NIS-Mediated Oxidative Lactonization of 2-Arylbenzoic Acids for the Synthesis of Dibenzopyranones under Metal-Free Conditions

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Abstract: A series of dibenzopyranones were synthesized from 2-arylbenzoic acids by a radical oxidative cyclization procedure mediated by *N*-iodosuccinimide (NIS). The methodology is distinguished by its practicality in terms of its wide substrate scope, good functional group tolerance, and mild reaction conditions without the use of transition metals.

Key words: lactones, oxidation, carboxylic acids, cyclization, radical reaction

Dibenzopyranones and their derivatives are an important scaffold that can be found in many natural products and pharmaceuticals because of their remarkable biological and pharmacological activity.[1a–d] The dibenzopyranone structural unit has also found application as an intermediate in the synthesis of useful axially chiral compounds.[1e,f] As a consequence, extensive efforts have been made to develop methods for the synthesis of these compounds, including: cyclization of hydroxyl carboxylic acids;[2] Baeyer–Villiger oxidation of fluorenones;[3] transition-metal-catalyzed C–O bond formation from 2′-halobiphenyl-2-carboxylic acids;[4] and C–H functionalization methodologies, such as, palladium-catalyzed intramolecular C–C bond formation of aryl 2-halobenzoates,[5] Pd/Cu or ruthenium-catalyzed C–H activation/carbonylation of biphenyl-2-ol derivatives in the presence of carbon monoxide,[6] and palladium or copper-catalyzed C–H activation/C–O bond formation from 2-arylbenzoic acids.[7] However, there are some problems associated with these methodologies, such as the reliance on transition-metal catalysts, the use of starting materials that are not readily available, and the requirement for prefunctionalization. To our knowledge, there are very few published methods for the transformation of 2-arylbenzoic acids into dibenzopyranones under metal-free conditions.[8] As a continuation of our efforts on the development of metal-free oxidative reactions,[9] we wished to develop simple methods for the preparation of dibenzopyranones; herein, we report a new method for the synthesis of dibenzopyranones from 2-arylbenzoic acids mediated by *N*-iodosuccinimide.

2-Phenylbenzoic acid (**1a**) was chosen as the model substrate to examine the reaction of 2-arylbenzoic acids in the presence of *N*-iodosuccinimide (Table 1). Reaction of 2-phenylbenzoic acid (**1a**) with 1.5 equivalents of *N*-iodosuccinimide as the oxidant in 1,2-dichloroethane at 75 °C gave the 6*H*-benzo[*c*]chromen-6-one (**2a**) in 61% yield (entry 1). Increasing the amount of *N*-iodosuccinimide increased yield of the **2a** and it was found 2.5 equivalents *N*-iodosuccinimide was optimum for the transformation (entries 2 and 3). Oxidants played a key role in the reaction; *N*-bromosuccinimide showed low reactivity and gave **2a** in 55%, while *N*-chlorosuccinimide gave no product (en-

Table 1 Optimization of Reaction Conditions[a]

1a **2a**

Entry	Oxidant (equiv)	Additive (equiv)	Solvent	Yield[b] (%)
1	NIS (1.5)	–	$(CH_2Cl)_2$	61
2	NIS (2.0)	–	$(CH_2Cl)_2$	78
3	NIS (2.5)	–	$(CH_2Cl)_2$	86
4	NBS (2.5)	–	$(CH_2Cl)_2$	55
5	NCS (2.5)	–	$(CH_2Cl)_2$	0
6	NIS (2.5)	Na_2CO_3 (1.0)	$(CH_2Cl)_2$	77
7	NIS (2.5)	NH_4Cl (1.0)	$(CH_2Cl)_2$	34
8	NIS (2.5)	2-iodobenzoic acid (0.2)	$(CH_2Cl)_2$	76
9	NIS (2.5)	AIBN (0.2)	$(CH_2Cl)_2$	53
10	NIS (2.5)	–	toluene	68
11	NIS (2.5)	–	MeOH	0[c]
12	NIS (2.5)	–	DMSO	0[c]
13	NIS (2.5)	–	CF_3CH_2OH	47
14	NIS (2.5)	–	$(CH_2Cl)_2$	51[d]
15	NIS (2.5)	–	$(CH_2Cl)_2$	38[e]

[a] Reaction conditions: **1a** (0.5 mmol), oxidant, solvent (3 mL), visible light, 75 °C, 4 h.
[b] Isolated yield.
[c] The reaction was carried out at reflux temperature.
[d] The reaction was carried out in darkness.
[e] The reaction was carried out at r.t.

