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# Visible-light induced trifluoromethylation/cyclization cascade to access CF<sub>3</sub>-containing pyrazolones by EDA complex

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The incorporation of trifluoromethyl groups into heterocycles often significantly enhances their bioactivity, thereby driving demand for efficient synthetic methods. Herein, a novel visible-light-driven, metal-free strategy is developed for the synthesis of trifluoromethylated pyrazolones *via* a radical addition/cyclization cascade. Under 390–400 nm irradiation, the reaction proceeds through an electron donor–acceptor (EDA) complex between Togni's reagent II and DABCO to generate CF<sub>3</sub> radicals, without the need for an external photocatalyst. A wide range of *N*-methacryloylhydrazones are converted into the corresponding products with good to excellent yields. This method is operationally simple and scalable, and exhibits high functional group tolerance, thus providing a sustainable route to functionalized heterocycles.

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## Introduction

Pyrazolone compounds, as an important class of five-membered heterocycles, have attracted widespread interest in pharmaceutical research and development owing to their diverse medicinal properties.<sup>1,2</sup> In particular, the 2-pyrazolin-5-one scaffold serves as a key structural motif in numerous bioactive molecules, and its derivatives are widely employed as intermediates for synthesizing functionalized pyrazoles (Fig. 1).<sup>2</sup> This versatility has motivated sustained efforts over recent decades to develop synthetic routes to construct the 2-pyrazolin-5-one cores. Conventional methods primarily rely on the Knorr condensation between  $\beta$ -ketoesters and substituted hydrazines.<sup>3</sup> Alternative strategies, such as palladium-catalyzed cyclocarbonylation of 1,2-diaza-1,3-butadienes,<sup>4</sup> palladium-catalyzed carbonylative coupling of  $\alpha$ -chloroketones with hydrazines,<sup>5</sup> and gold-catalyzed tandem reactions of *N*-propionyl hydrazones,<sup>6</sup> have also been explored. However, these approaches suffer from inherent limitations, including dependence on expensive noble metal catalysts, requirement for high-temperature conditions, and a narrow substrate scope. Therefore, the development of efficient and generalizable synthetic methods for 2-pyrazolin-5-ones remains an ongoing challenge in modern synthetic chemistry.

In recent years, photoredox catalysis has emerged as an exceptionally versatile tool in modern synthetic chemistry due

to its high efficiency and sustainability.<sup>7</sup> Visible-light-induced radical addition/cyclization cascade reactions are of great importance for the synthesis of nitrogen-containing heterocycles, owing to the unique redox properties of photocatalysts and their outstanding capability to generate diverse radical species (Scheme 1a).<sup>8</sup> Through this reaction strategy, various alkenes can undergo radical addition followed by subsequent intramolecular radical cascade cyclization, enabling the construction of diversely functionalized heterocyclic compounds under mild conditions.<sup>9</sup> However, most of these processes rely on noble Ru-/Ir-complexes or organic dyes as photocatalysts to drive the photoreactions, which imposes both economic and environmental burdens. In 2024, Murarka developed Ru-photoredox-catalyzed arylative and arylsulfonylative radical cascade reactions of *N'*-arylidene-*N*-acryloylhydrazides to access desired functionalized pyrazolones.<sup>10a</sup> Recently, Yu disclosed the visible-light-induced sulfamoylation/*5-endo-trig* cyclization between *N'*-arylidene-*N*-acryloylhydrazides and sulfamoyl chlorides using 4CzIPN as a photocatalyst.<sup>10b</sup> Therefore, the development of external catalyst-free visible-light-induced photocatalytic systems is of significant importance. Since 2000, the utilization of electron donor–acceptor (EDA) complexes, which formed electronically excited states in absence of organic and metal photocatalysts, has attracted significant interest.<sup>11</sup> In 2024, Murarka established photo-induced radical cascade between alkyl NHPI esters and *N*-acryloyl aldehyde hydrazones *via* the EDA complex, providing a route to alkylated 2-pyrazoline-5-ones with high efficiency.<sup>12</sup>

