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Ultrasonic-assisted Cu-catalyzed multicomponent synthesis of furo[3,4-*b*]pyrazolo[4,3-*f*] quinolinones

Abstract: A new series of 10-phenyl-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one derivatives has been synthesized through an efficient multicomponent reaction of tetronic acid, 5-aminoindazole and various aromatic aldehydes catalyzed by copper(II) triflate under ultrasonic irradiation.

Keywords: copper(II) triflate; furo[3,4-*b*]pyrazolo[4,3-*f*] quinolinones; one-pot.

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Introduction

Heterocycles are used as scaffolds for creating pharmacophores to yield potent and selective drugs. Pyrazole derivatives are known for their various biological activities, for example, pyrazolo[3,4-*b*]quinolines are potential antiviral [1] and antimalarial agents [2], and they lower serum cholesterol [3, 4]. Pyrazolo[3,4-*c*]pyrazoles are useful for the treatment of esophageal and gastrointestinal mucosa injury [5], brain injury [6] and are also immunostimulatory [7], antianginal and antitumor agents [8]. By contrast, quinolines are important building blocks in synthetic heterocyclic chemistry and their use in the preparation of pyrazolo[3,4-*b*]quinolines and benzo[*b*][1,8]naphthyridines derivatives has been reported recently [9–12]. In addition, 1*H*-pyrazolo[3,4-*b*]quinoline compounds act as sensors for the fluorescence detection of small inorganic cations such as lithium, sodium, barium and magnesium [13]. Furthermore, pyrazolo[3,4-*f*]quinoline derivatives are a novel class of immunostimulant compounds with potent *in vivo* effects in a murine infection model [5, 6]. Moreover, pyrazolo[4,3-*f*]quinoline derivatives, which are an important class of fused heterocyclic compounds, are antiviral [14] and antibacterial agents [15], acting as potent remedies for treating atherosclerosis or restenosis [16], inflammatory disorders, demyelinating disorders and cancers [17].

Multicomponent reactions (MCRs) play a crucial role in modern synthetic organic chemistry because they occur in a single pot involving the simultaneous molecular interaction of three or more components. They exhibit a high atom economy and selectivity to furnish diverse compounds [18, 19].

During the past few years, the outstanding potential of a variety of metal triflates (trifluoromethylsulfonates) has been realized, being widely used in organic synthesis due to their low toxicity, low cost, high stability and ease of handling [20]. Among the various metal triflates, copper(II) triflate [$Cu(OTf)_2$] plays an indispensable role in the discovery of novel and improved reaction processes [21]. In general, the triflate anion is both weakly nucleophilic and coordinating, rendering the metal counter ion more cationic, thus providing a stronger Lewis acid.

Recently, the synthesis of pyrazolo[4,3-*f*]quinoline derivatives has been reported by Shi and co-workers [22]. However, the synthesis of new heterocyclic compounds containing the furopyrazoloquinolinone scaffold and the development of more efficient entry to these heterocycles are strongly desired. During the course of our studies towards the development of new routes to the synthesis of novel heterocycle compounds using triflate salts [23, 24], herein, we wish to report a rapid and efficient synthesis of a new series of furo[3,4-*b*]pyrazolo[4,3-*f*]quinolinones via one-pot reaction of tetronic acid, aryl aldehydes and 5-aminoindazole under the influence of catalytic amount of copper(II) triflate under ultrasonic irradiation.

Results and discussion

To choose the most appropriate medium in this heterocyclization reaction, we examined the Cu-catalyzed reaction of tetronic acid, 5-aminoindazole and 4-nitrobenzaldehyde

as a model reaction to synthesize 10-(4-nitrophenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one in various solvents at 50°C under ultrasonic irradiation (Table 1). The solvents examined were chloroform, ethanol, tetrahydrofuran, 1,2-dichloroethane and acetonitrile. The reaction in $ClCH_2CH_2Cl$ afforded the target product in 80% yield. When the reaction was carried out in CH_3CN, the reaction was faster and the product was obtained in 90% yield (Table 1, entry 1); it turned out to be the best choice in terms of isolated yield. In the case of other solvents such as $CHCl_3$, THF and ethanol, the target product **8** was obtained in lower yields. Other copper salts including CuCl, CuCN and $Cu(OAc)_2$ were also employed for the model reaction. As shown in Table 1, among the copper salts screened, $Cu(OTf)_2$ showed excellent activity in terms of yield of the desired product and was more efficient than other catalysts (Table 1, entries 6–8). The recyclability of $Cu(OTf)_2$ was also investigated. The catalyst could be reused without any significant loss of activity at least four times.

