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β-Cyclodextrin as a Supramolecular Catalyst for the Synthesis of 1H-Pyrazolo[1,2-b]phthalazine-5,10-dione Derivatives in Water
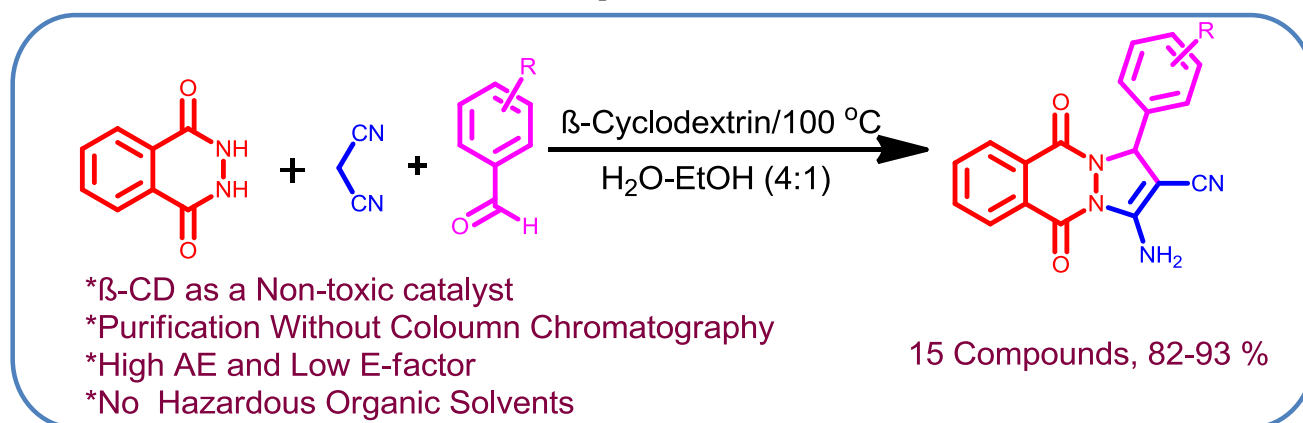
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Abstract An efficient and green method has been developed for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones derivatives by employing 20 mol% β-cyclodextrin via a one-pot multicomponent reaction of aldehydes, malononitrile and phthalhydrazide in H_2O–EtOH (4:1) at 100 °C for first time under neutral condition. The catalyst could be recovered and reused for four consecutive cycles without appreciable loss in catalytic activity.

Graphical Abstract

ß-Cyclodextrin/100 °C
H_2O-EtOH (4:1)

*ß-CD as a Non-toxic catalyst
*Purification Without Coloumn Chromatography
*High AE and Low E-factor
*No Hazardous Organic Solvents

15 Compounds, 82-93 %

1 Introduction

In recent years, environmental protection laws and global warming effects have impelled the researchers to utilize renewable sources for chemical processes with minimum of waste or zero discharge. Therefore, the development of eco-friendly processes is one of the great challenges for organic chemists. On the other hand, heterocycles containing phthalazine moiety are of interest because they show some pharmacological and biological activities [1–3].

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1H-Pyrazolo[1,2-b]phthalazine-5,10-diones derivatives constitute one of the privileged heterocyclic scaffolds known to exhibit important biological activities such as anticonvulsant [4], cardiotonic [5], vasorelaxant [6], cytotoxic [7, 8], antimicrobial [9], antifungal [10], anticancer [11], and anti-inflammatory activities [12]. In addition, the titled compounds, pyrazolo[1,2-b]phthalazine-diones, were also found to have analgesic, antihypoxic, and anti-pyretic activities [13].

In view of the importance of these heterocycles, number of methods have been developed for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones derivatives including PTSA in 1-butyl-3-methylimidazolium bromide {[bmim]Br} as solvent at 100 °C [14]. Recently, Malek Taher Meghsoodlou and co-workers reported the copper(II) acetate monohydrate as an efficient catalyst for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones derivatives under solvent free condition at 80 °C [15]. Some other methods for synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones has been also reported in the literature involving the use of dicationic 4,4′bipyridinium dichloride ordered mesoporous silica nanocomposite one-pot multicomponent method under solvent free condition [16], triethylamine (0.02 g, 20% mol) as catalyst in EtOH under ultrasonication [17], 1-butyl-3-methylimidazolium hydroxide ([Bmim]OH) under Microwave irradiation [18], CuI nanoparticles as catalyst under solvent-free conditions [19], Indium chloride (InCl$_3$) as catalyst under solvent-free conditions [20], Fe$_3$O$_4$ nanoparticles coated by (3-aminopropyl)-triethoxysilane as catalyst under solvent-free conditions [21], NiCl$_2$ as catalyst in ethanol under refluxing [22], N, N, N′, N′-tetrabromobenzene-1,3-disulfonamide [TBBDA] and poly(N-bromo-N ethylbenzene-1,3-disulfonamide) [PBBS] at 80–100 °C under solvent free conditions [23], Al-KIT-6 reflux in ethanol [24], Nanomagnetic basic catalyst of caesium carbonate supported on hydroxyapatite-coated NiO·5ZnO·5Fe$_2$O$_4$ magnetic nanoparticles (NiO·5ZnO·5Fe$_2$O$_4$@HAP-Cs$_2$CO$_3$) [25], protic ionic liquid at 80 °C [26], NaHCO$_3$ at 120 °C under solvent free condition [27], CAN in solvent PEG-400 [28], and deep eutectic mixture (choline chloride: malonic acid) [29] as a homogeneous catalyst at reflux temperature. These reported methodologies have shown good results in many instances. However, some of synthetic strategies also have limitations in terms of metal catalyst, expensive reagents, long reaction time, environmental hazard, harsh reaction conditions, tedious workup procedure, unsatisfactory yield, and use of homogeneous catalyst which are difficult in separation from reaction mixture and reuse. However, the development of green, mild and simpler procedures to eliminate the use and generation of hazardous substances is the foremost goal of green chemistry.

