

LACTAMIZATION OF ALKENYL C-H BONDS TO GENERATE 2-QUINOLINONES WITH TRIPHOSGENE

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Abstract – A simple and easy-going method is developed to synthesize the analogues of 2-quinolinones by using triphosgene (BTC) as the carbonyl source. In these reactions, both the toxic carbon monoxide (CO) and phosgene are avoided and the 2-quinolinones are obtained in moderate to good yields under mild conditions, all of which are anticipated to be meaningful in both industry and laboratory.

INTRODUCTION

2-Quinolinones and polyheterocycles are common motifs which widely appear in drugs, natural products and materials.¹⁻⁵ Multiple methods to generate such structures have been discovered; for examples, the intramolecular cyclization of β -keto anilides (Knorr synthesis) under the acid-mediated conditions,⁶ and intramolecular aldol condensation of 2-aminophenyl substituted carbonyl compounds under a base-mediated condition (Friedländer Reaction).⁷

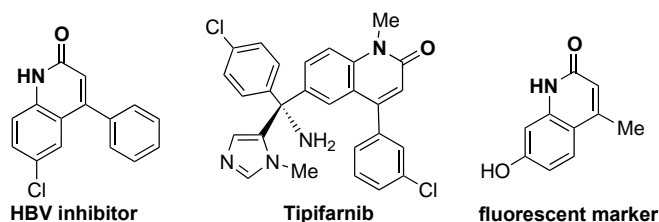
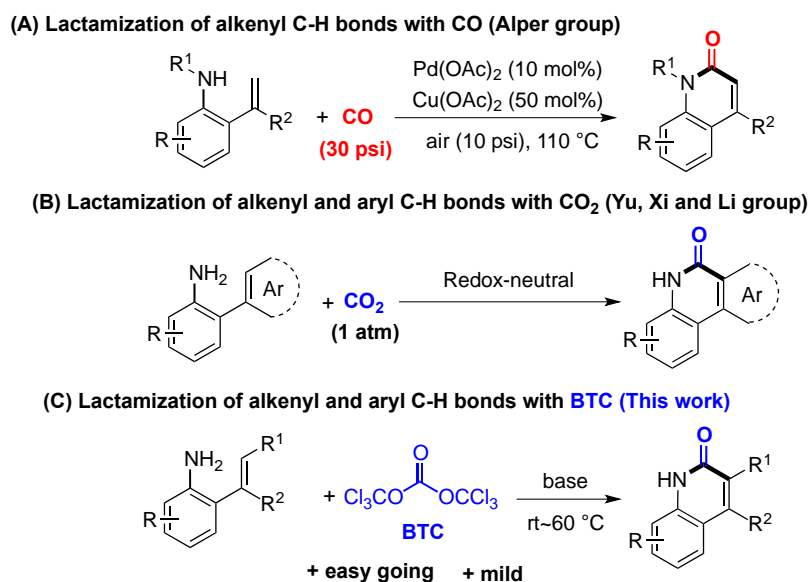


Figure 1. Selected useful compounds of 2-quinolinones

In recent decades, continuous research on the functionalization of C-H bond has provided new approaches for the synthesis of quinolinones. The direct carbonylation reaction on C-H bond can be utilized to efficiently construct quinolinone compounds. Furthermore, their substrates are relatively easy to be synthesized and the carbonyl group can be conveniently used for isotopic calibration, which may provide potential application in both drug research and development.

In recent years, the carbonylation reaction to generate quinolinones and their analogues has been achieved by using carbon monoxide (CO) or carbon dioxide (CO₂), such as Pd-catalyzed oxidative carbonylation reactions (Scheme 1A),⁸⁻¹¹ and lactamization reaction with CO₂ (Scheme 1B).¹²⁻¹⁴ In those reactions, gas and a high temperature are required. Thus, sometimes these methods are difficult to apply in both industry and laboratory due to inconvenience on operation. Therefore, it is significant to develop a simple and easy-going C-H bond carbonylation reaction to synthesize 2-quinolinones.

