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# 布朗斯特酸/可见光氧化还原接力催化构建喹唑啉(硫)酮

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**摘要** 报道了一种布朗斯特酸/可见光氧化还原接力催化合成喹唑啉(硫)酮的方法. 在温和的反应条件下, 原料 2-氨基苯乙酮和异(硫)氰酸酯先后经历了布朗斯特酸催化的[4+2]环化反应和光催化的碳-碳键断裂反应, 得到各种不同取代的喹唑啉-2,4-二酮和喹唑啉-2-硫酮-4-酮. 该合成方法具有能源和溶剂绿色、无金属催化剂、起始原料简单以及以空气作为绿色氧化剂等特点.

**关键词** 接力催化; 可见光催化; 绿色合成; 喹唑啉酮

# Construction of Quinazolin(thi)ones by Brønsted Acid/Visible-Light Photoredox Relay Catalysis

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**Abstract** A relay Brønsted acid/visible-light photoredox catalysis strategy for the synthesis of quinazolin(thi)ones is developed. In this procedure, 2-aminoacetophenones and iso(thio)cyanates undergo Brønsted acid catalyzed [4+2] annulation, followed by photocatalyzed C—C bond cleavage reaction. A wide range of diversely substituted quinazoline-2,4-diones and quinazoline-2-thione-4-ones were synthesized under mild conditions. The protocol features eco-benign energy source and solvent, metal-free catalysts, simple starting materials and air as green oxidant.

**Keywords** relay catalysis; visible-light catalysis; green synthesis; quinazolinone

## 1 Introduction

Quinazolinone is a highly important skeleton that widely exists in biologically active compounds such as naturally occurring products and drugs.[1] Specifically, quinazoline-2,4-dione and its derivatives represent the medicinally and pharmaceutically important class of compounds due to their wide range of biological activities including antihypertensive,[2] antidiabetic,[3] antimicrobial,[4] antimalarial[5] and anticancer activities.[6] Several representative examples of the biologically active quinazoline-2,4-diones are shown in Figure 1.[7] In view of their interesting properties, great progress has been made in the architecture of the quinazoline-2,4-dione frameworks.[8] Willis *et al.*[9] disclosed a tandem palladium-catalyzed arylation-ester amidation sequence to deliver quinazoline-2,4-dione products (Scheme 1, a). Tang *et al.*[10] demonstrated a $ZnCl_2$-catalyzed condensation of aromatic *o*-aminonitriles with *N*,*N*-dimethylformamide (DMF) to obtain quinazoline-2,4-diones (Scheme 1, b). Beller *et al.*[11] developed a novel three-component reaction from readily available 2-bromoanilines, RNC, and $CO_2$ affording $N_3$-subsituted quinazoline-2,4-(1*H*,3*H*)-diones in a single step (Scheme 1, c). Beutner *et al.*[12] reported a nickel(0)-catalyzed synthesis of quinazolinediones from readily available isatoic anhydrides and isocyanates (Scheme 1, d). Despite these impressive achievements, it should be pointed out that these methods involve the use of costly metals as catalysts and

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demand harsh conditions, including high temperature and high pressure for the synthesis of quinazoline-2,4-diones. As a consequence, there is continued effort on developing eco-friendly strategies to access quinazoline-2,4-diones in a metal-free and mild approach. Inspired by the advantages of photoredox catalysis[13] and in continuation to our own efforts on the construction of quinazolin(thi)one derivatives,[14] herein, we present a visible-light and Brønsted acid relay catalyzed construction of skeleton of quinazoline-2,4-dione and quinazoline-2-thione-4-one from readily available 2-aminoacetophenones and iso(thio)cyanates (Scheme 1, e).