Cyclodextrins are cyclic oligosaccharides possessing hydrophobic cavity, which binds substrate selectively and catalyze chemical reactions with high selectivity. They catalyze reaction by supramolecular catalysis involving reversible formation of host–guest complexes by noncovalent bonding as seen in enzymes [30, 31]. β-Cyclodextrin is a cyclic heptamer composed of seven glucose units jointed head to tail by α-1,4-links. It is widely accepted that the binding forces involved in the inclusion complex formation are Vander Waals interactions, hydrophobic interactions between guest molecules and β-CD [32]. Because of such behaviour β-CD is used as catalyst for varieties of organic reactions in aqueous medium [33–40]. To the best of our knowledge, the use of β-cyclodextrin for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones in aqueous medium has not been reported.

2 Results and Discussion

In continuation of our work on β-CD and development of newer methodologies [41–50], we disclosed a newer methodology for the synthesis of 1H-pyrazolo[1,2-b] phthalazine-5,10-diones derivatives as one-pot synthesis involving three component reaction of substituted aldehydes, malononitrile, and phthalhydrazide in the presence of β-cyclodextrin as a recyclable catalyst in aqueous media H$_2$O–EtOH (8:2) at 100 °C. The general scheme for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones is depicted in Scheme 1.

Scheme 1 General scheme for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones
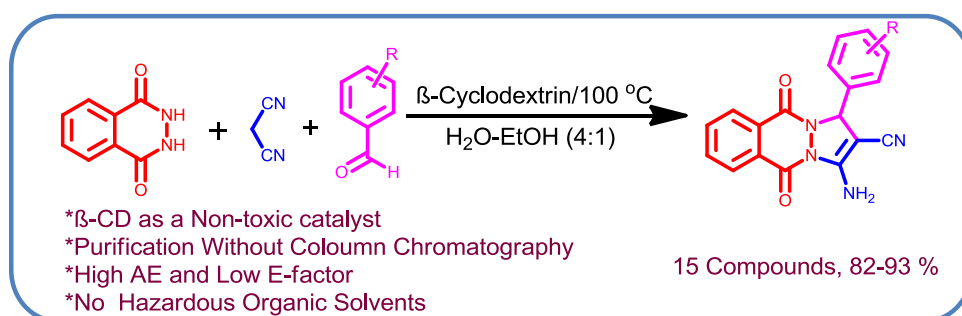
The catalytic role of β-CD for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones has been compared with various reported catalysts and found the present methodology developed green protocol for this transformation as well as the results found decent conversion of products within 2.5–5 h with good to excellent yields up to 82–93% and results are summarized in Table 1.

In order to optimize the reaction conditions and test the performance of β-CD as a catalyst for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones, we studied 4-chlorobenzaldehyde, malononitrile, and phthalhydrazide as a model reaction. The reaction proceeds in the absence of β-CD the reaction is sluggish to give lower yields (20%) with longer reaction times at higher temperature. The best result was obtained for 20 mol% of β-CD affording desired product without column chromatographic purification with 93% of yield within 2.5 h. The results indicate that a catalyst plays a critical role in this reaction. Encouraged by the initial success, we applied the optimal protocol to a variety of aldehydes. Generally, all the reactions were performed using 20 mol% of β-CD in H_2O–EtOH (4:1) at 100 °C and all the reaction completed within 2.5–5 h to give the desired products in good to excellent yields.

