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Caffeine: A green, natural and biodegradable catalyst for convenient and expedient eco-safe synthesis of 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives under solvent-free conditions

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A green, convenient, highly versatile and solvent-free synthetic route for caffeine catalyzed one-pot multi-component synthesis of biologically active 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives *via* one-pot four-component condensation reaction of phthalimide, hydrazine monohydrate, aryl aldehydes and malononitrile has been studied. The green, natural, biodegradable and inexpensive catalyst, eco-safe reaction, solvent-free conditions, avoidance of hazardous or toxic catalysts, simplicity of operation and work-up procedures with no necessity of chromatographic purification steps, the availability and ease of handling of this solid catalyst and good to high yields are the notable benefits for the highly efficient synthesis of these products.

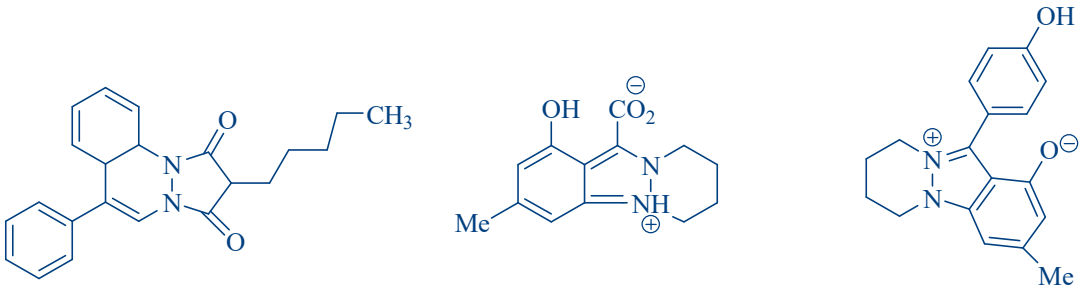
Keywords: Caffeine, naturally green, biodegradable catalyst, 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives, eco-safe procedure, solvent-free conditions

In the development of eco-safe synthetic routes for biologically active compounds, multi-component reactions (MCRs) have been recognized as a powerful tool during the past decades. These reactions comprises the formation of several bonds in a single operation with notable advantages such as simple work-up, atom-economy, mild and environmentally-friendly, low-cost, one-pot. Furthermore, in recent years, green chemistry, has become to one of the best approach for green and efficient preparation of organic compounds. The special benefits of green chemistry for the synthesis of heterocyclic compounds are using non-toxic substrate and environmentally benign nature and due to recent

catalyst[1-3] in the multi-component reactions[4-7].

Among the various nitrogen-containing heterocyclic compounds, 1*H*-pyrazolo [1,2-*b*] phthalazine-5, 10-dione derivatives have received considerable attention due to their various biological and pharmacological activities[8] (Figure 1) such as anticancer[9], anti-inflammatory[10], anti micrbiological[11] and they have been reported to possess vasorelaxant[12], cardiotonic[13], anticonvulsant[14] and antifungal[15].

Between the known procedures for the synthesis of 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives, the most straightforward method for synthesis of these systems involves a four-component tandem reaction of phthalimide/phthalic anhydrid, hydrazine monohydrate, aromatic aldehyde derivatives and malononitrile or three-component reaction of phthalhydrazide, aryl aldehyde derivatives and malononitrile utilizing a variety of homogeneous and heterogeneous catalysts, such as $Ce(SO_4)_2.4H_2O$[16], SBA-Pr-SO_3H[17], $InCl_3$[18], $NiCl_2.6H_2O$[19], [Bmim] OH[20], Ultrasound-assisted[21], *p*-TSA[22], STA[23], CuI nanoparticles[24], PTSA/[Bmim]Br[25], TBBAD[26]. Although these protocols find certain merits of their own, still they suffer from a number of demerits such as relying on multi-step conditions, use of toxic organic solvents or catalysts containing transition metals, tedious ne waste discarding, high reaction time, and low yields. Thus, a search for more eco-safe, general, clean, efficient, feasible, and high yielding routes to this class of 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives remains a valid exercise. Due to the above considerations and our interest for the development production of 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives the study of green and efficient catalyst for simple synthesis of these heterocyclic compounds is an important aim in our recent researches and finally, we have reported caffeine as a naturally green, biodegradable, inexpensive and cost effective catalyst