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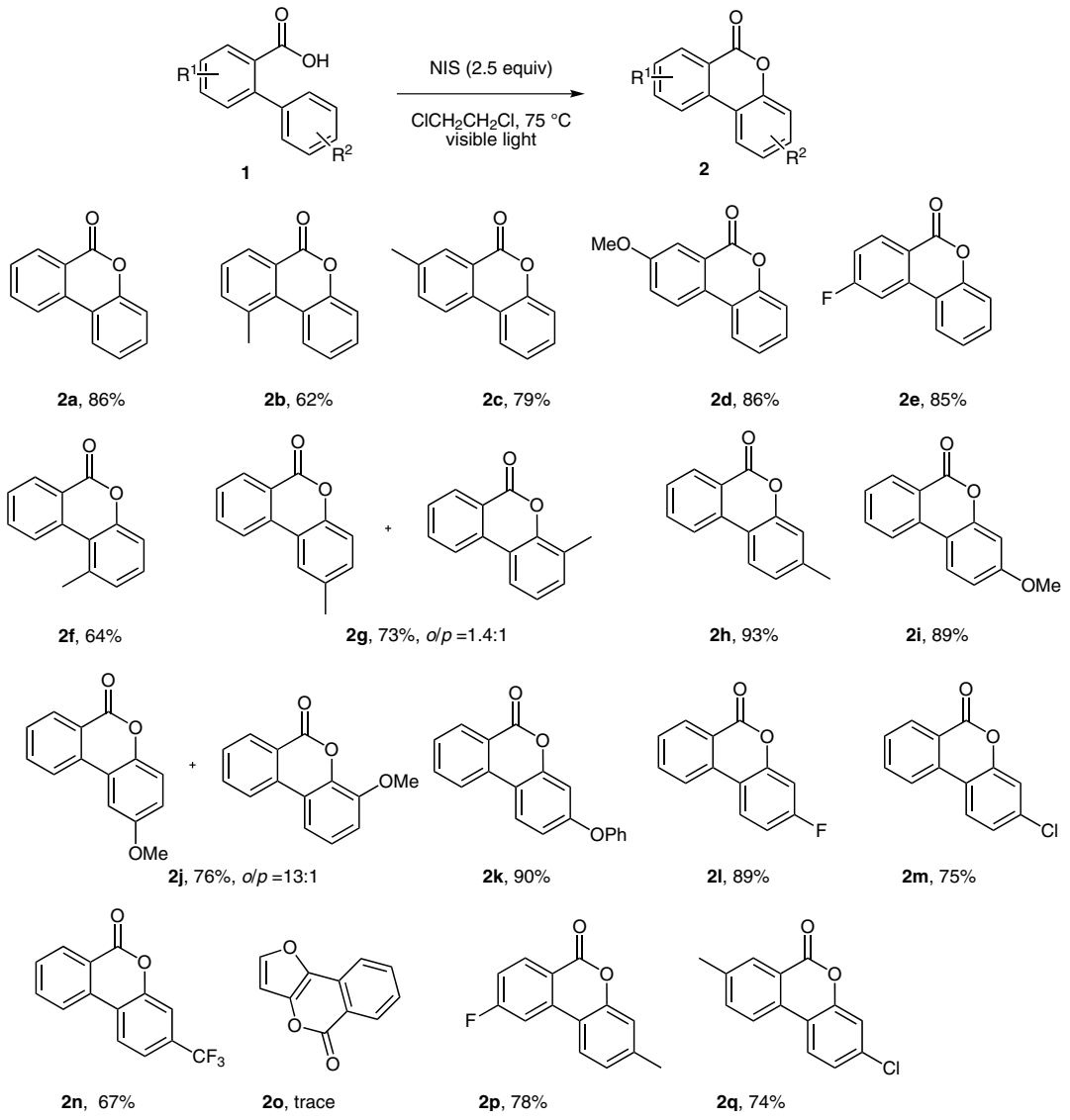
tries 4 and 5). The influence of other additives on the reaction was then studied. It was found that when sodium carbonate added as a base, the yield of **2a** decreased slightly (entry 6). To our surprise, the addition of ammonium chloride resulting in a dramatic decrease in the yield of **2a** to 34% (entry 7). The use of other additives, such as 2-iodobenzoic acid and 2,2′-azobisisobutyronitrile, also did not improve the yield of **2a** (entries 8 and 9). The use of various solvents was also examined, including 1,2-dichloroethane, toluene, methanol, dimethyl sulfoxide, and 2,2,2-trifluoroethanol; 1,2-dichloroethane was the best solvent for the reaction (entries 3 and 10–13). It should be noted that when the reaction was carried out in darkness, a lower yield of **2a** was obtained and this observation suggests that visible light may play a key role in this transformation (entry 14). Decreasing the reaction temperature from 75 °C to room temperature gave **2a** in only 38% yield (entry 15). Overall, the following optimized procedure was chosen: *N*-iodosuccinimide (2.5 equiv) as oxidant in 1,2-dichloroethane at 75 °C (entry 3).

