



Efficient solvent- and catalyst-free one-pot synthesis of novel trifluoromethylated pyrazole derivatives

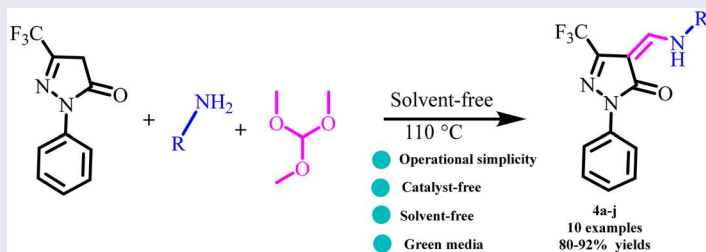
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ABSTRACT

A novel and efficient one-pot, three-component reaction was developed for the synthesis of 2-phenyl-4-((arylamino)methylene)-5-(trifluoromethyl)-2,4-dihydro-3H-pyrazol-3-one derivatives. The reaction proceeds via the condensation of 1-phenyl-3-(trifluoromethyl)-1H-pyrazol-5(4H)-one, aniline derivatives, and trimethyl orthoformate under solvent-free conditions at 110 °C. This green protocol integrates multiple pharmacophores, offering potential for biological applications. Key advantages include operational simplicity, high yields (80%–92%), and the absence of catalysts and solvents. IR, ¹H, ¹³C, ¹⁹F NMR, mass spectrometry, and elemental analysis confirmed product structures.

GRAPHICAL ABSTRACT



ARTICLE HISTORY

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Green chemistry; one-pot reaction; solvent-free; 1-phenyl-3-(trifluoromethyl)-1H-pyrazol-5(4H)-one; 2-phenyl-4-((arylamino)methylene)-5-(trifluoromethyl)-2,4-dihydro-3H-pyrazol-3-one

Introduction

Heterocycles are vital in natural and synthetic molecules, with broad applications in chemistry and biomedicine.^[1] Fluorine incorporation enhances these frameworks by improving molecular polarity, metabolic stability, and bioactivity.^[2,3] In drug design, the strong C–F bond boosts stability, bioavailability, and target affinity.^[4,5] Trifluoromethyl groups, in particular, significantly alter chemical and biological properties, contributing to the success of drugs like Prozac, Diflucan, and Casodex.^[6,7] Recently, fluorinated O- and N-heterocycles have gained attention for their promising bioactivities.^[8]

Pyrazolone, a five-membered ring with two nitrogen atoms and a carbonyl group,^[9] is a versatile scaffold known for its tautomerism—allowing dynamic interconversion

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between different structural forms. This property significantly affects its reactivity and physicochemical characteristics.^[10] Widely found in natural alkaloids, pyrazolone derivatives display a broad spectrum of pharmacological activities, including anti-inflammatory,^[11] anticancer,^[12] analgesic,^[13] antidiabetic,^[14] antimicrobial,^[15] and antioxidant effects.^[16] These diverse properties make pyrazolone a promising core structure for developing novel therapeutic agents (Figure 1).

Multicomponent reactions (MCRs) have emerged as a powerful and efficient approach for synthesizing complex heterocycles. By integrating multiple reagents into a single product, MCRs streamline synthesis, improving efficiency, selectivity, and atom economy. These reactions promote sustainable chemistry by reducing waste, raw material usage, and energy consumption. Additionally, MCRs enable the incorporation of diverse functional groups, facilitating the rapid creation of highly functionalized heterocycles with significant biological potential.^[17–21]

Given the biological significance of pyrazolones and the unique influence of fluorine in heterocycles, we hypothesize that fluorinated pyrazolone derivatives may possess enhanced or novel bioactivities. Building on our previous successful synthetic methodologies for heterocyclic compounds,^[22–24] we report here a novel multicomponent reaction between 1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-5(4*H*)-one, various aniline derivatives, and trimethyl orthoformate. This reaction affords a series of new heterocyclic compounds, specifically 2-phenyl-4-((arylamino)methylene)-5-(trifluoromethyl)-2,4-dihydro-3*H*-pyrazol-3-one, which may serve as valuable candidates for further biological evaluation (Scheme 1).

