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1,4-Disulfopiperazine-1,4-diium chloride ([Piper-$(SO_3H)_2$].2Cl) as an efficient ionic catalyst for synthesis of phthalazine derivatives

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ABSTRACT

This article describe the applicability of 1,4-disulfopiperazine-1,4-diium chloride ([Piper-$(SO_3H)_2$].2Cl) as a green, versatile and Brönsted acidic ionic catalyst in the promotion of the synthesis of phthalazine derivatives *via* one-pot three component reaction between aromatic aldehydes, 1,3-diketone derivatives and phthalhydrazide under solvent-free reaction conditions. The main advantages of this method are: (1) simplicity of the procedure, (2) solvent-free conditions, (3) availability of the starting materials, (4) high reaction rates and excellent yields, (5) reusability of the catalyst and (6) no column chromatographic of the products.

***Keywords**: Brönsted acidic ionic catalyst, Phthalazine derivatives, Solvent-free conditions.*

1. Introduction

Multicomponent reaction (MCR) shows significant advantages over classical stepwise methods [1,2]. MCR is a powerful tool in other to build novel and complex molecules, in particular, for the synthesis of biologically active heterocyclic compounds. MCR presents rapid and the convergent construction of molecules from commercially available starting materials without the need of isolation and purification of intermediates. Therefore MCR requires less manipulation time, cost and energy than conventional linear syntheses [3].

Among different types of Brönsted-acidic catalysts, *N*-sulfonic acidic catalysts have designed to replace traditional mineral liquid acids like sulfuric acid and hydrochloric acid. Besides**,** their polar natures make them frugal for use under solvent-free conditions [4].

Phthalazine derivatives are unique class of heterocyclic compounds with extraordinary properties that appear to be potential candidates for the pharmaceutical and biological activities [5]. Phthalazine derivatives were reported to possess some anticonvulsant [6], antimicrobial [7], anticancer [8], antifungal [9], cardiotonic [10] and vasorelaxant [11] activities.

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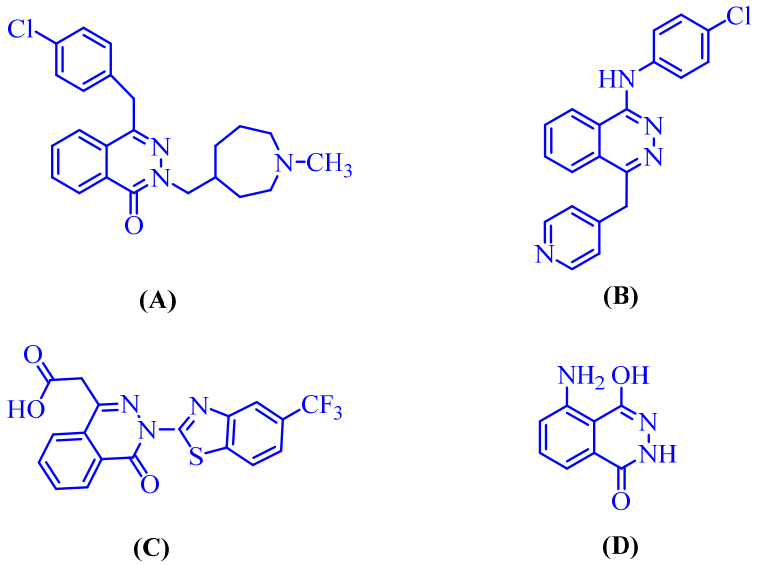
These compounds exhibited as new luminescent or fluorescence materials [12]. Phthalazine moieties are presented in the structure of a variety of potent commercial drugs, for example, azelastine (A) is an antihistamine used in the treatment of allergic rhinitis, Vatalanib (B) has been shown to serve as anticancer agent, zopolrestat (C) an aldose reductase inhibitor that may be useful for the treatment of complications of diabetes and Alpha-luminol (D) has shown profound anti-inflammatory and antioxidant effects in both experimental animal and human clinical studies (Scheme 1) [13-15].