Temperature plays an important role, as surprisingly, at the low temperature there was a formation of trace amounts of product and required longer reaction times. As the temperature increases from room temperature to 110 °C,

Table 2 Study of the effect of temperature on reaction time and yields

Entry	Temp. (°C)	Time (h)	Yield[a] (%)
1	Rt	12	Trace
2	40	12	30
3	60	8	60
4	80	3	85
5	**100**	**2.5**	**93**
6	110	2.5	93

Bold indicates the best result of present works

Reaction conditions: 4-chlorobenzaldehyde (2 mmol), malononitrile (2 mmol), phthalhydrazide (2 mmol), β-CD (0.4 mmol) and H_2O–EtOH (4:1, 10 mL) at 100 °C

[a]Isolated yield

the yields were found to increase while the reaction time decreases. We obtained the best results at 100 °C (Table 2, entry 5), hence all the reactions were performed at the temperature of 100 °C.

The amount of catalyst was evaluated in the model reaction (Scheme 2) at 100 °C in H_2O–EtOH (4:1). The best result was obtained for 20 mol% of β-CD (Table 3, entry 5), affording 93% of yield within 2.5 h and the use of excess catalyst had no impact either on the rate of reaction or on the yield of product (Table 3, entries 6 and 7).

Table 1 Comparison for preparation methods of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones with various reported catalysts
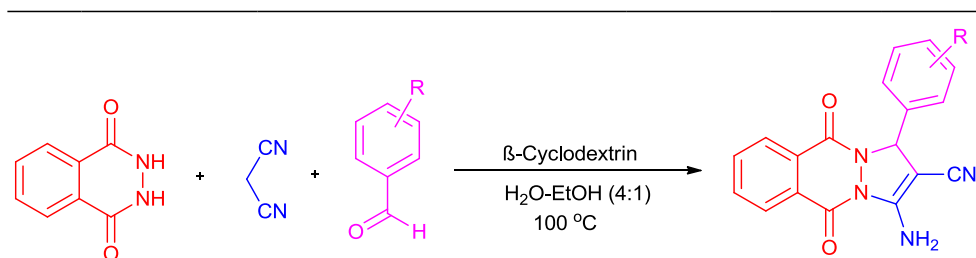
Entry	Catalyst	Reaction conditions	Time (h/min)	Yield (%)	References
1	PTSA-[bmim]Br	30 mol%, 0.30 g, 100 °C	3–5 h	75–97	[14]
2	Cu(OAc)$_2$·H$_2$O	20 mol%, 80 °C, SF	3–5 h	73–83	[15]
3	Et$_3$N	20 mol%, 50 °C, EtOH, U.S.	1 h	85–98	[17]
4	[Bmim]OH	0.2 mL, 45 °C, MW	4–5 min	89–97	[18]
5	CuI. NPs	10 mol%, 70 °C, SF	25–35 min	82–93	[19]
6	InCl$_3$	10 mol%, 80 °C, SF	25–35 min	85–94	[20]
7	NiCl$_2$·6H$_2$O	15 mol%, reflux in EtOH	2–6 h	80–94	[22]
8	(1) TBBDA	0.05 g, 100 °C, SF	10–65 min	63–89	[23]
	(2) PBBS	0.05 g, 100 °C, SF	10–140 min	53–85	
9	Al-KIT-6	300 mg, reflux in EtOH	4 h	84–93	[24]
10	**β-Cyclodextrin**	**20 mol%, 100 °C in H$_2$O:EtOH (4:1)**	**2.5 h**	**82–93**	**This work**

Bold indicates the best result of present works

U.S. ultrasonication, *SF* solvent free, *MW* microwave assisted

Scheme 2 Synthesis of 3-amino-1-(4-chlorophenyl)-5,10-dioxo-5,10-dihydro-1H-pyrazolo[1,2-b]phthalazine-2-carbonitrile
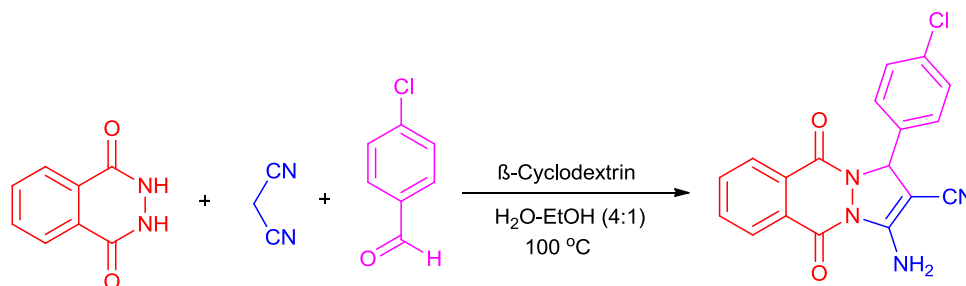

Table 3 3-Amino-1-(4-chlorophenyl)-5,10-dioxo-5,10-dihydro-1H-pyrazolo[1,2-b]phthalazine-2-carbonitrile formation using different amounts of catalyst at reflux in aqueous media

