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Vitamin B_{12} used as effective biocatalyst for the synthesis of pyrano[2,3-*c*]cromenes and pyrano[2,3-*d*]pyrimidines

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ABSTRACT

Synthesis of organic compounds using some natural compounds as catalysts has gained more attention in recent decades. With respect to the importance of these procedures, the synthesis of 3,4-dihydropyrano[2,3-*c*]chromene and pyrano[2,3-*d*]pyrimidine derivatives using vitamin B_{12} by a one-pot reaction of malononitrile, benzaldehydes and 4-hydroxycumarine or 1,3-dimethylbarbituric acid is reported.

***Keywords**: Vitamin B_{12}; Aldehydes; Pyrano[2,3-c]chromene; Pyrano[2,3-d]pyrimidine; Biocatalyst; Green route*

1. Introduction

Nowadays, the investigation on development of new efficient eco-friendly routes for the synthesis of organic compounds with high applications in medicine and material science is a main goal [1-4]. Vitamin B_{12} acts not only as a co-enzyme in a series of biochemical transformations but also is used as catalyst in chemical reactions for some proposes such as hydrogenation, reductive elimination, rearrangement and oxidation [5,6].

Because of the solubility in water, Vitamin B_{12} can be used as eco-friendly catalyst for the synthesis of new organic compounds [7-10]. Heterocyclic scaffolds bearing nitrogen and oxygen atoms such as chromene, pyrimidine, pyran and their other derivatives with fused rings have a key role in pharmaceutical and biochemical sciences [11-18]. There are many different synthetic procedures for generation of chromene, pyrane and their derivatives, but some of them are bearing some difficulties [19, 20]. One of the most interesting procedures to synthesize heterocyclic scaffolds such as chromene and pyrane derivatives is the use of multicomponent reactions (MCRs).

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Compared to other methods, multi-component reactions have advantages such as low solvent utilization, high efficiency, green protocol, short time

one-pot, and so on [21-25]. In continuation of our investigation on the synthesis of heterocyclic systems and development of efficient routs with mild conditions and eco-friendly catalyst [26], we have applied Vitamin B12 as a water soluble catalyst for the one-pot synthesis of heterocyclic scaffolds.

2.Experimental

2.1. Materials and methods

Melting points for synthesized compounds were measured using an Electrothermal 9100 apparatus and the IR spectra of the all compounds were recorded on a FT/IR Perkin Elmer Spectrum Two spectrometer. Further consideration on the structure of achieved molecules was conducted with ^{1}H and $^{13}CNMR$ spectroscopy. The spectra were obtained with a BRUKER DRX-300 AVANCE instruments using DMSO-d_6 as a solvent and TMS as an internal standard at 300 and 75.6 MHz, respectively. Elemental analyses for C, H and N were performed using a Heraeus CHN-O-Rapid analyzer. In addition, the mass spectra were recorded on an Agilent Technologies (HP) MS Model: 5975C VL MSD mass spectrometer operating at an

ionization potential of 70 eV. All the chemicals were purchased from Fluka, Merck and Sigma-Aldrich companies and were used without any further purification.

2.2.General Procedure for the Synthesis of 2-amino-4-(3-bromophenyl)-5-oxo-4H,5H-pyrano[3,2-c]chromene-3-carbonitriles (Exemplified by 4g)

To a magnetically stirred solution of 3-bromobenzaldehyde (1 mmol) and malononitrile (1 mmol) in the presence of concentrated vitamin B_{12} (7.35×10^{-5} mol%) as a catalyst in EtOH/H_2O (1:1) mixture (5 mL), a mixture of 4-hydroxycumarine (1 mmol) in EtOH/H_2O (1:1) mixture (0.5 mL) was added dropwise and kept in 5 min at room temperature. After the addition of 4-hydroxycumarine, the reaction mixture was heated to 50 ˚C. The progress of the reaction was monitored by TLC and after completion of the reaction, the mixture was cooled to room temperature and the resulting precipitate was filtered and washed with (3×2 mL) of ethanol. The crude product has been recrystallized from ethanol (5 mL) to afford the pure compound **4g**.