Cinnopentazone **Nigellicine derivatives**

Figure 1 — Biologically active compounds with two ring junction nitrogen atoms

Figure 2 — Structure of Caffeine

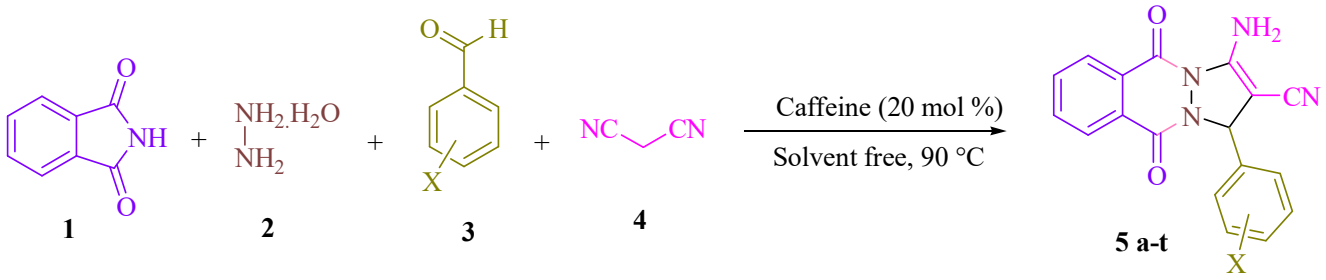
for one-pot four-component condensation of 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives under solvent-free conditions. Caffeine (trimethyl-xanthine alkaloid) (Figure 2) is chemically related to the adenine and guanine bases of deoxyribonucleic acid (DNA) and ribonucleic acid (RNA). It is found in the seeds, nuts, or leaves of a number of plants native to South America and East Asia and helps to protect them against predator insects and to prevent germination of nearby seeds. It is the world's most widely consumed psychoactive drug including Parkinson and Alzheimer's disease[27-29]. There are several known mechanisms of action to explain the effects of caffeine for example caffeine augments the antidepressant-like activity of mianserin and agomelatine in forced swim and tail suspension tests in mice[30]. The most prominent is that it reversibly blocks the action of adenosine on its receptor and consequently prevents the onset of drowsiness induced by adenosine. Caffeine also stimulates certain portions of the autonomic nervous system[31].

Caffeine has emerged as natural, green, cheap and efficient solid catalyst in various organic transformations[32]. The advantages of caffeine as solid catalyst in organic compounds synthesis is environment friendly, mild, easily available, non-toxic and biodegradable. Also caffeine can be successfully used in the type of carbon-carbon bonds as green and natural catalyst in organic synthesis. Furthermore, one of the sources of environmental pollution is the usage of organic solvents under reflux conditions and the need for column chromatography to purify the products. In this present work, the products were obtained through simple filtration with no need for column chromatographic separation.

Results and Discussion

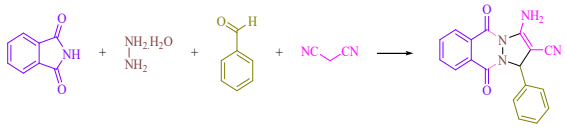
In the present study a series of 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives were prepared by condensing phthalimide (**1**, 1.0 mmol), hydrazine monohydrate (**2**, 1.0 mmol), aromatic aldehyde derivatives (**3**, 1.0 mmol) and malononitrile (**4**, 1.0 mmol) under conventional heating conditions using 20 mol% of caffeine as a naturally biodegradable and readily available catalyst without any solvent (Scheme I).