Entry	Catalyst (mol%)	Time (h)	Yield[a] (%)
1	–	12	20
2	β-CD (5 mol%)	8	60
3	β-CD (10 mol%)	5	80
4	β-CD (15 mol%)	3	85
5	**β-CD (20 mol%)**	**2.5**	**93**
6	β-CD (25 mol%)	2.5	93
7	β-CD (30 mol%)	2.5	92

Bold indicates the best result of present works

Reaction conditions: 4-chlorobenzaldehyde (2 mmol), malononitrile (2 mmol), phthalhydrazide (2 mmol), β-CD (0.4 mmol) and H_2O–EtOH (4:1, 10 mL) at 100 °C

[a]Isolated yield

Table 4 Optimization of solvents study for the synthesis of 3-amino-1-(4-chlorophenyl)-5,10-dioxo-5,10-dihydro-1H pyrazolo[1,2-b]phthalazine-2-carbonitrile

Entry	Catalyst (mol%)	Solvent	Time (h)	Yield[a] (%)
1	20	–	12	Trace
2	20	H_2O	12	60
3	20	THF	10	71
4	20	CH_3CN	6	77
5	20	DMF	8	81
6	20	EtOH	5	86
7	20	H_2O:EtOH (1:1)	4	88
8	**20**	**H_2O:EtOH (4:1)**	**2.5**	**93, 91, 88, 85[b]**

Bold indicates the best result of present works

Reaction conditions: 4-chlorobenzaldehyde (2 mmol), malononitrile (2 mmol), phthalhydrazide (2 mmol), β-CD (0.4 mmol) and H_2O–EtOH (4:1, 10 mL) at 100 °C
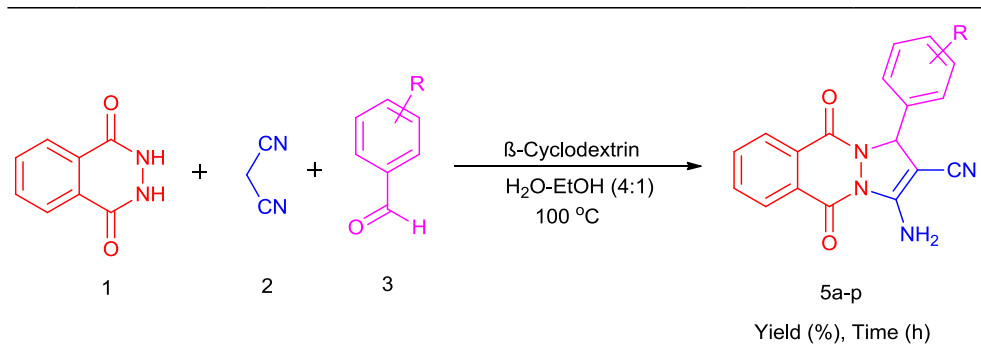
[a]Isolated yield

[b]Catalyst was reused four times

Solvents also play an important role in the studied transformation; we carried out the above reaction in various solvents in order to check catalytic response of β-CD. As shown in Table 4, when the reaction was carried out under solvent free conditions, the targeted product was obtained with very low yields. To find the best solvent for this transformation, the present one-pot three component reaction was screened in H_2O, CH_3CN, THF, DMF, EtOH, H_2O–EtOH (1:1), H_2O–EtOH (4:1) mixture. Among all these solvents, H_2O–EtOH (4:1) was found to play an effective role in this transformation affording highest yields (Table 4, entry 8). Therefore, H_2O–EtOH (4:1) was selected as the solvent system for this transformation.

After optimizing the reaction conditions, we extended this process to other substrates. The scope of the reactions is illustrated with respect to various aldehydes and the results are summarized in Table 5, entries 5a–5o. Both electron-rich and electron-deficient aromatic aldehydes such as 4-methyl, 4-cyano, and 4-nitro, 4-chlorobenzaldehydes reacted efficiently with phthalhydrazide and malononitrile to furnish the corresponding 1H-pyrazolo[1,2-b] phthalazine-5,10-diones derivatives in good yields (entries 5d, 5f, 5h and 5n, Table 5). The substituents present on the aromatic ring had shown some effect on the conversion. In all cases, the conversion was completed within 2.5–5 h with good to excellent yields of desired products without forming any by-products. The nature of functional group on the aromatic ring of aldehyde exerted a slight influence on the reaction time. The rate of reaction decreases in the case of arylaldehyde carrying an electron donating group in comparison to the unsubstituted. Similarly, hetero aromatic aldehydes like Thiophene-2-carboxaldehyde and Pyridine-3- carboxaldehyde reacted well to furnish the corresponding 1H-pyrazolo[1,2-b]phthalazine-5,10-diones in good yield (entry 5l and 5m, Table 5).

Table 5 Synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones in the presence of β-CD in H_2O–EtOH (4:1) at 100 °C.

