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$FeCl_3/ZnI_2$-Catalyzed regioselective synthesis of angularly fused furans†

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The $FeCl_3/ZnI_2$-catalyzed synthesis of angularly fused furans by intermolecular coupling between enols and alkynes has been developed in ambient air. The methodology is successfully applicable to 4-hydroxycoumarin, 4-hydroxyquinolinone and α-tetralone affording regioselective 2-aryl furans in good yields. The control experiments suggest the possibility of a radical reaction mechanism.

Introduction

Coumarin derivatives have great importance as a class of heterocyclic compounds and received major attention from organic chemists due to their vital medicinal values, as antivirals (including anti-HIV), anti-tumor agents, antimicrobials, anti-oxidants, anti-asthmatics, anti-coagulants and anti-inflammatories.[1] These bicyclic scaffolds show opto-electronic properties and lipid lowering activity.[2] They are also used in food additives and cosmetic products.[3]

Likewise, the furans are also privileged heterocyclic scaffolds, as these are ubiquitously present in a vast number of bio-active natural and unnatural compounds.[4] Furan derivatives also serve as building blocks of many pharmaceutical agents.[5] Because of the diversified applications of furan rings, the development of an efficient method for their synthesis has been highly attractive to synthetic chemists until now.[6]

A literature report reveals that the fusion of these two heterocyclic moieties introduces some unique biological and pharmacological properties in the organic molecules, for instance, antimicrobial, insecticidal, antiarrhythmic, antimalarial and sedative[7] (Fig. 1). However, a few number of procedures for achieving the structure of the above-mentioned furocoumarin derivatives have been reported,[8] and most of them either required pre-functionalized starting materials or had a restricted substrate scope. Accessibility of a more convenient and straightforward method for their direct synthesis is highly desirable. In addition, furoquinolinone as an angularly fused tricyclic compound has also some significant role in the field of medicinal chemistry.[9]

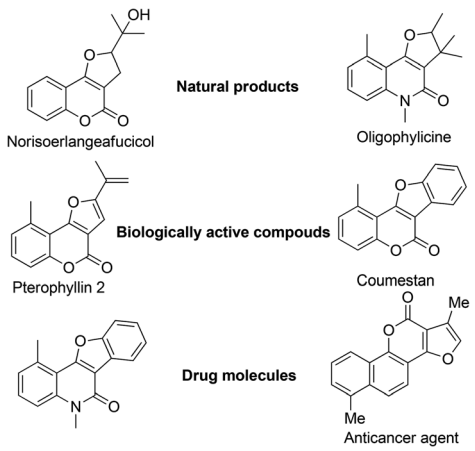

Fig. 1 Structure of bioactive molecules containing furocoumarin and furoquinolinone moieties.

Recently, our group has reported a DABCO-promoted synthesis of 2,3-disubstituted angularly fused furoquinolinones and furocoumarins using nitro-olefin as the Michael acceptor.[10] Based on our research experiences on furan synthesis,[11] we envisioned that alkynes might be good coupling partners with hydroxycoumarin derivatives. However, regioselective synthesis of furan, employing alkynes is a challenging task.[6a] Herein, we report a simple, atom-economical and concise synthetic approach for the preparation of angularly fused furan derivatives by coupling between arylacetylenes and enols in ambient air (Scheme 1).

Results and discussion

To find out the optimized reaction conditions, we began our study with commercially available 4-hydroxycoumarin and phenylacetylene as the model substrates using various catalysts, additives and solvents as summarized in Table 1. The

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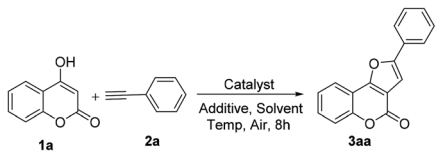
Scheme 1 Synthesis of angularly fused furans.

Table 1 Optimization of the reaction conditions[a]



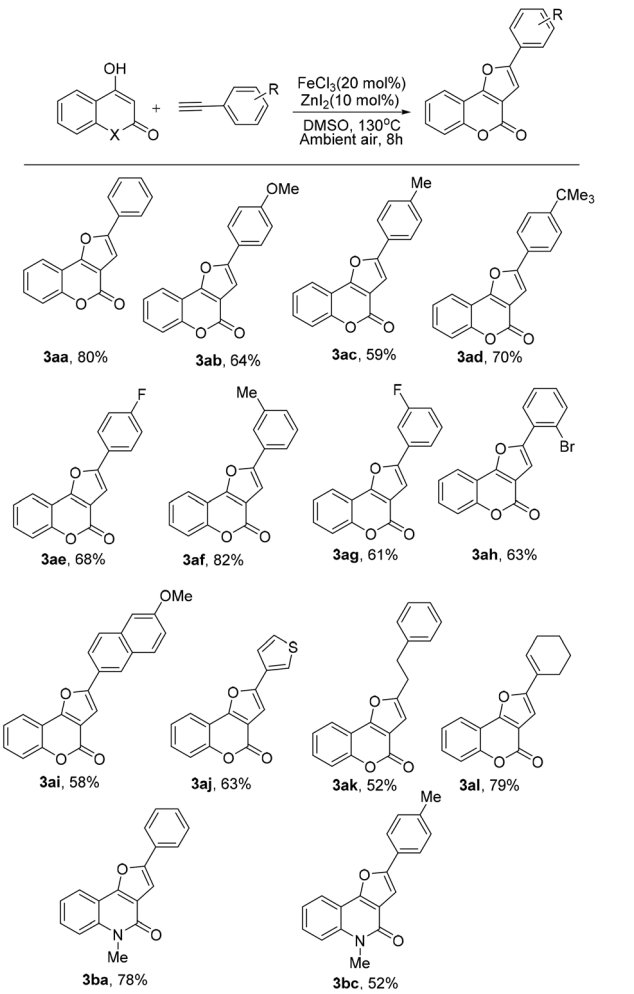
Entry	Catalyst [20 mol%]	Additive [10 mol%]	Solvent	Temp. [°C]	Yield [%]
1	CuI	—	DMSO	130	15
2	$Cu(OAc)_2·H_2O$	—	DMSO	130	35
3	$FeCl_3$	—	DMSO	130	65
4	$FeCl_3$	ZnI_2	DMSO	130	80
5	—	ZnI_2	DMSO	130	NR
6	$FeCl_3$	I_2	DMSO	130	67
7	$FeCl_3$	ZnI_2	Toluene	130	Trace
8	$FeCl_3$	ZnI_2	DMF	130	10
9	$FeCl_3$	ZnI_2	CH_3CN	130	30
10	$FeCl_3$	ZnI_2	1,2-DCB	130	16
11	$FeCl_3$	ZnI_2	DMSO	110	47
12	$FeCl_3$	ZnI_2	DMSO	130	Trace[b]

