigate the bifunctionality of the catalyst, a

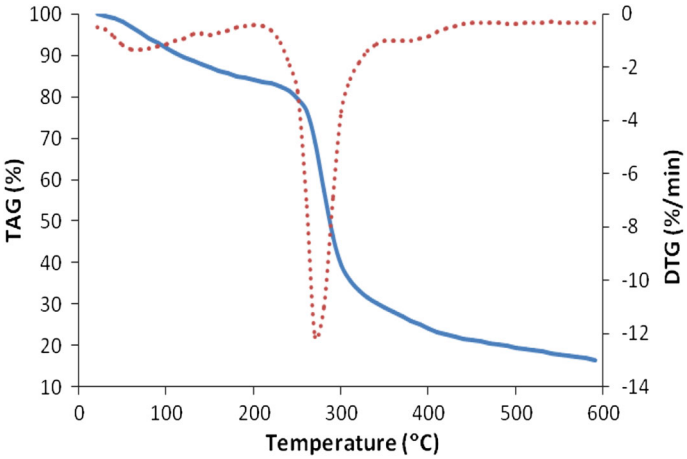
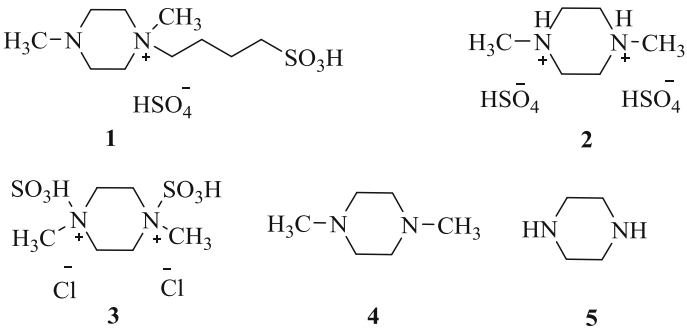


Fig. 1 TGA and DTG results for [DMSBP][HSO_4]



Scheme 2 Acidic and basic catalysts

series of catalysts with different acidic or basic properties were prepared and the effect and activity of the acidic and basic groups were investigated (Scheme 2).

The model reaction was performed with different amounts of **1**. The optimum result was obtained with 0.03 mmol catalyst at 80 °C and solvent-free conditions. Increasing the amount of catalyst or the temperature resulted in no substantial improvement of the yield (Table 1).

To confirm the presence of acidic and basic groups in **1**, another catalyst was prepared, for comparison, in which the two acidic protons were present on the nitrogen of the tertiary amine, resulting in an acidic catalyst. This 1,4-dimethylpiperazine-diium hydrogen sulfate catalyst (**2**), was synthesized by addition of **4** to sulfuric acid. The ^{1}H NMR spectrum of **2** contained three peaks at 2.48 (6H, $2CH_3$), 3.13 (8H, $4CH_2$), and 8.63 (2H, N–H) ppm whereas the ^{1}H NMR spectrum of **4** contained two peaks at 1.57 (6H, $2CH_3$) and 1.72 (8H, $4CH_2$) ppm. The proton shifts in the spectrum of **2**, compared with the raw material, and the important acidic

Table 1 Optimization of the amounts of different catalysts (**1**–**5**) under solvent-free conditions at 80 °C

Catalyst	Amount (mmol)			Time (min)	Isolated yield (%)		
	1st run	2nd run	3rd run		1st run	2nd run	3rd run
1	**0.03**	0.07	0.1	**5**	**94**	94	95
2	0.10	0.20	0.50	30	50	52	64
3	0.10	0.20	0.50	30	68	73	76
4	0.50	0.75	1.0	30	45	50	55
5	0.50	0.75	1.0	30	42	54	58

Bold values indicate the best condition

Reaction conditions: 4-chlorobenzaldehyde (1.0 mmol), dimedone (1.0 mmol), hydrazinium hydroxide (1.2 mmol), phthalic anhydride (1.0 mmol)

hydrogen peak at 8.63 ppm confirms the formation of **2**. When catalyst **2** was used to perform the model reaction the yield was poor.

