

# Original Article: Meglumine Catalysed Green Synthesis of Ethyl-6-amino-5-cyano-2-methyl-4-phenyl-4H-pyran-3-carboxylate Derivatives



Ganesh Baburao Punda<sup>a</sup> Sambhaji Tukaram Dhumal<sup>b</sup> | Madhav Janardan Hebade<sup>c</sup> | Mazahar Farooqui<sup>d</sup> | Bhagwansing Shivsing Dobhal<sup>c,\*</sup>

<sup>a</sup>Department of Chemistry, Dr. Rafiq Zakariya College for Women, Aurangabad (MS) India- 431001

<sup>b</sup>Department of Chemistry, Ramkrishna Paramhansa Mahavidyalaya, Osmanabad (MS) India- 431501

<sup>c</sup>Department of Chemistry, Badrinarayan Barwale Mahavidyalaya, Jalna (MS) India- 431213

<sup>d</sup>Department of Chemistry, Maulana Azad College of Arts Science and Commerce, Aurangabad, (MS) India- 431001

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## ABSTRACT

An efficient and simple one-pot synthetic protocol has been developed for the first time to synthesize the series of Ethyl-6-amino-5-cyano-2-methyl-4-phenyl-4H-pyran-3-carboxylates. This was achieved by the cyclocondensation of aromatic aldehydes, malononitrile, and ethylacetoacetate in the presence of the catalytic amount of Meglumine as a readily available, reusable, and biodegradable catalyst. This technique is very promising as it provides mild reaction conditions, an environmentally benign greener approach, easy workup process, high yield, less reaction time, low cost, and recycled up to five catalytic cycles without substantial loss of catalytic activity or product yield.

## Introduction

One of the most important challenges in organic synthesis is to design easy synthetic pathways for commonly used organic molecules using easily available reagents. Single-step, one-pot, and three-component condensation reactions are now widely used for the synthesis of various heterocyclic compounds in synthetic organic

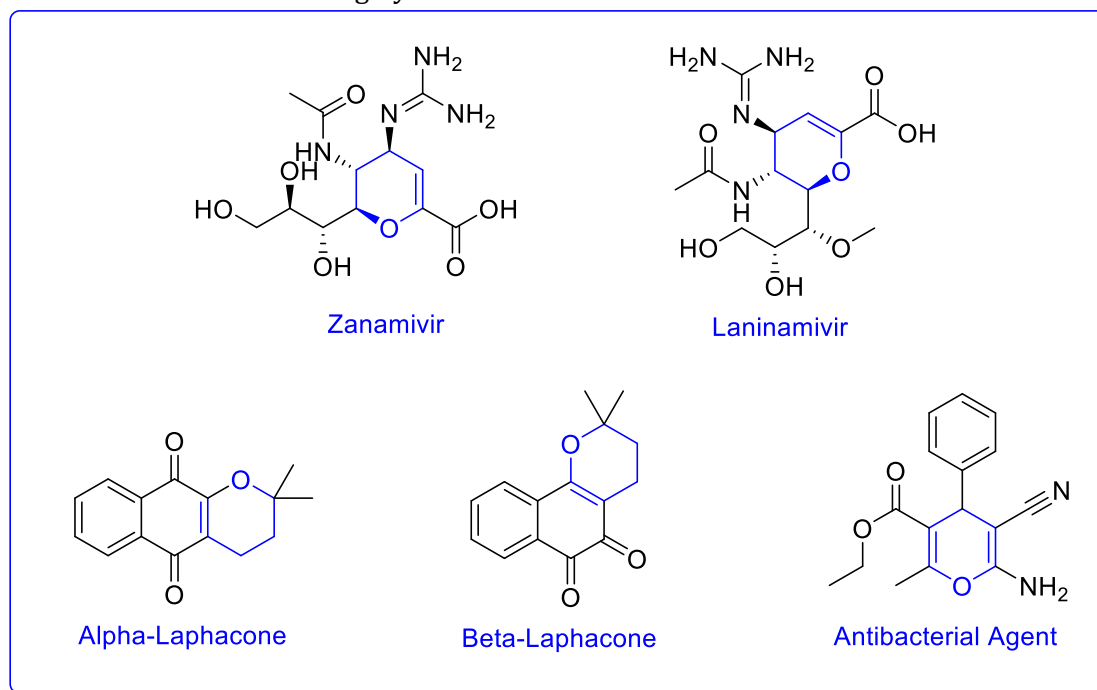
chemistry. As compared to multistep techniques, one-pot protocols are more efficient as it offers higher yield in shorter reaction time with easy workup processes.