[a] Reaction conditions: 0.5 mmol of **1a** and 0.5 mmol of **2a** in the presence of a catalyst and an additive in 2 mL solvent at mentioned temperature for 8 h in ambient air. [b] Under an argon atmosphere.

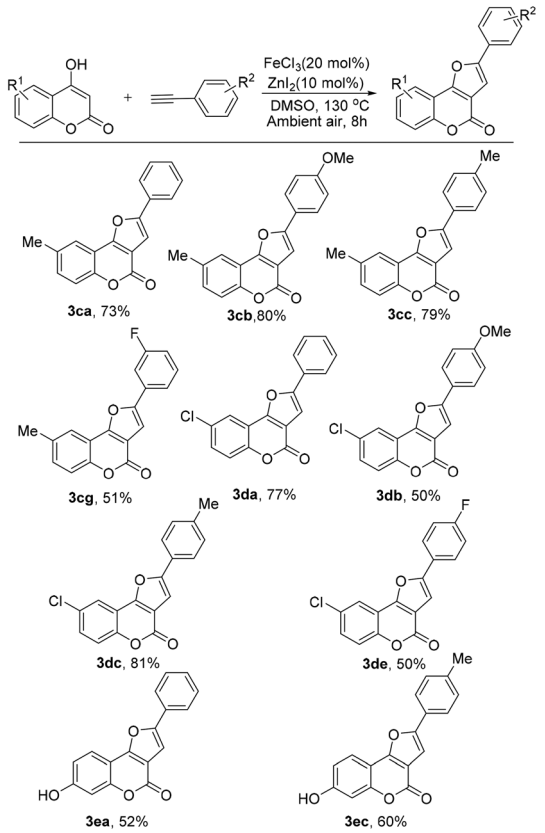
coupling product, 2-phenyl-4*H*-furo[3,2-*c*]chromen-4-one, was obtained as a single regioisomer with 15% yield in the presence of 20 mol% CuI in DMSO at 130 °C (Table 1, entry 1). The use of $Cu(OAc)_2·H_2O$ (20 mol%) increased the yield of the desired product up to 35% (Table 1, entry 2). Next we checked the catalytic activity of $FeCl_3$ as it is economical, environmentally benign and also highly abundant in nature.[12] Interestingly, the reaction with 20 mol% $FeCl_3$ afforded 65% yield (Table 1, entry 3). To our delight, the yield of **3aa** was improved to 80% using 10 mol% of ZnI_2 as the additive[13] (Table 1, entry 4). Significantly, the reaction did not proceed at all in the absence of $FeCl_3$ (Table 1, entry 5). Instead of ZnI_2 when I_2 (10 mol%) was used as the additive the yield was dropped to 67% (Table 1, entry 6). The other solvents like toluene, DMF, CH_3CN and 1,2-DCB were also screened. However, these are not so effective like DMSO (Table 1, entries 7–10). The yield was diminished by lowering the temperature (Table 1, entry 11). Moreover, only the formation of a trace amount of product was observed under an argon atmosphere (Table 1, entry 12). So, finally, carrying out the reaction employing 20 mol% of $FeCl_3$ and 10 mol% of ZnI_2 in DMSO at 130 °C is found to provide the optimized yield of the desired compound (Table 1, entry 4).

After having the optimized reaction conditions in hand, we began to investigate the substrate scope for the annulation of 4-hydroxycoumarin with various substituted phenylacetylenes (Scheme 2). For example, 4-substituted phenylacetylenes (-methoxy, -methyl, *-tert*-butyl, and -fluoro) worked well to give the desired furocoumarins (**3ab–3ae**) with 59–70% yields. 3-Substituted phenylacetylenes (-methyl and -fluoro) and 2-bromo phenylacetylene also provided good yields of the corresponding desired products (**3af–3ah**). Pleasingly, 2-ethynyl-6-methoxynaphthalene and 3-ethynylthiophene also responded well under the present reaction conditions (**3ai** and **3aj**). The methodology was also applied to aliphatic as well as alicyclic ethynyl groups to produce the corresponding furans **3ak** and **3al** in 52% and 79% yields respectively. 4-Hydroxy-1-methylquinolin-2-one also effectively coupled with phenylacetylene and 4-ethynyltoluene to provide the desired furoquinolinones **3ba** and **3bc** in good yields. However, 4-hydroxy-6-methylpyran-2-one did not work in this protocol.