Results and discussion

To optimize the reaction conditions, we selected 1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-5(4*H*)-one (**1**), 4-methoxyaniline (**2b**), and trimethyl orthoformate (**3**)

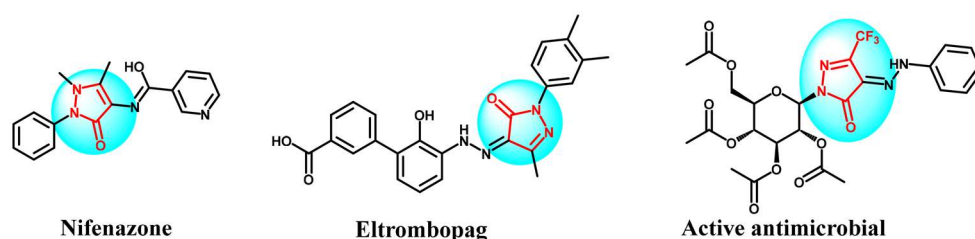
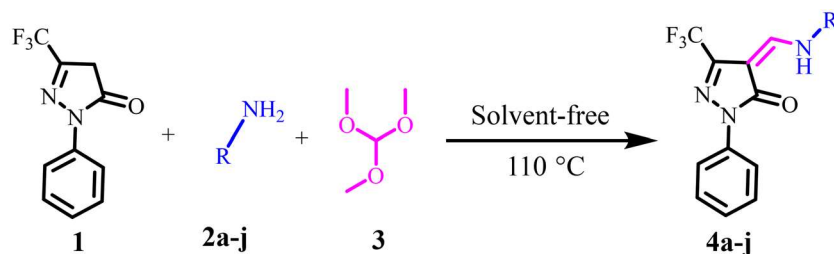


Figure 1. Drugs containing pyrazolone cores and investigational pyrazolone derivatives.



Scheme 1. Synthesis of new 4-((arylamino)methylene)-2,4-dihydro-3*H*-pyrazol-3-ones.

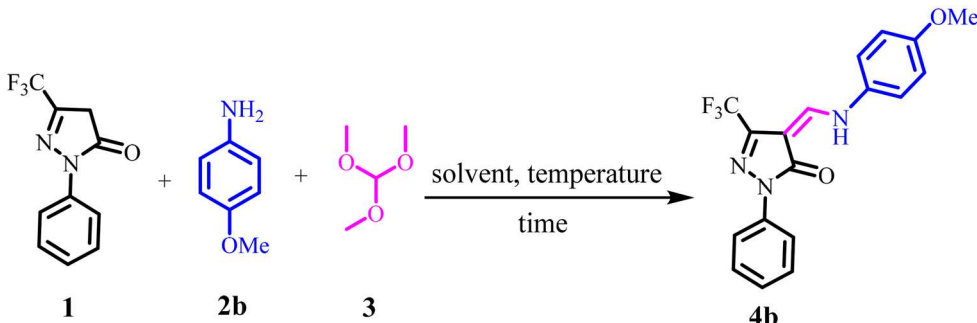
as the model substrates. The reaction was evaluated in various solvents, including H₂O, H₂O/EtOH (1:1), EtOH, MeOH, CH₃CN, DMF, PEG 400, DCM, as well as under solvent-free conditions (Table 1, entries 1–9). Among these, the solvent-free condition at 90 °C yielded the highest product formation, achieving a 75% yield (Table 1, entry 9).

Further optimization focused on the effect of temperature under solvent-free conditions, with reactions performed at 70 °C, 110 °C, and 130 °C. It was observed that reducing the reaction temperature to 70 °C led to a significant decrease in yield, while increasing the temperature beyond 110 °C did not enhance the product yield (Table 1, entries 10–12). These findings establish solvent-free conditions at 110 °C as the optimal parameters for achieving high product efficiency.