4H-pyranare oxygen-containing heterocyclic moiety, with several biological properties including antibacterial,[1],[2] anticancer,[3] antiviral,[4] antitumor,[5] antifungal, antioxidant[6], [7], and antimicrobial

\*Corresponding Author: Bhagwansing Shivsing Dobhal ([drbsdobhal@gmail.com](mailto:drbsdobhal@gmail.com))

activities[8] as well as shows anti-corrosion properties[9]. As illustrated in **Figure 1**, the pyran scaffold acts as a significant structural motif in various bioactive compounds such as beta-lapachone, alpha-lapachone, zanamivir, laninamivir. Correspondingly, derivatives of 4*H*-pyrans have been found as highly bioactive

compounds because of their biodegradable abilities. They are also found in cosmetics, dyes, pigments, and agrochemicals[10]. Consequently, the organic community has been encouraged to discover an excellent method for the synthesis of 4*H*-pyran.



**Figure 1.** Some pharmacologically active derivatives of pyran

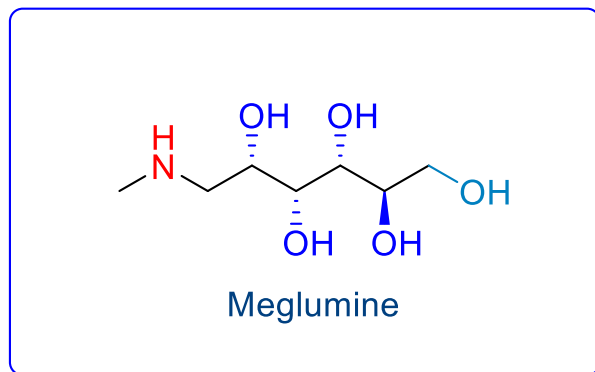
A review of the literature indicates that 4*H*-pyran could be synthesized by several different protocols, which include the two-step approach as well as the one-pot three-component system. Mostly, 4*H*-pyrans are synthesized by a one-pot cyclocondensation reaction between an aromatic aldehyde, malononitrile, and ethylacetoacetate in the presence of a basic catalyst. This synthetic approach was involved in the presence of various catalysts such as BaFe<sub>12</sub>O<sub>19</sub>@IM,[11] PPh<sub>3</sub>, [12] Potassium fluoride, [13] Mg/La mixed oxide, [14] tetramethylguanidine-[bmim][BF<sub>4</sub>], [15] potassium phthalimide, [16] molecular sieve-supported zinc, [17] BNFe<sub>3</sub>O<sub>4</sub>, [18] Silica Supported V<sub>2</sub>O<sub>5</sub>, [19] thiourea dioxide [20] and Baker's yeast. [21] However, these above-mentioned protocols have one or more disadvantages, and the majority of the required heating conditions and provide moderate yields

even after a considerable reaction time. This clearly indicates that there is still scope for improvement in terms of developing an effective and environmentally friendly protocol for the synthesis of 4*H*-pyrans.

Hence, we have planned to synthesize Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate by using Meglumine as a catalyst. The presence of hydroxyl groups and an amino group from the Meglumine tends to activate the nucleophilic as well as electrophilic centers, which helps in hydrogen bonding and electron donation. Meglumine shows fantastic properties such as low toxicity, biodegradability, physiological inertness, reusability, low cost, and non-corrosion nature. Because of the above findings and our ongoing efforts [22-25] to develop environment-friendly synthetic methods for various reactions, herein, we present Meglumine as a biodegradable catalyst

for one-pot synthesis of ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate by reacting aromatic aldehydes, malononitrile, and

ethylacetoacetate in Ethanol:Water at room temperature.