We also explored the general applicability of this methodology employing different substituted 4-hydroxycoumarins



Scheme 2 Substrate scope exploring different arylacetylenes. Reaction conditions: 0.5 mmol of **1** and 0.5 mmol of **2** in the presence of $FeCl_3$ (20 mol%) and ZnI_2 (10 mol%) in 2 mL DMSO at 130 °C for 8 h in ambient air.

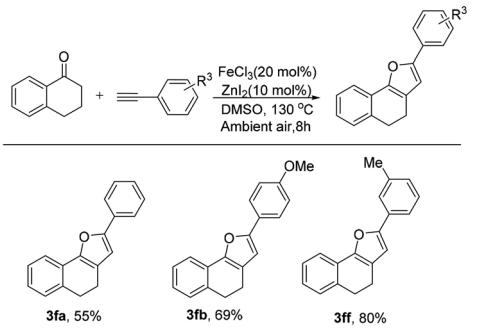


Scheme 3 Substrate scope exploring different 4-hydroxy coumarins and arylacetylenes. Reaction conditions: 0.5 mmol of **1** and 0.5 mmol of **2** in the presence of $FeCl_3$ (20 mol%) and ZnI_2 (10 mol%) in 2 mL DMSO at 130 °C for 8 h in ambient air.

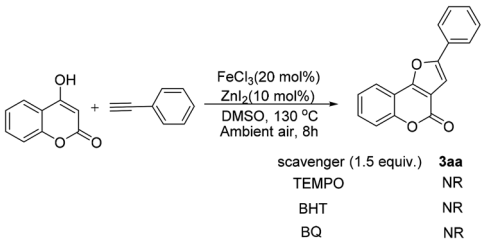
(Scheme 3). 4-Hydroxy-6-methylcoumarin reacted well with various substituted (4-methoxy, 4-methyl, and 3-fluoro) phenylacetylenes to give the desired derivatives smoothly (**3cb–3cg**). Similarly, reactions between 6-chloro-4-hydroxy-coumarin and arylacetylenes were also compatible and the desired furans (**3da**, **3db**, **3dc** and **3de**) were formed in good yields. The reactions of 4,7-dihydroxycoumarin with phenylacetylene and 4-ethynyltoluene were also successful to provide 52% and 60% yield of **3ea** and **3ec** respectively.

Next, we checked the feasibility of α-tetralone as the coupling partner with different arylacetylenes under the standard reaction conditions. The desired 2-phenyl-4,5-dihydronaphtho[1,2-*b*]furans (**3fa**, **3fb** and **3ff**) were obtained in high yields. It is worth mentioning that no aromatised product was obtained in this case.[14] Moreover, only 2-substituted furans were obtained for all the cases and internal alkynes did not take part under these reaction conditions (Scheme 4).

Few control experiments were carried out to understand the probable reaction mechanism, and the results are summarized in Scheme 5. No desired product was obtained on treatment with radical scavengers such as TEMPO (1.5 equiv.), BHT (1.5 equiv.) and BQ (1.5 equiv.). These results indicate the possibility of a radical pathway of the present reaction.

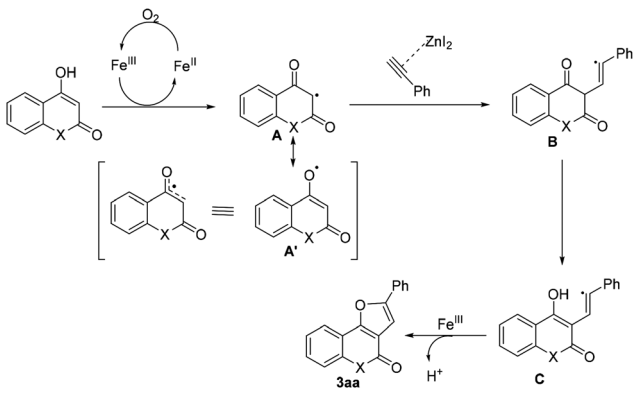


Scheme 4 Synthesis of 2-phenyl-4,5-dihydronaphtho[1,2-*b*]furans. Reaction conditions: 0.5 mmol of **1** and 0.5 mmol of **2** in the presence of $FeCl_3$ (20 mol%) and ZnI_2 (10 mol%) in 2 mL DMSO at 130 °C for 8 h in ambient air.



Scheme 5 Control experiments. Reaction conditions: 0.5 mmol of **1a** and 0.5 mmol of **2a** and 0.75 mmol scavenger in the presence of $FeCl_3$ (20 mol%) and ZnI_2 (10 mol%) in 2 mL DMSO at 130 °C for 8 h in ambient air.

A plausible reaction mechanism has been outlined in Scheme 6 on the basis of control experiments and literature reports.[6i,8g,15] First, the hydroxycoumarin is possibly converted into a carbon-centered radical **A** in the presence of Fe(III) and then the radical **A** reacted with phenylacetylene to form a vinyl radical intermediate **B**. Subsequently, it tautomerized to **C** which intramolecularly coupled with a hydroxy group to construct the furan **3aa**. Fe(III) is regenerated from Fe(II) in the presence of ambient air.



Scheme 6 The possible reaction pathway.

