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Copper(II) acetate monohydrate: an efficient and eco-friendly catalyst for the one-pot multi-component synthesis of biologically active spiropyrans and 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives under solvent-free conditions

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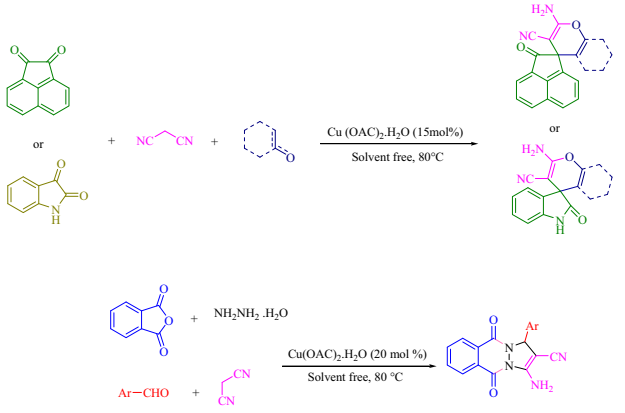
Abstract We have studied the catalytic ability of copper(II) acetate monohydrate as a mild, environmentally benign, natural and economical catalyst for the multi-component efficient synthesis of biologically active spiro-4*H*-pyran derivatives and 1*H*-pyrazolo[1,2-b]phthalazine-5,10-dione derivatives with excellent yields and short reaction times. The most important advantages of this procedure are its mild, non-toxic and inexpensive catalyst, one-pot synthesis, environmentally benign nature, solvent-free conditions, simple operational procedures, and highly efficient conditions.

Graphical Abstract



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Keywords Copper(II) acetate monohydrate · Spiro-4*H*-pyran derivatives · 1*H*-pyrazolo[1,2-b]phthalazine-5,10-dione derivatives · Solvent-free conditions

Introduction

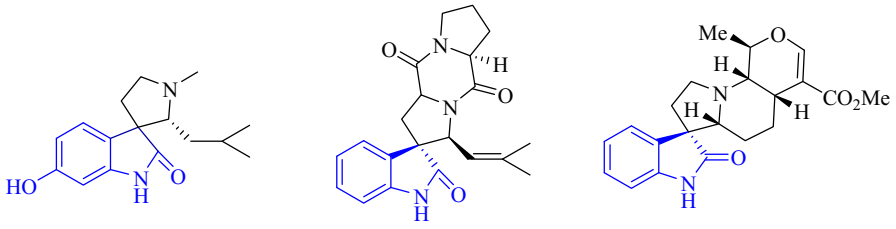
Nowadays, most research has been focused on the study of synthesising heterocyclic compounds. Organic compounds containing nitrogen heterocyclic rings are important compounds in medicinal chemistry. The synthesis of biologically active spiropyran derivatives (spirooxindole) and 1*H*-pyrazolo[1,2-b]phthalazine-5,10-dione derivatives have been given much attention because of their advantages in various biological (Figs. 1, 2) and pharmaceutical fields, for example, as anti-HIV [1], anti-tubercular [2], fungicidal [3], anticonvulsant [4] and anticancer [5, 6] agents; in addition, these spirocycles are MDM2 inhibitors [7] and progesterone receptor modulators [8].

In recent decades, a number of methodologies for preparation of these compounds have been reported that have included various catalysts such as [BMIm] BF_4 [9], urea–choline chloride [10], sulfated choline-based heteropolyanion [11], β-cyclodextrin [12], lipase [13], CsF [14], carbon–SO_3H [15], Et_3N [16], [Bmim]OH [17], ultrasound-assisted [18], $NiCl_2{\cdot}6H_2O$ [19], PTSA/[Bmim]Br [20], CuI nanoparticles [21], $InCl_3$ [22, 23], STA [24], SBA-Pr–SO_3H [25], PTSA [26], $Ce(SO_4)_2{\cdot}4H_2O$ [27] and $Cu(OAc)_2$/sodium L-ascorbate [28]. Some of these methodologies have limitations such as a difficult work-up, toxic and expensive catalysts and solvents, long reaction times and low yields.