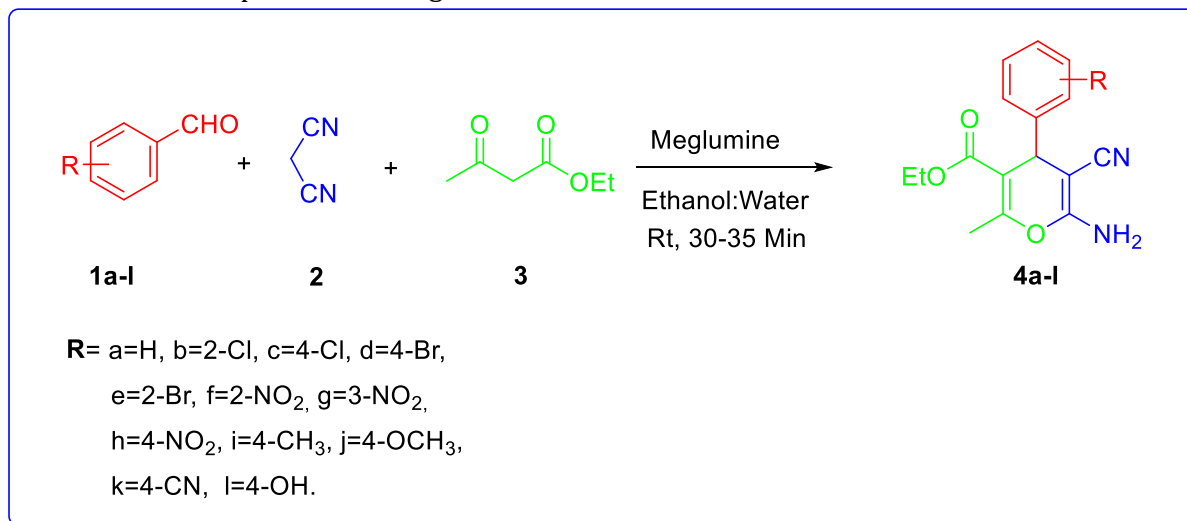


**Figure 2.** Structure of Meglumine Catalyst

### Results and Discussion

The synthesis of Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate derivatives was achieved by reacting substituted aromatic benzaldehydes (**1a-l**) with malononitrile (**2**) and ethylacetoacetate (**3**) in ethanol:water in the presence of Meglumine as a

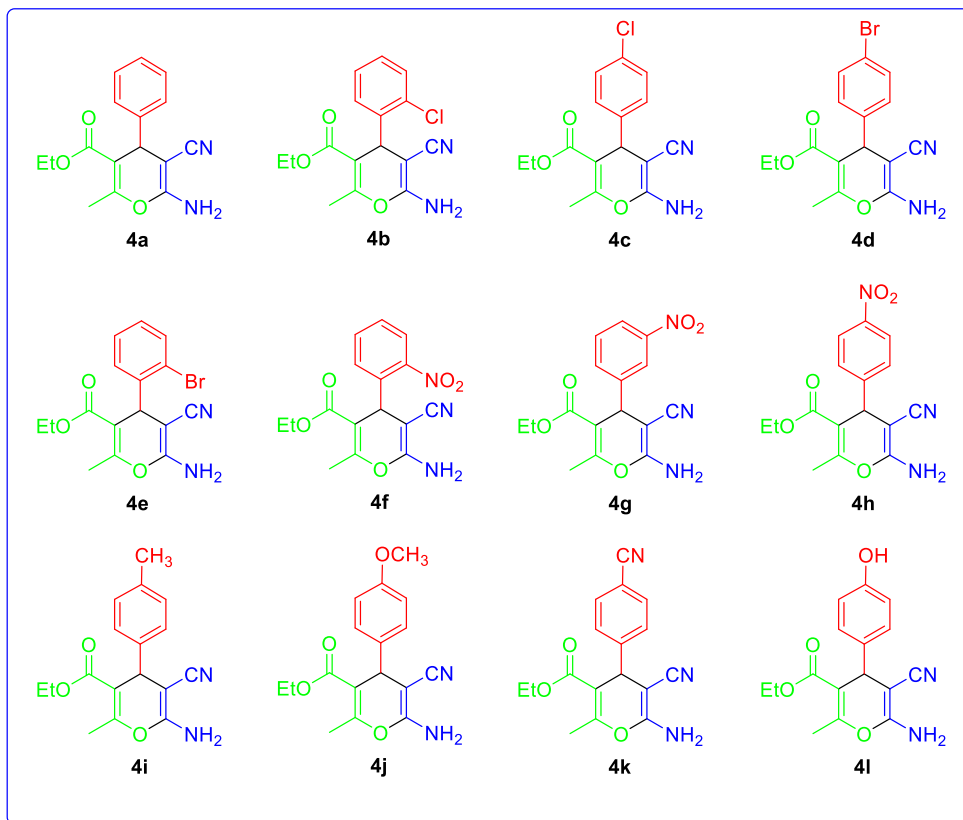
catalyst. The product was obtained after stirring the reaction mixture for 30-35 minutes at room temperature. Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate derivatives (**4a-l**) were obtained with high purity and better to excellent yields, as shown in **Scheme 1**.