At first, in order to optimize the amount of caffeine, the cyclocondensation reaction of phthalimide (1.0 mmol), hydrazine monohydrate (1.0 mmol), benzaldehyde (1.0 mmol) and malononitrile (1.0 mmol) was carried out under solvent-free conditions at 90°C in the presence of different quantities of caffeine as the catalyst (Table I). As shown in Table I, in the absence of the catalyst, the formation of product **4a** was not observed (Table I, entry 1). By adding catalyst (5 mol%) to the reaction mixture, the product was formed with relatively low yield (Table I, entry 2). The yield of product **5a** was improved as the amount of caffeine increased from 5 mol% to 15 and 20 mol% (Table I, entries 2-5). A further increase in mol% of caffeine (25 mol%) did not have any significant effect on the yield of the product or reaction time (Table I, entries 11). It was observed



Scheme I — Synthesis of 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives

Table I — Optimization of the reaction condition [a]



Entry	Caffeine (mol %)	Temperature (°C)	Time (h)	Isolated Yields (%)
1	Catalyst free	90	480	No product
2	5	90	5.5	32
3	10	90	4.5	52
4	15	90	3	74
5	20	90	3	89
6	20	rt	480	No product
7	20	40	6	30
8	20	60	4.5	62
9	20	80	3	78
10	20	100	3	89
11	25	90	3	90

[a]Reaction conditions: phthalimide, hydrazine monohydrate, aromatic aldehyde derivatives and malononitrile (1:1:1:1) and caffeine was heated at 90 °C for the appropriate time.

that 20 mol% loading of the catalyst provided the best yield. Therefore, 20 mol% was chosen as the optimal quantity of caffeine. Then, the same reaction was tested under different temperatures including RT, 40, 60, 80, 90 and 100°C. The maximum yield in shorter reaction time was achieved under solvent-free conditions at 90°C (Table I, entries 5). With the above-mentioned optimized conditions in hand, we then proceeded to probe the substrate diversity of this multi-component reaction by using readily available staring materials. It was found that the reaction proceeded efficiently and afforded the targeted products **5a-t** in high yields and the results are shown in Table II. Based on the results presented in Table II, it can be seen that the reaction of aryl aldehydes bearing both electron-donating and electron-withdrawing substituents with phthalimide (1.0 mmol), hydrazine monohydrate (1.0 mmol) and malononitrile worked well, and the corresponding 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives were obtained in reasonable yields.

Comparison of catalytic ability some of the catalysts reported in the literature for synthesis of 1*H*-pyrazolo[1,2-b]phthalazine-5,10-dione derivatives are shown in Table III. This study reveals that caffeine has shown its extraordinary potential to be an alternative green, biodegradable, mild, inexpensive and highly efficient catalyst for synthesis of these biologically active nitrogen-containing heterocyclic compounds, in addition to the use of solvent-free conditions with excellent yield and short reaction times in the reaction are the notable advantages of the present methodology.

Also, ^{1}H NMR data of the products have been compared with literature for synthesis of 1*H*-pyrazolo [1,2-*b*] phthalazine-5,10-dione derivatives are shown in Table IV.

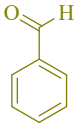
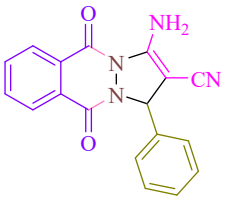
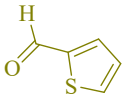
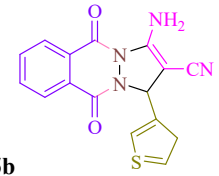
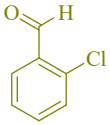
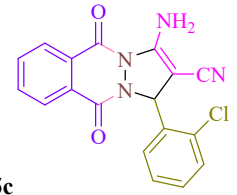
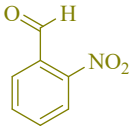
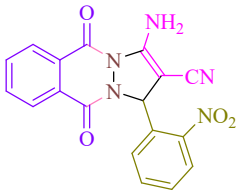
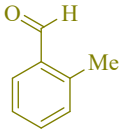
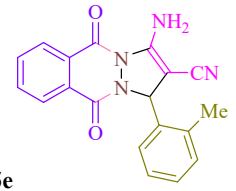
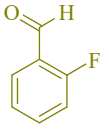
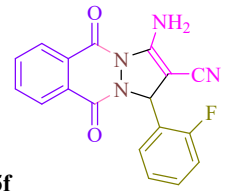
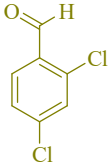
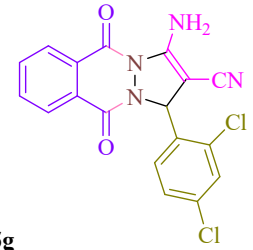
Experimental Section