Recently, increasing interest has been paid to the multi-component domino reactions (MCRs) [29–40]. Because of their notable advantages such as atom economy, environmental friendliness, low-cost, one-pot nature, simple work-up they have become powerful tools in the synthesis of organic compounds possessing biological and pharmaceutical properties.

Our recent research has focused on the development of clean and simple methodologies with environmentally benign natures. Therefore, given that one of the factors that reduces environmental pollution in synthesis of organics is solvent-free conditions, we have developed multicomponent reactions under solvent-free conditions.

In this regard, we have reported copper(II) acetate monohydrate for the synthesis of spiro-4*H*-pyran derivatives and 1*H*-pyrazolo[1,2-b]phthalazine-5,10-diones via



Elacomine Spirotryprostatin B Pteropodine

Fig. 1 Some alkaloids containing a heterocyclic spirooxindole unit

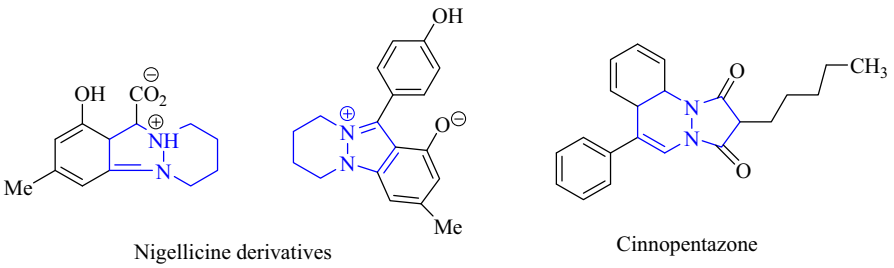


Fig. 2 Biologically active compounds with a two-ring junction nitrogen atom

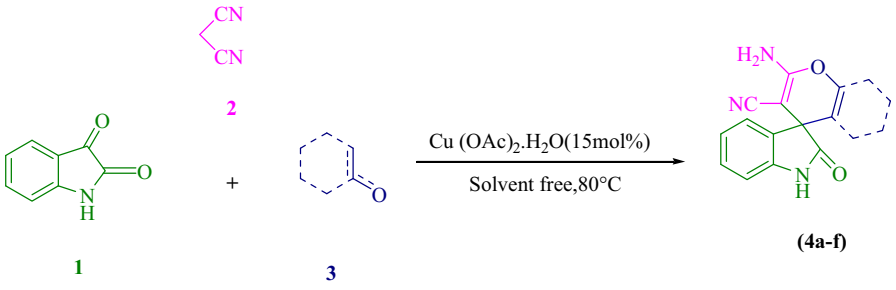
MCRs under thermal and solvent-free conditions. The advantages of copper(II) acetate monohydrate as a catalyst in organic compound synthesis are that it is environmentally benign, economical, mild, inexpensive, non-toxic and highly efficient [41–43].

Results and discussion

In this protocol, we have reported copper(II) acetate monohydrate as an efficient catalyst under thermal and solvent-free conditions for simple, environmentally benign synthesis of biologically active spiro-4*H*-pyran derivatives **4** from reaction between isatin **1**, malononitrile **2** and 1,3-dicarbonyl compounds/4-hydroxy-cumarin/naphthlol **3** (Scheme 1).

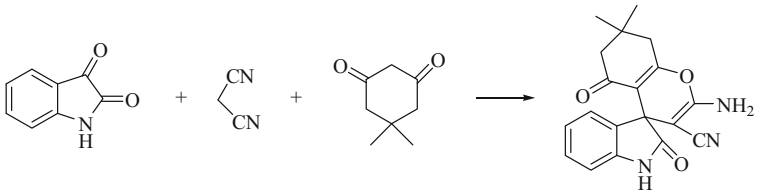
In order to optimize the reaction conditions, the synthesis of compound **4a** (Table 3, entry 1) was used as a model reaction. The effect of different amounts of catalyst on the reaction has been studied in this protocol. No product could be detected in the absence of the catalyst even after 10 h (Table 1, entry 1). Good yields were obtained in the presence of the catalyst. The best amount of catalyst was 15 mol% (0.03 g; Table 1, entry 4). The higher amount of catalyst did not increase the yields products (Table 1, entry 5).