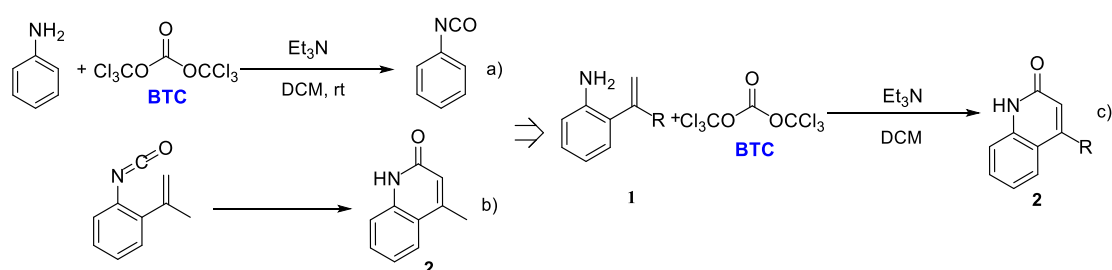


Scheme 1. Lactamization of C-H bonds

Triphosgene (BTC) is a much safer alternative to CO, phosgene and diphosgene. Moreover, BTC is solid, which is convenient for storage and transportation. During the previous application of BTC as the carbonylation reagent, it has already been proved that the reaction conditions are mild, the selectivity is good, and the final yield is always similar to phosgene.¹⁵ Due to the advantages of BTC, it is gradually applied in both laboratory and industry in carbonylation reactions.¹⁵ Herein, an easy-going method is reported to synthesize 2-quinolinones and polyheterocycles with BTC under mild conditions (Scheme 1C). The starting materials can be easily synthesized from the commercial reagents.

RESULTS AND DISCUSSION

It is well known that anilines can react with BTC to generate isocyanates,^{16,17} and 1-isocyanato-2-vinylbenzenes can be transferred to corresponding 2-quinolinones and polyheterocycles through a 6- π electrocyclization (Scheme 2a).^{12a,18-23} If there is an alkene group at the ortho position of aniline, after reacting with BTC, we anticipated that the 1-isocyanato-2-vinylbenzene can be obtained *in-situ* (Scheme 2b), and the 2-quinolinones can be obtained after the cyclization reaction. If so, we can easily synthesize the useful 2-quinolinones from the easy-to-get reagent,^{24,25} which should be useful in both for laboratory and industrial applications (Scheme 2c).



Scheme 2. Our design of lactamization of alkenyl C-H bonds with triphosgene

Based on the above design (Scheme 2c) and our previous reports (Table 1, entry 1),^{12a,12c} we have extended the reaction time to 12 h and the 2-quinolinones **2a** could be obtained in yield of 69% (Table 1, entry 2). When we reduced the amount of triphosgene to 0.5 equiv., the yield of **2a** has been increased to 77% (Table 1, entry 3).

Table 1. Optimization of the reaction conditions.^a

Reaction scheme for the synthesis of **2a**: **1a** (ortho-alkenyl aniline) reacts with BTC in the presence of Et₃N at 60 °C in CH₂Cl₂ to form **2a** (2-quinolinone).

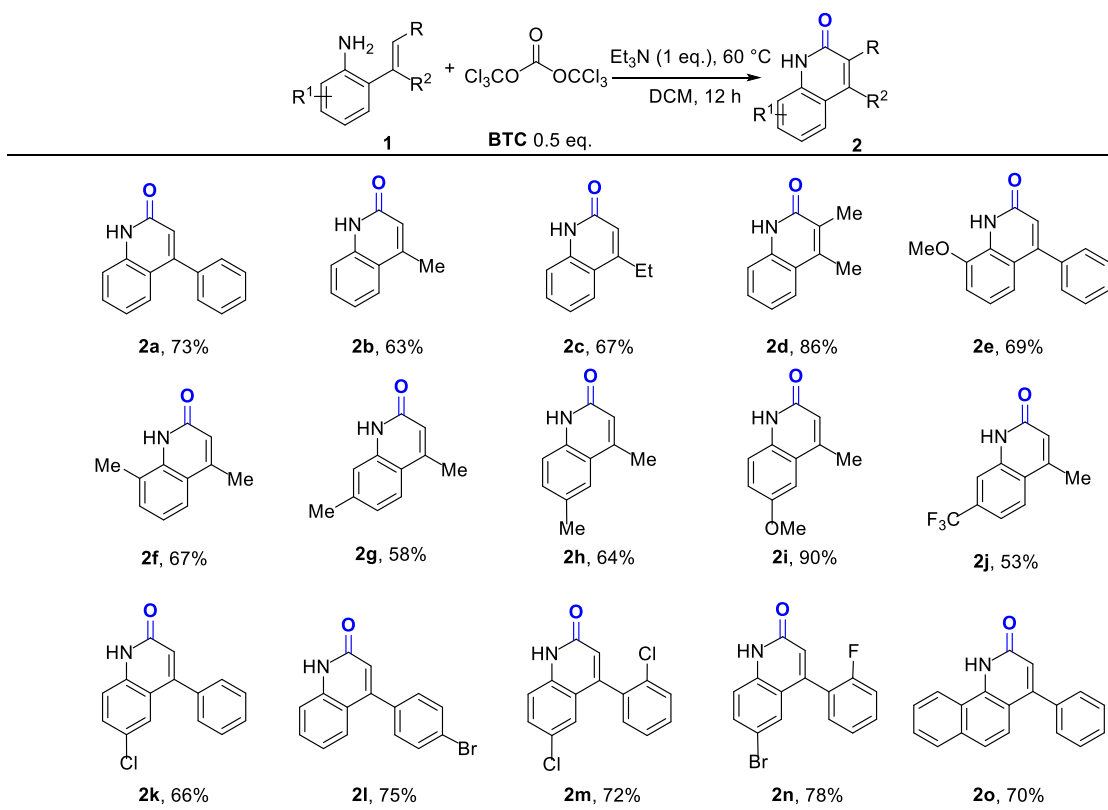
entry	BTC (eq.)	Et ₃ N (eq.)	T(°C)	t (h)	yield (%)
1	1.0	1.0	60	2	51 ^c
2	1.0	1.0	60	12	69
3	0.5	1.0	60	12	77 (73 ^b)