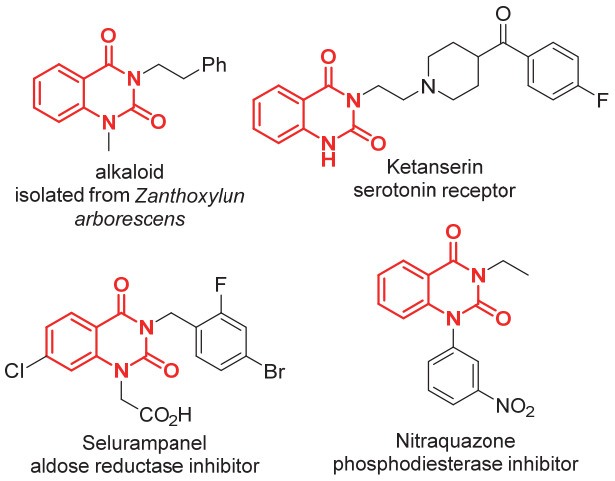
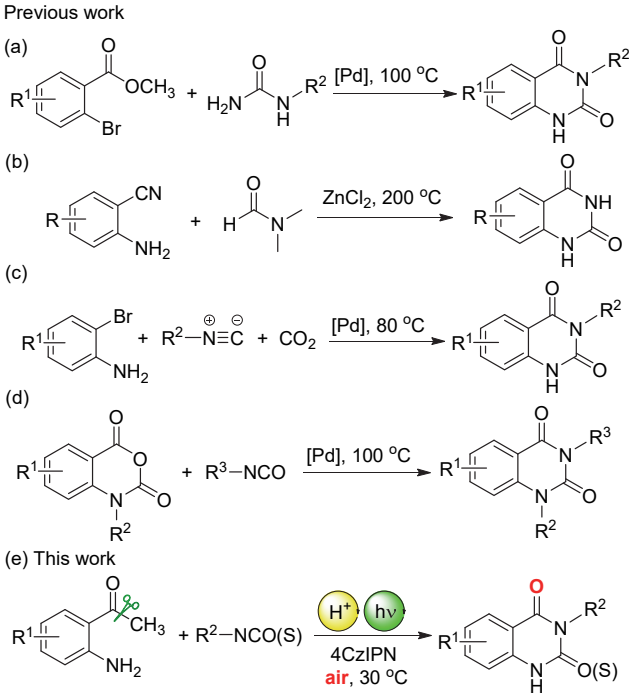



**Figure 1** Representative biologically active molecules containing the quinazoline-2,4-dione skeleton

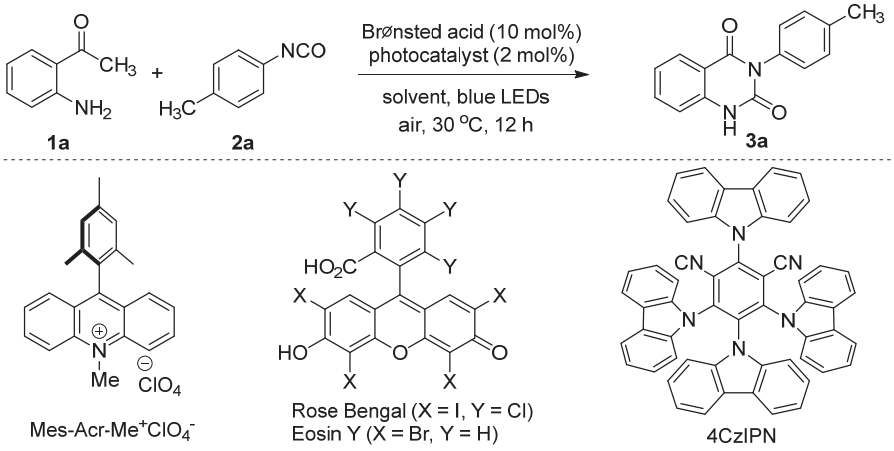



**Scheme 1** Previously reported routes for synthesis of quinazoline-2,4-dione frameworks and the strategy proposed herein

## 2 Results and discussion

The investigation was started with 2-aminoacetophenone (**1a**) and 4-tolyl isocyanate (**2a**) as the template substrates to evaluate the transformation (Table 1). Using TfOH as the