Also, the effect of temperature on the reaction has been investigated. At room temperature, the product was not detected (Table 2, entry 1). The reaction was



Scheme 1 Synthesis of spiro-4*H*-pyran derivatives

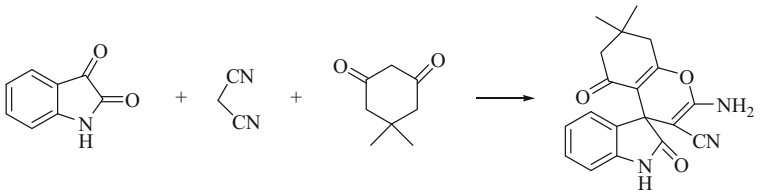
Table 1 Optimization of the reaction condition for the synthesis of spiro-4*H*-pyran **4a**



Entry	$Cu(OAc)_2 \cdot H_2O$ (mol%)	Time (h)	Product	Isolated yields (%)
1	Catalyst free	10	**4a**	–
2	5	7	**4a**	49
3	10	6	**4a**	71
4	15	4	**4a**	86
5	20	4	**4a**	88

Reaction condition: isatin, malononitrile, dimedone and copper(II) acetate monohydrate were heated at 80 °C for the appropriate time

Table 2 Effect of temperature on the synthesis of **4a**



Entry	Temperature (°C)	Time (h)	Isolated yields (%)
1	r.t.	10	–
2	40	6	41
3	60	5	67
4	80	4	86
5	100	4	87

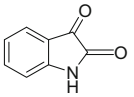
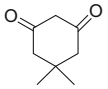
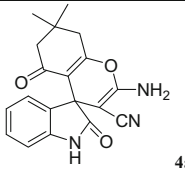
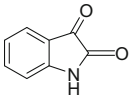
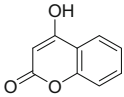
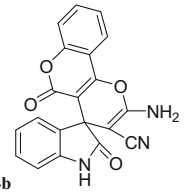
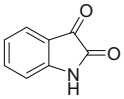
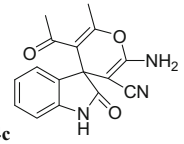
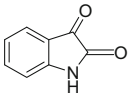
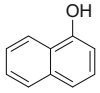
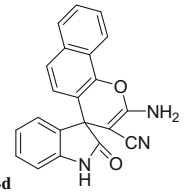
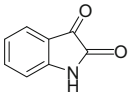
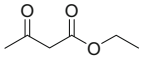
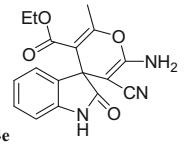
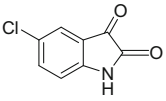
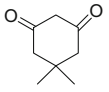
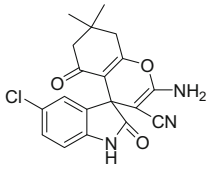
Reaction condition: isatin, malononitrile and dimedone (1:1:1) with copper(II) acetate monohydrate (15 mol%) were heated under various temperatures for the appropriate time and yields

investigated at various temperatures and a high yield of product was obtained at 80 °C (Table 2, entry 4).

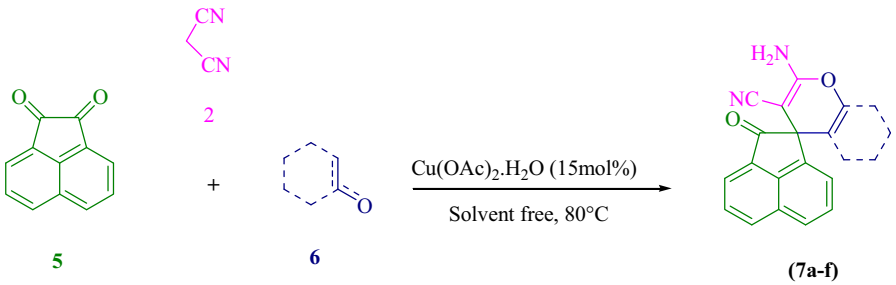
In order to study this procedure, we synthesized a one-pot, three-component condensation reaction of isatin (1.0 mmol), malononitrile (1.0 mmol) and compounds **3** (1.0 mmol) in the presence of copper(II) acetate monohydrate (15 mol%) as a mild catalyst under thermal and solvent-free conditions, and the results are shown in Table 3.

