An aqueous, catalyst-free and three-component synthesis of 6-amino-3-(trifluoromethyl)-1,4-dihydro-1-phenyl-4-arylpyrano[2,3-*c*]pyrazole-5-carbonitriles

Chenxia Yu · Changsheng Yao · Tuanjie Li · Xiangshan Wang

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Abstract A concise synthesis of 6-amino-4-aryl-3-(trifluoromethyl)-1,4-dihydro-1-phenylpyrano[2,3-c]pyrazole-5-carbonitriles was performed effectively in aqueous media without catalyst by the reaction of aryl aldehydes, malononitrile, and 1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-5(4*H*)-one. This method has the advantages of mild condition, avoidance of the use of catalysts, high yields, and environmentally benign procedure.

Keywords Aqueous synthesis \cdot Multi-component reaction \cdot Catalyst-free synthesis \cdot Trifluoromethylated heterocycles \cdot Pyrano[2,3-*c*]pyrazole

Introduction

The organofluorine compounds are of significant importance to the pharmaceutical and agrochemical industries, due to their often unique biological properties, such as increased membrane permeability, enhanced hydrophobic binding, and stability against metabolic oxidation [1–5]. The trifluoromethyl group (CF₃) is an important structural moiety in diverse classes of bioactive organic molecules. It has a bigger van der Waals radius than that of a CH₃ group and the same electronegativity as oxygen [6]. Thus, much effort has been exerted in the introduction of a

C. Yu · T. Li · X. Wang

C. Yao (🖂)

School of Chemistry and Engineering, Jiangsu Key Laboratory of Green Synthetic Chemistry for Functional Materials, Jiangsu Normal University, Xuzhou 221116, Jiangsu, People's Republic of China

School of Chemistry and Chemical Engineering, Key Laboratory of Biotechnology on Medical Plant, Jiangsu Normal University, Xuzhou 221116, Jiangsu, People's Republic of China e-mail: csyao@jsnu.edu.cn

trifluoromethyl group into organic molecules to tune their physical, chemical, and physiological properties [7–11].

The chemistry of condensed pyranopyrazoles has received considerable attention for their derivatives which display a broad range of bioactivities including antimicrobial, antibacterial, antifungal, antitubercular, anti-inflammatory, and cytotoxic [12–16]. Thus, the synthesis and biological investigation of fluorinated condensed pyranopyrazoles arouses great research interest in the fields of organic chemistry, medicinal chemistry, and agrochemistry. Very recently, Bhavanarushi et al. [17] reported the cytotoxic and DNA binding studies of fused trifluoromethylated pyranopyrazoles. This led us to pay much attention to the efficient synthesis of these biologically significant heterocyclic compounds.

Recently, aqueous multi-component reaction (MCR), which couples the advantages of MCR with that of aqueous synthesis, has become an attractive protocol for the diversity-oriented synthesis of heterocycles due to its prominent benefits including atom-economy, efficiency in bond formation, straightforward reaction design, and generation of products in an environmentally benign way [18–22]. To continue our work on the aqueous synthesis of heterocyclic compounds via MCR [23–25], we here disclose a catalyst-free synthesis of 6-amino-3-(trifluoromethyl)-1,4-dihydro-1-phenyl-4-arylpyrano[2,3-c]pyrazole-5-carbonitriles via the reaction of aryl aldehydes, malononitrile, and 1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-5(4*H*)-one in water (Scheme 1).

Results and discussion

Initially, the effect of temperature on the three-component synthesis was investigated using benzaldehyde **1a**, malononitrile **2**, and 1-phenyl-3-(trifluoromethyl)-1*H*pyrazol-5(4*H*)-one **3** as model substrates (Table 1). When the reaction was performed below 70 °C, no obvious transformation of the starting material was observed, as indicated by TLC. Later, the reaction proceeded well and gave the desired product in moderate to good yields at elevated temperatures. The final results showed 90 °C to be preferable.