**Table 1** Optimization of reaction conditions[a]




| Entry | Brønsted acid | Photocatalyst | Solvent | Yield[b]/% |
|---|---|---|---|---|
| 1 | TfOH | $Ru(bpz)_3PF_6$ | DMSO | 46 |
| 2 | TFA | $Ru(bpz)_3PF_6$ | DMSO | 53 |
| 3 | AcOH | $Ru(bpz)_3PF_6$ | DMSO | 59 |
| 4 | $H_2SO_4$ | $Ru(bpz)_3PF_6$ | DMSO | 35 |
| 5 | $HNO_3$ | $Ru(bpz)_3PF_6$ | DMSO | 28 |
| 6 | TsOH | $Ru(bpz)_3PF_6$ | DMSO | 63 |
| 7 | TsOH | $Ru(bpm)_3PF_6$ | DMSO | 58 |
| 8 | TsOH | $[Ir(dF(CF_3)ppy)_2dtbbpy]PF_6$ | DMSO | 72 |
| 9 | TsOH | Mes-Acr-$Me^+ClO_4^-$ | DMSO | 64 |

Continued

| Entry | Brønsted acid | Photocatalyst | Solvent | Yield[b]/% |
|---|---|---|---|---|
| 10 | TsOH | Rose Bengal | DMSO | 36 |
| 11 | TsOH | Eosin Y | DMSO | 49 |
| 12 | TsOH | 4CzIPN | DMSO | 73 |
| 13 | TsOH | 4CzIPN | DMF | 70 |
| 14 | TsOH | 4CzIPN | MeCN | 81 |
| 15 | TsOH | 4CzIPN | THF | 57 |
| 16 | TsOH | 4CzIPN | $CHCl_3$ | 74 |
| 17 | TsOH | 4CzIPN | EtOH | 84 |

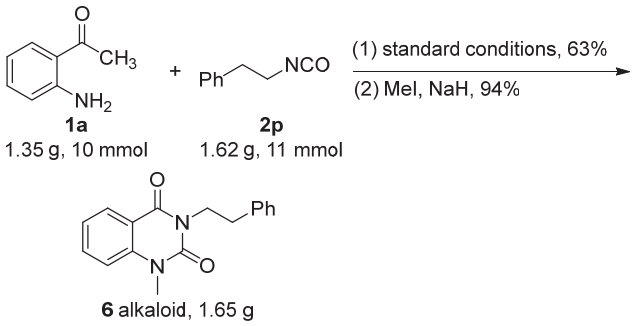
[a] Reaction conditions: **1a** (0.5 mmol), **2a** (0.55 mmol), Brønsted acid (0.05 mmol), photocatalyst (0.01 mmol), solvent (5 mL). [b]Isolated yield based on **1a**.

Brønsted acid and $Ru(bpz)_3PF_6$ as the photocatalyst, the model reaction was performed in dimethyl sulfoxide (DMSO) under blue light irradiation and open air. We were delighted to obtain the product 3-(*p*-tolyl)quinazoline-2,4(1*H*,3*H*)-dione (**3a**) in 46% yield after 12 h (Table 1, Entry 1). Subsequently, various Brønsted acids such as trifluoroacetic acid (TFA), AcOH, $H_2SO_4$, $HNO_3$ and TsOH were screened and TsOH was found to be the most effective Brønsted acid to obtain the target product in 63% yield (Entries 2～6). To our delight, the reaction proceeded with all used photosensitizers including transition-metal complexes such as $Ru(bpm)_3PF_6$ (58% yield, Entry 7) and [Ir(dF($CF_3$)ppy)$_2$dtbbpy]$PF_6$ (72% yield, Entry 8), as well as an organic photocatalyst (PC) such as Mes-Acr-$Me^+ClO_4^-$ (64% yield, Entry 9), rose bengal (36% yield, Entry 10), eosin Y (49% yield, Entry 11) and 4CzIPN (73% yield, Entry 12). 4CzIPN gave a similar result with [Ir(dF($CF_3$)ppy)$_2$dtbbpy]$PF_6$. To reduce the cost and enable a metal-free system, 4CzIPN was preferably chosen as the best PC. Then the reaction solvent was evaluated. *N*,*N*-Dimethylformamide (DMF) (Entry 13), acetonitrile (MeCN) (Entry 14), tetrahydrofuran (THF) (Entry 15) and chloroform ($CHCl_3$) (Entry 16) were found to be less effective to afford the product **3a** in 65% and 52% yields, respectively. Ethanol (EtOH) was found to be the best medium, producing quinazoline-2,4-dione in 84% yield (Entry 17).