After the successful synthesis of spiro-4*H*-pyran derivatives **4**, we turned our attention to the synthesis of spiro-4*H*-pyran derivatives from reaction between acenaphthoquinone **5**, malononitrile **2** and compounds **6** in the present of copper(II)

Table 3 Copper(II) acetate monohydrate-catalyzed synthesis of spiropyrans

Entry	Isatin	3	Product	Time (h)	Yield %[a]
1			 **4a**	4	86 [9]
2			 **4b**	6	79 [10]
3			 **4c**	5	81 [15]
4			 **4d**	5	83 [10]
5			 **4e**	4.5	89 [9]
6			 **4f**	4	81 [14]

[a] Isolated yield



Scheme 2 Synthesis of spiro-4*H*-pyran derivatives

acetate monohydrate (Scheme 2) and these compounds were synthesized under similar conditions in good yields. The results are shown in Table 4.

Then, we turned our attention toward the synthesis of pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives. In order to discern the optimal conditions, we investigated the four components of phthalic anhydride **8**, hydrazine monohydrate **9**, aromatic aldehydes derivatives **10** and malononitrile **2** in the presence of copper(II) acetate monohydrate as an efficient, environmentally benign and natural catalyst (Scheme 3).

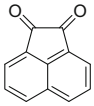
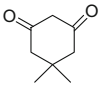
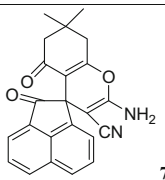
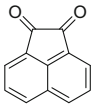
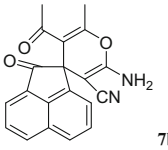
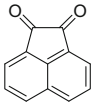
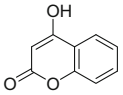
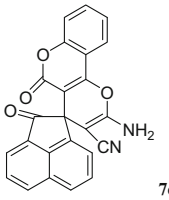
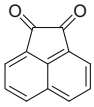
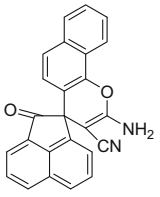
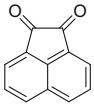
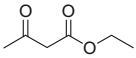
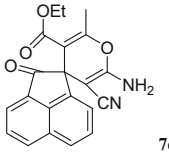
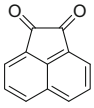
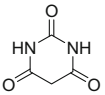
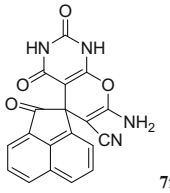
In order to optimize the reaction conditions, the synthesis of compound **11a** (Table 7, entry 1) was used as a model reaction. The effect of different amounts of catalyst on the reaction has been studied in this protocol. No product could be detected in the absence of the catalyst even after 10 h (Table 5, entry 1). Good yields were obtained in the presence of catalyst. The best amount of catalyst was 20 mol% (0.04 g; Table 5, entry 5). The higher amount of catalyst did not increase the yields products (Table 5, entry 6).

Also, the effect of temperature on the reaction has been investigated. At room temperature, the product was not detected (Table 6, entry 1). The reaction was investigated at various temperatures and a high yield of product was obtained at 80 °C (Table 6, entry 4).