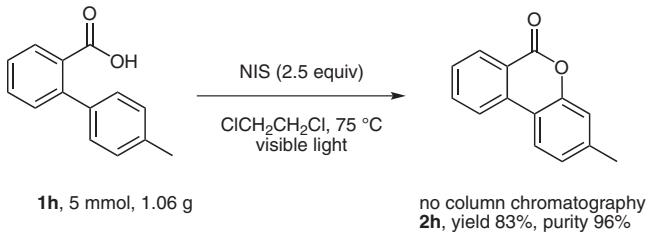
With the optimized conditions in hand, the scope of this *N*-iodosuccinimide-mediated oxidative lactonization was examined (Scheme 1). To our satisfaction, a variety of 2-arylbenzoic acids bearing electron-donating or electron-withdrawing groups in the aryl ring all reacted well and the corresponding products **2** were obtained in moderate to excellent yields. Compared to the substrates with electron-withdrawing substituents, such as chloro and trifluoromethyl, substrates with electron-donating substituents such as methoxy, phenoxy, and methyl, gave higher yields of products. Interestingly, cyclization of *meta*-substituted (R^2) 2-arylbenzoic acids gave two regioisomers with different ratios. When methyl was in *meta* position of the phenyl ring, the product ratio of *para* cyclization to *ortho* cyclization was **2g**/**2g′** 1.4:1. However, when methoxy was in the *meta* position of the phenyl ring, the product ratio of *para* cyclization to *ortho* cyclization was **2j**/**2j′** 13:1. These results suggest that an electron-rich ring was more reactive in this reaction. It was also found cyclization of *ortho*-substituted (R^1 or R^2) substrates, **1b** and **1f**,



Scheme 1 Substrate scope

gave relatively lower yields of the corresponding products, **2b** and **2f**, presumably because steric hindrance by the *ortho* substituents makes the coplanar form of the two benzene ring difficult to obtain. When 2-(furan-2-yl)benzoic acid (**1o**), a heterocyclic substrate, was employed in the reaction, the reaction was complex and only trace amounts of the product **2o** were detected.

For practical purposes, the synthesis of dibenzopyranone **2h** from the corresponding 2-(4-methylphenyl)benzoic acid (**1h**) was scaled up to a gram scale (Scheme 2). Since the byproduct, pyrrolidine-2,5-dione, is soluble in water, **2h** can be simply obtained by aqueous workup in 83% yield with 96% purity.



Scheme 2 Gram-scale preparation of **2h**

To gain insights into the reaction pathway, 2,2,6,6-tetramethylpiperidine *N*-oxide (2.5 equiv), a radical-trapping reagent, was added to the reaction system under the optimized conditions and none of desired product **2a** was formed. According to the present results and unique properties of *N*-halo-substituted compounds,[10] a radical cyclization mechanism was proposed (Scheme 3). Reaction of 2-arylbenzoic acid **1** with *N*-iodosuccinimide formed 2-arylbenzoic hypoiodous anhydride **A**. Homolytic cleavage of the O–I bond of **A** then occurred to provide oxygen-centered radical **B**. Intramolecular attack of the oxygen-centered radical on the phenyl ring led to the lactonization to provide the final product **2**.