^a Reaction conditions: the reaction was run in N₂, **1a** (0.3 mmol), CH₂Cl₂ (5 mL), BTC and Et₃N as above, detected by GC; ^b Yield of the isolated product. ^c Please see reference 12c, the SI of our study before; isolated product.

With the optimized condition in hand, we carried out reactions between the analogues of anilines with *ortho*-alkenyl substitutions and BTC (Table 2). Firstly, the substrate with a methyl (**1b**, 63%) or ethyl (**1c**,

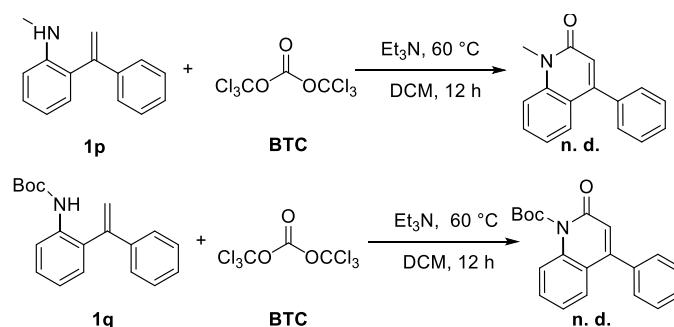
67%) group on the alkene showed good reactivity. We also found that both terminal alkenes (**1a**, 77%; **1b**) and internal alkenes (**1d**, 86%) could be applied in the reaction to give the products in moderate to good yields. Moreover, we tested a variety of substituents on the arene, and the steric hindrance did not hamper the reaction (**1e**, 69%; **1f**, 67%). Besides, we investigated the effect of the substituent on different positions. The anilines with electron-donating groups (EDGs) (**1g**, 58%; **1h**, 64%; **1j**, 90%) showed similar reactivity to those with electron-withdrawing groups (EWGs) (**1i**, 53% and **1k**, 66%). In addition, the substrates with one or two groups like F, Cl, Br could also well react with BTC (**1l**, 75%; **1m**, 72%; **1n**, 78%), which provided great opportunities for further functionalization. At last, polyheterocycles **2o** could be obtained in good yield (70%). Simply speaking, different kinds of functional groups were well tolerated.

Table 2. Reaction scope: synthesis of 2-quinolinones and polyheterocycles



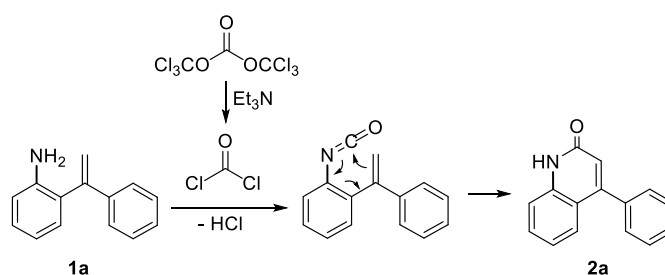
Reaction conditions: the reaction was run in N₂, **1** (0.3 mmol), CH₂Cl₂ (5 mL), BTC (0.15 mmol) and Et₃N (0.3 mmol), 60 °C, 12 h, isolated yield.