The introduction of a trifluoromethyl group (–CF<sub>3</sub>) into organic compounds significantly enhances their lipophilicity and metabolic stability.<sup>13</sup> This structural modification not only

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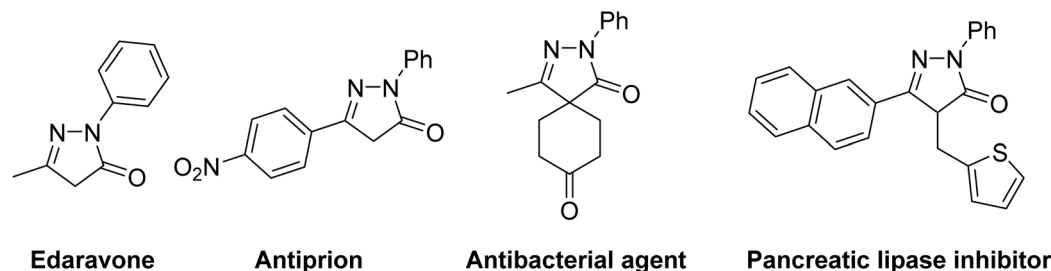


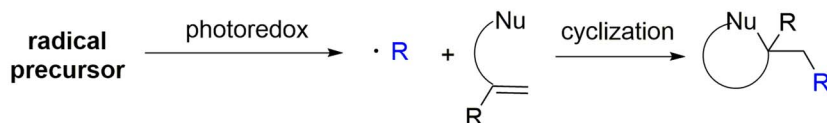
Fig. 1 Representative bioactive molecules containing 1-aryl-pyrazolin-5-one.

alters the electronic properties and three-dimensional conformation of the molecules, thereby improving the potency and selectivity of drug candidates, but also markedly affects key physicochemical properties such as boiling point and solubility.<sup>14</sup> Radical trifluoromethylation reactions have emerged as a crucial approach for CF<sub>3</sub> group incorporation, owing to their mild reaction conditions and excellent compatibility with diverse functional groups.<sup>15</sup> A widely adopted strategy involves generating CF<sub>3</sub> radicals from trifluoromethylation reagents, which subsequently undergo addition reactions with unsaturated systems such as alkenes or arenes, representing a mainstream methodology for CF<sub>3</sub> group installation.<sup>16</sup> Recently, Pan

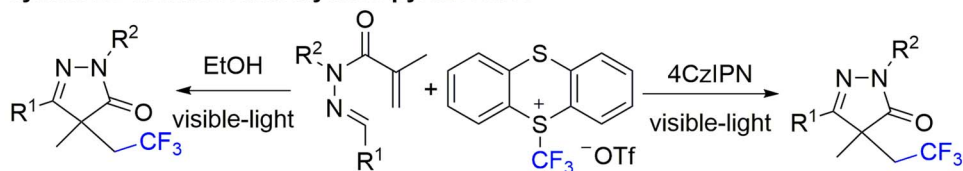
reported a photocatalytic trifluoromethylation/cyclization reaction of *N*-methacryloylhydrazone with trifluoromethyl thianthrenium triflate to afford yields (Scheme 1b).<sup>17</sup> Almost simultaneously, He developed a self-catalyzed phototandem trifluoromethylation/cyclization of *N*-methacryloylhydrazones with trifluoromethyl thianthrenium triflate in EtOH (Scheme 1b).<sup>18</sup> Building upon prior research, we aim to extend the applications of EDA complexes to more demanding and structurally complex chemical transformations. Although significant progress has been achieved in the trifluoromethylation of unsaturated bond using Togni's reagent II (Scheme 1c), the