Inspired by the abovementioned result, we next examined the generality of this method using several aryl aldehydes as substrates. The results are summarized in Table 2. The protocol showed good tolerance to the aryl aldehydes bearing both electron-withdrawing (Table 2, entries 3–4) and electron-donating (Table 2, entry 7)



Scheme 1 Catalyst-free aqueous synthesis trifluoromethylated pyrano[2,3-c]pyrazoles

groups, since they were subjected to the reaction and in nearly all cases furnished the expected trifluoromethylated heterocycles in excellent yields under the same conditions despite the elimination of a molecule of water from the reaction system. To our delight, terephthalaldehyde could also be involved in the three-component condensation and provided the product **4h** successfully incorporating two trifluoromethylated fused pyranopyrazole frameworks. Thus, we concluded safely that the electronic nature of the substituents on the aryl ring had no significant influence on the reaction yields and this highlighted the wide scope of the catalyst-free aqueous strategy.

All of the products were characterized by elemental analysis, FTIR, and 1 H NMR.

According to our results, the possible mechanism to account for the reaction was proposed (Scheme 2). One molecule of aryl aldehyde 1 was first condensed with malononitrile 2 to afford α -cyanocinnamonitrile derivative 5, which can be regarded as a fast knoevenagel condensation. The active methylene of 3 reacted with the electrophilic C–C double bond to give the intermediate 6, which underwent cyclization by the intramolecular nucleophilic attack of OH group on the cyano (CN) moiety to afford the intermediate 7. Finally, the expected products 4 were obtained through isomerization ($7 \rightarrow 8 \rightarrow 4$). To confirm this reaction mechanism, 2-(4-bromobenzylidene)malononitrile was synthesized and reacted with 3 in 2 h, which gave the corresponding product in similar yield as entry 2.

Table 1 Optimization of solvent and temperature for the three-component synthesis	Entry	Temp. (°C)	Time (h)	Yield (%)
	1	70	6	56
	2	80	5	62
	3	85	5	68
	4	90	4	79
	5	95	3.5	78
	6	100	3.5	79

Entry	Compound	Ar	Time (h)	Yield (%)
1	4a	C ₆ H ₅	4.0	79
2	4b	$4-BrC_6H_4$	3.5	86
3	4c	3-NO ₂ C ₆ H ₄	3.0	82
4	4d	$4-NO_2C_6H_4$	3.5	90
5	4e	$4-ClC_6H_4$	5.0	87
6	4f	$4-FC_6H_4$	4.0	81
7	4g	3,4,5-(MeO) ₃ C ₆ H ₂	3.5	84
8	4h	$4-HCOC_6H_4$	4.0	84
9	4i	$3-FC_6H_4$	5.0	78
10	4j	$2-FC_6H_4$	3.0	87
11	4k	4-HO-3-NO ₂ C ₆ H ₃	4.5	81

Table 2	Aqueous three-
componen	t synthesis of
trifluorom	ethylated
pyrano[2.3	3-clpvrazoles



Scheme 2 A proposed plausible reaction pathway for the aqueous synthesis

Conclusion

In summary, we have developed an efficient, economical, safe, and eco-friendly method to synthesize 6-amino-4-aryl-3-(trifluoromethyl)-1,4-dihydro-1-phenylpyr-ano[2,3-c]pyrazole-5-carbonitrile by the catalyst-free reaction of aryl aldehydes, malononitrile, and 1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-5(4*H*)-one in aqueous media. The result showed that our strategy for the syntheses of these trifluoromethylated heterocyclic molecules is of great importance in both the organofluorine and organic synthetic fields.

Experimental

Common reagents and materials were purchased from commercial sources and purified by recrystallization or distillation. The starting material, 1-phenyl-3-trifluoromethylpyrazol-5-one **3**, was prepared according to Ref. [26]. Infrared (IR) spectra were recorded on a TENSOR 27 spectrophotometer in KBr pellet and are reported in terms of frequency of absorption (cm⁻¹). NMR spectra were recorded on

a Bruker AV-400; data for ¹H are reported as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; and br, broad), coupling constant (Hz) and integration. Elemental analyses were performed on Yanaco-CHN CORDER elementary analyzer.

General procedure for the synthesis of 6-amino-3-(trifluoromethyl)-1,4-dihydro-1-phenyl-4-arylpyrano[2,3-*c*]pyrazole-5-carbonitrile (**4a–4k**)

Aryl aldehydes (1.0 mmol), malononitrile (1.0 mmol; for compound **4h**, 2.0 mmol was used.), 1-phenyl-3-(trifluoromethyl)-1*H*-pyrazol-5(4*H*)-one (1.0 mmol; for compound **4h**, 2.0 mmol was used.), and 3 mL water were added to a 25-mL round-bottom flask and were stirred at 90 °C for several hours (monitored by TLC). Then the mixture was cooled and filtered. The solid was further recrystallized from ethanol to give pure products.