Conclusions

In conclusion, we have demonstrated a convenient and versatile method for the synthesis of angularly fused furans from arylacetylenes and different enols, including 4-hydroxycoumarins and 4-hydroxyquinolinones with good yields using $FeCl_3/ZnI_2$ catalysts *via* an oxidative radical process. The present protocol is also able to produce furan from α-tetralone. High regioselectivity, cost-effectiveness, atom-efficiency, broad substrate scope and availability of starting materials are the notable advantages of this method which provide a novel synthetic route to create a library of angularly fused furan derivatives.

Experimental section

General information

^{1}H NMR spectra were determined on a 400 MHz spectrometer in $CDCl_3$ or DMSO-d_6 solution. Chemical shifts are expressed in parts per million (δ) and are referenced to tetramethylsilane (TMS) as an internal standard and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet), dd (doublet of doublets) and coupling constants J were given in Hz. Proton-decoupled $^{13}C\{^1H\}$ NMR spectra were recorded at 100 MHz in $CDCl_3$ solution or in DMSO-d_6 solution. Chemical shifts are referenced to $CDCl_3$ (δ = 7.26 for ^{1}H and δ = 77.16 for $^{13}C\{^1H\}$ NMR) and DMSO-d_6 (δ = 2.50 for ^{1}H and δ = 39.52 for $^{13}C\{^1H\}$ NMR) as internal standards. TLC was monitored with aluminium backed silica gel 60 (HF_{254}) plates (0.25 mm). Silica gel (60–120 mesh) was used for column chromatography. Petroleum ether (PE) refers to the fractional boiling in the range of 60–80 °C unless otherwise mentioned. All solvents were dried and distilled before use. Commercially available substrates were freshly distilled before the reaction. All reactions involving moisture sensitive reactants were executed using oven dried glassware.

Experimental procedure

A mixture of **1** (0.5 mmol), **2** (0.5 mmol), $FeCl_3$ (20 mol%, 16 mg) and ZnI_2 (10 mol%, 16 mg) in DMSO (2 mL) was placed in an oven dried reaction tube. Then the reaction mixture was stirred at 130 °C for 8 h in ambient air. After completion of the reaction (TLC), the reaction mixture was cooled to room temperature and extracted with ethyl acetate (EA). The organic phase was dried over anhydrous Na_2SO_4. The crude residue was obtained after evaporation of the solvent under vacuum and was purified by column chromatography on silica gel (60–120 mesh) using petroleum ether as the eluent to afford a pure product.

2-Phenyl-4*H*-furo[3,2-*c*]chromen-4-one (3aa).[8h] White solid (80%, 105 mg), R_f = 0.4 (PE : EA = 8 : 2), mp 186–187 °C (lit. mp 180–181 °C), IR (KBr): ν 1737, 1629, 1604, 1528, 1499 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (dd, J = 8.0, 1.6 Hz, 1H), 7.84–7.81 (m, 2H), 7.54–7.46 (m, 4H), 7.42–7.39 (m, 2H), 7.19 (s, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 158.4, 152.7, 141.3, 130.7, 129.3, 129.1, 128.7, 124.75, 124.71, 124.6, 120.9, 117.5, 117.2, 112.9, 102.8.

2-(4-Methoxyphenyl)-4*H*-furo[3,2-*c*]chromen-4-one (3ab).[8i] White solid (64%, 93 mg), R_f = 0.4 (PE : EA = 7 : 3), mp 188–189 °C, IR (KBr): ν 3108, 2359, 1739, 1628, 1490, 754 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (dd, J = 7.6, 1.6 Hz, 1H), 7.76–7.73 (m, 2H), 7.54–7.49 (m, 1H), 7.47–7.44 (m, 1H), 7.39–7.35 (m, 1H), 7.04 (s, 1H), 7.01–6.99 (m, 2H), 3.87 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 160.5, 156.9, 152.6, 140.7, 130.4, 130.0, 126.3, 124.6, 124.5, 121.9, 120.8, 117.5, 114.6, 114.1, 101.1, 55.5; HRMS (ESI-TOF) m/z: $[M + H]^+$ calcd for $C_{18}H_{13}O_4$: 293.0808; found: 293.0809.

2-(*p*-Tolyl)-4*H*-furo[3,2-*c*]chromen-4-one (3ac).[8f] White solid (59%, 81 mg), R_f = 0.4 (PE : EA = 7 : 3), mp 190–192 °C, IR (KBr): ν 3060, 2921, 1737, 1605, 1187 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (dd, J = 8.0, 1.6 Hz, 1H), 7.70 (d, J = 8.0 Hz, 2H), 7.54–7.50 (m, 1H), 7.47–7.44 (m, 1H), 7.39–7.35 (m, 1H), 7.28 (d, J = 8.0 Hz, 2H), 7.12 (s, 1H), 2.41 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 158.5, 157.0, 156.7, 152.6, 139.5, 130.6, 129.8, 129.7, 126.4, 124.7, 120.9, 117.5, 112.9, 112.6, 102.0, 21.5.

2-(4-(*tert*-Butyl)phenyl)-4*H*-furo[3,2-*c*]chromen-4-one (3ad).[8i] Yellow solid (70%, 111 mg), R_f = 0.4 (PE : EA = 9 : 1), mp 191–192 °C, IR (KBr): ν 3110, 2959, 2357, 1742, 1625, 1494, 747 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (dd, J = 8.0, 1.6 Hz, 1H), 7.76–7.73 (m, 2H), 7.52–7.49 (m, 3H), 7.46–7.44 (m, 1H), 7.40–7.36 (m, 1H), 7.13 (s, 1H), 1.36 (s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 158.4, 157.0, 156.7, 152.67, 152.64, 130.5, 129.0, 126.3, 126.0, 124.6, 124.5, 123.8, 120.8, 117.4, 112.6, 102.1, 34.9, 31.3.