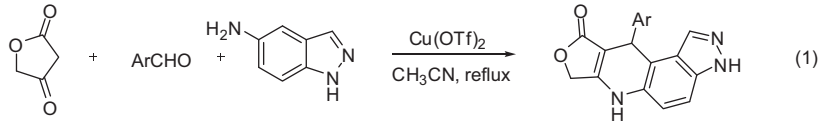
Subsequently, the effect of the amount of $Cu(OTf)_2$ was examined. The optimum molar ratios of tetronic acid, 5-aminoindazole and 4-nitrobenzaldehyde to $Cu(OTf)_2$ were found to be 1:1:1.1:0.1. Under optimized conditions, the reaction was conducted in acetonitrile under ultrasonication. As can be seen from Table 1, the reaction was completed in minutes in the presence of a copper catalyst. It should be noted that in the absence of a catalyst no reaction occurred after 6 h under otherwise similar conditions.

Table 1 Influence of solvent on ultrasonic assisted, Cu-catalyzed (10 mol%) synthesis of 10-(4-nitrophenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (8).

Entry	Catalyst (10 mol%)	Solvent	Reaction time (min)	Yield[a,b]
1	$Cu(OTf)_2$	CH_3CN	10.5	90, 88, 88, 85, 80[c]
2	$Cu(OTf)_2$	THF	20	55
3	$Cu(OTf)_2$	EtOH	19	75
4	$Cu(OTf)_2$	$ClCH_2CH_2Cl$	17	80
5	$Cu(OTf)_2$	$CHCl_3$	20	55
6	CuCl	CH_3CN	20	Trace
7	CuCN	CH_3CN	20	32
8	$Cu(OAc)_2$	CH_3CN	20	~10

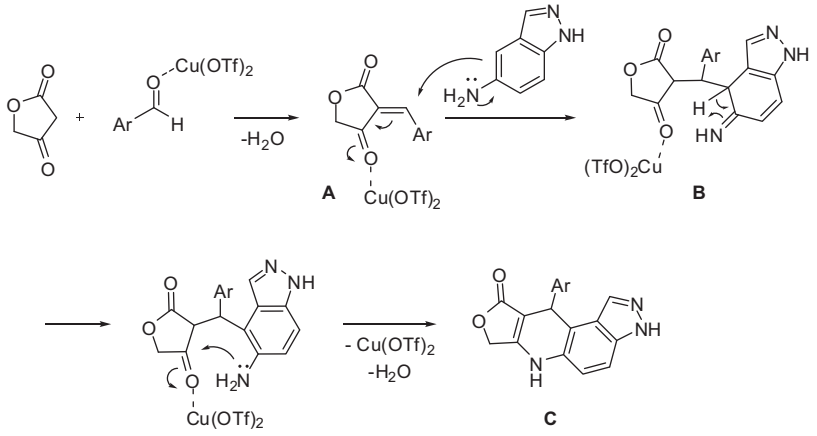
[a]All reactions were carried out at 50°C under ultrasonication.
[b]Isolated yields. [c]Catalyst was reused at least four times.

Various aromatic aldehydes were allowed to react with tetronic acid and 5-aminoindazole, and all these reactions proceeded smoothly to give the corresponding 10-aryl-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one products 1–10 in good to excellent yields (Eq. 1). The electronic nature of substituents on the aromatic aldehydes does not affect the reaction time significantly.



1-10

1: Ar = Ph
2: Ar = 4-$MeOC_6H_4$
3: Ar = 4-MeC_6H_4
4: Ar = 4-BrC_6H_4
5: Ar = 2-BrC_6H_4
6: Ar = 4-ClC_6H_4
7: Ar = 2-ClC_6H_4
8: Ar = 4-$NO_2C_6H_4$
9: Ar = 3-$NO_2C_6H_4$
10: Ar = 2,4-$Cl_2C_6H_3$



Scheme 1

A plausible mechanism for the synthesis of pyrazolo[4,3-*f*]quinolinones is shown in Scheme 1. It can be suggested that in the first step the Knoevenagel condensation generates an intermediate product A. Then, Michael-type addition of 5-aminoindazole to A generates another intermediate product B. Intramolecular cyclization of B followed by dehydration furnishes the observed product C.

Conclusion

A rapid, direct and efficient synthetic route to 10-phenyl-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one was developed. This class of compounds may prove to be of interest for biomedical screening.