### Previous work

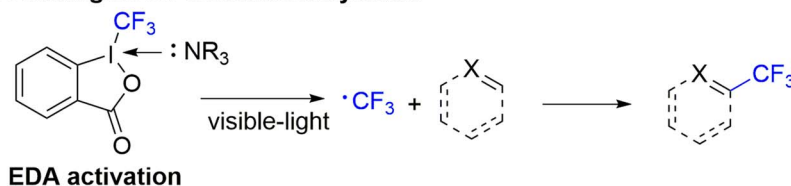
#### a) Photocatalytic radical addition/cyclization reaction



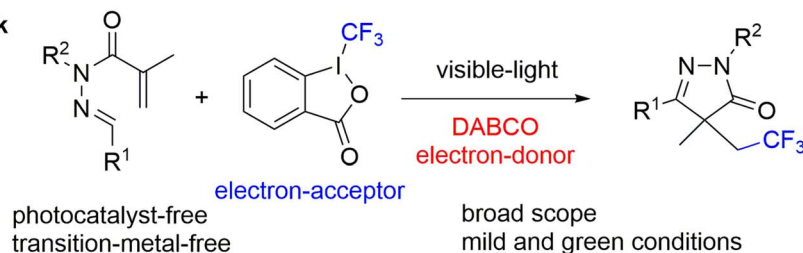
#### b) Synthesis of trifluoromethylated pyrazolones



#### c) EDA strategies for trifluoromethylation



#### d) This work



Scheme 1 Strategies for CF<sub>3</sub>-substituted pyrazolones.



direct incorporation of the trifluoromethyl group into heterocyclic frameworks remains a substantial challenge in modern organic synthesis.<sup>19</sup> Inspired by these elegant precedents, we present a visible-light-driven radical addition/cyclization cascade of *N*-methacryloylhydrazones with Togni's reagent II by an EDA complexes, which affords a range of trifluoromethylated pyrazolones derivatives (Scheme 1d).

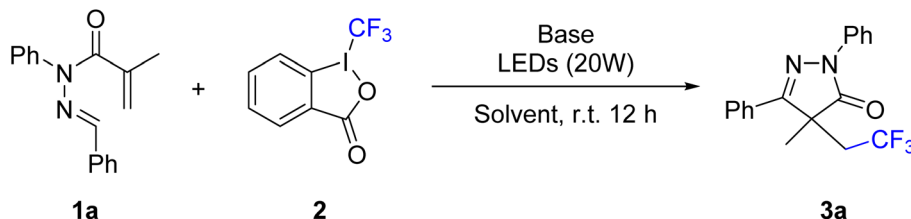
## Results and discussion

Initially, (*E*)-*N'*-benzylidene-*N*-phenylmethacrylohydrazone **1a** was selected as the model substrate and Togni's reagent II **2** as the trifluoromethyl radical source to explore the cyclization reaction under visible-light conditions (Table 1). When the reaction was carried out in MeCN in the presence of 1,4-diazabicyclo[2.2.2]octane (DABCO) under irradiation with a 420–430 nm LED for 12 hours, the CF<sub>3</sub>-substituted pyrazolone **3a** was obtained in 67% yield (entry 1). The influence of irradiation wavelength was then evaluated using LEDs of different emission ranges (entries 2–5). The highest reaction efficiency was achieved with a purple 390–400 nm LED. Screening of various bases, including Cs<sub>2</sub>CO<sub>3</sub>, DBU, and *t*-BuOK, showed that these alternatives were less effective (entries 6–8). Replacing MeCN with other solvents, such as THF, MeOH, EtOH, DMSO, or DMF, did not significantly improve the product yield (entries 9–13). Reducing the amount of DABCO to 0.2 mmol or 0.04 mmol led

to diminished yields of 68% and 41%, respectively (entries 14 and 15). Control experiments confirmed that both light irradiation and DABCO are essential for the trifluoromethylation/cyclization process (entries 16 and 17). Finally, conducting the reaction under an air atmosphere resulted in a significantly reduced product yield (entry 18).