2-Amino-4,5-dihydro-4-(3-bromophenyl)-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4g)

Pale white solid; M.p.: 252-254 °C; IR (KBr) υ: 3400 and 3327 (NH_2), 2202 (CN), 1707, 1672 (CO) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ: 4.51 (s, 1H, CH), 7.30-7.92 (m, 8H, Ar and NH_2). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 37.1, 57.8, 103.7, 113.5, 117.1, 119.6, 122.2, 123.1, 125.2, 127.4, 130.6, 131.0, 131.2, 133.5, 145.5, 152.7, 154.2, 158.4, 160.1. MS (*m*/*z*, %): 395 (M^++2, 62), 350.2 (M^+, 65), 316. (22), 283 (18), 249 (38), 239 (100), 118 (33), 92 (15), 66 (7). Anal. Calcd for $C_{19}H_{11}BrN_2O_3$ (395.21) C, 57.74; H, 2.81; N, 7.09. Found: C, 57.92; H, 2.89; N, 7.16%.

2-Amino-4,5-dihydro-4-(3-fluorophenyl)-5-oxopyrano[3,2-c]chromene-3-carbonitrile (4m)

Pale white solid; M.p.: 236-238 °C; IR (KBr) υ: 3393 and 3325 (NH_2), 2199 (CN), 1703, 1675 (CO) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ: 4.52 (s, 1H, CH), 7.06-7.92 (m, 10H, Ar and NH_2). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 37.1, 57.9, 103.7, 113.5, 114.3, 114.6, 114.9, 115.2, 117.1, 119.6, 123.0, 124.3, 125.1, 130.8, 130.9, 133.5, 146.7, 146.8, 152.8, 154.2, 158.4, 160.1, 161.1, 164.3. MS (*m*/*z*, %): 334 (M^+, 5), 318 (9), 290 (24), 225 (39), 183 (19), 238 (100), 158 (9) 66 (11). Anal. Calcd for $C_{19}H_{11}FN_2O_3$ (334.31) C, 68.26; H, 3.32; N, 8.38. Found: C, 68.35; H, 3.27; N, 8.43%.

2.3.General Procedure for the Synthesis of 7-amino-1,3-dimethyl-5-(3-nitrophenyl)-2,4-dioxo-1,3,4,5-tetrahydro-2H-pyrano[2,3-d]pyrimidine-6-carbonitriles (Exemplified by 6g)

To a magnetically stirred solution of 3-nitrobenzaldehyde (1 mmol) and malononitrile (1 mmol) in the presence of concentrated vitamin B_{12} (7.35×10^{-5} mol%) as a catalyst in EtOH/H_2O (1:1) mixture (5 mL), a mixture of 1,3-dimethylbarbituric acid (1 mmol) in EtOH/H_2O (1:1) mixture (0.5 mL) was added dropwise and kept in 5 min at room temperature. After the addition of 1,3-dimethylbarbituric acid, the reaction mixture was heated to 60 ˚C. The progress of the reaction was monitored by TLC and after completion of the reaction, the mixture was cooled to room temperature and the resulting precipitate was filtered and washed with (3×2 mL) of ethanol. The crude product has been recrystallized from ethanol (5 mL) to afford the pure compound **6g**.

7-Amino-1,3-dimethyl-5-(3-nitrophenyl)-2,4-dioxo-2,3,4,5-tetra-hydro-1H-pyrano[2,3-d]pyrimidine-6-carbonitrile (6g)

Light yellow solid; M.p.: 204-207 °C; IR (KBr) υ: 3364 and 3337, (NH_2), 2201 (CN), 1726, 1698 and 1683 (CO) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ: 3.09 and 3.39 (6H, 2s, 2 NCH_3), 4.60 (1H, s, CH), 7.51 (2H, brs, NH_2), 7.60 (1H, t, Ar), 7.78 (1H, d, Ar), 8.08-8.13 (2H, m, Ar).