In summary, a simple and convenient *N*-iodosuccinimide-mediated oxidative lactonization procedure was developed. Broad substrate scope, good functional group tolerance, and mild reaction conditions make the protocol a potential approach for the synthesis of dibenzopyranones. Further work to understand the mechanism and extend the scope of the protocol is currently under investigation in our laboratory.

All solvents and reagents were obtained from commercial sources and used without further purification. NMR spectra were recorded on a Bruker Avance 500 spectrometer. HRMS were recorded on a Waters Micromass GCT Premier using ESI-TOF (EI-ToF). IR spectra were recorded on a Nicolet-10 FTIR instrument. Melting points were determined on Yamato melting point apparatus Model MP-21. Silica gel (200–300 mesh) was used for column chromatographic separations and purifications. Petroleum ether (PE) refers to the fraction boiling in the 60–90 °C range.

Benzo[*c*]chromen-6-ones 2; General Procedure

To a solution of 2-arylbenzoic acid **1** (0.5 mmol) in $ClCH_2CH_2Cl$ (3 mL) was added NIS (1.25 mmol) in one portion; the mixture was heated to 75 °C for 4 h. After completion of the reaction, EtOAc (10 mL) was added and the mixture was washed with sat. $Na_2S_2O_3$ (5 mL), then with H_2O (2 × 5 mL). The organic layer was dried (anhyd Na_2SO_4) and concentrated in vacuo. The crude products were purified by column chromatography (silica gel, PE–EtOAc, 50:1) to provide the product **2**. All the products were confirmed by ^1H and ^{13}C NMR spectra and new compounds were reconfirmed additionally by HRMS.

6*H*-Benzo[*c*]chromen-6-one (2a)[7a]

White solid; yield: 84.3 mg (86%); mp 91–92 °C.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.39 (dd, J_1 = 1.2 Hz, J_2 = 7.9 Hz, 1 H), 8.11 (d, J = 8.1 Hz, 1 H), 8.05 (dd, J_1 = 1.3 Hz, J_2 = 8.0 Hz, 1 H), 7.80–7.83 (m, 1 H), 7.55–7.59 (m, 1 H), 7.45–7.48 (m, 1 H), 7.31–7.36 (m, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.1, 151.3, 134.8, 134.7, 130.5, 130.4, 128.9, 124.5, 122.8, 121.7, 121.3, 118.0, 117.7.

10-Methyl-6*H*-benzo[*c*]chromen-6-one (2b)[7a]

White solid; yield: 65.1 mg (62%); mp 122–124 °C.

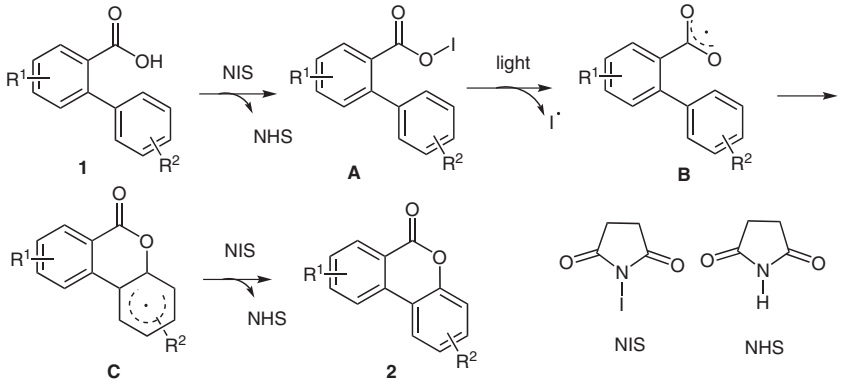
^1H NMR (500 MHz, $CDCl_3$): δ = 8.37 (d, J = 7.8 Hz, 1 H), 8.31 (d, J = 8.2 Hz, 1 H), 7.65 (d, J = 7.4 Hz, 1 H), 7.46–7.49 (m, 2 H), 7.40 (dd, J_1 = 1.3 Hz, J_2 = 8.2 Hz, 1 H), 7.31–7.34 (m, 1 H), 2.90 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.7, 151.3, 139.0, 135.0, 133.6, 129.6, 129.2, 128.2, 127.2, 124.0, 122.9, 119.7, 118.0, 25.3.