In order to confirm the isocyanate as the intermediate in the reaction, we investigated the substrate with the substituents on the aniline nitrogen. Neither alkyl (**1p**) nor Boc (**1q**) substituted anilines could afford the target products (Scheme 3).



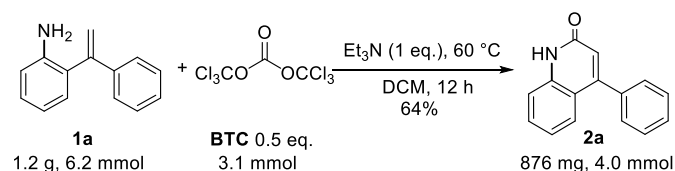
Scheme 3. Control experiments

Based on the above results, we proposed the following pathway (Scheme 4). BTC was initiated by triethylamine to generate phosgene *in-situ* and **1a** subsequently reacted with the phosgene to form the isocyanate which could further undergo the following cyclization reaction to form the desired 2-quinolinones **2a**.



Scheme 4. Possible pathway

After developing this methodology, we further demonstrated its utility (Scheme 5). We found that such a reaction could be carried out in larger scale with similar yield and efficiency.



Scheme 5. Reaction in larger scale

In summary, an easy-going method has been developed to synthesize 2-quinolinones and polyheterocycles containing a free (NH)-lactam motif, and most of the yields of the transformations are good. The reactions of lactamization are realized with BTC as the carbonyl source and this approach may be further applied in both lab and industry.

EXPERIMENTAL

All reactions were set up with glovebox and carried out under a N₂ atmosphere in Schlenk tubes. Solvent (including diglyme, DMA, THF, DMSO, 1,4-dioxane, DCM and DMF, 99.8%, Water < 0.005%) were purchased from J&K Scientific Ltd., and used as received. Commercially available chemicals were obtained from J&K Scientific Ltd., Adamas, Acros Organics, Aldrich Chemical Co., Alfa Aesar, Chengdu Research Accelerators Technology Co., Ltd. and BT Reagent and used as received unless otherwise stated. ¹H NMR spectra were recorded on a Bruker Advance 400 spectrometer (1H: 400 MHz). Chemical shifts (δ) for ¹H NMR spectra are given in ppm relative to TMS. The residual solvent signals were used as references for ¹H spectra and the chemical shifts converted to the TMS scale (CDCl₃: δH = 7.26 ppm, (CD₃)₂SO: δH = 2.50 ppm). GC-MS was obtained using electron ionization (Agilent Technologies 7890B/GC-System and 5977A/MSD). TLC was performed using commercially prepared 100-400 mesh silica gel plates (GF254), and visualization was afforded at 254 nm. ESI-MS are obtained on a Thermo-LTQ.

General Procedure of Products (2). An oven-dried Schlenk tube (25 mL) containing a stirring bar was charged with the substrate (0.3 mmol). The Schlenk tube was then introduced in a glovebox where it was charged with triphosgene (44.5 mg, 0.15 mmol, 0.5 equiv.). The tube was taken out of the glovebox and connected to a vacuum line where it was evacuated and back-filled under N₂ flow for at least 3 times. The DCM (5 mL) and Et₃N (0.3 mmol, 1 equiv.) were added under N₂ flow. Once added, the tube was closed at atmospheric pressure of N₂ (1 atm) and stirred for 12 h at 60 °C. Then, the mixture was cooled to room temperature, quenched with 2 mL water, then concentrated in vacuo. The residue was purified by silica gel flash chromatography (petroleum ether/AcOEt 3/1) to give the pure target product **2**. (the procedure of control experiments and the reaction in larger scale are similar.)

4-Phenylquinolin-2(1H)-one (2a)^{12a}: 48.7 mg, 0.220 mmol, 73%; pale yellow solid; R_f (PE/EA 1/1): 0.34; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 11.90 (s, 1H), 7.54 (m, 4H), 7.48 (dd, *J* = 7.5, 1.9 Hz, 2H), 7.39 (m, 2H), 7.15 (m, 1H), 6.40 (s, 1H); ESI-MS: calculated *m/z* for [C₁₅H₁₁NOH]⁺: 222.1, measured 222.1.