3. Results and Discussion

It has been found that the reaction of benzaldehyde **1**, malononitrile **2** and 4-hydroxycumarine **3** in the presence of catalytic amounts of vitamin B_{12} in the mixture of EtOH/H_2O (1:1) leads to 2-amino-4-phenyl-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitrile **4** in good yields (**Scheme 1**).

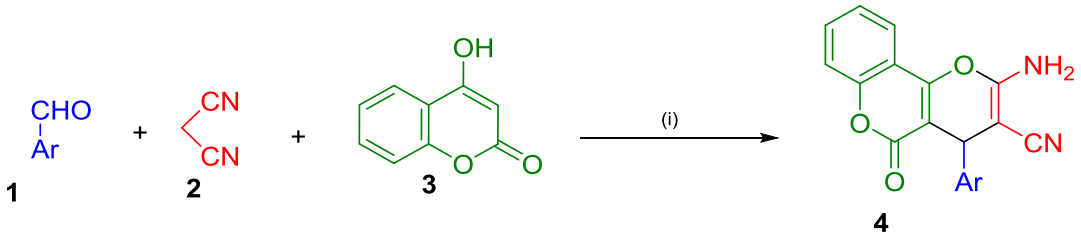
Synthesis of pyrano[3,2-*c*]chromenes has been reported in many reports under different conditions, but in the current work, vitamin B_{12} has been used as a natural biocatalyst for synthesis of pyrano[3,2-*c*]chromene derivatives.

The reaction was performed with no catalyst and the reaction did not show acceptable results. In the presence of vitamin B_{12}, the reaction of benzaldehyde **1a**, malononitrile **2** and 4-hydroxycumarine **3** produced pyrano[3,2-*c*]chromene **4a** in short reaction time with high yield.

At the first step, the reaction was carried out in H_2O, EtOH and mixture of EtOH/H_2O with various proportions to realize the most appropriate solvent for

this reaction. Among the mentioned solvents, a mixture of EtOH/H_2O with (1:1) proportion was selected as the best solvent through its high efficiency. The results are shown in **Table 1**.

After solvent optimization, to find the best reaction conditions and to evaluate the catalytic efficiency of vitamin B_{12}, a model study was conducted for the synthesis of 2-amino-4-phenyl-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitrile **4a** (**Table 2**). Under the optimized reaction conditions, a number of 2-amino-4-phenyl-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitriles, shown in **Table 3**, were synthesized.



Ar: C_6H_5, 4-ClC_6H_4, 4-FC_6H_4, 4-BrC_6H_4, 4-MeC_6H_4, 4-CNC_6H_4, 4-$OMeC_6H_4$, 3,4-$(Me)_2C_6H_3$, 2-$NO_2C_6H_4$, 3-OHC_6H_4, 3-ClC_6H_4, 3-FC_6H_4, 3-BrC_6H_4, 3-$NO_2C_6H_4$, 2-ClC_6H_4, 2,6-$(Cl)_2C_6H_3$

(i) Reaction Conditions: benzaldehyde (1, 1 mmol), malononitrile (2, 1 mmol), 4-hydroxycumarine (3, 1mmol), vitamin B12 (7.35×10-5 mol%) as catalyst in EtOH/H2O (1:1) mixture (5 mL) at 50 °C.

Scheme 1. Synthesis of 2-amino-4-(aryl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitrile derivatives.

Table 1. Optimization of solvent in synthesis of 4a

Entry	Solvent	Temperature (°C)	Time (min)	Yield (%)[a]
1	H_2O	50	60	71
2	EtOH/H_2O (1:1)[b]	50	20	90
3	EtOH/H_2O (1:2)	50	40	88
4	EtOH/H_2O (3:1)	50	45	85
5	EtOH/H_2O (1:3)	50	45	86
6	EtOH	50	60	82