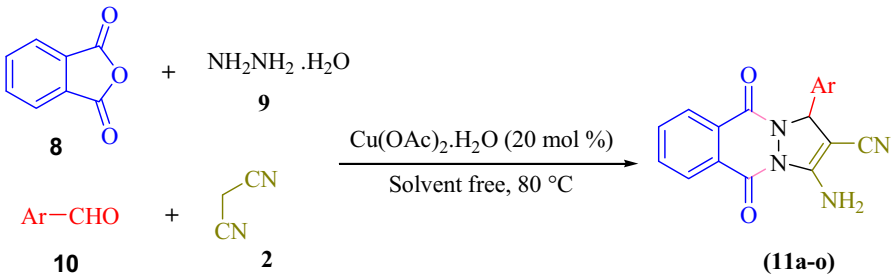
In order to study this procedure, we synthesized a series of compounds with various types of electron-donating and electron-withdrawing aldehyde derivatives such as Cl, Br, NO_2, OH, OMe,….substituted banzaldehydes which gave excellent yields. Also, the generality of this four-component condensation reaction was studied by using copper(II) acetate monohydrate (20 mol%) via phthalic anhydride (1.0 mmol), hydrazine monohydrate (1.0 mmol), the various aldehyde derivatives (1.0 mmol) and malononitrile (1.0 mmol) under thermal and solvent-free conditions, and the results are shown in Table 7.

Comparison of the catalytic ability of some catalysts reported in the literature for the synthesis of spiro-4*H*-pyran and 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives are shown in Tables 8 and 9. This study reveals that copper(II) acetate monohydrate has shown its extraordinary potential to be an alternative cheap, cost-effective, eco-friendly, efficient catalyst for the synthesis of these compounds. In

Table 4 Copper(II) acetate monohydrate-catalyzed synthesis of spiro-4*H*-pyran

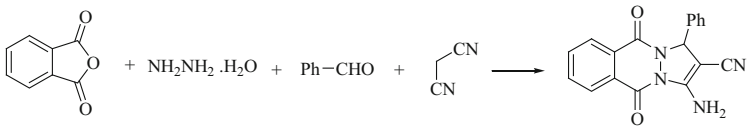
Entry	Acenaphthoquinone	**7**	Product	Time (h)	Yield %[a]
1			 **7a**	5	84 [16]
2			 **7b**	6	81 [16]
3			 **7c**	7	79 [16]
4			 **7d**	4	82 [16]
5			 **7e**	6	87 [16]
6			 **7f**	6	78 [16]

[a] Isolated yield



Scheme 3 Synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-dione derivatives

Table 5 Optimization of the reaction condition for the synthesis of pyrazolo[1,2-b]phthalazine-5,10-dione **11a**



Entry	$Cu(OAc)_2{\cdot}H_2O$ (mol%)	Time (h)	Isolated yields (%)
1	Catalyst-free	10	–
2	5	7	46
3	10	5	58
4	15	4	72
5	20	3	83
6	25	3	84

Reaction conditions: phthalic anhydride, hydrazine monohydrate, aromatic aldehydes derivatives and malononitrile (1:1:1:1) and copper(II) acetate monohydrate were heated at 80 °C for the appropriate time

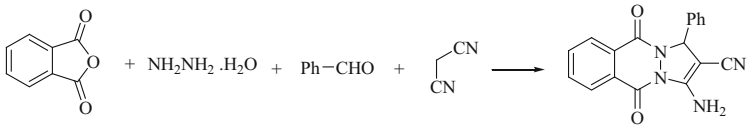
addition, the use of solvent-free conditions with excellent yields and short reaction times are notable advantages of this presented methodology.

Experimental

General

Melting points of all compounds were determined using an Electrothermal 9100 apparatus. Also, proton nuclear magnetic resonance (^{1}H NMR) analyses were recorded on a Bruker DRX-400 Avance instrument with deuterated dimethyl sulfoxide (DMSO-d_6) as a solvent. In this article, all reagents and solvents were purchased from Merck, Fluka and Acros chemical companies and were used without further purification.