4-Methylquinolin-2(1H)-one (2b)^{12a}: 29.9 mg, 0.188 mmol, 63%; White powder; R_f (PE/EA 1/1): 0.10; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 11.59 (s, 1H), 7.71 (dd, *J* = 8.1, 1.2 Hz, 1H), 7.50 (t, *J* = 8.0 Hz, 1H), 7.30 (dd, *J* = 8.2, 0.8 Hz, 1H), 7.20 (t, *J* = 8.0 Hz, 1H), 6.40 (s, 1H), 2.42 (s, 3H); ESI-MS: calculated *m/z* for [C₁₀H₉NOH]⁺: 160, found: 160.

4-Ethylquinolin-2(1H)-one (2c)^{12a}: 34.6 mg, 0.200 mmol, 67%; light yellow solid; R_f (PE/EA 1/1): 0.29; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 11.60 (s, 1H), 7.76 (d, *J* = 7.3 Hz, 1H), 7.49 (t, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 8.2, 1H), 7.19 (t, *J* = 8.0 Hz, 1H), 6.36 (s, 1H), 2.83 (q, *J* = 7.4 Hz, 2H), 1.24 (t, *J* = 7.4 Hz, 3H); ESI-MS: calculated *m/z* for [C₁₁H₁₁NOH]⁺: 174, measured 174.

3,4-Dimethylquinolin-2(1H)-one (2d)^{12a}: 44.6 mg, 0.258 mmol, 86%; Pale yellow solid; Rf (PE/EA 1/1): 0.42; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 11.63 (s, 1H), 7.74 (d, *J* = 8.1 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.28 (d, *J* = 9.1 Hz, 1H), 7.18 (t, *J* = 8.0 Hz, 1H), 2.41 (s, 3H), 2.12 (s, 3H); ESI-MS: calculated *m/z* for [C₁₁H₁₁NOH]⁺: 174, measured 174.

8-Methoxy-4-phenylquinolin-2(1H)-one (2e)^{12a}: 52.3 mg, 0.208 mmol, 69%, pale yellow solid, Rf (PE/EA 1/1): 0.28; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 10.90 (s, 1H), 7.58-7.47 (m, 5H), 7.21 (d, *J* = 8.0 Hz, 1H), 7.10 (t, *J* = 8.0 Hz, 1H), 6.97 (d, *J* = 8.0 Hz, 1H), 6.43 (s, 1H), 3.94 (s, 3H); ESI-MS: calculated *m/z* for [C₁₆H₁₃NO₂H]⁺: 252, found: 252.

4,8-Dimethylquinolin-2(1H)-one (2f)^{12a}: 34.7 mg, 0.201 mmol, 67%; pale yellow solid; Rf (PE/EA 1/1): 0.33; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 10.69 (s, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.11 (t, *J* = 8.0 Hz, 1H), 6.42 (s, 1H), 2.42 (6H); ESI-MS: calculated *m/z* for [C₁₁H₁₁NOH]⁺: 174, measured 174.

4,7-Dimethylquinolin-2(1H)-one (2g)^{12a}: 30.1 mg, 0.174 mmol, 58%; white solid; Rf (PE/EA 1/1): 0.22; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 11.50 (s, 1H), 7.59 (d, *J* = 8.2 Hz, 1H), 7.09 (s, 1H), 7.02 (d, *J* = 9.3 Hz, 1H), 6.31 (s, 1H), 2.39 (d, *J* = 1.0 Hz, 3H), 2.37 (s, 3H); ESI-MS: calculated *m/z* for [C₁₁H₁₁NOH]⁺: 174, measured 174.

4,6-Dimethylquinolin-2(1H)-one (2h)^{12a}: 33.2 mg, 0.192 mmol, 64%; pale yellow solid; Rf (PE/EA 1/1): 0.19; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 11.52 (s, 1H), 7.50 (s, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.20 (d, *J* = 4.0 Hz, 1H), 6.36 (s, 1H), 2.40 (s, 3H), 2.36 (s, 3H); ESI-MS: calculated *m/z* for [C₁₁H₁₁NOH]⁺: 174, measured 174.

6-Methoxy-4-methylquinolin-2(1H)-one (2i)^{12a}: 51.3 mg, 0.271 mmol, 90%; White solid; Rf (PE/EA 1/1): 0.13; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 11.48 (s, 1H), 7.25 (d, *J* = 8.0 Hz, 1H), 7.17 (dd, *J* = 8.0, 4.0 Hz, 1H), 7.13 (s, 1H), 6.39 (s, 1H), 3.81 (s, 3H), 2.42 (s, 3H); ESI-MS: calculated *m/z* for [C₁₁H₁₁NO₂H]⁺: 190, measured 190.