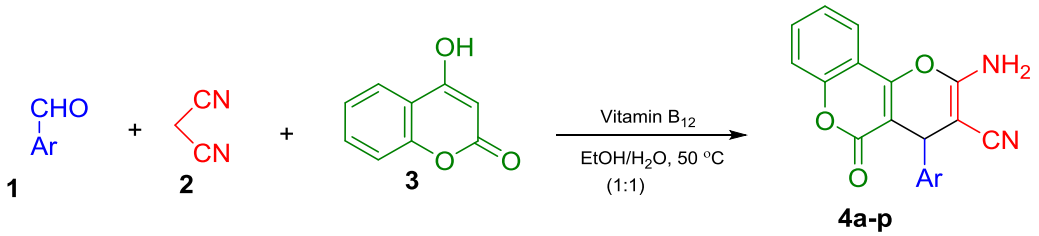
[a] Isolated yield; [b] Selected solvent with high performance in the reaction.

Table 2. Optimization of catalyst loading in synthesis of 4a

Entry	Solvent	Catalyst (mol%)	Temperature (°C)[a]	Time (min)	Yield (%)[b]
1	EtOH/H_2O (1:1)	4.90×10^{-5}	50	60	86
2	EtOH/H_2O (1:1)	7.35×10^{-5}	50	40	90
3	EtOH/H_2O (1:1)	9.80×10^{-5}	50	45	84
4	EtOH/H_2O (1:1)	12.25×10^{-5}	50	50	84

[a] Selected temperature; [b] Isolated yield.

Table 3. Synthesis of 2-amino-4-(aryl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitriles with optimized conditions for selected aldehydes in the presence of vitamin B_{12} at 50 °C.



Entry	Ar	Time (min)	Yield (%)[a]	M.p. (°C)	M.p. [Ref.][b]
4a	C_6H_5	20	90	254-257	256-258 [27]
4b	4-MeC_6H_4	30	87	257-259	258-260 [28]
4c	3,4-$(Me)_2C_6H_4$	35	84	233-235	231-232 [29]
4d	3-HOC_6H_4	15	93	265-268	268-270 [30]
4e	4-$OMeC_6H_4$	40	90	244-247	247-249 [30]
4f	4-BrC_6H_4	20	82	252-255	254-256 [31]
4g	3-BrC_6H_4	12	86	252-254[c]	--------
4h	4-ClC_6H_3	25	87	262-264	263-265 [32]
4i	2-ClC_6H_4	25	97	263-265	266-268 [33]
4j	3-ClC_6H_4	15	81	240-243	240-242 [31]
4k	2,6-$(Cl)_2C_6H_4$	30	88	297-299	300-301 [34]
4l	4-FC_6H_4	35	82	261-264	262-263 [31]
4m	3-FC_6H_4	20	79	236-238[c]	--------
4n	4-CNC_6H_3	10	91	279-282	281-283 [30]
4o	3-$NO_2C_6H_4$	25	98	255-257	257-258 [35]
4p	2-$NO_2C_6H_4$	10	86	254-256	257-259 [30]

[a] Isolated yield; [b] Reported melting points; [c] New compound.

Because of the nature of vitamin B_{12} structure shown in **Fig. 1**, this molecule is able to give hydrogen bonding with some functional groups of other molecules to have a catalyst role.

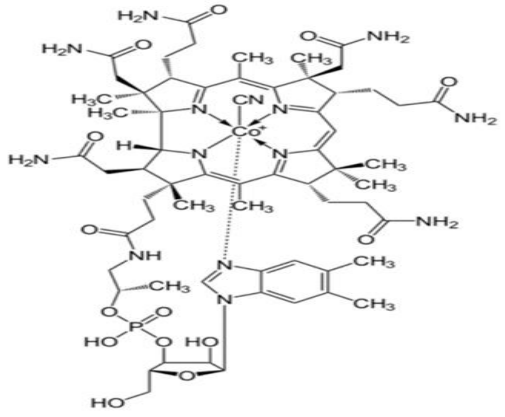


Fig. 1. Chemical structure of vitamin B_{12}

An illustrative mechanism for the synthesis of 2-amino-4-(3-nitrophenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitriles **4** is shown in **Scheme 2**. At first step in mechanism, the activation of arylaldehyde toward the synthesis of dicyanoalkene **A** as a conjugated electron deficient system can be completed which can be attacked by nucleophilic systems such as enol system (**3**) to obtain compound **B**. Then, the intramolecular attack of oxygen atom on active nitrile group leading to pyran ring synthesis can be converted to final compound **4** by a 1,3-proton shift.