After optimizing the reaction conditions, the substrate scope was explored for the reaction. As shown in Table 2, initially, a variety of aromatic and aliphatic isocyanates were evaluated. Electron-withdrawing and electron-donating substituted aromatic isocyanates were well-tolerated, giving the corresponding quinazoline-2,4-diones in 68%～91% yields (**3a**～**3o**). Various *para*-substituted aromatic isocyanates were all good substrates for this transformation, affording the desired products in moderate to excellent yields (**3a**～**3e**). Notably, fluoro- and chloro-substituted isocyanates were successfully transformed into the expected products **3c** and **3d** in 88% and 91% yields, respectively, leaving a chemical handle for further elaboration. *meta*-Substituted aromatic isocyanates could also be converted to the corresponding quinazoline-2,4-diones **3f**～**3h** in 86%～89% yields. Notably, this reaction was found to be sensitive to the steric hindrance. For instance, decreased yields were achieved when *ortho*-substituted phenyl isocyanates ($CH_3$, **2i**; $OCH_3$, **2j**; F, **2k**; Br, **2l**) were examined under the standard conditions, affording the corresponding products **3i**～**3l** in lower yields (68%～77%). Multisubstituted aromatic isocyanates were also successfully involved in the reaction, giving the quinazoline-2,4-diones **3m**～**3o** in 82%～89% yields. Delightfully, alkyl isocyanates could smoothly furnish the desired products **3p** and **3q**, albeit in moderate yields (63% and 61%, respectively). In addition, treatment of 4-tolyl isocyanate (**2a**) with 1-(2-amino-5-chlorophenyl)ethan-1-one (**1r**) or 1-(2-amino-4,5-dimethoxyphenyl)ethan-1-one (**1s**) under the established reaction conditions could afford the target products in good yields (**3r** and **3s**, 85% and 93%).

For the construction of quinazoline-2-thione-4-ones, the isothiocyanates (**4**) was next applied to react with **1a** to further examine the generality of this transformation (Table 3). Pleasingly, the reactions proceeded well under the optimal reaction conditions and various functional groups were well tolerated on the phenyl ring of isothiocyanates including F (**4a**), $CF_3$ (**4b**), CN (**4c**), Cl (**4d** and **4e**) and $OCH_3$ (**4f**) groups. The disubstituted isothiocyanate bearing two electron-withdrawing groups (3,5-ditrifluoromethyl) was also compatible with the protocol developed herein, delivering **5g** in 89% yield. Additionally, the alkyl isothiocyanate (**4h**) also proceeded smoothly to produce 3-benzyl-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one (**5h**) in 68% yield.

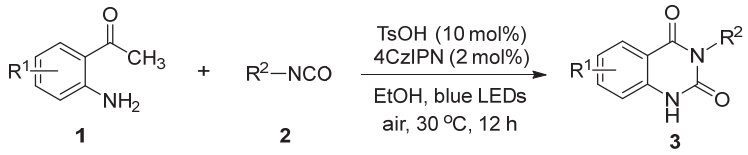
Furthermore, the methodology was applied for the synthesis of natural product **6**, isolated from *Zanthoxylun arborescens* (Scheme 2).[7a] Subjecting **1a** with **2p** to the standard conditions led to the isolation of 3-phenethylquinazoline-2,4(1*H*,3*H*)-dione (**3p**) with 63% yield. Methylation with methyl iodide and sodium hydride provided the product **6** in 94% yield.