With the optimized conditions established (Table 1, entry 2), we next evaluated the substrate scope of the photoinduced trifluoromethylation/cyclization tandem reaction (Scheme 2). As expected, the current metal-free photocatalytic system exhibited broad applicability across a diverse range of *N*-methacryloyl hydrazones using Togni's reagent II. Aryl aldehyde-derived hydrazones bearing a methyl group at the *ortho*-, *meta*-, or *para*-position of the benzene ring underwent the cascade reaction smoothly, affording products **3b–3d** in good to excellent yields. Notably, both electron-donating (methoxy and phenyl) and electron-withdrawing (bromo, chloro, fluoro, and trifluoromethyl) substituents on the phenyl ring were well tolerated, delivering products **3e–3i** in 51–83% yields. Replacement of the benzene ring with thiophene or pyridine heterocycles also proceeded efficiently, affording the corresponding trifluoromethylated pyrazolones **3m** and **3n** in 67% and 64% yield, respectively. Furthermore, *N*-methacryloyl hydrazones derived from alkyl aldehydes containing cycloalkyl (cyclopropyl and cyclohexyl) or alkyl (*tert*-butyl and isopropyl) groups were also compatible, yielding the corresponding products **3o–3r**.

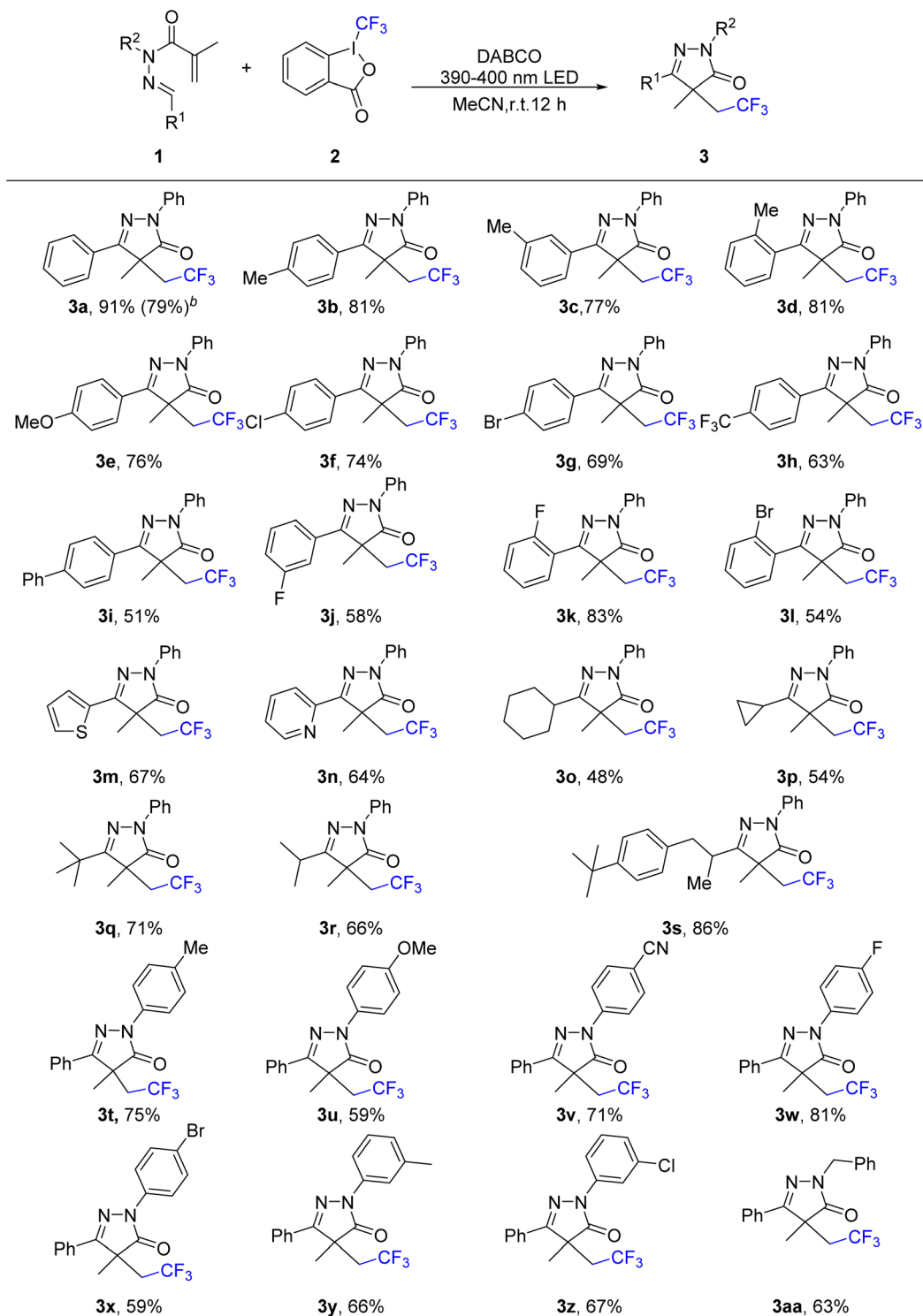
Table 1 Optimization of reaction conditions<sup>a</sup>