6-Amino-3-(trifluoromethyl)-1,4-dihydro-1,4-diphenylpyrano[2,3-c]pyrazole-5-carbonitrile (**4***a*)

M.p.: 190–191 °C (Lit. [27].: 185–187 °C); IR (potassium bromide) (v, cm⁻¹): 3,325, 2,200, 1,663, 1,595, 1,518, 1,481, 1,456, 1,389, 1,231, 1,184, 1,145, 1,036, 759, 697; ¹H NMR (400 MHz, DMSO- d_6): 4.79 (s, 1H, C⁴-H), 7.25–7.40 (m, 7H, ArH + NH₂), 7.48 (t, J = 7.6 Hz, 1H, ArH), 7.58 (t, J = 7.6 Hz, 2H, ArH), 7.85 (d, J = 8.0 Hz, 2H, ArH); Anal. calcd. For C₂₀H₁₃F₃N₄O: C, 62.83; H, 3.43; N, 14.65. Found: C, 62.75; H, 3.54; N, 14.74.

6-Amino-4-(4-bromophenyl)-3-(trifluoromethyl)-1,4-dihydro-1-phenylpyrano[2,3c]pyrazole-5-carbonitrile (**4b**)

M.p.: 188–189 °C; IR (potassium bromide) (v, cm⁻¹): 3,441, 3,324, 3,207, 2,198, 1,658, 1,595, 1,578, 1,520, 1,483, 1,458, 1,396, 1,310, 1,277, 1,233, 1,200, 1,144, 1,074, 1,038, 1,024, 1,011, 912, 893, 827, 807, 759, 734, 696; ¹H NMR (400 MHz, DMSO- d_6): 4.81 (s, 1H, C⁴-H), 7.22 (d, J = 8.4 Hz, 2H, ArH), 7.38 (s, 2H, NH₂), 7.45–7.59 (m, 5H, ArH), 7.82 (d, J = 8.4 Hz, 2H, ArH); Anal. calcd. For C₂₀H₁₂BrF₃N₄O: C, 52.08; H, 2.62; N, 12.15. Found: C, 52.15; H, 2.48; N, 12.24.

6-Amino-3-(trifluoromethyl)-1,4-dihydro-4-(3-nitrophenyl)-1-phenylpyrano[2,3-c]pyrazole-5-carbonitrile (**4***c*)

M.p.: 192–193 °C; IR (potassium bromide) (v, cm⁻¹): 3,355, 2,194, 1,655, 1,595, 1,530, 1,482, 1,458, 1,394, 1,353, 1,312, 1,233, 1,188, 1,146, 1,038, 1,023, 763, 687; ¹H NMR (400 MHz, DMSO- d_6): 5.12 (s, 1H, C⁴-H), 7.50 (s, 2H, NH₂), 7.52 (s, 1H, ArH), 7.58 (t, J = 8.0 Hz, 2H, ArH), 7.65 (t, J = 7.8 Hz, 1H, ArH), 7.82 (d, J = 8.4 Hz, 1H, ArH), 7.86 (d, J = 7.6 Hz, 2H, ArH), 8.16 (d, J = 8.4 Hz, 1H,

ArH), 8.21 (s, 1H, ArH); Anal. calcd. For $C_{20}H_{12}F_3N_5O_3$: C, 56.21; H, 2.83; N, 16.39. Found: C, 56.15; H, 2.68; N, 16.34.

6-Amino-3-(trifluoromethyl)-1,4-dihydro-4-(4-nitrophenyl)-1-phenylpyrano[2,3c]pyrazole-5-carbonitrile (**4d**)

M.p.: 195–196 °C; IR (potassium bromide) (v, cm⁻¹): 3,425, 3,328, 3,213, 2,202, 1,666, 1,596, 1,582, 1,520, 1,483, 1,458, 1,397, 1,350, 1,311, 1,233, 1,196, 1,145, 1,039, 1,025, 817, 760; ¹H NMR (400 MHz, DMSO- d_6): 5.06 (s, 1H, C⁴-H), 7.51 (s, 3H, NH₂ + ArH), 7.59 (t, J = 8.4 Hz, 4H, ArH), 7.85 (d, J = 7.6 Hz, 2H, ArH), 8.21 (d, J = 8.4 Hz, 2H, ArH); Anal. calcd. For C₂₀H₁₂F₃N₅O₃: C, 56.21; H, 2.83; N, 16.39. Found: C, 56.18; H, 2.78; N, 16.46.