Table 6 Effect of the reaction temperature on the synthesis of **11a**



Entry	Temperature (°C)	Time (h)	Isolated yields (%)
1	r.t.	12	–
2	40	8	47
3	60	5	69
4	80	3	83
5	100	3	83

Reaction conditions: phthalic anhydride, hydrazine monohydrate, aromatic benzaldehyde and malononitrile (1:1:1:1) with copper(II) acetate monohydrate (20 mol%) were heated under various temperatures for the appropriate time

Table 7 Copper(II) acetate monohydrate-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	M.p. °C Ref.
1	C_6H_5	**11a**	3	83	269–271	270–272 [21]
2	4-Br–C_6H_4	**11b**	5	76	265–267	265–267 [18]
3	2-Cl–C_6H_4	**11c**	5	81	256–258	257–259 [20]
4	3-Me–C_6H_4	**11d**	3	85	250–252	250–252 [21]
5	3-F–C_6H_4	**11e**	3	89	265–267	264–266 [22]
6	3-O_2N–C_6H_4	**11f**	5	85	271–273	269–271 [20]
7	C_4H_3S	**11g**	5	78	244–246	244–246 [21]
8	3,4,5-$(OMe)_3$–C_6H_2	**11h**	4	82	254–256	253–255 [20]
9	4-F–C_6H_4	**11i**	3	86	265–267	263–265 [18]
10	4-O_2N–C_6H_4	**11j**	4	83	228–230	228–229 [20]
11	3-Br–C_6H_4	**11k**	5	74	268–270	270–272 [18]
12	3-OMe–C_6H_4	**11l**	3	86	247–249	248–251 [18]
13	4-Me–C_6H_4	**11m**	3	87	251–253	253–255 [21]
14	4-OH–C_6H_4	**11n**	5	73	272–274	270–272 [21]
15	3-Cl–C_6H_4	**11o**	4	78	266–268	266–267 [18]

General procedure for preparation of spiro-4*H*-pyran derivatives (4a–f) and (8a–f)

A mixture of isatin/acenaphthoquinone (1.0 mmol), malononitrile (1.0 mmol) and 1,3-dicarbonyl compounds/4-hydroxycumarin/naphthlol **3** (1.0 mmol) in the presence of copper(II) acetate monohydrate as a mild, environmentally benign and natural catalyst under thermal and solvent-free conditions was heated for the

Table 8 Comparison of the catalytic ability of some catalysts reported in the literature for the synthesis of spiro-4*H*-pyran **4a**

Entry	Catalyst	Conditions	Time/yield (%)	References
1	β-Cyclodextrin	H_2O, heating	5 h/90	[12]
2	Lipase	EtOH, heating	3 h/94	[13]
3	Carbon–SO_3H	EtOH, reflux	3 h/81	[15]
4	Et_3N	EtOH, reflux	4 h/80	[16]
5	$InCl_3$	MeCN, reflux	90 min/75	[23]
6	$Cu(OAc)_2{\cdot}H_2O$	Solvent-free, heating	4 h/86	This work

Based on the three-component reaction of isatin, malononitrile and dimedone

Table 9 Comparison of the catalytic ability of some catalysts reported in the literature for the synthesis of 1*H*-pyrazolo[1,2-b]phthalazine-5,10-dione derivatives

Entry	Catalyst	Conditions	Time/yield (%)	References
1	$NiCl_2{\cdot}6H_2O$	EtOH, reflux	3 h/87	[19]
2	CuI nanoparticles	MeCN, reflux	27 min/91	[21]
3	$InCl_3$	Water, reflux	1.5 h/85	[22]
4	STA	Solvent-free, heating	20 min/94	[24]
5	PTSA	[Bmim]Br, heating	3 h/94	[26]
6	$Cu(OAc)_2{\cdot}H_2O$	Solvent-free, heating	3 h/83	This work

Based on the four-component reaction of benzaldehyde, phthalic anhydride, hydrazine monohydrate and malononitrile

appropriate time. After completion of the reaction [as determined by thin layer chromatography (TLC)], the mixture was cooled to room temperature (rt), the solid products were filtered and then were recrystallized from ethanol to give pure compounds (**4a–f**) and (**7a–f**). All products were characterized by comparison of spectroscopic data (^{1}HNMR). Spectra data of selected and known products are represented below:

7-Amino-1,3-dimethyl-5-nitro-2,2,4-trioxo-1,2,3,4-tetra-hydrospiro[indoline-3,5-pyrano[2,3-d]pyrimidine]-6-carbonitrile (**4a)** mp: 288–290 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 1.00 (3H, s, CH_3), 1.03 (3H, s, CH_3), 2.07–2.19 (2H, m, CH_2), 2.50–2.57 (2H, m, CH_2), 6.79 (1H, d, $J = 7.2$ Hz, ArH), 6.89 (1H, t, $J = 7.2$ Hz, ArH), 6.98 (1H, d, $J = 6.8$ Hz, ArH), 7.13 (1H, t, $J = 6.4$ Hz, ArH), 7.22 (2H, s, NH_2), 10.38 (1H, s, NH).