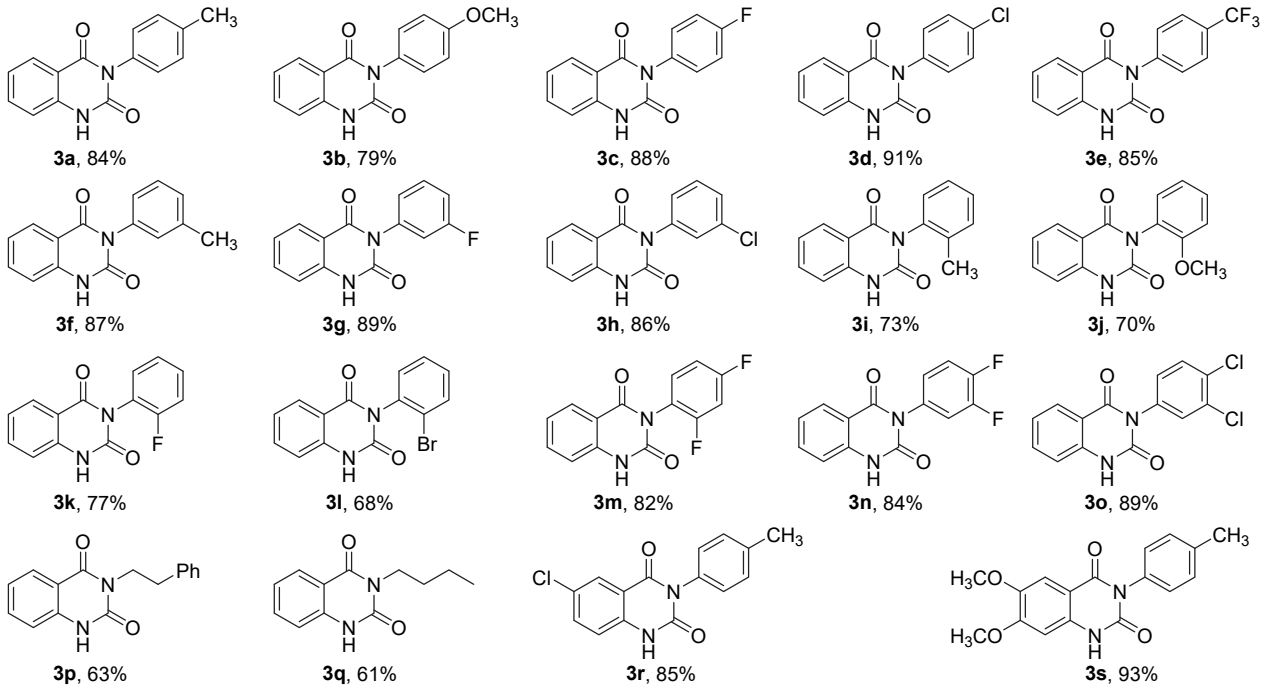

**Scheme 2** Synthesis of natural product **6**

**Table 2** Substrate scope expansion for quinazoline-2,4-dione synthesis[a,b]

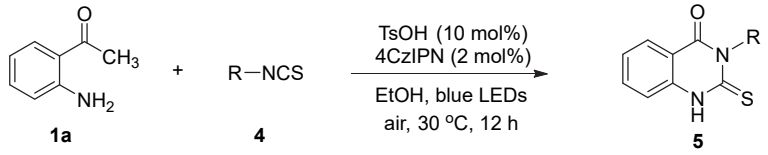



1 2 3

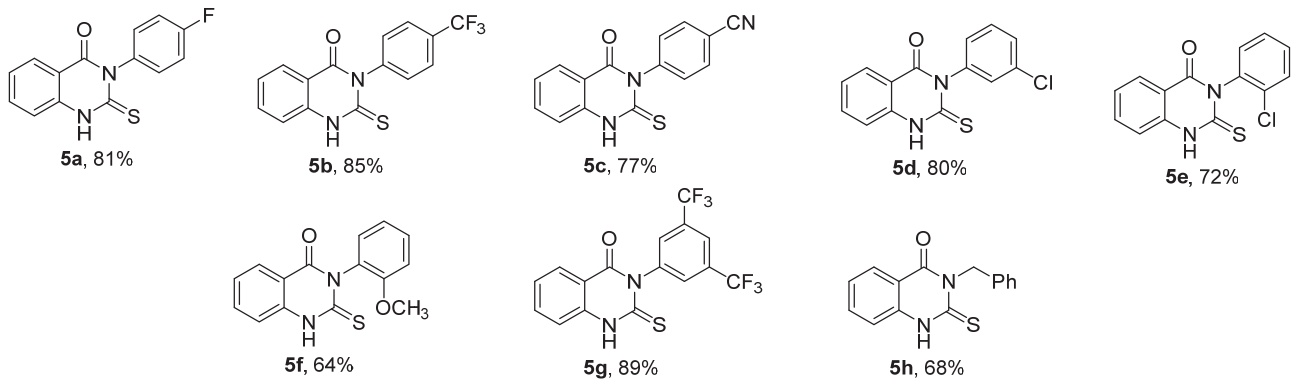



[a] Reaction conditions: **1** (0.5 mmol), **2** (0.55 mmol), TsOH (0.05 mmol), 4CzIPN (0.01 mmol), EtOH (5 mL). [b] Isolated yield based on **1**.