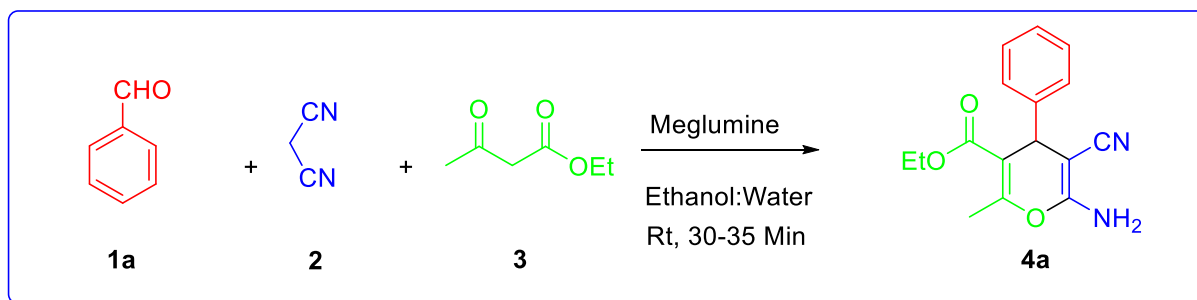
**Scheme 1.** Synthesis of 4*H*-pyran using Meglumine catalyst

The structures of all the newly synthesized Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4*H*-pyran-3-carboxylates (**4a-l**) are depicted in **Figure 3**.

Optimization of the reaction parameters was performed by model reaction of benzaldehyde (**1a**), malononitrile (**2**), and ethylacetoacetate (**3**) as shown below.



**Figure 3.** Structures of the synthesized Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4*H*-pyran-3-carboxylate derivatives (**4a-l**)



**Scheme 2.** Model reaction for Synthesis of 4*H*-pyran

Firstly, we have considered solvent parameters and observed reactions in different solvents like water, methanol, PEG-400, and ethanol as protic solvents as well as DCM, DMF, and acetonitrile as aprotic solvents. We observed that the solvent has an important role in the progress of the reaction. The reaction with ethanol:water gave the corresponding product

in good yields, where as the findings with other solvents such as DCM, DMF, and acetonitrile, yielded the product **4a** in fewer quantities, neither PEG-400 nor water were particularly given good results. Further, it has been decided the impressive and ideal solvent for this conversion was aqueous ethanol (water:ethanol, 9:1, v/v).