To further optimize the reaction, we investigated the influence of various catalysts, including Et₃N, DABCO, L-proline, and P-TAS, under solvent-free conditions at 110 °C. Surprisingly, our results demonstrated that the highest yield of the desired product was achieved without the use of any catalyst, under the same solvent-free conditions (Table 2, entries 1–9). This finding underscores the reaction's efficiency and simplicity, eliminating the need for extra catalytic agents.

Following the optimization of reaction conditions, we systematically investigated the scope and versatility of the protocol by utilizing a diverse range of aniline derivatives. This exploration successfully yielded a series of 2-phenyl-4-((arylamino)methylene)-5-(trifluoromethyl)-2,4-dihydro-3H-pyrazol-3-one derivatives (**4a–j**) with impressive yields ranging from 85% to 92%, as summarized in Table 3.

Table 1. Optimization of reaction conditions of **4b**^a.



Entry	Solvent	Temperature (°C)	Time	Yield (%) ^b
1	H ₂ O	Reflux	12h	20
2	EtOH/ H ₂ O (1:1)	Reflux	12h	29
3	EtOH	Reflux	12h	62
4	MeOH	Reflux	12h	38
5	CH ₃ CN	Reflux	8h	39
6	DMF	Reflux	8h	42
7	PEG 400	110	8h	53
8	CH ₂ Cl ₂	r.t	5h	25
9	Solvent-free	90	1h	75
10	Solvent-free	70	6h	30
11	Solvent-free	110	1h	89
12	Solvent-free	130	1h	89

^aReaction conditions: 1-phenyl-3-(trifluoromethyl)-1H-pyrazol-5(4H)-one (1.0 mmol), 4-methoxyaniline (1.2 mmol), and trimethyl orthoformate (1.0 mmol), in solvent (5 ml).

^bIsolated yield.

Table 2. Catalyst effect for the three-component synthesis of **4b**^a.

Entry	Catalyst (mol%)	Solvent	Time (hours)	Yield (%) ^b
1	Et ₃ N (5)	–	6	65
2	Et ₃ N (10)	–	6	68
3	DABCO (5)	–	6	60
4	DABCO (10)	–	6	62
5	L-proline (5)	–	6	64
6	L-proline (10)	–	6	65
7	<i>p</i> -TSA (5)	–	6	53
8	<i>p</i> -TSA (10)	–	6	53
9	–	–	1	89

^aReaction conditions: 1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-5(4*H*)-one (1.0 mmol), 4-methoxyaniline (1.2 mmol), and trimethyl orthoformate (1.0 mmol) in solvent-free condition at 110 °C.

^bIsolated yield.

The protocol exhibited broad applicability, accommodating both electron-donating groups (e.g., Me, OMe, OEt) and electron-withdrawing groups (e.g., NO₂, Cl, Br) on the aromatic ring. All reactions proceeded efficiently, delivering the desired products with consistently high yields. Remarkably, the use of 2-aminothiazole as a heterocyclic amine resulted in a high yield of 85% (Table 3, **4g**).

The protocol exhibited limited compatibility with aliphatic amines like ethylamine, propylamine, and butylamine, yielding only trace amounts of the desired products under optimized conditions.

The structures of all synthesized compounds (**4a–j**) were comprehensively elucidated using IR, ¹H, ¹³C NMR spectroscopy, mass spectrometry, and elemental analysis (See Supplemental Data).