8-Methyl-6*H*-benzo[*c*]chromen-6-one (2c)[7a]

White solid; yield: 83.0 mg (79%); mp 126–128 °C.

^1H NMR (500 MHz, $CDCl_3$): δ = 8.18 (s, 1 H), 7.98–8.02 (m, 2 H), 7.62 (dd, J_1 = 1.4 Hz, J_2 = 8.2 Hz, 1 H), 7.42–7.46 (m, 1 H), 7.29–7.35 (m, 2 H), 2.49 (s, 3 H).



Scheme 3 Proposed reaction mechanism

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.4, 151.0, 139.2, 136.0, 132.2, 130.4, 129.9, 124.5, 122.5, 121.7, 121.1, 118.2, 117.7, 21.3.

8-Methoxy-6*H*-benzo[*c*]chromen-6-one (2d)[7b]
White solid; yield: 97.2 mg (86%); mp 159–161 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.01 (d, *J* = 8.8 Hz, 1 H), 7.95 (dd, J_1 = 1.1 Hz, J_2 = 7.9 Hz, 1 H), 7.78 (d, *J* = 2.8 Hz, 1 H), 7.28–7.42 (m, 4 H), 3.92 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.2, 160.0, 150.5, 129.3, 128.1, 124.6, 124.2, 123.4, 122.4, 122.1, 118.2, 117.6, 111.3, 55.8.

9-Fluoro-6*H*-benzo[*c*]chromen-6-one (2e)
White solid; yield: 91.0 mg (85%); mp 171–172 °C.

FT-IR: 1730, 1610, 1280, 1200, 1100 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.42 (m, 1 H), 7.94 (d, *J* = 7.5 Hz, 1 H), 7.72 (dd, J_1 = 2.3 Hz, J_2 = 9.6 Hz, 1 H), 7.51 (m, 1 H), 7.35 (m, 2 H), 7.26 (td, J_1 = 2.1 Hz, J_2 = 8.6 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.0 (d, $J_{C\text{-}F}$ = 255.4 Hz), 160.2, 151.7, 137.7 (d, $J_{C\text{-}F}$ = 9.8 Hz), 134.0 (d, $J_{C\text{-}F}$ = 10.1 Hz), 131.3, 124.7, 123.0, 117.9, 117.8, 117.4, 117.0 (d, $J_{C\text{-}F}$ = 22.8 Hz), 108.1 (d, $J_{C\text{-}F}$ = 4.7 Hz).

HRMS (EI): *m*/*z* $[M]^+$ calcd for $C_{13}H_7FO_2$: 214.0430; found: 214.0428.

1-Methyl-6*H*-benzo[*c*]chromen-6-one (2f)[7a]
White solid; yield: 67.2 mg (64%); mp 130–132 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.37 (d, *J* = 7.9 Hz, 1 H), 8.31 (d, *J* = 8.1 Hz, 1 H), 7.64 (d, *J* = 7.4 Hz, 1 H), 7.47 (m, 2 H), 7.40 (dd, J_1 = 1.3 Hz, J_2 = 8.1 Hz, 1 H), 7.33 (m, 1 H), 2.90 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.2, 149.7, 135.2, 134.7, 131.8, 130.5, 128.7, 127.1, 124.0, 121.8, 121.1, 120.4, 117.8, 16.0.

2-Methyl-6*H*-benzo[*c*]chromen-6-one (2g) and 4-Methyl-6*H*-benzo[*c*]chromen-6-one (2g′)[7b]
White solid; yield: 76.7 mg (73%); mp 93–95 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.38 (m, 1.68 H), 8.08 (t, *J* = 7.3 Hz, 1.75 H), 7.87 (d, *J* = 7.9 Hz, 0.74 H), 7.77–7.81 (m, 2.67 H), 7.53–7.57 (m, 1.73 H), 7.31 (d, *J* = 7.3 Hz, 0.77 H), 7.17–7.26 (m, 2.86 H), 2.48 (s, 2.21 H), 2.44 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.3, 161.2, 149.7, 149.4, 135.1, 134.8, 134.7, 134.1, 131.8, 131.3, 130.6, 130.5, 128.7, 127.0, 124.0, 122.7, 121.6, 121.3, 121.1, 120.4, 117.7, 117.6, 117.5, 21.1, 16.0.