2*H*-Indazolo[2,1-*b*]phthalazinetriones, the significant derivatives of phthalazine, can be synthesized *via* three-component condensation of phthalhydrazide, dimedone and aromatic aldehydes. A variety of reagents have been reported for the promotion of the synthesis of these derivatives which of them *p*-TSA [16], silica sulfuric acid [17], Mg $(HSO_4)_2$ [18], I_2/EtOH [19], TCT [20], HPA/IL [21], PPA/SiO_2 [22], Me_3SiCl [23] $H_{14}[NaP_5W_{30}O_{110}]$/$SiO_2$ [24] and (MTSA) [25] are examples. These methods although useful but most of them are accompanied with limitations such as use of expensive catalysts or toxic organic solvents, intense acidic conditions and rough reaction conditions. Therefore, the development of new synthetic methods for the effective preparation of these heterocyclic compounds is an interesting challenge.



Scheme 1. Medicinal structures of some phthalazine derivatives.

Recently use of dicationic catalysts due to their great variety of harmonious interactions, high thermal stabilities and broader selectivity became a rapidly growing field in organic chemistry [26,27].

More recently, we have reported the preparation and identification of some *N*-sulfonic acidic catalysts, which in their structure SO_3H group is bonded to positive nitrogen atom. These compounds were successfully used as catalysts and regents in organic transformations [28-42]. Herein and in continuation of these studies, we wish to report the applicability of [Piper-$(SO_3H)_2$].2Cl [43] as one of these catalysts in the synthesis of phthalazine derivatives.

2. Experimental

2.1. Material

Chemical materials were purchased from Merck, Fluka, and Aldrich Chemical Companies. Products were characterized by their physical constants, NMR and FT-IR spectroscopy. The purity percent of the substrate and reaction monitoring were determined by TLC on silics-gel polygram SILG/UV 254 plates.

2.2. Characterization techniques

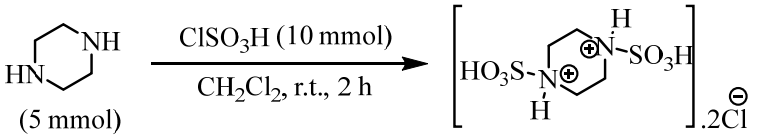
The ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) were run on a Bruker AVANCE^{III}-400 spectrometer instrument using TMS as an internal reference (δ in ppm) in DMSO solvent. The FT-IR spectra were run on a VERTEX 70 Bruker company (Germany).

2.3. Catalyst preparation

A round-bottomed flask (50 mL) was charged with a solution of piperazine (0.43 g, 5 mmol) in dry CH_2Cl_2 (30 mL), and then chlorosulfonic acid (1.21 g, 10.4 mmol) was gradually added during 10 minutes at room temperature. After 2 hours a white solid was produced. Afterward the solvent was decanted, the residue was triturated with dry Et_2O and dried under vacuum to give [Piper-$(SO_3H)_2$].2Cl as a white puffy solid at 98 % yield (m.p. 220 °C) (Scheme 2).

2.4. General procedure

A mixture of the requested aldehyde (1.0 mmol), dimedone and/ or 1,3-cyclohexadione (1.0 mmol), phthalhydrazide (1.0 mmol) and [Piper-$(SO_3H)_2$].2Cl (12.5 mol%) was heated in an oil bath (100 °C) under solvent-free conditions for the appropriate time. The reaction was monitored by TLC [*n*-hexane: ethylacetate (4:1)]. After termination, the reaction mass was cooled to room temperature and washed with water for the separation of the catalyst. The solid product was carefully purified in aqueous EtOH (25%) by re-crystallization.



Scheme 2. Preparation of [Piper-$(SO_3H)_2$].2Cl.

Selected spectral data

*13-(2-Chlorophenyl)-2,3,4,13-tetrahydro-1H-indazolo[1,2-b]phthalazine-1,6,11-trione (**s**):*

Yellow solid. Yield: 94%. m.p.= 259-261 °C. FT-IR (KBr): $\bar{\nu}$= 3019, 2888, 1661, 1478, 1363 cm^{-1}.^{1}HNMR (DMSO-d_6, 400 MHz): δ= 2.13-2.14 (m, 2H), 2.32-2.36 (m, 2H), 3.29-3.45 (m, 2H), 6.61 (s,1H), 7.27-7.31 (m, 2H), 7.39-7.41 (m, 1H), 7.52-7.54 (m, 1H), 7.97-8.01 (m, 2H), 8.08-8.11 (m, 1H), 8.28-8.31 (m, 1H) ppm.