Table 5 Synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones in the presence of β-CD in H$_2$O–EtOH (4:1) at 100 °C

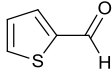| Entry	Aldehyde	Product	Time (h)	Yield (%)	Mp (°C) [Lit.]
5a			2.5	86	278–280
					276–278 [14]
5b			3	92	256–258
					259–262 [14]
5c			3	90	268–270
					266–267 [17]
5d			2.5	93	270–272
					270–272 [14]
5e			3	89	256–258
					260–262 [20]

Table 5 (continued)

5a–p
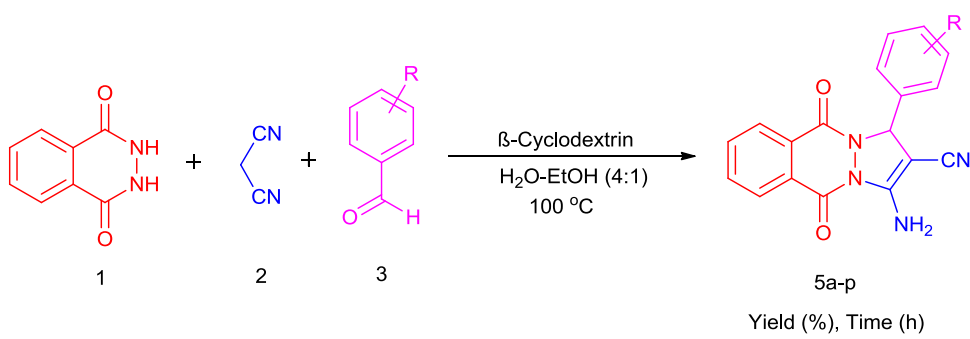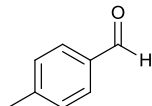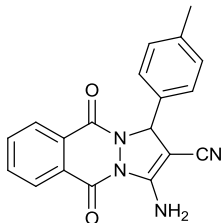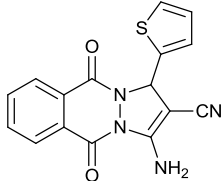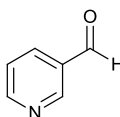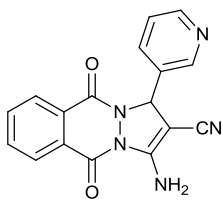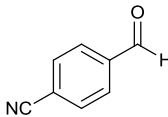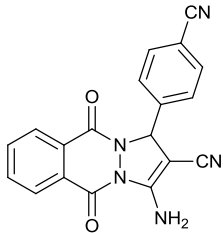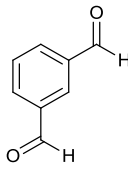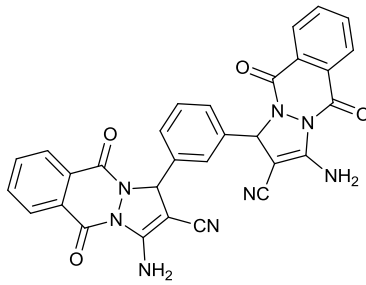
Yield (%), Time (h)

Entry	Aldehyde	Product	Time (h)	Yield (%)	Mp (°C) [Lit.]
5 f			2.5	93	260–262
					262–264 [18]
5 g			3.5	88	270–272
					270–272 [18]
5h			3	86	264–266
					267–268 [20]
5i			3	88	266–268
					268–270 [20]
5j			3	82	262–264
					263–265 [17]

Table 5 (continued)

Entry	Aldehyde	Product	Time (h)	Yield (%)	Mp (°C) [Lit.]
5k			5	86	250–252
					253–255 [19]
5l			4	84	246–248
					244–246 [19]
5 m			3	88	232–234
					230–232 [26]
5n			2.5	89	290–292
5o			4.5	85	288–290

Experimental conditions: aldehyde (2 mmol), phthalhydrazide (2 mmol), malononitrile (2 mmol) catalyst (20 mol%), H$_2$O–EtOH (4:1, 10 mL) at 100 °C

Scheme 3 Synthesized 1,1′-(1,3-phenylene)bis(3-amino-5,10-dioxo-5,10-dihydro-1H-pyrazolo [1,2b]phthalazine-2-carbonitrile) derivative
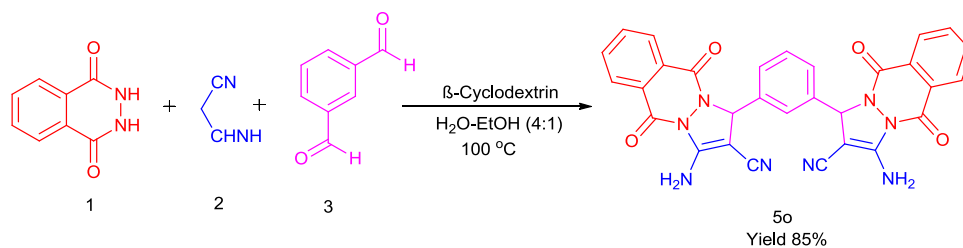
Inspired by the above results, we synthesized 1,1′-(1,3-phenylene)bis(3-amino-5,10-dioxo-5,10-dihydro-1H-pyrazolo[1,2b]phthalazine-2-carbonitrile) derivative from isopthalaldehyde, malononitrile and phthalhydrazide at the same reaction conditions (Scheme 3). The compound was characterized by ^{1}H-NMR, ^{13}C-NMR, Mass and the results obtained are summarized in Table 5, entry 5o.