The **Scheme 2** indicates a proposed mechanism for the synthesis of compounds **4** and **6** concomitantly. All synthesized compounds have stable structures which have been proved by spectroscopic data.

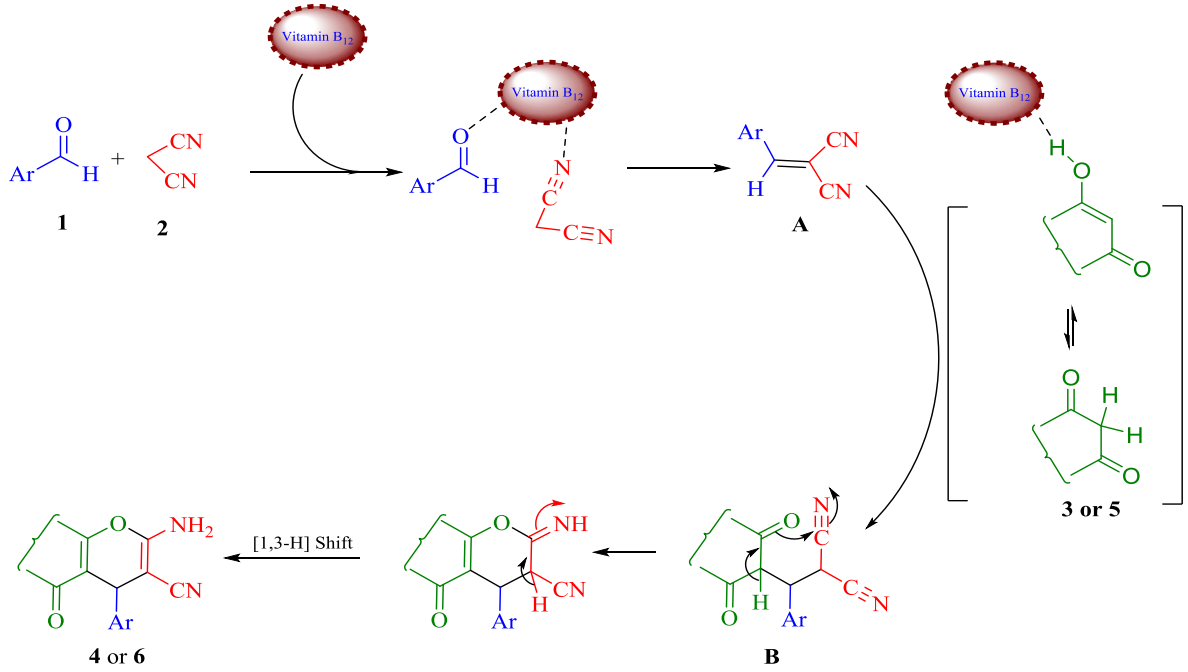
To confirm the catalysis effect on the synthesis of heterocyclic systems, another reaction was performed in according to undergoes reaction. The reaction of benzaldehyde **1**, malononitrile **2** and 1,3-dimethylbarbituric acid **3** in the presence of vitamin B_{12} as a catalyst led to 7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine-6-carbonitrile **6** in high yields (**Scheme 3**).

For the selection of best solvent, the procedure was the same as the synthesis of compound **4** and a mixture of EtOH/H_2O with (1:1) proportion has been selected as a solvent for synthesis of 7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine -6-carbonitriles **6** (see **Table 4**).