These results therefore illustrate that the N-protonated amino group was poorly active in catalyzing the model reaction and the acidic proton must be in the proximity of the sulfonate group for catalysis of the reactions. These comparisons adequately confirm cooperation between Brønsted acidic and Lewis basic sites and confirm the structure of **1**.

We also wished to investigate the position in which the sulfonate group was most active in promotion of this reaction. Thus, for comparative purposes, we prepared another acidic catalyst (**3**). To prepare this catalyst, **4** was added to chlorosulfonic acid at low temperature. When the base is added dropwise to the acid (ratio 1:2), there is no excess amount of base which avoids formation of by-products. Moreover, according to the literature [34, 35], in the reaction of amines with chlorosulfonic acid at low temperature, the mechanism is nucleophilic substitution (substitution of the Cl of $ClSO_3H$ by the nitrogen of the tertiary amine), not an acid–base reaction [36]. In addition, peaks of the acidic hydrogen of **3** and $ClSO_3H$ were observed at 8.99 and 13.45 ppm, respectively. The difference between the positions of the peaks of the acidic hydrogens in **3** and $ClSO_3H$ confirmed that the peak observed at 8.99 ppm in the ^{1}H NMR spectrum of **3** is correctly assigned to the SO_3H group of acidic catalyst **3**. The model reaction did not proceed to completion with acidic catalyst **3**, even when the amount of the catalyst was increased (Table 1). The low activity of catalysts **2** and **3** was presumably because of steric hindrance of the tertiary amine. To summarize, although the sulfonic acid group (SO_3H) is a highly acidic group suitable for catalysis of the reaction, the position of the group in the structure of the catalyst is very important. When the SO_3H group is oriented away from the crowded center, it acts as an efficient acidic center for catalysis of the reaction.

Also, when the model reaction was repeated under similar conditions with the basic homogeneous analogues **4** and **5**, the reactions were sluggish with low yields of products (Table 1). This means that basic catalysts can not promote this reaction.

Finally, it was found that **1** catalyzes the reaction very efficiently, in a short reaction time, with high yield. The results indicate that the crucial factor responsible

Table 2 Synthesis of indazolophthalazinetrione derivatives (**5a**–**m**) catalyzed with **1** at 80 °C under solvent-free conditions

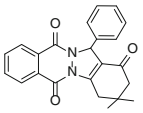
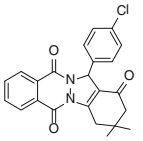
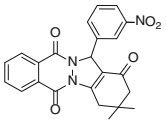
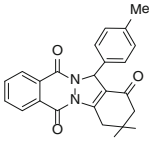
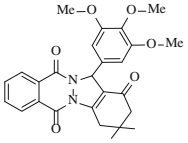
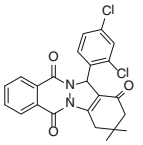
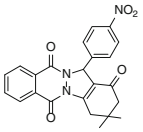
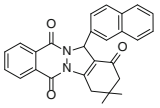
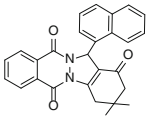
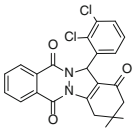
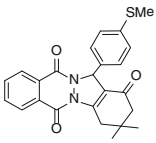
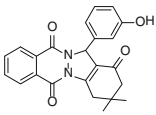
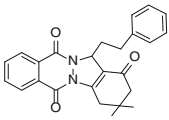
Entry	R	Product		Time (min)	Yield[a] (%)	m.p. °C	
						Found	Reported [28, 30, 31]
1	C_6H_5		**5a**	7	92	205–208	204–206
2	4-ClC_6H_4		**5b**	5	94	264–262	264–262
3	3-$O_2NC_6H_4$		**5c**	5	94	271–268	272–270
4	4-MeC_6H_4		**5d**	10	85	228–226	229–227
5	3,4,5-$(OMe)_3C_6H_4$		**5e**	15	88	236–234	232–234
6	2,4-$Cl_2C_6H_3$		**5f**	7	93	219–221	219–221
7	4-$O_2NC_6H_4$		**5g**	7	89	222–225	223–225
8	2-naphthyl		**5h**	12	90	253–251	251–252
9	1-naphthyl		**5i**	15	87	275–278	273–275