**Table 1.** Screening of reaction conditions with respect to solvent and catalyst loading **4a**<sup>a</sup>

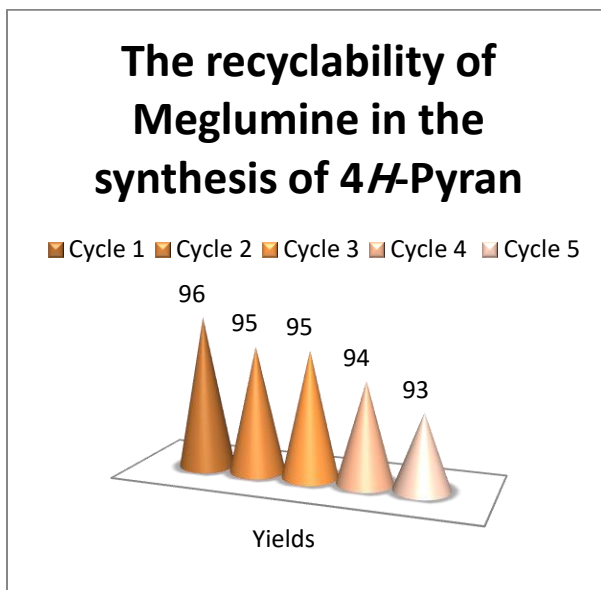
Entry	Solvent	Catalyst(mol%)	Yield <sup>b</sup> (%)
1	Water	10% Meglumine	N R
2	Methanol	10% Meglumine	74
3	Ethanol	10% Meglumine	79
4	PEG-400	10% Meglumine	58
5	DCM	10% Meglumine	26
6	DMF	10% Meglumine	29
7	Acetonitrile	10% Meglumine	31
8	Ethanol:Water(1:1)	10% Meglumine	79
9	Ethanol:Water(9:1)	5% Meglumine	90
10	Ethanol:Water(9:1)	10% Meglumine	96
11	Ethanol:Water(9:1)	15% Meglumine	93
12	Ethanol:Water(9:1)	No catalyst	09

<sup>a</sup>**Reaction conditions:** Benzaldehyde (1mmol), Malononitrile (1mmol), Ethylacetoacetate(1mmol), 10mol% Meglumine in 5 mL ethanol:water, at room temperature for 30-35 min. <sup>b</sup>Isolated yields, NR: No Reaction

However, in the model reaction, the influence of catalyst loading was also investigated. The results revealed that a catalyst concentration of 10mol% was a great choice for this process. Increasing the catalyst concentration 10 to 15mol% resulted in a low effect on yield and not be further increased. When the reaction was conducted with reducing amounts of catalyst, the yield of **4a** could not be further increased. Under optimized conditions when the standard reaction was performed in absence of Meglumine there was less conversion of reactants to products after stirring at room temperature (Table 1, entry 12).

This result motivates us to investigate the methods for synthesis of Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4*H*-pyran-3-carboxylates from substituted benzaldehydes, malononitrile, and ethylacetoacetate using a 10mol% Meglumine catalyst and ethanol:water as a solvent in an optimized reaction condition.