*13-(2-Nitrophenyl)-2,3,4,13-tetrahydro-1H-indazolo[1,2-b]phthalazine-1,6,11-trione (**t**):*

Yellow solid. Yield: 91%. m.p.= 249-251 °C. FT-IR (KBr): $\bar{\nu}$= 3028, 2886, 1664, 1529, 1361 cm^{-1}. ^{1}HNMR (DMSO-d_6, 400 MHz): δ= 2.10-2.17 (m, 2H), 2.31-2.38 (m, 2H), 3.25-3.45 (m, 2H), 7.18 (s, 1H), 7.54 (t, J = 7.2 Hz, 1H), 7.630 (t, J = 6.8 Hz, 1H), 7.71 (d, J = 8 Hz , 1H), 7.99 (m, 3H), 8.1 (m, 1H), 8.3 (m, 1H) ppm. ^{13}CNMR (DMSO-d_6, 100 MHz): δ= 22.3, 24.5, 36.7, 59.3, 117.6, 124.8, 127.3, 128, 128.8, 129.6, 129.8, 131.25, 131.8, 134.1, 134.4, 135, 149.2, 154.2, 154.4, 155.8, 192.7 ppm.

3. Results and Discussion

Very recently the use of [Piper-$(SO_3H)_2$].2Cl in the acceleration of the *N*-Boc protection of amines is reported by our research group [43]. On the basis of these results we were interested in investigating the applicability of [Piper-$(SO_3H)_2$].2Cl in the acceleration of the synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-1,6,11(13*H*)-triones.

In order to optimize the reaction conditions, the synthesis of 13-(4-chlorophenyl)-3,3-dimethyl-2,3,4,13-tetrahydro-1*H*-indazolo[1,2-*b*]phthalazine-1,6,1-trione by using of different amounts of [Piper-$(SO_3H)_2$].2Cl was selected as a model reaction (Table 1). As it can be seen, 40 mg of [Piper-$(SO_3H)_2$].2Cl is suitable for produces of high yields of the product in short reaction times (Table 1, entry 4). Increasing of the amount of catalyst does not significantly affect in the yield of product (Table 1, entry 5, 6). Next, the influence of different temperatures was tested that the best result was obtained at 100 °C (Table 2, entry 3). It is worthy of notice that, the reaction did not progress even after 2 h at the room temperature (Table 2, entry 1).

The condensation of 4-chlorobenzaldehyde with dimedone and phthalhydrazide in the presence of 12.5 mol% [Piper-$(SO_3H)_2$].2Cl was also monitored in different solvents and in the absence of solvent at 100°C (Table 3). The obtained result showed that the yield of the product was high in solvent-free circumstances. Using these data, the optimized conditions are selected as shown in (Scheme 3).

After optimization of the reaction conditions and in order to show the efficiency of this method, various aromatic aldehydes were subjected to the same reaction under the optimized condition (Table 4).

Table 1. Effect of amount of the catalyst on the synthesis of 13- (4-chlorophenyl)- 3,3- dimethyl- 2,3,4,13- tetrahydro-1*H*-indazolo[1,2-*b*]phthalazine-1,6,1-trione.

Entry	Catalyst (mg)	Time(min)	Yield (%)[a]
1	10	90	68
2	20	50	75
3	30	20	83
4	40	10	96
5	50	10	96
6	60	10	97

[a]Isolated yield.

Table 2. Effect of temperature on the synthesis of 13-(4-chlorophenyl)-3,3-dimethyl-2,3,4,13-tetrahydro-1*H*-indazolo [1,2-*b*]phthalazine-1,6,1-trione.[a]

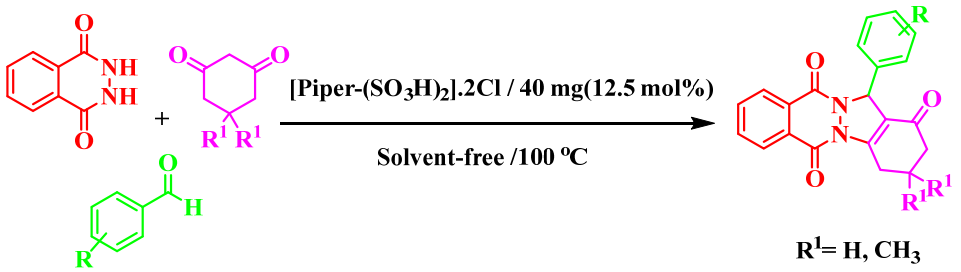
Entry	Temperature	Time (min)	Yield (%)[b]
1	25	120	20
2	70	45	80
3	100	10	97
4	120	10	98

[a]Reaction conditions: 4-Chlorobenzaldehyde (1 mmol), dimedone (1 mmol) and phthalhydrazide (1 mmol); catalyst: [Piper-$(SO_3H)_2$].2Cl (12.5 mol%).
[b]Isolated yields.