Next, the green and sustainability factors such as atom economy and E-factor of present method were calculated for the model reaction between 4-chlorobenzaldehyde, malononitrile, and phthalhydrazide to afford the 3-amino-1-(4-chlorophenyl)-5,10-dioxo-5,10-dihydro-1-H pyrazolo[1,2-b]phthalazine-2-carbonitrile (Table 5, entry 5d). The results indicate that the present method follows green chemistry principles with smaller E-factor as 0.13 and high atom economy (AE) as 91% (see the Supporting Information for calculations).

To account for the very efficient catalysis by β-CD of this multicomponent reaction, wherein supramolecular catalyzed reactions are involved, it is proposed that β-CD with its seven free primary –OH groups acting synergistically behaves as an efficient host and supramolecular catalyst Fig. 1. In the first step, the aldehyde binds to the β-CD cavity. Activation of a proton from malononitrile by β-CD catalyzes its Knoevenagel condensation with the carbonyl group to give the ylidenemalononitrile(I). The co-operative enzyme-like binding of these intermediates which ensure their tighter fit into the cavity facilitates further reactions, namely Michael addition of phthalhydrazide(II) to ylidenemalononitrile (by activation and of a proton from phthalhydrazide), its undergo cyclization to form desire product.

^{1}H NMR study of the complex: to gain further evidence we propose a supramolecular catalysis for the formation of pyranopyrazole product by inclusion complex of phthalhydrazide β-CD and 4-cyanobenzaldehyde Fig. 2. The formation of inclusion complex can be explained by ^{1}H NMR studies. The ^{1}H NMR spectra of the catalyst β-CD and its inclusion complex with one of the reactant 4-cyanobenzaldehyde is recorded in D$_2$O and are given in Fig. 3 and Table 6, it is clear that there is upfield shift from 3.8959 to 3.8482 (0.0477) of H3

and 3.7886–3.7204 (0.0682) of H5 protons of β-CD-4-cyanobenzaldehyde complex compared to β-CD, indicating the complex formation. These observations clearly demonstrate that the aldehyde is ideally located for the condensation with malononitrile and phthalhydrazide in the CD cavity. On the basis of the above results and previous studies [51, 52], we suggest that the internal hydrophobic cavity of β-CD forms inclusion complex with aldehyde more effectively and enhances the rate of reaction with increase in the yield of product and decreases the reaction time.

2.1 General Procedure for Synthesis of 1H-Pyrazolo[1,2-b]phthalazine-5,10-diones

The mixture of carbonyl compound aldehyde (2 mmol), phthalhydrazide (2 mmol) and malononitrile (2 mmol) were added in β-cyclodextrin (0.4 mmol) solution containing H$_2$O–EtOH (4:1, 10 mL). The resulting mixture was stirred at 100 °C. After completion of the reaction (monitored by TLC), the reaction mixture was poured into 20 mL water. Filtered the product and washed with hot water. The crude product was recrystallized with 30% aqueous ethanol to afford the desired product. The filtered aqueous layer was cooled at 5 °C to recovery of β-CD by filtration. The recovered β-CD was reused for 3–4 consecutive runs in this reaction without any significant loss in yield and activity.