Entry	Base	LEDs	Solvent	Yield <sup>b</sup> (%)
1	DABCO	420–430 nm	MeCN	67
2	DABCO	390–400 nm	MeCN	91
3	DABCO	380–390 nm	MeCN	75
4	DABCO	400–800 nm	MeCN	47
5	DABCO	520–530 nm	MeCN	Trace
6	Cs <sub>2</sub> CO <sub>3</sub>	390–400 nm	MeCN	52
7	DBU	390–400 nm	MeCN	35
8	<i>t</i> -BuOK	390–400 nm	MeCN	21
9	DABCO	390–400 nm	THF	78
10	DABCO	390–400 nm	MeOH	56
11	DABCO	390–400 nm	EtOH	82
12	DABCO	390–400 nm	DMSO	37
13	DABCO	390–400 nm	DMF	46
14 <sup>c</sup>	DABCO	390–400 nm	MeCN	68
15 <sup>d</sup>	DABCO	390–400 nm	MeCN	41
16	—	390–400 nm	MeCN	Trace
17	DABCO	Dark	MeCN	0
18 <sup>e</sup>	DABCO	390–400 nm	MeCN	18

<sup>a</sup> Reaction conditions: **1a** (0.2 mmol), **2** (0.4 mmol), base (0.4 mmol), in dry solvent (2.0 mL) under N<sub>2</sub> at room temperature with the irradiation of LED lamps for 12 h. <sup>b</sup> Isolated yield based on **1a**. <sup>c</sup> DABCO (0.2 mmol) was added. <sup>d</sup> DABCO (0.04 mmol) was added. <sup>e</sup> Under air. DABCO, 1,4-diazabicyclo[2.2.2]octane; DBU, 8-diazabicyclo[5.4.0]undec-7-ene; THF, tetrahydrofuran; DMSO, dimethyl sulfoxide; DMF, *N,N*-dimethylformamide.