Table 3. Effect of various solvents and solvent-free conditions on the synthesis of 13-(4-chlorophenyl)-3,3-dimethyl-2,3,4,13-tetrahydro-1*H*-indazolo[1,2-*b*]phthalazine-1,6,1-trione.[a]

Entry	Solvent	Time(min)	Yield (%)[b]
1	C_2H_5OH	75	40
2	H_2O	60	45
3	CH_3CN	80	20
4	CH_2Cl_2	60	30
5	Solvent free	10	98

[a]Reaction conditions: 4-Chlorobenzaldehyde (1 mmol), dimedone (1 mmol), phthalhydrazide (1 mmol) and catalyst: [Piper-$(SO_3H)_2$].2Cl (12.5 mol%), was used in various solvents (5 ml) at 100 °C.
[b]Isolated yields.



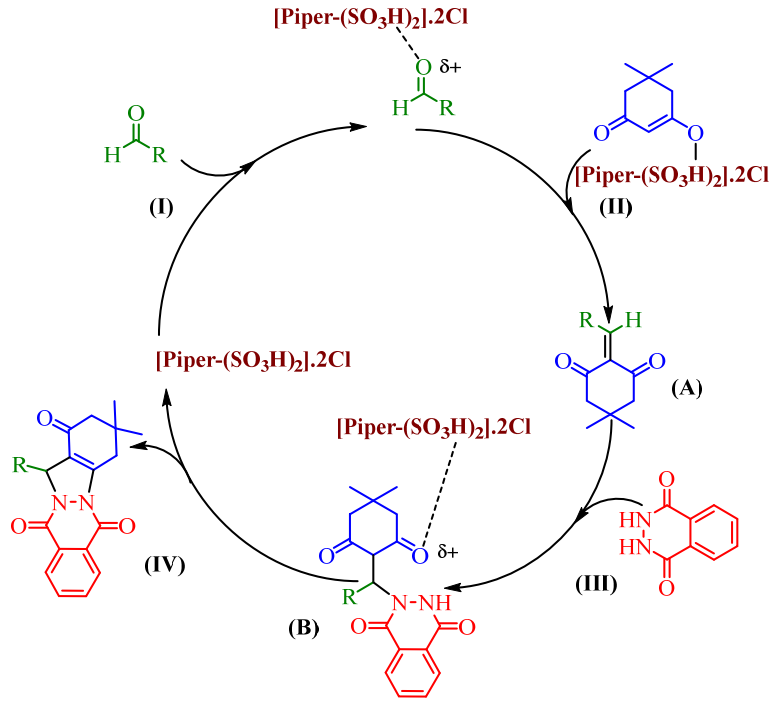
Scheme 3. [Piper-$(SO_3H)_2$].2Cl catalyzed the synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-1,6,11(13*H*)-triones derivatives.

As it is clear from this table the *ortho* Cl, OMe, NO_2 and OH substituted aldehydes are reacted slower than the aromatic aldehydes containing the same functional groups on the other positions because of the steric or electronic effects of the substituents (entries 2-5, 19 and 20). In a similar manner, the reaction of 1,3-cyclohexanedione for the synthesis of 2*H*-indazolo[1,2-*b*]phthalazine-triones was examined and the desired products were obtained in good yields (Table 4, entries 12-20).

A plausible mechanism for the reaction is shown in (Scheme 4). At the beginning of reaction, aldehyde is activated by the proton from [Piper-$(SO_3H)_2$].2Cl which is attacked by the nucleophilic cyclohexanedione to produce intermediate (A). After that the reaction of phthalhydrazide with (A) gives the intermediate (B), which undergoes intramolecular cyclization by the participation of the OH and NH groups to afford the desired product.