2-(4-Fluorophenyl)-4*H*-furo[3,2-*c*]chromen-4-one (3ae).[8f] White solid (68%, 95 mg), R_f = 0.4 (PE : EA = 8 : 2), mp 210–215 °C, IR (KBr): ν 3111, 2352, 1748, 1496, 1233, 754 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (dd, J = 8.0, 1.6 Hz, 1H), 7.82–7.78 (m, 2H), 7.56–7.52 (m, 1H), 7.47–7.45 (m, 1H), 7.41–7.37 (m, 1H), 7.20–7.16 (m, 2H), 7.12 (s, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 163.4 (J_{C-F} = 219 Hz), 157.0, 155.8, 152.7, 131.7, 130.8, 126.6 (J_{C-F} = 8 Hz), 125.4, 124.7, 121.9, 120.9, 117.5, 116.3 (J_{C-F} = 22 Hz), 112.7 (J_{C-F} = 16 Hz), 102.5.

2-(*m*-Tolyl)-4*H*-furo[3,2-*c*]chromen-4-one (3af). White solid (82%, 113 mg), R_f = 0.4 (PE : EA = 8 : 2), mp 168–170 °C, IR (KBr): ν 3792, 2321, 1729, 1606, 1479 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (dd, J = 8.0, 1.6 Hz, 1H), 7.60 (d, J = 8.0 Hz, 2H), 7.54–7.50 (m, 1H), 7.46–7.44 (m, 1H), 7.39–7.34 (m, 2H), 7.20 (d, J = 7.6 Hz, 1H), 7.15 (s, 1H), 2.44 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 158.4, 156.9, 152.7, 138.9, 130.6, 130.1, 129.0, 128.9, 125.2, 124.6, 121.9, 121.4, 120.9, 117.5, 117.3, 112.9, 102.6, 21.6; anal. calcd for $C_{18}H_{12}O_3$: C, 78.25; H, 4.38%; found: C, 78.15; H, 4.41%.

2-(3-Fluorophenyl)-4*H*-furo[3,2-*c*]chromen-4-one (3ag). Yellow solid (61%, 85 mg), R_f = 0.4 (PE : EA = 8 : 2), mp 188–190 °C, IR (KBr): ν 3050, 2362, 1733, 1620, 1488, 1176, 754 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (dd, J = 8.0, 1.6 Hz, 1H), 7.59–7.48 (m, 3H), 7.47–7.37 (m, 3H), 7.20 (s, 1H), 7.11–7.06 (m, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 163.2 (J_{C-F} = 245 Hz), 157.7 (J_{C-F} = 88 Hz), 155.3, 152.8, 131.0, 130.9 (J_{C-F} = 8 Hz), 124.8,

124.6, 120.4, 120.6 (J_{C-F} = 63 Hz), 117.5, 116.1 (J_{C-F} = 22 Hz), 112.6 (J_{C-F} = 11 Hz), 111.7, 111.5, 103.9; anal. calcd for $C_{17}H_9FO_3$: C, 72.86; H, 3.24%; found: C, 72.99; H, 3.22%.

2-(2-Bromophenyl)-4*H*-furo[3,2-*c*]chromen-4-one (3ah). White solid (63%, 107 mg), R_f = 0.45 (PE : EA = 8 : 2), mp 188–190 °C, IR (KBr): ν 3423, 2912, 1734, 1591, 1184, 755 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 7.96 (dd, J = 8.0, 1.6 Hz, 1H), 7.70–7.67 (m, 2H), 7.63–7.60 (m, 2H), 7.53–7.51 (m, 1H), 7.48–7.46 (m, 1H), 7.41–7.37 (m, 1H), 7.20 (s, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 174.9, 162.3, 157.3, 137.1, 133.9, 132.4, 131.9, 131.0, 126.1, 124.8, 120.9, 119.7, 117.9, 117.6, 111.4, 103.4; anal. calcd for $C_{17}H_9BrO_3$: C, 59.85; H, 2.66%; found: C, 59.99; H, 2.59%.

2-(6-Methoxynaphthalen-2-yl)-4*H*-furo[3,2-*c*]chromen-4-one (3ai).[8f] Yellow solid (58%, 99 mg), R_f = 0.35 (PE : EA = 7 : 3), mp 170–172 °C, IR (KBr): ν 3110, 2357, 1735, 1620, 1487, 1180, 753 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 7.99 (s, 1H), 7.92 (dd, J = 8.0, 1.6 Hz, 1H), 7.69 (t, J = 9.6 Hz, 2H), 7.52 (dd, J = 8.4, 1.6 Hz, 2H), 7.26 (s, 1H), 7.17 (dd, J = 8.8, 2.4 Hz, 1H), 7.11 (d, J = 2.4 Hz, 1H), 7.03–7.00 (m, 1H), 6.95–6.91 (m, 1H), 3.93 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 162.3, 158.8, 139.6, 137.0, 134.7, 133.2, 132.9, 131.2, 131.0, 130.9, 129.5, 129.3, 128.4, 127.1, 122.1, 121.0, 119.9, 119.78, 119.70, 117.9, 106.0, 55.5.