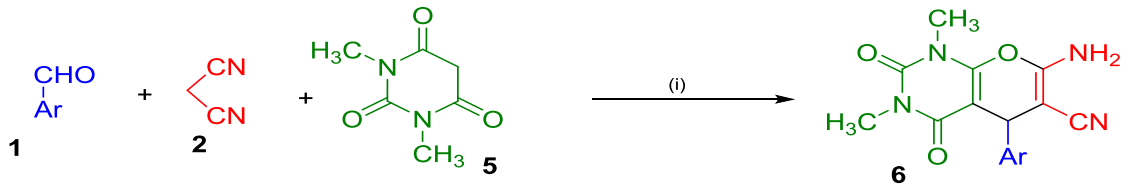
After the solvent optimization, to find the best reaction conditions and to evaluate the catalytic efficiency of vitamin B_{12}, a model study was conducted for the synthesis of 7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine -6-carbonitriles **6** (**Table 5**). As shown in **Table 5**, unlike reaction for the synthesis of compound **4,** the reaction performance is better at 60 °C for the synthesis of compound **6**.

Reaction with some aldehydes bearing electron donating and withdrawing groups for the synthesis of 7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine-6-carbonitriles **6** was carried out and the results were summarized in **Table 6**.

Some methods for the synthesis of compounds **4** and **6** have been shown in the **Table 7**. As shown in the **Table 7**, the current work has many advantages in comparison to reported procedures such as non-toxic, short reaction time, and easy workup.



Scheme 2. Proposed mechanism for Synthesis of 2-amino-4-(aryl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitriles **4** or 7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine-6-carbonitriles **6**.



Ar: C_6H_5, 4-ClC_6H_4, 4-FC_6H_4, 4-BrC_6H_4, 4-CNC_6H_4, 4-$OMeC_6H_4$, 2-$NO_2C_6H_4$,3-$NO_2C_6H_4$,

(i) Reaction Conditions: benzaldehyde (1, 1 mmol), malononitrile (2, 1 mmol), 1,3-dimethybarbituric acid (5, 1mmol), vitamin B12 (7.35×10-5 mol%) as catalyst in EtOH/H2O (1:1) mixture (5 mL) at 60 °C.

Scheme 3. Synthesis of 7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine-6-carbonitriles.

Table 4. Optimization of solvent in synthesis of 6a

Entry	Solvent	Time (min)	Yield (%)[a]
1	H_2O	60	79
2	EtOH/H_2O (1:1)[b]	15	96
3	EtOH/H_2O (1:2)	15	94
4	EtOH/H_2O (3:1)	30	90
5	EtOH/H_2O (1:3)	30	94
6	EtOH	60	88

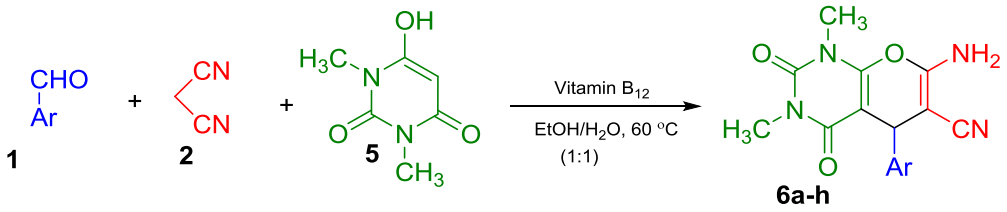
[a] Isolated yield; [b] Selected solvent with high performance in the reaction.

Table 5. Optimization of catalyst loading in synthesis of 6a

Entry	Solvent	Catalyst (mol%)	Temperature (°C)[a]	Time (min)	Yield (%)[b]
1	EtOH/H_2O (1:1)	4.90×10^{-5}	60	45	94
2	EtOH/H_2O (1:1)	7.35×10^{-5}	60	30	96
3	EtOH/H_2O (1:1)	9.80×10^{-5}	60	35	87
4	EtOH/H_2O (1:1)	12.25×10^{-5}	60	35	79

[a] Selected temperature; [b] Isolated yield.