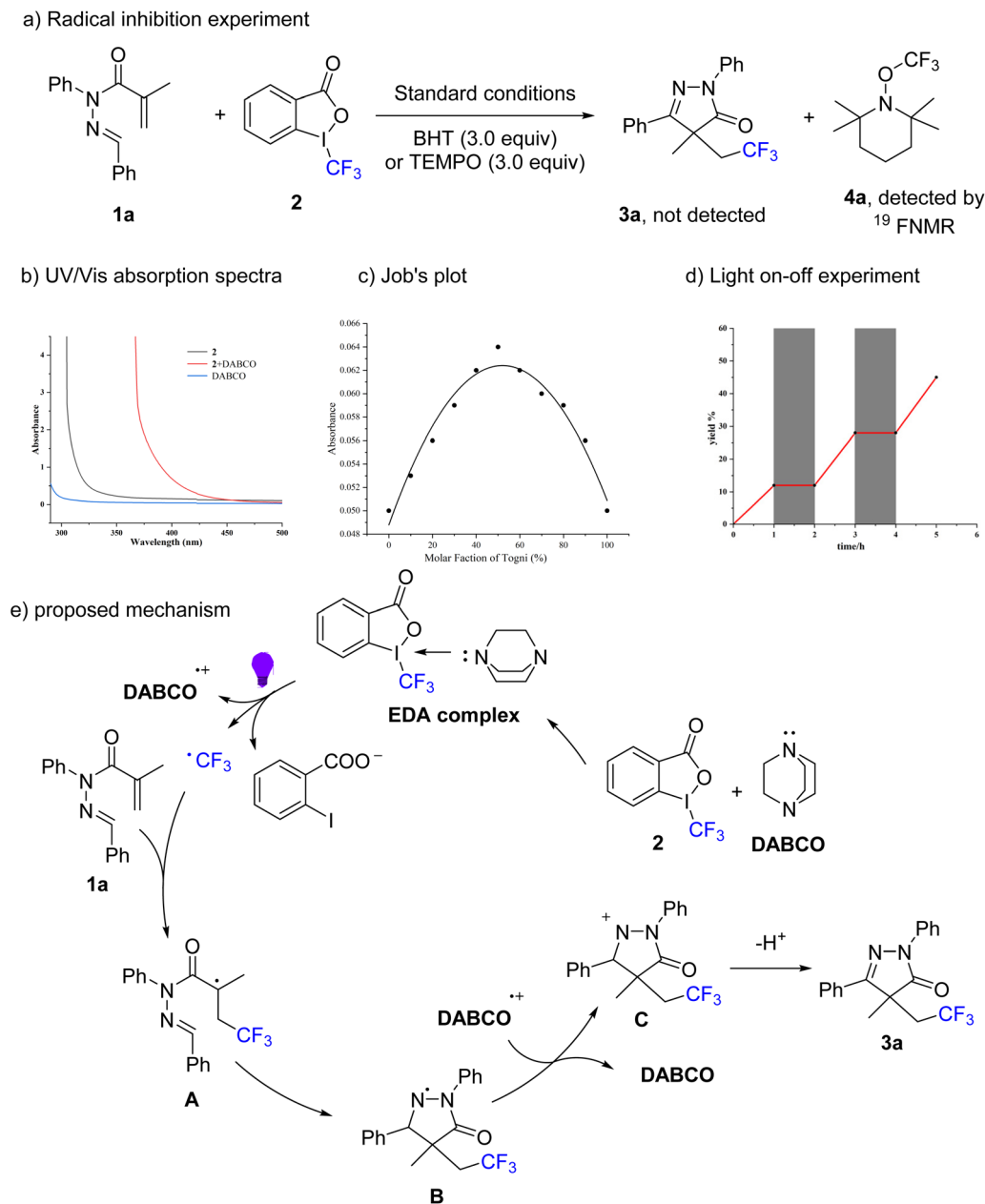


**Scheme 2** <sup>a</sup>Substrate scope for the trifluoromethylation/cyclization cascade. Reaction conditions: **1a** (0.2 mmol), **2** (0.4 mmol), DABCO (0.4 mmol), in dry MeCN (2.0 mL) under N<sub>2</sub> at room temperature with the irradiation of 390–400 nm LED lamps for 12 h. Isolated yield. <sup>b</sup>2 mmol scale reaction.