Melting points of all compounds were determined using an Electro thermal 9100 apparatus. Also, nuclear magnetic resonance, ^{1}H NMR spectra were recorded on a Bruker DRX-400 Avance instrument with DMSO-d_6 as solvents. For the present work, all reagents and solvents were purchased from Merck, Fluka and Acros chemical companies and used without further purification.

General procedure for preparation of pyrazolo [1,2-*b*]phthalazine-5,10-dione derivatives, 5a-t

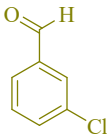
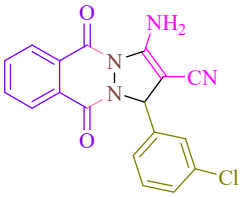
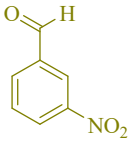
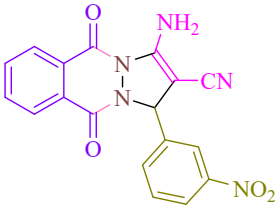
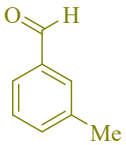
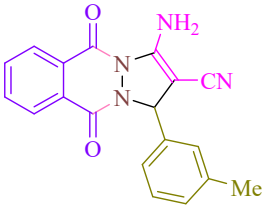
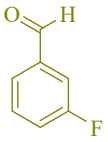
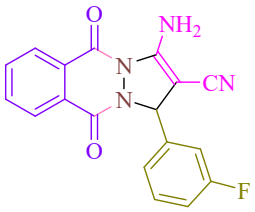
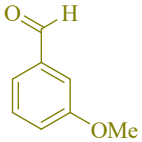
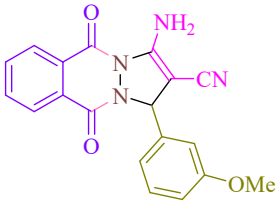
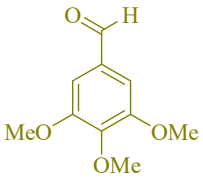
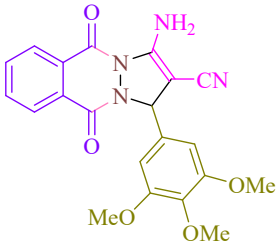
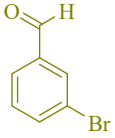
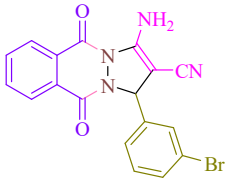
A mixture of phthalimide (**1**, 1.0 mmol), hydrazine monohydrate (**2**, 1.0 mmol) and caffeine (20 mol %) was heated for 2 h at 90°C. Then aromatic aldehyde (**3**, 1.0 mmol) and malononitrile (**4**, 1.0 mmol) were added and the mixture was heated for the appropriate time. After completion of the reaction (checked by Thin Layer Chromatography, TLC) the mixture was cooled to RT, the solid products were filtered and then recrystallized from ethanol to give pure compounds **5a-t**. Products have been characterized by melting points and ^{1}H NMR spectroscopy. Spectra data of all products are presented as follows.

Table II — Caffeine catalyzed synthesis of pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives under solvent-free conditions.