3-Methyl-6*H*-benzo[*c*]chromen-6-one (2h)[7a]
White solid; yield: 97.7 mg (93%); mp 136–138 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.36 (d, *J* = 7.8 Hz, 1 H), 8.05 (d, *J* = 8.1 Hz, 1 H), 7.90 (d, *J* = 8.1 Hz, 1 H), 7.77 (m, 1 H), 7.52 (t, *J* = 7.7 Hz, 1 H), 7.13 (m, 2 H), 2.42 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.4, 151.4, 141.3, 136.0, 134.7, 130.6, 128.4, 126.7, 122.5, 121.4, 121.0, 117.9, 115.5, 21.4.

3-Methoxy-6*H*-benzo[*c*]chromen-6-one (2i)[7a]
White solid; yield: 100.6 mg (89%); mp 141–143 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.38 (d, *J* = 8.0 Hz, 1 H), 8.02 (d, *J* = 8.1 Hz, 1 H), 7.96 (d, *J* = 8.8 Hz, 1 H), 7.80 (t, *J* = 7.7 Hz, 1 H), 7.52 (t, *J* = 7.6 Hz, 1 H), 6.93 (dd, J_1 = 2.4 Hz, J_2 = 8.8 Hz, 1 H), 6.88–6.90 (m, 1 H), 3.90 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.6, 161.4, 152.7, 135.2, 134.8, 130.6, 127.7, 123.8, 121.1, 120.1, 112.5, 112.2, 101.7, 55.7.

2-Methyl-6*H*-benzo[*c*]chromen-6-one (2j) and 4-Methyl-6*H*-benzo[*c*]chromen-6-one (2j′)[7b]
White solid; yield: 85.9 mg (76%); mp 156–158 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.37 (dd, J_1 = 0.9 Hz, J_2 = 7.9 Hz, 1 H), 8.00–8.06 (m, 1.07 H), 7.77–7.81 (m, 1.06 H), 7.54–7.59 (m, 1.14 H), 7.44 (d, *J* = 2.9 Hz, 1 H), 7.21–7.27 (m, 1.11 H), 7.01 (dd, J_1 = 2.9 Hz, J_2 = 9.0 Hz, 1.05 H), 3.96 (s, 0.22 H), 3.89 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.2, 160.5, 156.4, 145.6, 134.7, 134.6, 130.6, 128.9, 121.7, 121.4, 118.6, 118.5, 117.1, 114.1, 112.3, 106.4, 56.2, 55.9.

3-Phenoxy-6*H*-benzo[*c*]chromen-6-one (2k)
White solid; yield: 129.6 mg (90%); mp 148–150 °C.

FT-IR: 1730, 1600, 1270, 1070, 813 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.34 (dd, J_1 = 0.8 Hz, J_2 = 7.9 Hz, 1 H), 7.98 (q, *J* = 8.5 Hz, 1 H), 7.78 (m, 1 H), 7.52 (t, *J* = 7.9 Hz, 1 H), 7.39–7.43 (m, 2 H), 7.21 (t, *J* = 7.4 Hz, 1 H), 7.1 (d, *J* = 7.7 Hz, 1 H), 6.98 (dd, J_1 = 2.5 Hz, J_2 = 7.5 Hz, 1 H), 6.90 (d, *J* = 2.4 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 161.2, 159.8, 155.6, 152.4, 134.9, 134.8, 130.6, 130.1, 128.2, 124.6, 124.0, 121.3, 120.3, 120.0, 114.8, 113.0, 106.4.

HRMS (EI): *m*/*z* $[M]^+$ calcd for $C_{19}H_{12}O_3$: 288.0785; found: 288.0784.

3-Fluoro-6*H*-benzo[*c*]chromen-6-one (2l)[7a]
White solid; yield: 95.2 mg (89%); mp 159–161 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.33 (d, *J* = 7.9 Hz, 1 H), 7.99 (t, *J* = 7.2 Hz, 2 H), 7.80 (t, *J* = 7.7 Hz, 1 H), 7.54 (t, *J* = 7.6 Hz, 1 H), 7.02–7.07 (m, 2 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 163.5 (d, $J_{C\text{-}F}$ = 248.3 Hz), 160.7, 152.1 (d, $J_{C\text{-}F}$ = 12.0 Hz), 135.1, 134.2, 130.6, 128.7, 124.3 (d, $J_{C\text{-}F}$ = 9.7 Hz), 121.5, 120.4, 114.6, 112.4 (d, $J_{C\text{-}F}$ = 22.3 Hz), 105.0 (d, $J_{C\text{-}F}$ = 25.1 Hz).