3 Catalyst Recovery

The catalyst recovery and reusability were studied by four cycles including the use of fresh catalyst for the synthesis of 3-amino-1-(4-chlorophenyl)-5,10-dioxo-5,10-dihydro-1H-pyrazolo[1,2-b]phthalazine-2-carbonitrile (Table 5, entry 5d). In every cycle, the catalyst was almost quantitatively recovered and after third and fourth use of catalyst decreasing yield is not much more significant which is shown in Fig. 4.
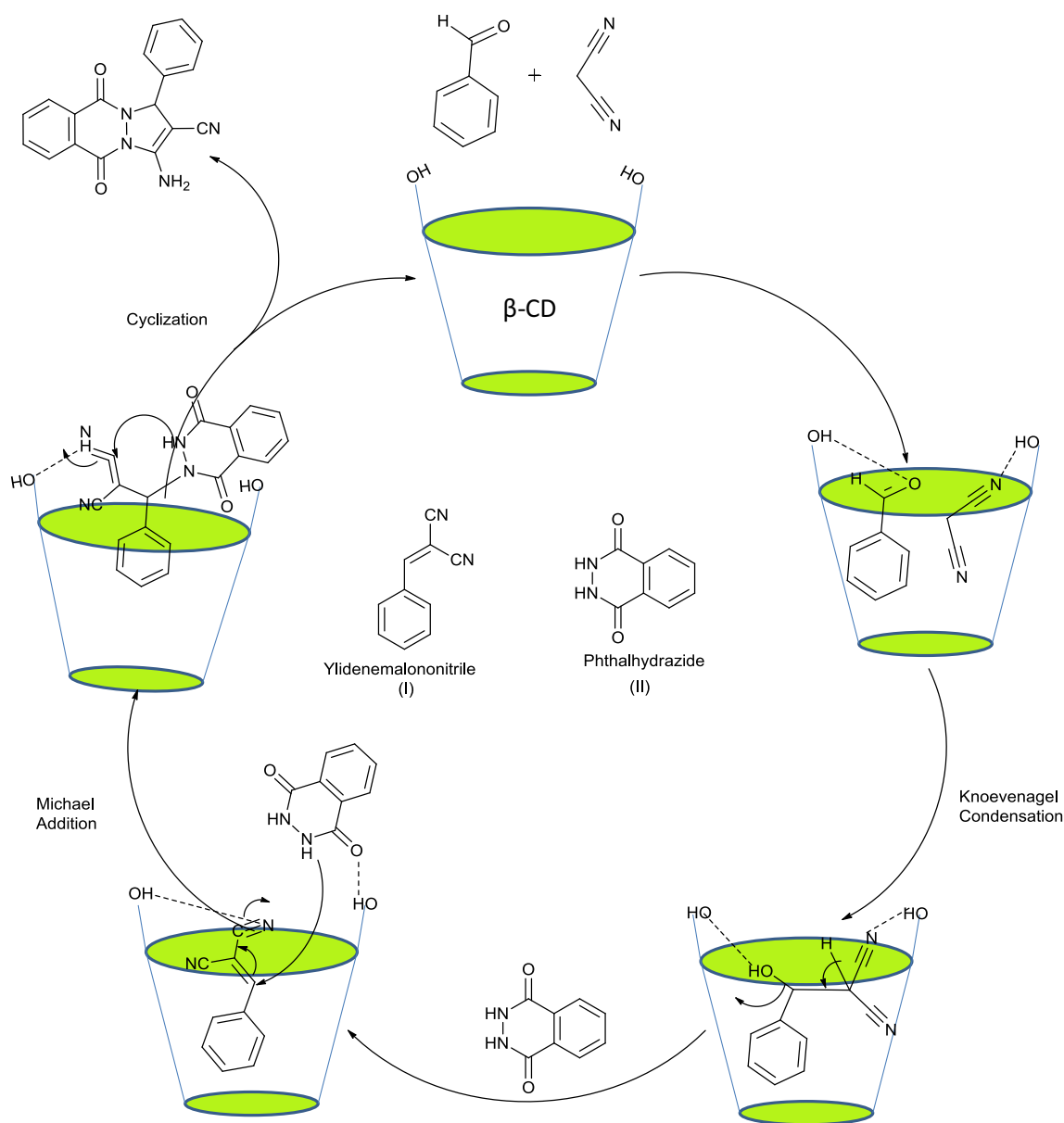
Fig. 1 Plausible mechanism for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones derivatives

4 Conclusion

In summary, we have developed a one-pot, three-component ecofriendly protocol for the synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones derivatives catalyzed by supramolecular, biodegradable and reusable catalyst β-cyclodextrin. The salient features of the present methods are environmentally benign [low E-factor (0.13) and high atom economy (AE) = 91%], mild neutral reaction conditions, good to excellent yields of products, simple and clean workup of the desired product without column chromatography and easy recovery as well as reuse of the catalyst.

Fig. 2 **a** Structure of
β-cyclodextrin and **b** inclusion
complex of β-cyclodextrin with
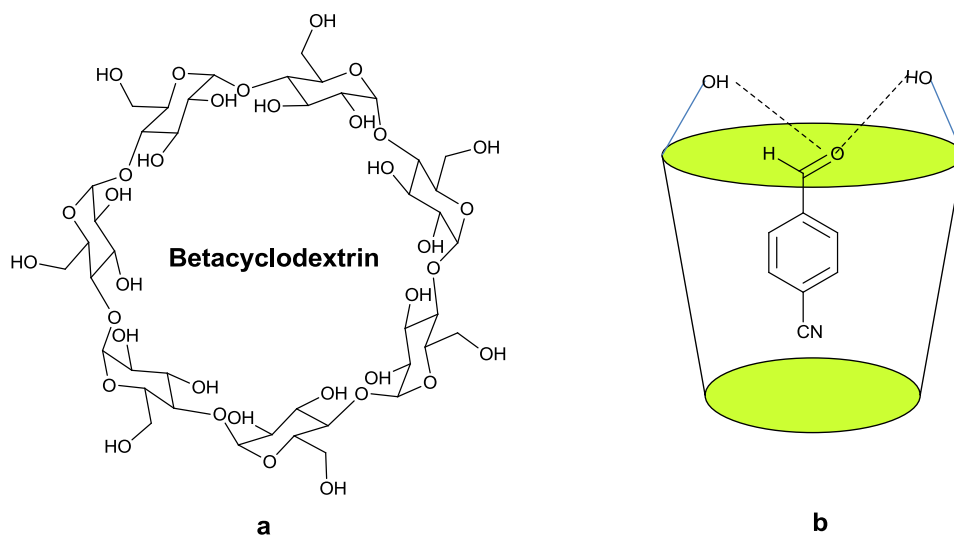4-cyanobenzaldehyde