The hydrazone derived from Lily aldehyde also afforded product **3s** in 86% yield. Subsequently, we investigated the reactivity of arylhydrazine-derived hydrazones under the optimized conditions. This method proved effective, delivering the target products **3t–3z** in moderate to good yields, irrespective of

whether the aryl ring bore electron-donating or electron-withdrawing substituents. In addition, the benzyl hydrazine-derived substrate was also compatible under the standard conditions, affording the corresponding product **3aa** in 63% yield. Finally, to demonstrate the utility of this photoinduced





Scheme 3 Investigation of the reaction mechanism.

cascade method, the reaction between **1a** and **2** can scale-up to 2 mmol with the isolation of **3a** in 79% yield.

To gain deeper mechanistic insights into the visible-light-driven trifluoromethylation/cyclization cascade, a series of control experiments were carried out. In first, the addition of 3.0 equivalents of the radical scavengers 2,6-di-*tert*-butyl-4-methylphenol (BHT) or 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO) under standard conditions completely inhibited the reaction (Scheme 3a). The formation of the radical adduct **4a** was detected by  $^{19}\text{F}$  NMR spectroscopy, providing strong evidence for the involvement of radical intermediates in this transformation. Since the reaction proceeded efficiently in the absence of any exogenous photocatalyst, we sought to

investigate the potential involvement of an EDA complex as the initiating species in the radical mechanism. Consistent with the findings of previous studies, a distinct bathochromic shift was detected in the UV-vis absorption spectrum when Togni's reagent II **2** and DABCO were mixed at a 1:1 molar ratio in acetonitrile solution, indicating the formation of an EDA complex between the two reactants (Scheme 3b). Furthermore, subsequent analysis *via* the Job's plot method confirmed the formation of a 1:1 EDA complex between Togni's reagent II **2** and DABCO (Scheme 3c). The possibility of a radical chain mechanism operating in this EDA complex system was ruled out by a light on-off experiment conducted under standard conditions between *N*-methacryloyl hydrazone **1a** and **2** (Scheme 3d).



According to the aforementioned mechanistic studies and previous reports,<sup>17–19</sup> a plausible reaction mechanism was illustrated in Scheme 3e. Initially, Togni's reagent II **2** formed an EDA complex with DABCO, which underwent dissociation under purple light irradiation to generate a trifluoromethyl radical, a DABCO radical cation, and an 2-iodobenzoate anion. Subsequently, the electrophilic CF<sub>3</sub> radical attacked the acryl double bond in **1a** to afford the radical intermediate **A**. Then, this key intermediate preferred to add the more electron-rich C=N double bond of the hydrazone *via* 5-*endo-trig* cyclization, generating the *N*-centered radical intermediate **B**. Next, the SET oxidation of intermediate **B** by the DABCO radical cation concurrently regenerated DABCO and produced cationic intermediate **C**. Ultimately, this cationic intermediate then underwent deprotonation to furnish the target product **3a**.

## Conclusions

In summary, we have developed a novel visible-light-promoted, metal-free radical addition/cyclization cascade strategy for the efficient construction of trifluoromethylated pyrazolone derivatives. Under optimized conditions using commercially available Togni's reagent II and DABCO as the radical source under 390–400 nm light irradiation, a variety of *N*-methacryloylhydrazones were smoothly transformed into the desired products in good to excellent yields. Mechanistic studies revealed that a key EDA complex formed between the trifluoromethylation reagent and DABCO facilitates efficient radical generation without the need for an external photocatalyst. This method is operationally simple, readily scalable, and exhibits broad substrate scope with good functional group tolerance, offering a green and sustainable approach to the functionalization of heterocyclic compounds.

## Author contributions

Zilin Liu: investigation, data curation, and methodology. Minxi Hu: investigation and data curation. Shuo Gao: data curation. Pifeng Wei: data curation. Mengmeng Zhao: supervision and writing – review & editing. Yunqiang Sun: writing – original draft. Zhen-Hua Zhang: writing – review & editing, supervision, funding acquisition, and conceptualization.

## Conflicts of interest

There are no conflicts to declare.

## Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Supplementary information (SI) is available. See DOI: <https://doi.org/10.1039/d5ra08028b>.

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