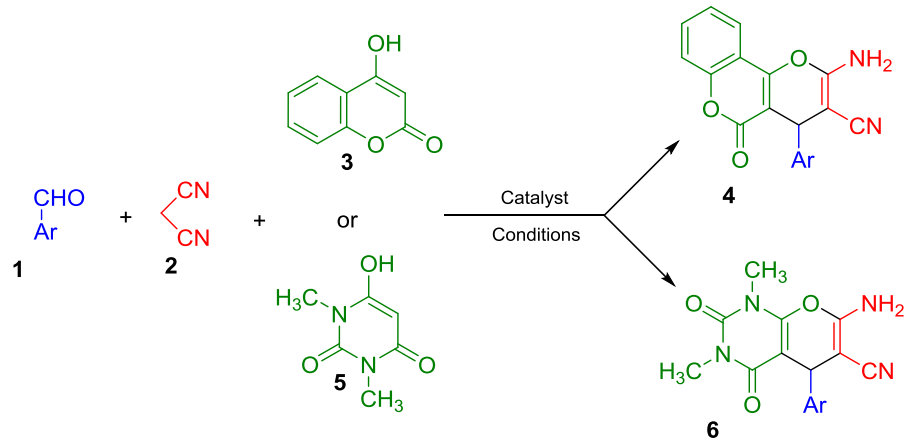
Table 6. Synthesis of 7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine -6-carbonitriles with optimized conditions for selected aldehydes in the presence of vitamin B_{12} at 60 °C.



Entry	Ar	Time (min)	Yield (%)[a]	M.p. (°C)	M.p. [Ref.][b]
6a	C_6H_5	20	90	220-223	219-222 [36]
6b	4-OMeC_6H_4	30	96	223-225	225-227 [37]
6c	4-BrC_6H_4	15	94	234-236	235-237 [38]
6d	4-ClC_6H_4	20	83	236-238	234-237 [39]
6e	4-FC_6H_4	25	79	227-230	230-232 [40]
6f	4-CNC_6H_4	10	93	203-205	202-204 [3]
6g	3-$NO_2C_6H_4$	20	87	204-207	204 [41]
6h	2-$NO_2C_6H_4$	10	97	207-209	208-209 [42]

[a] Isolated yield; [b] Reported melting points.

Table 7. Synthesis of 2-amino-4-(aryl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitriles and 7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine -6-carbonitriles in reported various procedures.



Entry[a]	Catalyst	Conditions	Time (min)	Yield (%)	[Ref.]
1	Method A: diammonium hydrogen phosphate Method B: (*S*)-proline	Method A: H_2O, EtOH (1:1), r.t Method B: H_2O, EtOH (1:1), reflux	120	Method A: 81-95 Method B: 72-88	[27]
2	Nano aluminum hydroxide	EtOH, 78 °C	120	48	[43]
3	4-(dimethylamino)pyridine	EtOH, reflux	90-300	64-94	[28]
4	KF-Montmorillonite	DMF, 90 °C	360-660	80-94	[29]
5	Fe_3O_4@SiO_2-imid-PMA	H_2O, reflux	6-12	88-97	[30]
6	Vitamin B_{12}	EtOH,H_2O (1:1), 50 °C	10-40	79-98	Current work
7	SBA-Pr-SO_3H	Solventless, 140 °C	5-45	30-90	[36]
8	*L*-Proline	H_2O:EtOH, r.t	30-90	68-86	[44]
10	Fe_3O_4@SiO_2-Propyl-Pip-SO_3H.HSO_4	H_2O, 60 °C	10-35	92-98	[45]
11	Vitamin B_{12}	EtOH,H_2O (1:1), 60 °C	10-30	83-97	Current work

[a] Entry 1-6 refers to the synthesis of 2-amino-4-(3-nitrophenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitriles and entry 7-11 refers to the synthesis of7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine -6-carbonitriles.

4. Conclusions

In conclusion, we demonstrate the use of vitamin B_{12} as an eco-friendly nontoxic catalyst by a three-component condensation reaction for the synthesis of 2-amino-4-(aryl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitriles **4** and 7-amino-1,3-dimethyl-5-(aryl)-2,4-dioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine-6-carbonitriles.

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