Table 2 continued

Entry	R	Product		Time (min)	Yield[a] (%)	m.p. °C	
						Found	Reported [28, 30, 31]
10	2,3-$Cl_2C_6H_3$		**5j**	8	89	264–266	266–268
11	4-$MeSC_6H_4$		**5k**	10	88	227–230	229–231
12	3-HOC_6H_4		**5l**	10	85	265–268	268–270
13	$C_6H_4(CH_2)_2$		**5m**	20	87	172–175	171–173
14	Octanal	–	**5n**	24 h	–	–	–

[a] Isolated yield

for the efficiency of **1** is, apparently, its cooperative behavior, which results in the most active catalyst used in this study.

Under these optimized conditions, a variety of substituted aldehydes were used for preparation of the corresponding 1*H*-indazolo[1,2-*b*]phthalazinetriones (**5a–m**) in the presence of **1** (Table 2).

Aromatic aldehydes carrying either electron-donating or electron-withdrawing substituents reacted efficiently giving excellent yields. Benzaldehyde and aromatic aldehydes bearing electron-withdrawing groups, for example nitro or halogen (entries 2 and 3) require a shorter reaction times to give higher yields than those bearing electron-donating groups, for example methoxy or methyl (entries 4 and 5). All the synthesized compounds are known and were characterized by comparison of their melting points and spectral data with those of authentic samples.

One major advantage of this procedure is the simple work up and purification of the product, because the ionic liquid is entirely miscible in water whereas the products are not. After separation of the products, the catalyst was easily recovered by removal of the water. The reusability of **1** was investigated in different runs. The catalyst was recovered from the aqueous medium, dried under vacuum, and reused in five successive runs without substantial loss of activity (Fig. 2).

A plausible mechanism for synthesis of the indazolophthalazinetriones in the presence of [DMSBP][HSO_4] is shown in Scheme 3. This mechanism is proposed

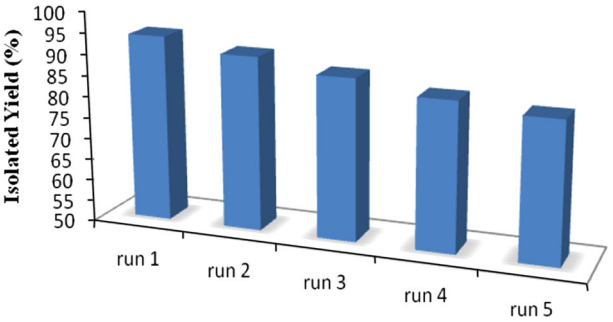
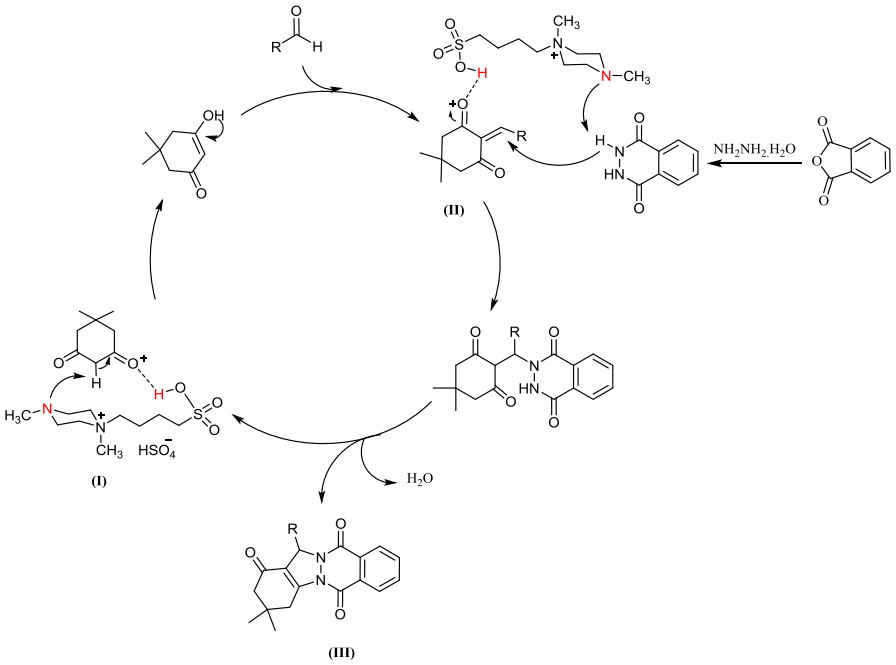