3-Chloro-6*H*-benzo[*c*]chromen-6-one (2m)[7a]
White solid; yield: 86.3 mg (75%); mp 160–161 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.36 (d, *J* = 8.0 Hz, 1 H), 8.04 (d, *J* = 8.1 Hz, 1 H), 7.95 (d, *J* = 8.5 Hz, 1 H), 7.81 (m, 1 H), 7.57 (t, *J* = 7.6 Hz, 1 H), 7.35 (d, *J* = 1.8 Hz, 1 H), 7.29 (dd, J_1 = 2.1 Hz, J_2 = 8.5 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 160.5, 151.6, 136.0, 135.1, 134.0, 130.8, 129.2, 126.0, 123.8, 121.7, 121.0, 118.0, 116.7.

3-(Trifluoromethyl)-6*H*-benzo[*c*]chromen-6-one (2n)[7a]
White solid; yield: 88.5 mg (67%); mp 146–147 °C.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.45 (dd, J_1 = 0.94 Hz, J_2 = 7.9 Hz, 1 H), 8.18 ((t, *J* = 9.1 Hz, 2 H), 7.88–7.91 (m, 1 H), 7.66–7.70 (m, 1 H), 7.63 (s, 1 H), 7.59 (d, *J* = 8.3 Hz, 1 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 160.2, 151.0, 135.2, 133.4, 132.3 (d, $J_{C\text{-}F}$ = 33.0 Hz), 130.9, 130.1, 124.4, 123.6, 122.2, 121.7, 121.1, 115.3 (d, $J_{C\text{-}F}$ = 3.1 Hz).

9-Fluoro-3-methyl-6*H*-benzo[*c*]chromen-6-one (2p)
White solid; yield: 88.9 mg (78%); mp 220–221 °C.

FT-IR: 3090, 1730, 1610, 1300, 1190, 856 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.40 (m, 1 H), 7.81 (d, *J* = 7.9 Hz, 1 H), 7.68 (dd, J_1 = 2.4 Hz, J_2 = 9.7 Hz, 1 H), 7.22 (td, J_1 = 2.3 Hz, J_2 = 8.5 Hz, 1 H), 7.15–7.17 (m, 2 H), 2.46 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.0 (d, $J_{C\text{-}F}$ = 254.9 Hz), 160.5, 151.7, 142.3, 138.0 (d, $J_{C\text{-}F}$ = 9.7 Hz), 133.9 (d, $J_{C\text{-}F}$ = 10.1 Hz), 125.8, 122.7, 118.0, 117.4, 116.5 (d, $J_{C\text{-}F}$ = 22.9 Hz), 114.8, 107.8 (d, $J_{C\text{-}F}$ = 23.4 Hz), 21.5.

HRMS (EI): *m*/*z* $[M]^+$ calcd for $C_{14}H_9FO_2$: 228.0587; found: 228.0592.

3-Chloro-8-methyl-6*H*-benzo[*c*]chromen-6-one (2q)
White solid; yield: 90.3 mg (74%); mp 176–178 °C.

FT-IR: 3080, 2920, 1730, 1620, 1300, 1100, 859 cm^{-1}.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.12 (s, 1 H), 7.89 (m, 2 H), 7.60 (dd, J_1 = 1.2 Hz, J_2 = 8.1 Hz, 1 H), 7.24–7.30 (m, 2 H), 2.47 (s, 3 H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 160.7, 151.2, 139.6, 136.2, 135.3, 131.4, 130.5, 124.9, 123.5, 121.6, 120.7, 117.8, 115.8, 21.3.

HRMS (EI): *m*/*z* $[M]^+$ calcd for $C_{14}H_9ClO_2$: 244.0289; found: 244.0290.

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Supporting Information for this article is available online at http://www.thieme-connect.com/ejournals/toc/synthesis.

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