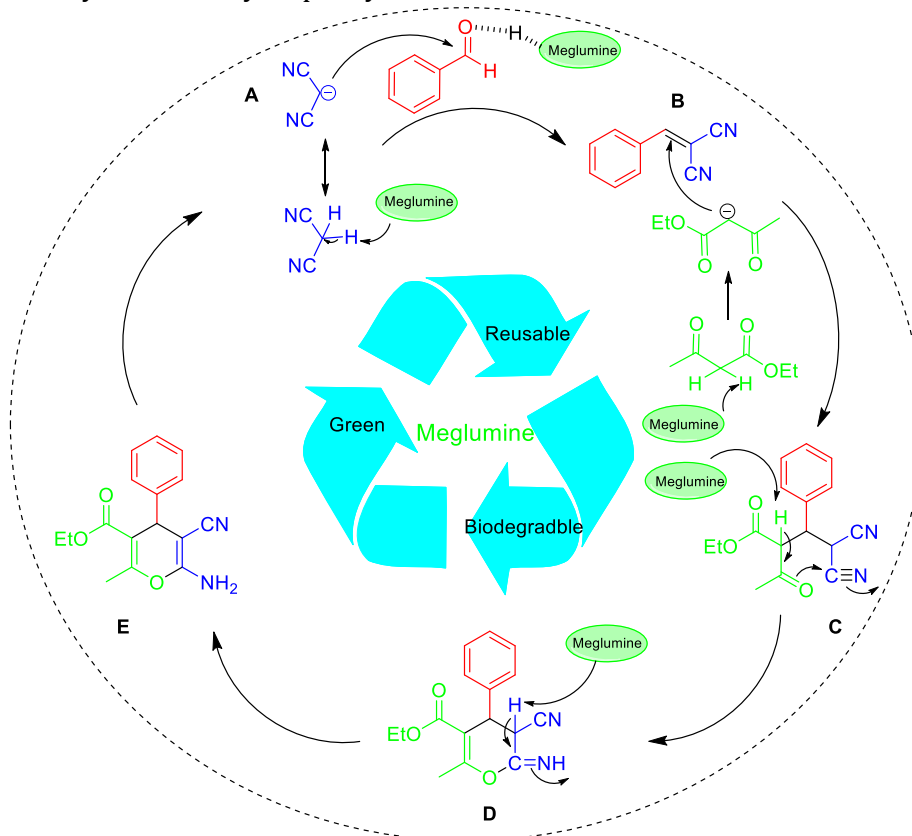
The recyclability of the meglumine catalyst was further examined for the standard reaction of benzaldehyde (**1a**), malononitrile (**2**), and ethylacetoacetate(**3**) in ethanol:water solvent at room temperature for 30-35 minutes. The results are shown in Figure 4.

**Figure 4.** The recyclability of Meglumine<sup>a</sup> in the synthesis of 4*H*-pyran

After the completion of the reaction, the catalyst was recovered and the filtrate was dried. The recovered catalyst was reused after drying for the next run. The catalyst was reused for four runs, and the target compounds were formed in yields (96% to 94%) in their corresponding reaction periods in each and every reaction.

The plausible mechanism for the synthesis of Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4H-

pyran-3-carboxylate(**4a**) as shown in **Scheme 3**, involves the proton abstraction of the active methylene group of malononitrile by the Meglumine indicated as **A**, later it reacts with benzaldehyde involves the dehydration resulting in the formation of **B**, which on the attack of active methylene group of ethylacetoacetate to form **C**, after that abstraction of hydrogen by Meglumine from **C** and **D** to form desired product **E**.



**Scheme 3.** Plausible mechanism for the synthesis of Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4H-pyran-3-carboxylate derivative(**4a**)

## Conclusion

Finally, by utilizing Meglumine as a green and recyclable catalyst, we designed a mild, rapid, and environmentally sustainable synthesis process for Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4H-pyran-3-carboxylate (**4a-I**) from aromatic benzaldehyde, malononitrile, and ethylacetoacetate. Simple reaction conditions, no side reactions, and high yield product formation are all essential features of the

technique. For the synthesis of Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4H-pyran-3-carboxylate, the current technique is an alternative to traditional methods. The catalyst was retrieved several times without losing catalytic activity, resulting in a cost-effective method.

## Experimental



*General experimental procedure for the synthesis of ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4H-pyran-3-carboxylate*

In a dry and clean 50 mL round bottom flask, a mixture of substituted benzaldehyde (1 mmol), malononitrile (1 mmol), and ethylacetoacetate (1 mmol) was stirred in 5 mL ethanol:water as a solvent along with Meglumine (10 mole%) as a catalyst at room temperature for 30–35 min. The progress of the reaction was monitored by thin-layer chromatography. After the completion of the reaction, the reaction mixture was poured on crushed ice, which was then filtered. The crude product was crystallized using ethanol to yield pure 6-amino-5-cyano-2-methyl-4-phenyl-4H-pyran-3-carboxylate. The melting points of the products are in good agreement with those described in the literature [26–32].