Experimental

The reagents were either prepared in our laboratories or purchased from Merck, Fluka and Aldrich Chemical Companies. All yields refer to isolated products. The IR spectra were recorded in KBr disks on a Shimadzu-IR 470 spectrophotometer. The ^{1}H NMR spectra were recorded on a Bruker 100-MHz spectrometer in DMSO-d_6. Flash column chromatography was performed with 300- and 400-mesh silica gel, and analytical thin layer chromatography (TLC) was performed on precoated silica gel plates (60F-254). Sonication was performed in a Shanghai Branson-CQX ultrasonic cleaner with a frequency of 40 kHz and a nominal power of 100 W. Elemental analyses were performed on a Thermo Finnigan EA1112 elemental analyzer.

General procedure for the synthesis of pyrazolo[4,3-*f*]quinolinone derivatives 1–10

A mixture of tetronic acid (0.10 g, 1 mmol), 5-aminoindazole (0.13 g, 1 mmol), aldehyde (1 mmol) and $Cu(OTf)_2$ (0.036 g, 0.1 mmol) in acetonitrile (8 mL) was placed in a 100 mL conical flask and the mixture was stirred at 50°C under ultrasonic irradiation. The course of the reaction was followed by TLC. Upon completion of the reaction, the mixture was extracted with ethyl acetate (20 mL) and the extract was washed with dilute aqueous solution of $NaHCO_3$ (2×10 mL). The aqueous layer containing the catalyst was separated and concentrated under reduced pressure to afford the catalyst which was dried at 75°C for 4 h before a subsequent reuse. The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure to give the crude product, which was purified by column chromatography on silica gel using ethyl acetate-hexane (1:9) as eluent.

10-Phenyl-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (1) After 12 min the yield was 90%; IR: ν 3324, 3155, 1710, 1650, 1573, 1377, 1205, 1144, 1082, 930 cm^{-1}; ^{1}H NMR: δ 11.15 (s, 1H, NH), 9.85 (s, 1H, NH), 8.11 (s, 1H, ArH), 7.25–7.05 (m, 6H, ArH), 6.75 (d, 1H, J = 8 Hz, ArH), 5.60 (s, 1H, CH), 4.95–4.87 (m, 2H, CH_2). Anal. Calcd for $C_{18}H_{13}N_3O_2$: C, 71.28; H, 4.32; N, 13.85. Found: C, 71.19; H, 4.24; N, 13.74.

10-(4-Methoxyphenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (2) After 13 min the yield was 85%; IR: ν 3355, 3183, 1710, 1633, 1545, 1406, 1361, 1233, 1166, 944 cm^{-1}; ^{1}H NMR: δ 11.46 (s, 1H, NH), 10.10 (s, 1H, NH), 7.83 (s, 1H, ArH), 7.35–7.20 (m, 2H, ArH), 7.10–6.96 (m, 2H, ArH), 6.95–6.80 (m, 2H, ArH), 5.37 (s, 1H, CH), 4.94–4.88 (m, 2H, CH_2), 3.62 (s, 3H, OCH_3). Anal. Calcd for $C_{19}H_{15}N_3O_3$: C, 68.46; H, 4.54; N, 12.61. Found: C, 68.22; H, 4.48; N, 12.60.

10-(4-Methylphenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (3) After 12 min the yield was 86%; IR: ν 3314, 3109, 1721, 1657, 1577, 1488, 1372, 1201, 1089, 955 cm^{-1}; ^{1}H NMR: δ 12.73 (s, 1H, NH), 10.32 (s, 1H, NH), 7.90 (s, 1H, ArH), 7.45–7.10 (m, 3H, ArH), 7.04–6.96 (m, 3H, ArH), 5.43 (s, 1H, CH), 4.90–4.84 (m, 2H, CH_2), 2.14 (s, 3H, CH_3). Anal. Calcd for $C_{19}H_{15}N_3O_2$: C, 71.91; H, 4.76; N, 13.24. Found: C, 71.68; H, 4.69; N, 13.12.

10-(4-Bromophenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (4) After 11.5 min the yield was 90%; IR: ν 3313, 3119, 1701, 1671, 1548, 1486, 1306, 1246, 1110, 973 cm^{-1}; ^{1}H NMR: δ 12.65 (s, 1H, NH), 10.80 (s, 1H, NH), 8.10 (s, 1H, ArH), 7.42–7.25 (m, 5H, ArH), 7.04 (d, 1H, J = 7.6 Hz, ArH), 5.70 (s, 1H, CH), 5.06–4.98 (m, 2H, CH_2). Anal. Calcd for $C_{18}H_{12}BrN_3O_2$: C, 56.56; H, 3.16; N, 10.99. Found: C, 56.77; H, 3.21; N, 11.09.