Entry	Ar	Product	Time (h)	Isolated Yields (%)	m.p. (°C)	Lit. m.p. (°C)
1		 **5a**	3	89	269-270	270-272[24]
2		 **5b**	3.5	85	243-245	244-246[24]
3		 **5c**	4	84	254-257	257-259[23]
4		 **5d**	3	86	266-268	265-266[17]
5		 **5e**	3	90	247-249	248-250[24]
6		 **5f**	2	92	268-269	268-270[18]
7		 **5g**	4.5	85	243-245	242-244[17]

(*Contd.*)

Table II — Caffeine catalyzed synthesis of pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives under solvent-free conditions. (*Contd.*)

Entry	Ar	Product	Time (h)	Isolated Yields (%)	m.p. (°C)	Lit. m.p. (°C)
8		 **5h**	4	82	264-266	266-267[17]
9		 **5i**	3	83	270-272	269-271[25]
10		 **5j**	3	88	250-251	250-252[24]
11		 **5k**	2.5	89	264-266	263-265[23]
12		 **5l**	3.5	86	249-251	248-251[21]
13		 **5m**	4	85	254-255	253-255[18]
14		 **5n**	4	81	271-273	270-272[17]

(*Contd.*)

Table II — Caffeine catalyzed synthesis of pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives under solvent-free conditions. (*Contd.*)

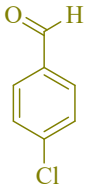
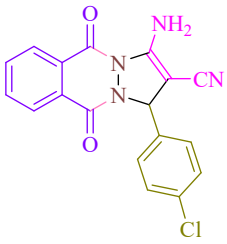
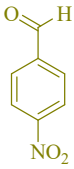
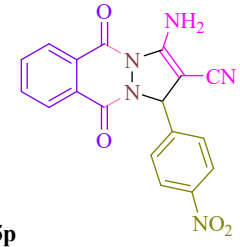
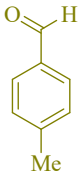
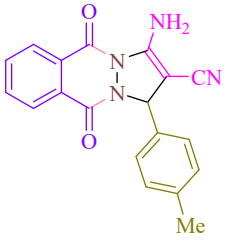
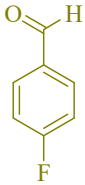
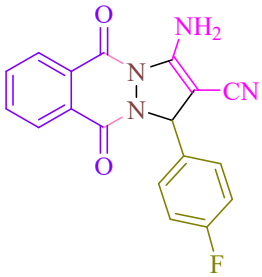
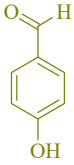
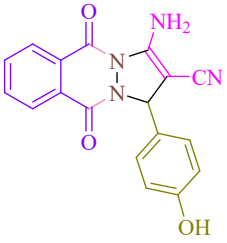
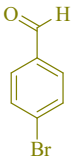
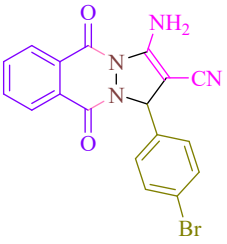
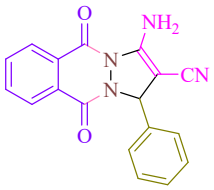
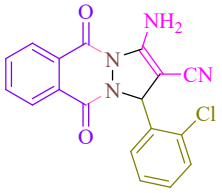
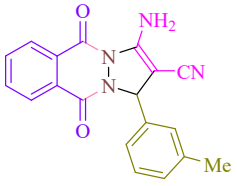
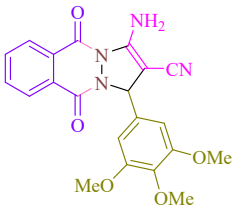
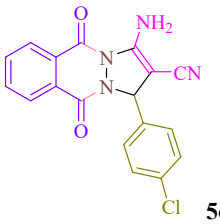
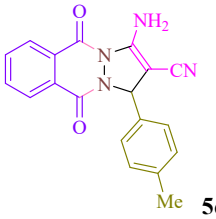
Entry	Ar	Product	Time (h)	Isolated Yields (%)	m.p. (°C)	Lit. m.p. (°C)
15		 **5o**	4	83	272-274	270-272[25]
16		 **5p**	3.5	84	227-229	228-229[23]
17		 **5q**	3	89	251-253	253-255[24]
18		 **5r**	2.5	87	264-265	263-265[17]
19		 **5s**	4.5	81	271-273	270-272[18]
20		 **5t**	4.5	84	264-266	265-267[17]

Table III — Comparison of catalytic ability some of catalysts reported in the literature for synthesis of 1*H*-pyrazolo [1,2-*b*]phthalazine-5,10-dione derivatives [a]