The recyclability of the catalyst was checked in the reaction of 4-chlorobenzaldehyde, dimedone and phthalhydrazide under optimized reaction. When the reaction was completed, water was added and [Piper-$(SO_3H)_2$].2Cl was separated by filtration. Then the filtrate was dried at 80 °C to obtain the recycled catalyst and reused for the same reaction. This procedure was repeated at least for five runs (The yields were 98, 97, 95, 93, and 95%, respectively at reaction time 10-15 min) and each time the product was obtained by the recovered catalyst without considerable change in the reaction yield and time.

Exists of typical absorption bands in the FT-IR spectra of [Piper-$(SO_3H)_2$].2Cl such as 3245, 1289, 1172, 1070, 1006, 881, 857 and 587 cm^{-1}, which are acidic peak, demonstrate that in the course of recovery, quality of the catalyst was remained (Fig. 1).



Scheme 4. Proposed mechanism for the synthesis of 2*H*-indazolo[2,1-*b*]phthalazine-1,6,11(13*H*)-trione derivatives in the presence of [Piper-$(SO_3H)_2$].2Cl.

Table 4. Preparation of 2*H*-indazolo[2,1-*b*]phthalazine-1,6,11(13*H*)-triones derivatives using [Piper-$(SO_3H)_2$].2Cl as the catalyst.

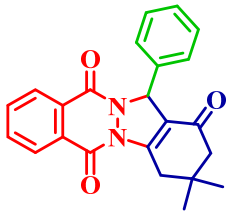
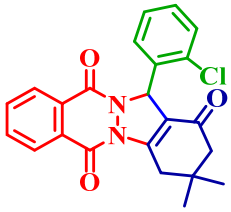
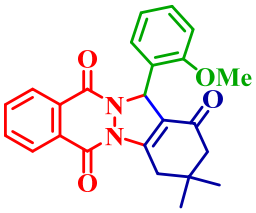
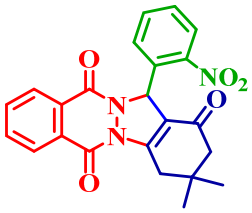
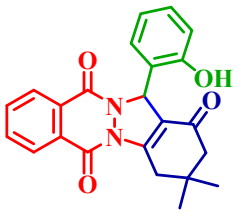
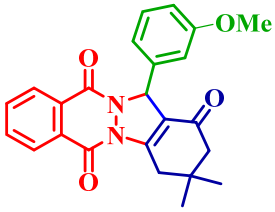
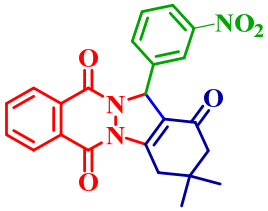
Entry	Product		Time (min)	Yield (%)[a]	m.p. (°C)		Ref.
					Found	Reported	
1		(a)	8	92	201-203	204-206	[18]
2		(b)	12	90	262-264	266-268	[44]
3		(c)	17	89	238-240	242-243	[44]
4		(d)	18	90	232-234	236-238	[44]
5		(e)	25	88	183-185	185-187	[45]
6		(f)	16	92	208-210	206-208	[44]
7		(g)	15	92	268-270	270-272	[44]

Table 4. (*Continued*).