2-(Thiophen-3-yl)-4*H*-furo[3,2-*c*]chromen-4-one (3aj). White solid (63%, 84 mg), R_f = 0.4 (PE : EA = 7 : 3), mp 188–190 °C, IR (KBr): ν 3423, 2912, 1734, 1591, 1184, 755 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, J = 7.6 Hz, 1H), 7.719–7.712 (m, 1H), 7.53–7.48 (m, 1H), 7.44–7.40 (m, 3H), 7.36 (t, J = 7.6 Hz, 1H), 6.98 (s, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 167.7, 158.3, 156.5, 153.4, 152.7, 130.6, 128.9, 127.2, 124.8, 124.6, 121.9, 120.8, 117.4, 112.8, 112.4, 102.4; anal. calcd for $C_{15}H_8O_3S$: C, 67.15; H, 3.01%; found: C, 66.99; H, 2.94%.

2-Phenyl-4*H*-furo[3,2-*c*]chromen-4-one (3ak). Yellow solid (52%, 75 mg), R_f = 0.55 (PE : EA = 8 : 2), mp 170–172 °C, IR (KBr): ν 3110, 2359, 1741, 754 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.15 (dd, J = 8.4, 1.6 Hz, 1H), 8.06 (dd, J = 8.0, 1.6 Hz, 1H), 7.82 (s, 1H), 7.67–7.63 (m, 1H), 7.61–7.56 (m, 1H), 7.51–7.49 (m, 1H), 7.46–7.42 (m, 1H), 7.28–7.25 (m, 1H), 7.11–7.09 (m, 1H), 7.05–7.01 (m, 1H), 1.67–1.50 (m, 2H), 0.87–0.83 (m, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 163.4, 159.9, 157.4, 153.9, 137.2, 132.9, 130.9, 128.2, 127.7, 125.2, 122.1, 119.6, 119.0, 118.3, 117.9, 22.7, 14.2; anal. calcd for $C_{19}H_{14}O_3$: C, 78.61; H, 4.86%; found: C, 78.82; H, 4.72%.

2-(Cyclohex-1-en-1-yl)-4*H*-furo[3,2-*c*]chromen-4-one (3al).[8i] White solid (79%, 105 mg), R_f = 0.5 (PE : EA = 9 : 1), mp 168–170 °C, IR (KBr): ν 2946, 2832, 1743, 1645, 1498, 754 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 7.87 (dd, J = 8.0, 1.6 Hz, 1H), 7.49–7.47 (m, 1H), 7.44–7.42 (m, 1H), 7.36–7.32 (m, 1H), 6.67 (s, 1H), 6.63–6.60 (m, 1H), 2.37–2.26 (m, 4H), 1.83–1.67 (m, 4H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 158.2, 152.6, 136.3, 132.3, 130.4, 126.8, 124.5, 123.1, 120.8, 117.4, 116.5, 113.0, 101.2, 29.1, 24.9, 22.2, 19.3.

5-Methyl-2-phenylfuro[3,2-*c*]quinolin-4(5*H*)-one (3ba).[8g] White solid (78%, 107 mg), R_f = 0.35 (PE : EA = 7 : 3), mp 202–203 °C (lit. mp 205 °C), IR (KBr): ν 3582, 2316, 1733, 1606, 1174, 753 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.11–8.09 (m, 1H), 7.86–7.83 (m, 2H), 7.57–7.54 (m, 1H), 7.48–7.44 (m, 3H), 7.38–7.34 (m, 2H), 7.29 (s, 1H), 3.81 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 159.5, 155.7, 154.6, 138.3, 131.8, 131.0, 129.5, 129.0, 128.7, 124.6, 122.5, 121.3, 115.2, 114.2, 103.0, 29.6.

5-Methyl-2-(*p*-tolyl)furo[3,2-*c*]quinolin-4(5*H*)-one (3bc). White solid (52%, 75 mg), R_f = 0.4 (PE : EA = 8 : 2), mp >250 °C, IR (KBr): ν 3389, 2309, 1738, 1614, 1101, 755 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 8.11 (dd, J = 8.0, 1.6 Hz, 1H), 7.74 (d, J = 8.4 Hz, 2H), 7.61–7.56 (m, 2H), 7.47 (d, J = 8.4 Hz, 1H), 7.39–7.35 (m, 1H), 7.29 (d, J = 8.0 Hz, 1H), 7.24 (s, 1H), 3.82 (s, 3H), 2.41 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 138.8, 138.5, 131.0, 129.7, 129.4, 125.4, 124.6, 124.5, 123.9, 122.6, 122.5, 121.2, 115.2, 114.3, 102.2, 30.2, 21.5; anal. calcd for $C_{19}H_{15}NO_2$: C, 78.87; H, 5.23; N, 4.84%; found: C, 79.09; H, 5.31; N, 4.68%.

8-Methyl-2-phenyl-4*H*-furo[3,2-*c*]chromen-4-one (3ca).[8i] Yellow solid (73%, 101 mg), R_f = 0.45 (PE : EA = 8 : 2), mp 187–188 °C, IR (KBr): ν 1737, 1638, 1565, 1510, 1428, 755 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 7.82–7.80 (m, 2H), 7.74 (s, 1H), 7.49–7.45 (m, 2H), 7.41–7.37 (m, 1H), 7.35–7.30 (m, 2H), 7.16 (s, 1H), 2.48 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 158.6, 157.0, 156.5, 150.9, 134.5, 131.8, 131.0, 129.2, 129.1, 128.9, 124.6, 120.6, 117.2, 112.5, 102.8, 21.1.