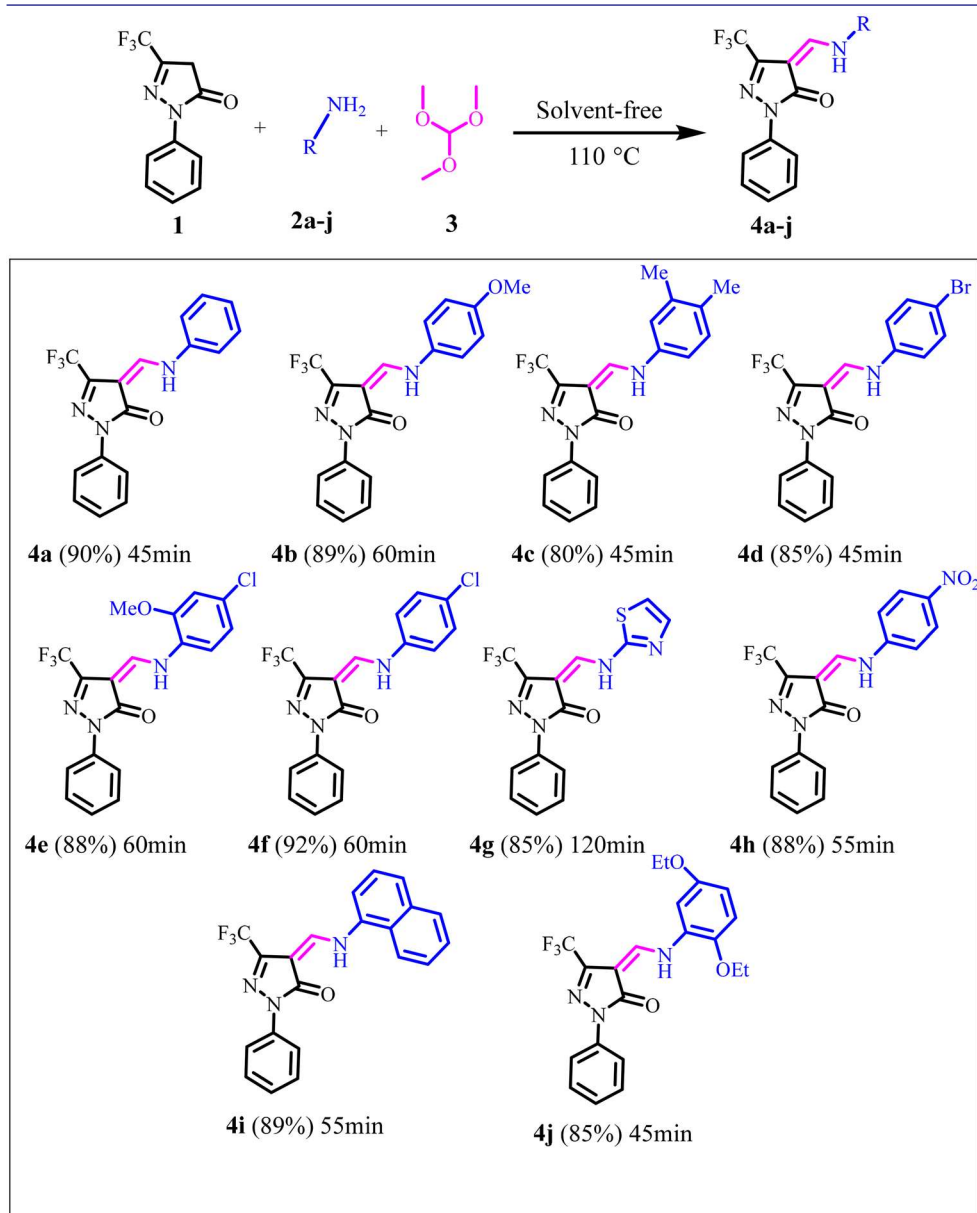
For example, the IR spectrum of compound **4b** exhibited characteristic absorption bands at 3223 cm^{−1} (NH stretching), 1674 cm^{−1} (C=O stretching), 1625 cm^{−1} (C=N stretching), and 1496 cm^{−1} (C–F stretching). The ¹H NMR spectrum of **4b** displayed a singlet at δ = 3.75 ppm (3H, OCH₃), a doublet at δ = 6.86 ppm (*J* = 9 Hz), a doublet of doublets at δ = 7.13 ppm (*J* = 16.4 Hz), a multiplet between δ = 7.30–7.38 ppm, and a doublet at δ = 7.90 ppm (*J* = 9.1 Hz), corresponding to aromatic protons (9H). Additionally, signals at δ = 8.01 ppm and δ = 11.98 ppm were attributed to vinyl and NH₂ groups, respectively.