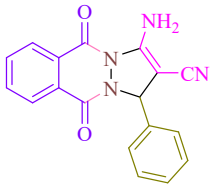
Entry	Catalyst	Conditions	Time/Yield (%)/References
1	$InCl_3$	Water, Reflux	1.5 h/85[18]
2	$NiCl_2.6H_2O$	EtOH, Reflux	3 h/87[19]
3	*p*-TSA	[Bmim]Br, 100°C	3 h/94[22]
4	STA	Solvent-free, 70°C	20 min/94[23]
5	CuI nanoparticles	MeCN, Reflux	27 min/91[24]
6	TBBAD	Solvent-free, 80-100°C	15 min/89[26]
7	Caffeine	Solvent-free, 90°C	3 h/89[This work]

[a] Based on the four-component reaction of benzaldehyde, phthalimide, hydrazine monohydrate and malononitrile.

Table IV — Comparison of ^{1}H NMR data for synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives.

Entry	Product	H Shift (found)	H Shift (lit)	References
1	 **5a**	6.14 (1H, s, $H_{benzylic}$) 7.33-7.48 (5H, m, H_{Ar}) 7.97-8.29 (6H, m, NH_2 and H_{Ar})	6.12 (1H, s, $H_{benzylic}$) 7.29-7.47 (5H, m, H_{Ar}) 7.80-8.3 (6H, m, NH_2 and H_{Ar})	26
2	 **5c**	6.47 (1H, s, $H_{benzylic}$) 7.39-7.65(4H, m, H_{Ar}) 7.91-8.31 (6H, m, NH_2 and H_{Ar})	6.46 (1H, s, $H_{benzylic}$) 7.33-7.62 (4H, m, H_{Ar}) 7.87-8.30 (4H, m, H_{Ar}) 8.15 (2H, s, NH_2)	25
3	 **5j**	2.30 (3H, s, CH_3) 6.08 (1H, s, $H_{benzylic}$) 7.14-7.26 (4H, m, H_{Ar}) 7.97-8.29 (6H, m, NH_2 and ArH).	2.27 (3H, s, CH_3) 6.05 (1H, s, $H_{benzylic}$) 7.12-7.24 (4H, m, H_{Ar}) 7.96-8.26 (6H,m,Ar and NH_2)	24
4	 **5m**	3.66 (3H, s, OCH_3) 3.76 (6H, s, 2×OCH_3) 6.07 (1H, s, $H_{benzylic}$) 6.78 (2H, s, H_{Ar}) 7.89- 8.29 (6H, m, NH_2 and H_{Ar}).	3.64-3.73 (9H, s, OCH_3) 6.05 (1H, s, $H_{benzylic}$) 6.75 (2H, s, ArH) 7.94- 8.26 (6H, m, NH_2 and H_{Ar}).	26
5	 **5o**	6.15 (1H, s, $H_{benzylic}$) 7.43 (2H, d, J = 11.2 Hz, H_{Ar}) 7.54 (2H, d, J = 11.2 Hz, H_{Ar}) 7.88-8.28 (6H, m, NH_2 and H_{Ar})	6.14 (1H, s, $H_{benzylic}$) 7.39-7.52 (4H, m, H_{Ar}) 7.94-8.26 (6H, m, NH_2 and H_{Ar})	24
6	 **5q**	2.30 (3H, s, CH_3) 6.10 (1H, s, $H_{benzylic}$) 7.18 (2H, d, J = 8.0 Hz, H_{Ar}) 7.34 (2H, d, J = 8.0 Hz, H_{Ar}) 7.97-8.28 (6H, m, NH_2 and H_{Ar})	2.28 (3H, s, CH_3) 6.07 (1H, s, $H_{benzylic}$) 7.14-7.33 (4H, m, H_{Ar}) 7.94-8.25 (6H, m, NH_2 and H_{Ar})	24

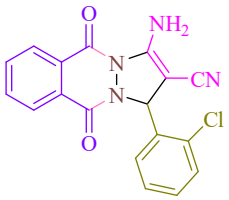
3-Amino-1-(phenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile, 5a



5a

Yield: 89%. m.p.269-270°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ 6.14 (1H, s, $H_{benzylic}$), 7.33-7.48 (5H, m, H_{Ar}), 7.97-8.29 (6H, m, NH_2 and H_{Ar}).