6-Amino-4-(4-chlorophenyl)-3-(trifluoromethyl)-1,4-dihydro-1-phenylpyrano[2,3c]pyrazole-5-carbonitrile (**4e**)

M.p.: 215–217 °C (Lit. [27].: 210–211 °C); IR (potassium bromide) (v, cm⁻¹): 3,448, 3,324, 3,208, 2,202, 1,661, 1,595, 1,580, 1,521, 1,482, 1,458, 1,396, 1,311, 1,233, 1,200, 1,145, 1,090, 1,038, 1,024, 830, 810, 760, 735; ¹H NMR (400 MHz, DMSO- d_6): 4.85 (s, 1H, C⁴-H), 7.31 (d, J = 8.4 Hz, 2H, ArH), 7.39–7.41 (m, 4H, NH₂ + ArH), 7.50 (t, J = 7.2 Hz, 1H, ArH), 7.58 (t, J = 8.0 Hz, 2H, ArH), 7.84 (d, J = 7.6 Hz, 2H, ArH); Anal. calcd. For C₂₀H₁₂ClF₃N₄O: C, 57.64; H, 2.90; N, 13.44. Found: C, 57.58; H, 2.88; N, 13.36.

6-Amino-3-(trifluoromethyl)-4-(4-fluorophenyl)-1,4-dihydro-1-phenylpyrano[2,3c]pyrazole-5-carbonitrile (**4***f*)

M.p.: 218–219 °C; IR (potassium bromide) (v, cm⁻¹): 3,459, 3,327, 3,213, 2,205, 1,666, 1,597, 1,583, 1,523, 1,510, 1,483, 1,459, 1,393, 1,311, 1,278, 1,228, 1,200, 1,145, 1,038, 1,026, 816, 761, 735, 687; ¹H NMR (400 MHz, DMSO- d_6): 4.84 (s, 1H, C⁴-H), 7.14 (t, J = 8.4 Hz, 2H, ArH), 7.31–7.35 (m, 2H, ArH), 7.36 (s, 2H, NH₂), 7.48 (t, J = 7.4 Hz, 1H, ArH), 7.58 (t, J = 7.8 Hz, 2H, ArH), 7.84 (d, J = 8.4 Hz, 2H, ArH); Anal. calcd. For C₂₀H₁₂F₄N₄O: C, 60.00; H, 3.02; N, 14.00. Found: C, 60.08; H, 2.88; N, 13.86.

6-Amino-3-(trifluoromethyl)-1,4-dihydro-4-(3,4,5-trimethoxyphenyl)-1phenylpyrano[2,3-c]pyrazole-5-carbonitrile (**4g**)

M.p.: 149–150 °C; IR (potassium bromide) (v, cm⁻¹): 3,394, 3,325, 3,215, 2,193, 1,654, 1,593, 1,518, 1,482, 1,469, 1,458, 1,427, 1,394, 1,324, 1,311, 1,231, 1,149, 1,130, 1,038, 1,023, 813, 761, 736, 686; ¹H NMR (400 MHz, DMSO- d_6): 3.66 (s, 3H, CH₃O), 3.73 (s, 6H, 2 × CH₃O), 4.78 (s, 1H, C⁴-H), 6.54 (s, 2H, ArH), 7.32 (s, 2H, NH₂), 7.47 (t, J = 7.4 Hz, 1H, ArH), 7.57 (t, J = 7.8 Hz, 2H, ArH), 7.86 (d, J = 8.0 Hz, 2H); Anal. calcd. For C₂₃H₁₉F₃N₄O₄: C, 58.48; H, 4.05; N, 11.86. Found: C, 58.38; H, 3.98; N, 11.76.