Fig. 2 Reusability of **1** in the model reaction



Scheme 3 Proposed mechanism for synthesis of 1*H*-indazolo[1,2-*b*]phthalazineriones with [DMSBP][HSO_4] as catalyst

on the basis of the assumption of acid–base cooperation in the ionic liquid. According to the mechanism, the first step of this reaction (**I**) is the interaction between the basic site of the ionic liquid and dimedone forming intermediate **II** by Knoevenagel condensation of dimedone and aromatic aldehyde in the presence of the catalyst. In the next step, cooperation of the acid and basic sites of this ionic liquid leads to Michael-type addition of formed phthalhydrazide to intermediate **II**. Finally, cyclocondensation and dehydration of these intermediates occur to provide the corresponding products (**III**).

Table 3 Comparison of **1** with other catalysts for synthesis of indazolophthalazinetriones

Entry	Catalyst and conditions	Time (min)	Isolated yield (%)	TON (min^{-1})	TOF (min^{-1})
1	PEG-OSO_3H (8 mol %), 80 °C [37]	10	92	11.5	1.15
2	Cellulose-SO_3H (4 mol %), solvent-free, 80 °C [38]	6	88	22	3.6
3	**1** (3 mol %), solvent-free, 80 °C [present work]	**5**	**94**	**31.3**	**6.3**
4	H_2SO_4, [bmim]BF_4 (15 mol %), H_2O–EtOH, reflux [31][a]	25	88	6	0.23
5	Silica–sulfuric acid (6.5 mol %), solvent-free, 100 °C [39][a]	7	91	14	2
6	HPA (3 mol %), solvent-free, 100 °C [32][a]	8	92	30.6	4

Bold values indicate the best condition

[a] Based on reaction of 4-chlorobenzaldehyde, dimedone, and phthalhydrazide

In addition, to compare the capability and efficiency of **1** with those of previous methods, results from use of different catalysts for synthesis of 1*H*-indazolo[1,2-*b*]phthalazinetriones by the model reaction were studied. The values of the turnover number (TON) and turnover frequency (TOF) confirm that **1** is a suitable catalyst with regard to reaction times and product yield (Table 3).

Conclusion

In summary, we report the synthesis of a new reusable acid–base bifunctional ionic liquid catalyst (**1**). This homogeneous catalyst is a good solvent for promotion of one-pot multicomponent condensation of phthalic anhydride, hydrazinium hydroxide, dimedone, and aromatic aldehydes.

The main advantage of the method is easy product isolation, because the catalyst is immiscible with most organic solvents and can be separated by extraction with water. Other advantages of the method include high efficiency, low cost, short reaction times, and clean reaction with fewer side-products. Finally, it conforms with the principles of green chemistry.

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