The ¹³C NMR spectrum, recorded with proton decoupling, confirmed the proposed structure of **4b** by displaying 14 distinct peaks. Notably, two quartet peaks at δ = 119.6 ppm (¹*J*_{CF} = 271.2 Hz) and δ = 137.8 ppm (²*J*_{CF} = 40 Hz) indicated the presence of a CF₃ group. The mass spectrum further corroborated the structure with a molecular ion peak at *m/z* = 361.

The ¹⁹F NMR spectrum of compound **4f** displayed a singlet at δ = −62.9 ppm, consistent with the CF₃ group. Scheme 2).

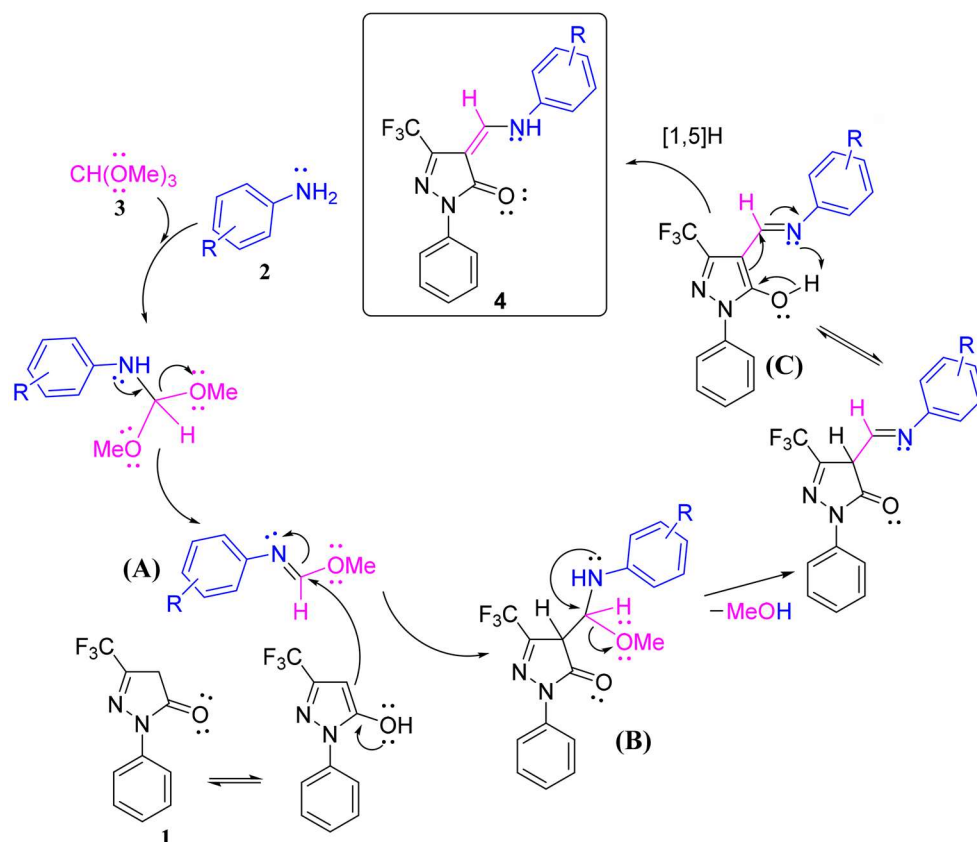
Although the reaction mechanism has yet to be experimentally verified, we propose a plausible pathway for the formation of product **4**, as outlined in Scheme 2, which is supported by prior studies.^[25] Initially, the *in-situ* formation of intermediate (**A**) via the nucleophilic addition of amines (**2**) to trimethyl orthoformate (**3**), accompanied by the elimination of two molecules of CH₃OH. Subsequently, the anion generated from 1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-5(4*H*)-one (**1**) stabilized by resonance