2-Amino-7,7-dimethyl-2,5,6,7,8-tetrahydro-2*H*-spiro[acenaphthylene-1,4-chromene]-3-carbonitrile (**7a**) mp: 270–272 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 1.02 (3H, s, CH_3), 1.04 (3H, s, CH_3), 2.04–2.13 (1H, m, CH_2), 2.50–2.51 (1H, m, CH_2), 2.63 (2H, s, CH_2), 7.32 (2H, s, NH_2), 7.37–7.85 (6H, m, ArH).

General procedure for preparation of pyrazolo[1,2-b]phthalazine-5,10-dione derivatives (11a–o)

A mixture of phthalic anhydride (**8**, 1.0 mmol), hydrazine monohydrate (**9**, 1.0 mmol) and copper(II) acetate monohydrate was heated for 2 h at 80 °C. Then, aromatic aldehyde (**10**, 1.0 mmol) and malononitrile (**12**, 1.0 mmol) were added and the mixture was heated for the appropriate time. After completion of the reaction (as per TLC), the mixture was cooled to rt, the solid products were filtered and then were recrystallized from ethanol to give pure compounds (**11a–o**). All products were characterized by comparison of spectroscopic data (^{1}HNMR). Spectra data of selected and known products are represented below:

3-Amino-1-(2-thenaldehyde)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-b]phthalazine-2-carbonitrile (**11g**) mp: 244–246 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 6.09 (1H, s, CHAr), 6.88–7.30 (4H, m, ArH), 7.96–8.28 (6H, m, NH_2 and ArH).

3-Amino-1-(3,4,5-trimethoxyphenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-b]phthalazine-2-carbonitrile (**11h**) mp: 254–256 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 3.66 (3H, s, OCH_3), 3.76 (6H, s, 2 OCH_3), 6.07 (1H, s, CHAr), 6.78 (2H, s, ArH), 7.89–8.29 (6H, m, NH_2 and ArH).

3-Amino-1-(4-fluorophenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-b]phthalazine-2-carbonitrile (**11i**) mp: 265–267 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 6.17 (1H, s, CHAr), 7.20 (2H, t, $J = 8.8$ Hz, ArH), 7.53–7.57 (2H, m, ArH), 7.96–8.26 (6H, m, NH_2 and ArH).

3-Amino-1-(3-methoxyphenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1,2-b]phthalazine-2-carbonitrile (**11l**) mp: 247–249 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 3.34 (3H, s, OCH_3), 6.09 (1H, s, CHAr), 6.88–7.30 (4H, m, ArH), 7.83–8.26 (6H, m, NH_2 and ArH).

3-Amino-1-(4-methylphenyl)-5,10-dihydro-5,10-dioxo-1*H*-pyrazolo[1, 2-b]phthalazine-2-carbonitrile (**11m**) mp: 251–253 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 2.30 (3H, s, CH_3), 6.10 (1H, s, CHAr), 7.18 (2H, d, $J = 8.0$ Hz, ArH), 7.34 (2H, d, $J = 8.0$ Hz, ArH), 7.97–8.28 (6H, m, NH_2 and ArH).

Conclusion

In summary, we have studied an efficient, environmentally benignm, natural and economical catalyst for the one-pot, multi-component synthesis of spiro-4*H*-pyran derivatives and pyrazolo[1,2-b]phthalazine-5,10-dione derivatives. Copper(II) acetate monohydrate catalyzed synthesis of these bioactive compounds under solvent-free and thermal conditions. The notable advantages of this methodology are its use of a mild, non-toxic and inexpensive catalyst, its excellent yields and short reaction times, its highly efficient yields, environmentally benign nature and one-pot and solvent-free conditions.

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