Fig. 3 ^{1}H-NMR of spec-
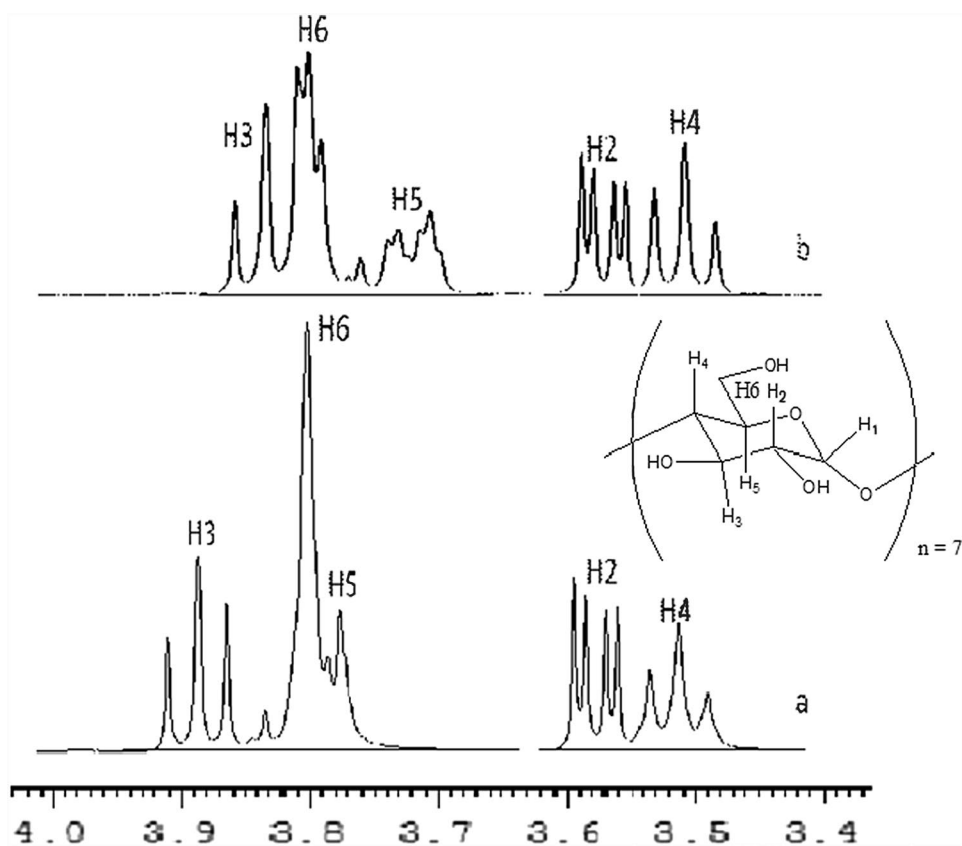tra **a** β-CD and **b** β-CD-4-
cyanobenzaldehyde inclusion
complex

Table 6 400 MHz ^{1}H-NMR
chemical shifts of β-CD protons
in the free and inclusion
complex in D$_2$O

Proton	H1	H2	H3	H4	H5	H6a,b
β-CD	5.0021	3.5815	3.8959	3.5154	3.7886	3.8094
Complex	4.9940	3.5743	3.8482	3.5107	3.7204	3.8032
Δδ	0.0081	0.0072	**0.0477**	0.0047	**0.0682**	0.0062

Bold indicates the best result of present works
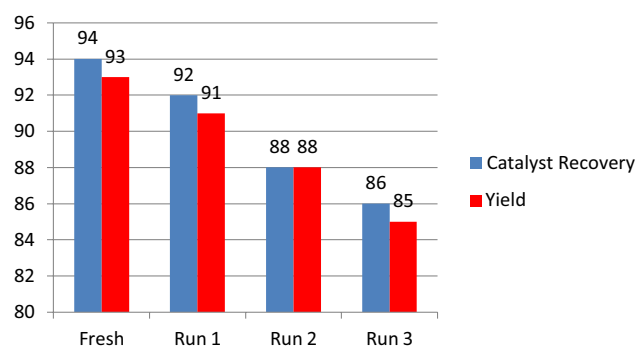
Chemical shift expressed in ppm.

Fig. 4 Reuse and recovery of β-CD and its effect on yield

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