10-(2-Bromophenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (5) After 15 min the yield was 90%; IR: ν 3302, 3109, 1711, 1663, 1567, 1371, 1263, 1217, 1108, 895 cm^{-1}; ^{1}H NMR: δ 12.15 (s, 1H, NH), 10.24 (s, 1H, NH), 8.07 (s, 1H, ArH), 7.63 (d, 1H, J = 8.2 Hz, ArH), 7.50–7.25 (m, 4H, ArH), 7.05 (d, 1H, J = 8.7 Hz, ArH), 5.72 (s, 1H, CH), 4.90–4.85 (m, 2H, CH_2). Anal. Calcd for $C_{18}H_{12}BrN_3O_2$: C, 56.56; H, 3.16; N, 10.99. Found: C, 56.46; H, 3.09; N, 10.91.

10-(4-Chlorophenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (6) After 11.5 min the yield was 87%; IR: ν 3315, 3127, 1704, 1671, 1555, 1475, 1382, 1255, 1201, 1123, 924 cm^{-1}; ^{1}H NMR: δ 12.10 (s, 1H, NH), 10.70 (s, 1H, NH), 8.13 (s, 1H, ArH), 7.52–7.35 (m, 4H, ArH), 7.15–6.97 (m, 2H, ArH), 5.55 (s, 1H, CH), 5.00–4.95 (m, 2H, CH_2). Anal. Calcd for $C_{18}H_{12}ClN_3O_2$: C, 64.01; H, 3.58; N, 12.44. Found: C, 63.87; H, 3.50; N, 12.37.

10-(2-Chlorophenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (7) After 14.5 min the yield was 91%; IR: ν 3372, 3097, 1717, 1653, 1559, 1535, 1337, 1223, 1183, 1070, 956 cm^{-1}; ^{1}H NMR: δ 12.22 (s, 1H, NH), 10.67 (s, 1H, NH), 8.05 (s, 1H, ArH), 7.60–7.25 (m, 4H, ArH), 7.25–7.12 (m, 2H, ArH), 5.48 (s, 1H, CH), 4.97–4.87 (m, 2H, CH_2). Anal. Calcd for $C_{18}H_{12}ClN_3O_2$: C, 64.01; H, 3.58; N, 12.44. Found: C, 63.86; H, 3.51; N, 12.35.

10-(4-Nitrophenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (8) After 10.5 min the yield was 92%; IR: ν 3311, 3106, 1722, 1660, 1561, 1551, 1339, 1263, 1160, 1083, 973 cm^{-1}; ^{1}H NMR: δ 11.75 (s, 1H, NH), 10.34 (s, 1H, NH), 8.10 (s, 1H, ArH), 7.94 (d, 2H, J = 6.8 Hz, ArH), 7.55–7.45 (m, 2H, ArH), 7.30–7.10 (m, 2H, ArH), 5.55 (s, 1H, CH), 4.87–4.80 (m, 2H, CH_2). Anal. Calcd for $C_{18}H_{12}N_4O_4$: C, 62.07; H, 3.47; N, 16.09. Found: C, 61.93; H, 3.40; N, 15.96.

10-(3-Nitrophenyl)-7,9-dihydro-3*H*-furo[3,4-*b*]pyrazolo[4,3-*f*]quinolin-9-one (9) After 12 min the yield was 88%; IR: ν 3312, 3097, 1717, 1653, 1559, 1535, 1337, 1223, 1183, 1070, 956 cm^{-1}; ^{1}H NMR: δ 12.83

(s, 1H, NH), 10.12 (s, 1H, NH), 8.15 (s, 1H, ArH), 8.05–7.90 (m, 2H, ArH), 7.70–7.65 (m, 2H, ArH), 7.35–7.17 (m, 2H, ArH), 5.65 (s, 1H, CH), 4.90–4.82 (m, 2H, CH_2). Anal. Calcd for $C_{18}H_{12}N_4O_4$: C, 62.07; H, 3.47; N, 16.09. Found: C, 61.82; H, 3.39; N, 15.98.

10-(2,6-Dichlorophenyl)-7,9-dihydro-3*H*-furo[3,4-*b*] pyrazolo[4,3-*f*]quinolin-9-one (10) After 17 min the yield was 86%; IR: ν 3305, 3122, 1712, 1667, 1541, 1444, 1275, 1155, 1102, 965 cm^{-1}; ^1H NMR: δ 11.65 (s, 1H, NH), 10.07 (s, 1H, NH), 8.03 (s, 1H, ArH), 7.63 (s, 1H, ArH), 7.44 (m, 3H, ArH), 6.95 (d, 1H, *J* = 7.8 Hz, ArH), 5.74 (s, 1H, CH), 4.93–4.85 (m, 2H, CH_2). Anal. Calcd for $C_{18}H_{11}Cl_2N_3O_2$: C, 58.08; H, 2.98; N, 11.29. Found: C, 57.85; H, 2.93; N, 11.18.

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