*Ethyl 6-amino-5-cyano-2-methyl-4-phenyl-4H-pyran-3-carboxylate (4a)*

The compound (**4a**) was synthesized with Meglumine catalyzed reaction in between benzaldehyde (**1a**), malononitrile (**2**) and ethylacetoacetate (**3**) as white solid; yield 96%; Mp 190–192 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 1.00–1.03 (t, 3H,  $\text{CH}_3\text{-CH}_2\text{-}$ ), 2.31 (s, 3H,  $\text{CH}_3$ ), 3.92–3.99 (m, 2H,  $\text{CH}_2\text{-CH}_3$ ), 4.37 (s, 1H, pyran-H), 4.40 (s, 2H,  $\text{NH}_2$ ), 7.12–7.24 (m, 5H, Ar-H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 12.86, 17.37, 37.74, 59.65, 76.00, 106.98, 117.80, 126.17, 126.49, 127.56, 142.73, 155.76, 156.39, 164.84; LCMS (ESI<sup>+</sup>) calcd. for  $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_3(\text{M}+\text{H})^+$ : 285.12; found 285.14.

*Ethyl 6-amino-4-(4-chlorophenyl)-5-cyano-2-methyl-4H-pyran-3-carboxylate (4c)*

The compound (**4c**) was synthesized with Meglumine catalyzed reaction in between 4-chlorobenzaldehyde (**1c**), malononitrile (**2**) and ethylacetoacetate (**3**) as light yellow solid; yield 94%; mp 138–139 °C;  $^1\text{H}$ -NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 1.03–1.05 (t, 3H,  $\text{CH}_3\text{-CH}_2\text{-}$ ), 2.30 (s, 3H,  $\text{CH}_3$ ), 3.96–4.00 (q, 2H,  $\text{CH}_2\text{-CH}_3$ ), 4.36 (s, 1H, pyran-H), 4.49 (s, 2H,  $\text{NH}_2$ ), 7.06–7.08 (d, 2H, Ar-H), 7.19–7.20 (d, 2H, Ar-H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 12.77, 17.30, 37.16, 59.62, 75.88, 106.46, 117.52, 127.59, 127.76, 131.80,

141.24, 155.90, 156.37, 164.49; LCMS (ESI<sup>+</sup>) calcd. for  $\text{C}_{16}\text{H}_{15}\text{ClN}_2\text{O}_3(\text{M}+\text{H})^+$ : 319.08; found 319.09.

*Ethyl 6-amino-5-cyano-2-methyl-4-(2-nitrophenyl)-4H-pyran-3-carboxylate (4f)*

The compound (**4f**) was synthesized with Meglumine catalyzed reaction in between 2-nitrobenzaldehyde (**1f**), malononitrile (**2**) and ethylacetoacetate (**3**) as yellowish solid; yield 94%; Mp 178–179 °C;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 0.88–0.91 (t, 3H,  $\text{CH}_3\text{-CH}_2\text{-}$ ), 2.31 (s, 3H,  $\text{CH}_3$ ), 3.85–3.87 (q, 2H,  $\text{CH}_2\text{-CH}_3$ ), 4.56 (s, 2H,  $\text{NH}_2$ ), 5.16 (s, 1H, pyran-H), 7.17–7.72 (m, 4H, Ar-H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 12.51, 17.30, 31.83, 59.79, 75.61, 106.15, 117.00, 122.91, 126.76, 129.46, 132.08, 137.95, 147.95, 156.91, 157.09, 163.89; LCMS (ESI<sup>+</sup>) calcd. for  $\text{C}_{16}\text{H}_{15}\text{N}_3\text{O}_5(\text{M}+\text{H})^+$ : 330.31; found 330.35.

*Supporting Information*

Full experimental detail, LCMS,  $^1\text{H}$ , and  $^{13}\text{C}$  NMR spectra. This material can be found via the “Supplementary Content” section of this article’s webpage.

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*Conflict of Interest*

The authors declare no conflict of interest.

Orcid:

Ganesh Baburao Pund  
<https://orcid.org/0000-0003-1560-6171>  
 Sambhaji Tukaram Dhumal  
<https://orcid.org/0000-0003-3018-6179>  
 Madhav Janardan Hebade  
<https://orcid.org/0000-0003-0008-8834>  
 Mazahar Farooqui  
<https://orcid.org/0000-0003-2236-6639>  
 Bhagwansing Shivsing Dobhal

<https://orcid.org/0000-0002-9469-3869>

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