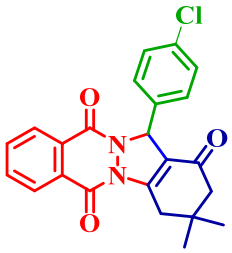
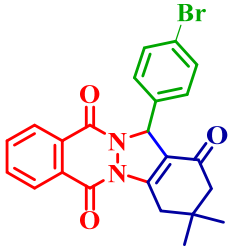
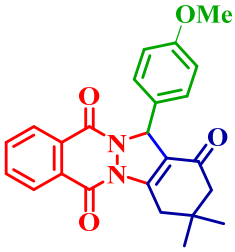
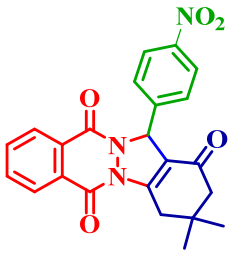
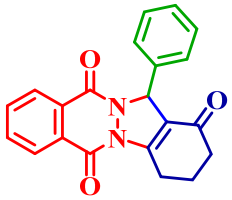
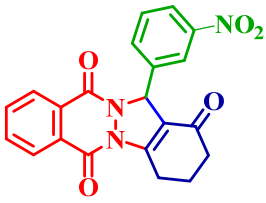
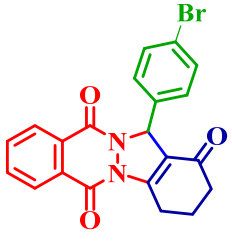
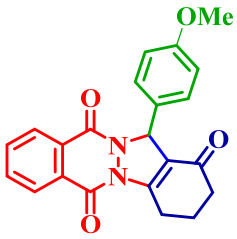
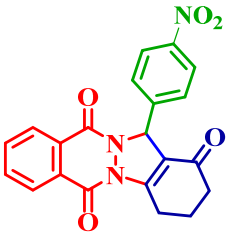
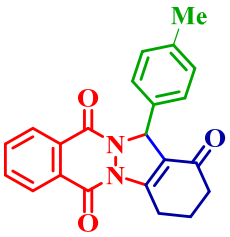
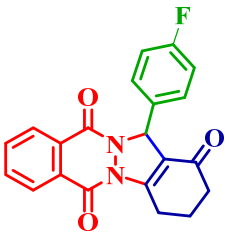
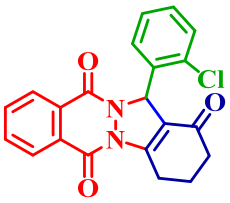
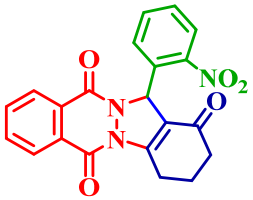
8		(h)	10	97	264-266	262-264	[19]
9		(i)	10	96	264-266	266-268	[21]
10		(j)	12	95	219-221	220-222	[44]
11		(k)	17	94	223-225	226-228	[21]
12		(l)	8	95	221-223	223-224	[44]
13		(m)	15	90	224-226	228-230	[46]
14		(n)	10	93	282-284	278-281	[44]

Table 4. (*Continued*).

15		(o)	10	93	253-255	251-255	[44]
16		(p)	20	88	249-251	252-254	[44]
17		(q)	12	91	246-248	244-246	[44]
18		(r)	8	95	256-258	259-261	[44]
19		(s)	25	94	259-261	232-234	[47]
20		(t)	25	91	249-251	248-250	[47]

[a]Isolated yield.

Furthermore the SEM micrographs of the fresh catalyst (a) and recycled catalyst after 5th run (b) showed that the structure of the catalyst is not changed during the course of the reaction (Fig. 2).

In order to show the merit of this manner the efficiency of [Piper-$(SO_3H)_2$].2Cl in the synthesis of desired product (h) is compared with the other results by different catalysts (Table 5). As shown in this Table, the newly developed manner avoids some of disadvantages such as harsh reaction circumstances, high temperature, use of toxic or moisture sensitive catalyst, use of higher amounts of the catalysts and recoverability of the catalyst.

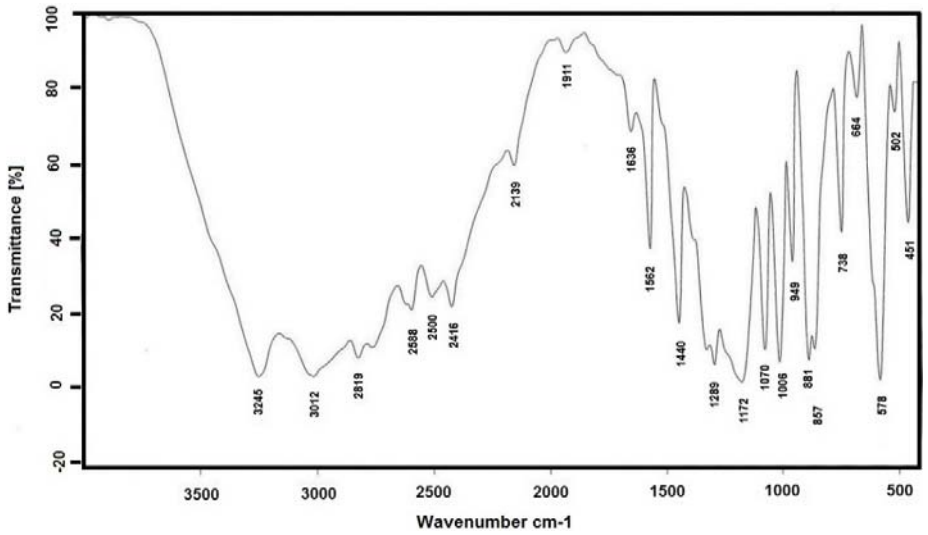


Fig. 1. FT-IR spectra of recovered [Piper-$(SO_3H)_2$].2Cl after 5 run.