6-Amino-3-(trifluoromethyl)-1,4-dihydro-4-(4-(6-amino-5-cyano-3-(trifluoromethyl)-1,4-dihydro-1-phenylpyrano[2,3-c]pyrazol-4-yl)phenyl)-1phenylpyrano[2,3-c]pyrazole-5-carbonitrile (**4**h)

M.p.: 132–133 °C; IR (potassium bromide) (v, cm⁻¹): 3,475, 3,326, 2,200, 1,655, 1,596, 1,522, 1,482, 1,458, 1,389, 1,310, 1,233, 1,185, 1,143, 1,038, 1,022, 801, 759, 685; ¹H NMR (400 MHz, DMSO- d_6): 4.76 (s, 2H, 2 × C⁴-H), 7.20 (d, J = 5.2 Hz, 4H, ArH), 7.33 (s, 4H, 2 × NH₂), 7.49–7.51 (m, 3H, ArH), 7.58–7.62 (m, 4H, ArH), 7.82 (t, J = 8.4 Hz, 4H); Anal. calcd. For C₃₄H₂₀F₆N₈O₂: C, 59.48; H, 2.94; N, 16.32. Found: C, 59.38; H, 2.98; N, 16.47.

6-Amino-3-(trifluoromethyl)-4-(3-fluorophenyl)-1,4-dihydro-1-phenylpyrano[2,3c]pyrazole-5-carbonitrile (**4i**)

M.p.: 211–212 °C; IR (potassium bromide) (v, cm⁻¹): 3,476, 3,335, 3,216, 2,198, 1,661, 1,613, 1,594, 1,581, 1,522, 1,483, 1,458, 1,393, 1,329, 1,309, 1,277, 1,233, 1,198, 1,146, 1,132, 1,119, 1,077, 1,039, 1,024, 943, 890, 835, 795, 783, 763, 733, 695, 679; ¹H NMR (400 MHz, DMSO- d_6): 4.86 (s, 1H, C⁴-H), 7.08–7.19 (m, 3H, ArH), 7.36–7.40 (m, 3H, NH₂ + ArH), 7.48 (t, J = 7.4 Hz, 1H, ArH), 7.58 (t, J = 7.6 Hz, 2H, ArH), 7.85 (d, J = 8.0 Hz, 2H); Anal. calcd. For C₂₀H₁₂F₄N₄O: C, 60.00; H, 3.02; N, 14.00. Found: C, 60.15; H, 3.17; N, 14.06.

6-Amino-3-(trifluoromethyl)-1,4-dihydro-4-(4-hydroxy-3-nitrophenyl)-1phenylpyrano[2,3-c]pyrazole-5-carbonitrile (**4j**)

M.p.: 196–198 °C; IR (potassium bromide) (v, cm⁻¹): 3,416, 3,329, 2,196, 1,662, 1,630, 1,583, 1,483, 1,458, 1,432, 1,396, 1,313, 1,278, 1,233, 1,177, 1,145, 1,076, 1,039, 912, 886, 825, 763, 735, 698, 682, 601; ¹H NMR (400 MHz, DMSO- d_6): 5.10 (s, 1H, C⁴-H), 7.16–7.21 (m, 2H, ArH), 7.31 (q, J = 7.6 Hz, 2H, ArH), 7.44 (s, 2H, NH₂), 7.48 (t, J = 7.4 Hz, 1H, ArH), 7.58 (t, J = 7.8 Hz, 2H, ArH), 7.83 (d, J = 8.0 Hz, 2H, ArH); Anal. calcd. For C₂₀H₁₂F₄N₄O: C, 60.00; H, 3.02; N, 14.00. Found: C, 59.88; H, 3.08; N, 14.17.

6-Amino-3-(trifluoromethyl)-4-(2-fluorophenyl)-1,4-dihydro-1-phenylpyrano[2,3c]pyrazole-5-carbonitrile (**4k**)

M.p.: 212–214 °C; IR (potassium bromide) (v, cm⁻¹): 3,484, 3,335, 2,201, 1,657, 1,615, 1,596, 1,583, 1,522, 1,490, 1,458, 1,393, 1,311, 1,279, 1,232, 1,190, 1,144, 1,102, 1,076, 1,038, 1,021, 912, 896, 863, 757, 741, 697; ¹H NMR (400 MHz, DMSO- d_6): 4.90 (s, 1H, C⁴-H), 7.09 (d, J = 8.8 Hz, 1H, ArH), 7.41 (s, 2H, NH₂), 7.47–7.51 (m, 2H, ArH), 7.57 (t, J = 8.0 Hz, 2H, ArH), 7.84 (d, J = 8.0 Hz, 3H, ArH), 11.01 (s, 1H, OH); Anal. calcd. For C₂₀H₁₂F₃N₅O₄: C, 54.18; H, 2.73; N, 15.80. Found: C, 54.05; H, 2.87; N, 15.68.

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