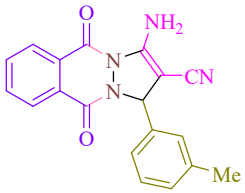
3-Amino-1-(2-chlorophenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile, 5c



5c

Yield: 84%. m.p.254-257°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ 6.47 (1H, s, $H_{benzylic}$), 7.39-7.65 (4H, m, H_{Ar}), 7.91-8.31 (6H, m, NH_2 and H_{Ar}).

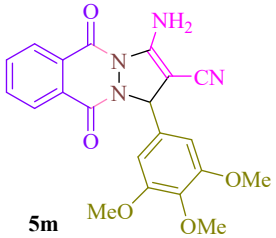
3-Amino-1-(3-methylphenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile, 5j



5j

Yield: 88%. m.p.250-251°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.30 (3H, s, CH_3), 6.08 (1H, s, $H_{benzylic}$), 7.14-7.26 (4H, m, H_{Ar}), 7.97-8.29 (6H, m, NH_2 and H_{Ar}).

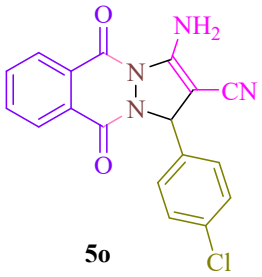
3-Amino-1-(3,4,5-trimethoxyphenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile, 5m



5m

Yield: 85%. m.p. 254-255°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ 3.66 (3H, s, OCH_3), 3.76 (6H, s, 2×OCH_3), 6.07 (1H, s, $H_{benzylic}$), 6.78 (2H, s, H_{Ar}), 7.89-8.29 (6H, m, NH_2 and H_{Ar}); ^{13}C NMR (100 MHz, DMSO-d_6): δ 56.5, 60.3, 61.7, 63.8, 104.6, 116.1, 127.1, 127.7, 129.2, 129.4, 134.1, 134.6, 135.0, 137.7, 151.0, 152.8, 153.9, 157.2; EI-MS: *m/z* (%) 406 (M^+, 22), 389 (25), 366 (9), 275 (9), 239 (100), 162 (12), 145 (9), 130 (20), 104 (51), 76 (46), 43 (28).

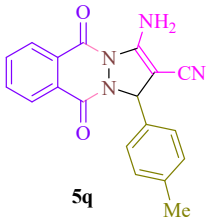
3-Amino-1-(4-chlorophenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile, 5o



5o

Yield: 83%. m.p. 272-274°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ 6.15 (1H, s, $H_{benzylic}$), 7.43 (2H, d, J = 11.2 Hz, H_{Ar}), 7.54 (2H, d, J = 11.2 Hz, H_{Ar}), 7.88-8.28 (6H, m, NH_2 and H_{Ar}).

3-Amino-1-(4-methylphenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-*b*]phthalazine-2-carbonitrile, 5q



5q

Yield: 89%. m.p. 251-253°C. ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.30 (3H, s, CH_3), 6.10 (1H, s, $H_{benzylic}$), 7.18 (2H, d, J = 8.0 Hz, H_{Ar}), 7.34 (2H, d, J = 8.0 Hz, H_{Ar}), 7.97-8.28 (6H, m, NH_2 and H_{Ar}).

Conclusion

In conclusion, we developed a cost effective, green and biodegradable caffeine catalyzed, one-pot, four-component protocol for synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives *via* condensation of phthalimide (1.0 mmol), hydrazine monohydrate (1.0 mmol) and the type of aldehyde derivatives (1.0 mmol), malononitrile (1.0 mmol) under solvent-free conditions. The advantages of this method are high yields, short reaction times, naturally green

and biodegradable catalyst, simple experimental and work-up procedures with no necessity of chromatographic purification steps, mild and environmentally friendly reaction conditions, and economic availability of the catalyst.

Acknowledgements

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