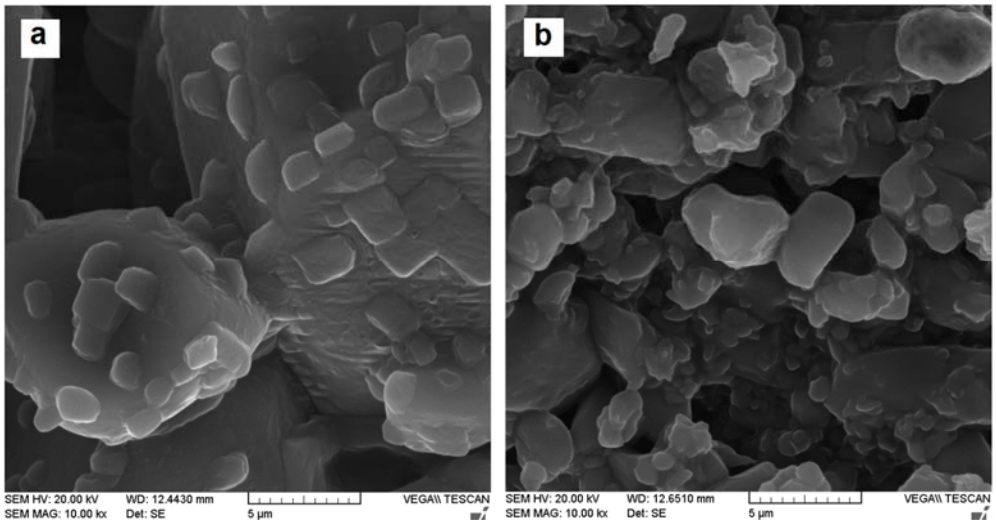


Fig. 2. The SEM micrographs of fresh catalyst (a) and recycled catalyst after 5th run (b)

Table 5. Compared performance of various catalysts with [Piper-$(SO_3H)_2$].2Cl in the synthesis of 13-(4-chlorophenyl)-3,3-dimethyl-2,3,4,13-tetrahydro-1*H*-indazolo[1,2-*b*]phthalazine-1,6,11-trione.

Entry	Catalyst	Conditions	Time (min)	Yield (%)[a]	Ref.
1	—	Solvent-free/ 100 °C	3 hr	>30	This work
2	Cyanuric chloride (3 mol%)	Neat/ 100 °C	15	97	[20]
3	$Mg(HSO_4)_2$ (11.5 mol%)	Solvent-free/ 100 °C	4	88	[18]
4	Iodine (10 mol%)	Sonic bath (35 kHz)	10	92	[46]
5	H_2SO_4 (15 mol%)	[bmim]BF_4/ r.t.	25	88	[47]
6	[bmim]BF_4 & H_2SO_4 (15 mol%)	H_2O-EtOH/ Reflux	30	88	[48]
7	CAN (5 mol%)	PEG/50 °C	120	90	[49]
8	PPA-SiO_2 (0.1 g)	Solvent-free/ 100 °C	6	93	[22]
9	*p*-TSA (30 mol%)	Solvent-free/ 80 °C	10	93	[16]
10	$H_{14}[NaP_5W_{30}O_{110}]/SiO_2$ (50 mol%)	Reflux	7	87.5	[24]
11	[Piper-$(SO_3H)_2$].2Cl (12.5 mol%)	Solvent-free/ 100 °C	10	97	This work

[a]Isolated yield.

4. Conclusions

In summary, we have introduced the [Piper-$(SO_3H)_2$].2Cl as a good catalyst for the synthesis of phthalazine derivatives. The procedure has several advantages including high rates of reaction, ease of preparation and handling of the catalyst, simple experimental procedure and excellent yields.

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