2-(4-Methoxyphenyl)-8-methyl-4*H*-furo[3,2-*c*]chromen-4-one (3cb). Yellow solid (80%, 122 mg), R_f = 0.4 (PE : EA = 7 : 3), mp 170–172 °C, IR (KBr): ν 3006, 2333, 1738, 1626, 1182, 754 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 7.76–7.73 (m, 3H), 7.35–7.31 (m, 2H), 7.03 (s, 1H), 7.01–6.98 (m, 2H), 3.87 (s, 3H), 2.48 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 160.4, 156.7, 150.8, 138.2, 134.5, 134.0, 131.5, 130.7, 130.4, 126.2, 120.5, 117.1, 114.5, 113.8, 101.1, 55.6, 21.1; anal. calcd for $C_{19}H_{14}O_4$: C, 74.50; H, 4.61%; found: C, 74.79; H, 4.57%.

8-Methyl-2-(*p*-tolyl)-4*H*-furo[3,2-*c*]chromen-4-one (3cc). White solid (79%, 114 mg), R_f = 0.45 (PE : EA = 9 : 1), mp 185–187 °C, IR (KBr): ν 3761, 2920, 1733, 1603, 1181, 756 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 7.73–7.68 (m, 3H), 7.32–7.25 (m, 4H), 7.09 (s, 1H), 2.47 (s, 3H), 2.41 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 158.6, 156.8, 150.9, 139.4, 134.5, 131.6, 129.8, 126.4, 124.6, 120.6, 117.2, 112.6, 112.5, 102.0, 21.5, 21.1; anal. calcd for $C_{19}H_{14}O_3$: C, 78.61; H, 4.86%; found: C, 78.47; H, 4.93%.

2-(3-Fluorophenyl)-8-methyl-4*H*-furo[3,2-*c*]chromen-4-one (3cg). Yellow solid, (51%, 75 mg), R_f = 0.45 (PE : EA = 8 : 2), mp 199–200 °C, IR (KBr): ν 3107, 2923, 2357, 1733, 1592, 1488, 772 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ 7.75 (s, 1H), 7.60–7.57 (m, 1H), 7.54–7.51 (m, 1H), 7.47–7.42 (m, 1H), 7.35 (s, 2H), 7.21 (s, 1H), 7.11–7.06 (m, 1H), 2.49 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 134.7, 132.1, 130.9 (J_{C-F} = 8 Hz), 120.7, 120.3 (J_{C-F} = 2 Hz), 119.4, 117.3, 117.0 (J_{C-F} = 22 Hz), 116.1, 115.9, 112.4, 111.7, 111.5, 103.9, 21.1; anal. calcd for $C_{18}H_{11}FO_3$: C, 73.47; H, 3.77%; found: C, 73.68; H, 3.72%.

8-Chloro-2-phenyl-4*H*-furo[3,2-*c*]chromen-4-one (3da).[8j] Yellow solid (77%, 114 mg), R_f = 0.35 (PE : EA = 7 : 3), mp 174–175 °C, IR (KBr): ν 3414, 3095, 1738, 1603, 1192 cm^{-1}.

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, J = 2.4 Hz, 1H), 7.83–7.81 (m, 2H), 7.49–7.46 (m, 3H), 7.44–7.39 (m, 2H), 7.19 (s, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 157.8, 157.4, 155.6, 132.6, 131.0, 130.6, 129.5, 129.3, 129.2, 128.9, 128.5, 124.8, 120.4, 118.9, 102.8.

8-Chloro-2-(4-methoxyphenyl)-4*H*-furo[3,2-*c*]chromen-4-one (3db).[8f] White solid (50%, 82 mg), R_f = 0.35 (PE : EA = 7 : 3), mp 178–179 °C, IR (KBr): ν 3582, 2316, 1733, 1606, 1174, 753 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, J = 2.4 Hz, 1H), 7.77–7.73 (m, 2H), 7.45–7.43 (m, 1H), 7.39 (d, J = 8.8 Hz, 1H), 7.04 (s, 1H), 7.02–6.99 (m, 2H), 3.88 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 163.5, 160.7, 157.4, 134.1, 132.7, 130.6, 130.4, 130.2, 126.3, 120.2, 118.8, 114.6, 114.3, 113.7, 101.0, 55.5.

8-Chloro-2-(*p*-tolyl)-4*H*-furo[3,2-*c*]chromen-4-one (3dc). White solid (81%, 125 mg), R_f = 0.5 (PE : EA = 8 : 2), mp 212–214 °C, IR (KBr): ν 2962, 1749, 1489, 981, 817 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, J = 2.4 Hz, 1H), 7.71–7.69 (m, 2H), 7.47–7.44 (m, 1H), 7.39 (d, J = 8.8 Hz, 1H), 7.29 (d, J = 8.0 Hz, 2H), 7.12 (s, 1H), 2.42 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 157.6, 155.4, 150.9, 139.8, 130.4, 130.2, 129.9, 129.6, 126.0, 124.7, 121.2, 120.3, 118.9, 113.9, 102.0, 21.5; anal. calcd for $C_{18}H_{11}ClO_3$: C, 69.58; H, 3.57%; found: C, 69.77; H, 3.48%.