4-Methyl-7-(trifluoromethyl)quinolin-2(1H)-one (2j)^{12a}: 36.3 mg, 0.160 mmol, 53%; white solid; Rf (PE/EA 1/1): 0.42; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 11.86 (s, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.62 (s, 1H), 7.51 (d, *J* = 8.4 Hz, 1H), 6.57 (s, 1H), 2.46 (d, *J* = 1.1 Hz, 3H); ESI-MS: calculated *m/z* for [C₁₁H₈F₃NOH]⁺: 228, measured 228.

6-Chloro-4-phenylquinolin-2(1H)-one (2k)^{12a}: 50.9 mg, 0.200 mmol, 66%; white solid; Rf (PE/EA 1/1): 0.40; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 12.05 (s, 1H), 7.62 – 7.39 (m, 7H), 7.33 – 7.16 (m, 1H), 6.47 (s, 1H); ESI-MS: calculated *m/z* for [C₁₅H₁₀ClNOH]⁺: 256, measured 256.

4-(4-Bromophenyl)quinolin-2(1H)-one (2l)^{12a}: 67.0 mg, 0.224 mmol, 75%; pale yellow solid; Rf (PE/EA 1/1): 0.33; ¹H NMR (400 MHz, DMSO-*d*₆): δ = 11.90 (s, 1H), 7.74 (d, *J* = 8.0 Hz, 2H), 7.54 (t, *J* = 8.0 Hz, 1H), 7.45 (d, *J* = 12.0 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 1H), 7.15 (t, *J* =

8.0 Hz, 1H), 6.42 (d, $J = 1.6$ Hz, 1H); ESI-MS: calculated m/z for $[C_{15}H_{10}BrNOH]^+$: 300.00, measured 300.00.

6-Chloro-4-(2-chlorophenyl)quinolin-2(1H)-one (2m)^{12a}: 62.9 mg, 0.218 mmol, 72%; pale yellow solid; Rf (PE/EA 1/1): 0.29; ¹H NMR (400 MHz, DMSO-*d*₆): $\delta = 12.12$ (s, 1H), 7.68 (dd, $J = 7.8, 1.3$ Hz, 1H), 7.61 – 7.52 (m, 3H), 7.48 (dd, $J = 7.4, 1.8$ Hz, 1H), 7.43 (d, $J = 8.8$ Hz, 1H), 6.85 (d, $J = 2.3$ Hz, 1H), 6.49 (s, 1H); ESI-MS: calculated m/z for $[C_{15}H_9Cl_2NOH]^+$: 290.0, measured 290.0.

6-Bromo-4-(2-fluorophenyl)quinolin-2(1H)-one (2n)^{12a}: 74.1 mg, 0.234 mmol, 78%; pale yellow solid; Rf (PE/EA 1/1): 0.45; ¹H NMR (400 MHz, DMSO-*d*₆): $\delta = 12.12$ (s, 1H), 7.72 (dd, $J = 8.8, 2.2$ Hz, 1H), 7.67 – 7.56 (m, 1H), 7.53 – 7.36 (m, 4H), 7.18 (t, $J = 2.2$ Hz, 1H), 6.56 (s, 1H); Exact Mass: calculated m/z for $[C_{15}H_9BrFNOH]^+$: 317.9930, found: 317.9922.

4-Phenylbenzo[h]quinolin-2(1H)-one (2o)^{12a}: 57.1 mg, 0.211 mmol, 70%; yellow solid; Rf (PE/EA 1/1): 0.58; ¹H NMR (600 MHz, DMSO-*d*₆) δ 12.19 (s, 1H), 8.94 (d, $J = 7.6$ Hz, 1H), 7.95 (d, $J = 7.6$ Hz, 1H), 7.70 – 7.63 (m, 2H), 7.62 – 7.48 (m, 6H), 7.38 (d, $J = 8.6$ Hz, 1H), 6.52 (s, 1H); ESI-MS: calculated m/z for $[C_{19}H_{13}NOH]^+$: 272.1, found: 272.1.

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SUPPORTING INFORMATION

Supplementary data (MOs calculated by DFT method) associated with this article can be found, in the online version, at <https://www.heterocycles.jp/newlibrary/downloads/PDFsi/26727/100/4>.

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