Table 3. Synthesis of novel 2-phenyl-4-((arylamino)methylene)-5-(trifluoromethyl)-2,4-dihydro-3H-pyrazol-3-one derivatives.^a



a) Reaction conditions: 1-phenyl-3-(trifluoromethyl)-1H-pyrazol-5(4H)-one (1.0 mmol), aromatic amines (1.2 mmol), and trimethyl orthoformate (1.0 mmol) in solvent-free conditions at 110 °C.

with the carbonyl group and the strong electron-withdrawing effect of the trifluoromethyl group reacts with the imine intermediate (A). This interaction leads to the formation of intermediate (B), which undergoes a final elimination of another CH₃OH molecule, yielding the desired product (4).



Scheme 2. Proposed reaction mechanism for the synthesis of novel 2-phenyl-4-((arylamino)methylene)-5-(trifluoromethyl)-2,4-dihydro-3H-pyrazol-3-one derivatives.

Conclusion

We have successfully developed a new and facile approach for the synthesis of 2-phenyl-4-((arylamino)methylene)-5-(trifluoromethyl)-2,4-dihydro-3H-pyrazol-3-one derivatives through a one-pot three-component reaction of primary amines under solvent-free condition. The advantages of this work include operational simplicity, no need for solvent and catalyst, the use of readily and simply available starting materials, and short reaction time. The findings indicate that our approach to synthesizing these trifluoromethylated heterocyclic compounds is significant for the organofluoride heterocyclic compounds.

Experimental

General

The solvents and chemicals were sourced from Merck (Germany) and Sigma-Aldrich (Buchs, Switzerland) and were used without further purification. 1-Phenyl-3-(trifluoromethyl)-1H-pyrazol-5(4H)-one (1) was synthesized following a previously established method.^[26] Melting points were determined using an Electrothermal 9100

apparatus in open capillary tubes. The products were characterized by FT-IR spectroscopy using KBr disks on a Nicolet Avatar 370 FT-IR Thermo instrument. In all cases, ^{13}C and ^1H NMR spectra were recorded on a Bruker DRX-300 Avance spectrometer at 75.47 and 300.13 MHz, respectively, with CDCl_3 as the solvent, and chemical shifts were reported in ppm. Mass spectra were obtained using a Varian MAT CH-7 spectrometer at 70 eV. Elemental analysis (C, H, N) was performed using a Thermo Finnigan Flash EA.

General procedure for synthesis of 2-phenyl-4-((arylamino)methylene)-5-(trifluoromethyl)-2,4-dihydro-3H-pyrazol-3-one derivatives 4

In a sealed tube, a mixture of 1-phenyl-3-(trifluoromethyl)-1H-pyrazol-5(4H)-one (1, 1 mmol), aniline derivatives (2, 1.2 mmol), and trimethyl orthoformate (3, 1 mmol) was stirred under solvent-free conditions at 110 °C for 45–120 min. The reaction progress was monitored by TLC (Hexane/Ethyl acetate, 7/3). Upon completion, the mixture was allowed to cool to room temperature, and the precipitated product was isolated via simple filtration. The crude product was washed with a 1:1 mixture of ethanol and water (5 mL) and then purified by flash chromatography (Hexane/Ethyl acetate, 9/1). IR, ^1H NMR, ^{13}C NMR, ^{19}F NMR, mass spectrometry, and CHN analysis confirmed the structures of all synthesized products (Supporting Information).

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Declaration of interest

The authors have no relevant financial or non-financial interests to disclose. The authors have no competing interests to declare that they are relevant to the content of this article. All authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.

Authors contributions

CRediT: **Zhila Zharf Zaki**: Conceptualization, Investigation, Methodology, Writing – original draft; **Abbas Ali Esmaili**: Conceptualization, Investigation, Project administration, Supervision, Writing – review & editing.

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Data availability statement

The data underlying this study, are available in the published article and its Supporting Information.

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