8-Chloro-2-(4-fluorophenyl)-4*H*-furo[3,2-*c*]chromen-4-one (3de). Yellow solid (50%, 78 mg), R_f = 0.4 (PE : EA = 7 : 3), mp 234 °C, IR (KBr): ν 3080, 2960, 1757, 1496, 1235, 982, 756 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, J = 2.4 Hz, 1H), 7.83–7.78 (m, 2H), 7.48 (dd, J = 8.8, 2.4 Hz, 1H), 7.40 (d, J = 8.8 Hz, 1H), 7.22–7.16 (m, 2H), 7.13 (s, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 163.4 (J_{C-F} = 249 Hz), 151.0, 136.6 (J_{C-F} = 147 Hz), 132.4, 130.7, 130.3, 127.2, 126.8 (J_{C-F} = 8 Hz), 125.1, 120.4, 119.0, 118.7, 116.5 (J_{C-F} = 22 Hz), 113.6 (J_{C-F} = 45 Hz), 102.6; anal. calcd for $C_{17}H_8ClFO_3$: C, 64.88; H, 2.56%; found: C, 65.05; H, 2.62%.

7-Hydroxy-2-phenyl-4*H*-furo[3,2-*c*]chromen-4-one (3ea). White solid (52%, 72 mg), R_f = 0.4 (PE : EA = 8 : 2), mp >250 °C, IR (KBr): ν 3218, 2357, 1731, 1593, 1270, 680 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.62 (s, 1H), 7.95–7.90 (m, 3H), 7.58 (s, 1H), 7.53–7.49 (m, 2H), 7.43–7.38 (m, 1H), 6.92 (dd, J = 8.8, 2.4 Hz, 1H), 6.88 (d, J = 2.0 Hz, 1H); $^{13}C\{^1H\}$ NMR (100 MHz, DMSO-d_6): δ 160.6, 154.6, 154.0, 131.5, 129.0, 128.8, 128.7, 128.6, 124.1, 122.2, 113.6, 104.2, 103.1, 103.0, 71.1; anal. calcd for $C_{17}H_{10}O_4$: C, 73.38; H, 3.62%; found: C, 73.17; H, 3.69%.

7-Hydroxy-2-(*p*-tolyl)-4*H*-furo[3,2-*c*]chromen-4-one (3ec). White solid (60%, 87 mg), R_f = 0.45 (PE : EA = 8 : 2), mp >250 °C, IR (KBr): ν 3307, 2358, 1701, 1629, 1456, 1265, 757 cm^{-1}. ^{1}H NMR (400 MHz, DMSO-d_6): δ 10.57 (s, 1H), 7.88 (d, J = 8.4 Hz, 1H), 7.81 (d, J = 8.0 Hz, 2H), 7.48 (s, 1H), 7.31 (d, J = 8.0 Hz, 2H), 6.91 (dd, J = 8.8, 2.4 Hz, 1H), 6.86 (d, J = 2.4 Hz, 1H), 2.35 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, DMSO-d_6): δ 160.5, 157.6, 157.1, 154.9, 153.9, 138.5, 129.6, 126.0, 124.1, 122.2, 113.5, 108.8, 104.2, 103.0, 102.2, 20.9; anal. calcd for $C_{18}H_{12}O_4$: C, 73.97; H, 4.14%; found: C, 74.12; H, 4.19%.

2-Phenyl-4,5-dihydronaphtho[1,2-*b*]furan (3fa).[11a] Colorless oil (55%, 68 mg), R_f = 0.6 (PE), IR (KBr): ν 3056, 2932, 2357, 1488, 757 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06–8.02 (m, 1H), 7.84–7.81 (m, 2H), 7.51–7.47 (m, 2H), 7.37–7.33 (m, 2H), 7.29 (d, J = 6.8 Hz, 1H), 7.24–7.20 (m, 1H), 6.73 (s, 1H), 3.09 (t, J = 8.0 Hz, 2H), 2.87 (t, J = 8.4 Hz, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 153.3, 149.8, 134.8, 128.9, 128.8, 128.0, 127.2, 126.8, 126.4, 124.8, 123.7, 121.6, 119.2, 106.6, 102.6, 29.1, 21.1.

2-(4-Methoxyphenyl)-4,5-dihydronaphtho[1,2-*b*]furan (3fb). Colorless oil (69%, 95 mg), R_f = 0.5 (PE : EA = 9 : 1). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59–7.55 (m, 2H), 7.45 (d, J = 8.8 Hz, 1H), 7.17–7.14 (m, 1H), 7.10 (d, J = 7.6 Hz, 1H), 7.04–7.00 (m, 1H), 6.87–6.83 (m, 2H), 6.40 (s, 1H), 3.75 (s, 3H), 2.89 (t, J = 8.0 Hz, 2H), 2.66 (t, J = 8.4 Hz, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 159.0, 153.4, 149.1, 134.5, 128.2, 128.0, 126.8, 126.1, 125.1, 124.2, 121.6, 119.0, 114.4, 114.2, 105.1, 55.45, 29.1, 21.1; anal. calcd for $C_{19}H_{16}O_2$: C, 82.58; H, 5.84%; found: C, 82.42; H, 5.80%.

2-(*m*-Tolyl)-4,5-dihydronaphtho[1,2-*b*]furan (3ff).[11a] Colorless oil (80%, 104 mg), R_f = 0.55 (PE). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49–7.42 (m, 3H), 7.20–7.13 (m, 2H), 7.09–7.96 (m, 3H), 6.49 (s, 1H), 2.88 (t, J = 8.4 Hz, 2H), 2.65 (t, J = 8.4 Hz, 2H), 2.30 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 153.4, 149.6, 138.3, 134.7, 128.7, 128.1, 128.0, 126.8, 126.42, 126.40, 124.3, 121.5, 120.9, 119.2, 106.5, 102.4, 29.1, 21.6, 21.1.

Conflicts of interest

There are no conflicts to declare.

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