**Table 3** Substrate scope expansion for quinazoline-2-thione-4-ones synthesis[a,b]




1a 4 5



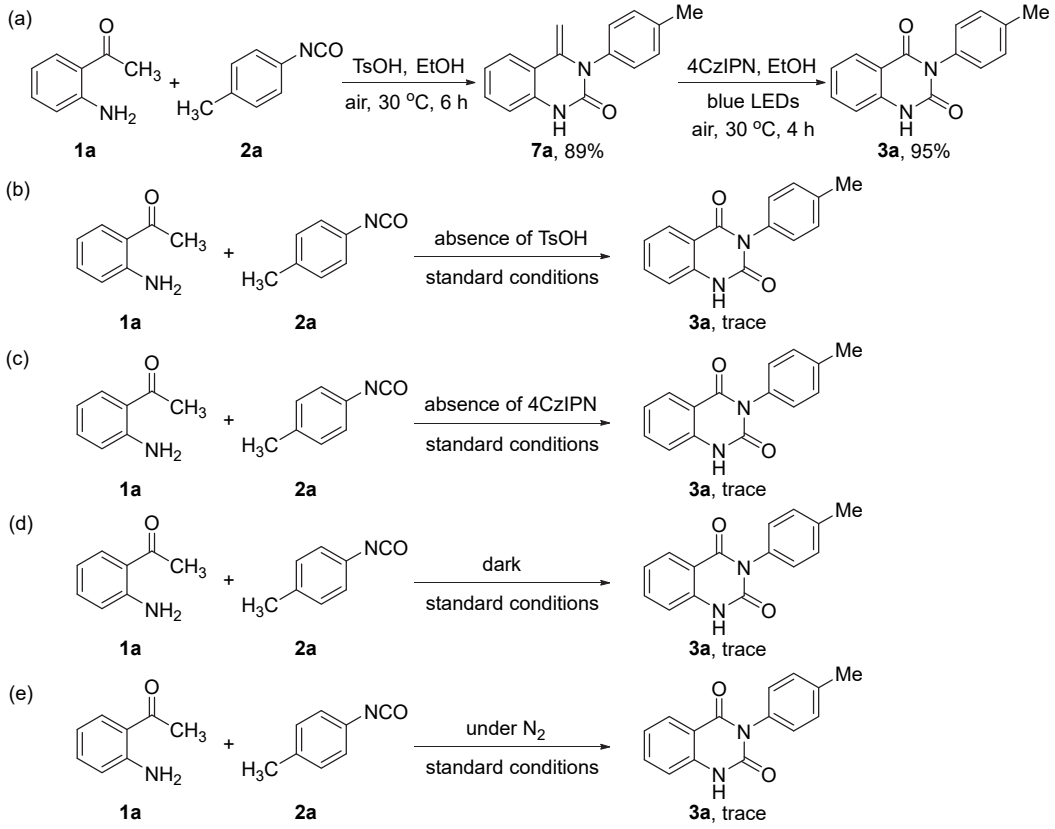

[a] Reaction conditions: **1a** (0.5 mmol), **4** (0.55 mmol), TsOH (0.05 mmol), 4CzIPN (0.01 mmol), EtOH (5 mL). [b] Isolated yield based on **1a**.

To gain insight into the reaction mechanism, several control experiments were conducted (Scheme 3). First, the reaction of 2-aminoacetophenone (**1a**) and 4-tolyl isocyanate (**2a**) was carried out in the presence of TsOH. [4+2] cycloaddition was observed, with the formation of 4-methylene-quinazolinone (**7a**) in 89% yield (Scheme 3, a). Furthermore, with 4CzIPN under visible light irradiation, the compound **7a** could be converted into the target product **3a** in 95% yield. These results suggested that **7a**, which was generated *in situ*, might be the key intermediate in this transformation. Additionally, in the absence of TsOH, 4CzIPN or visible light, the reactions failed to pro-

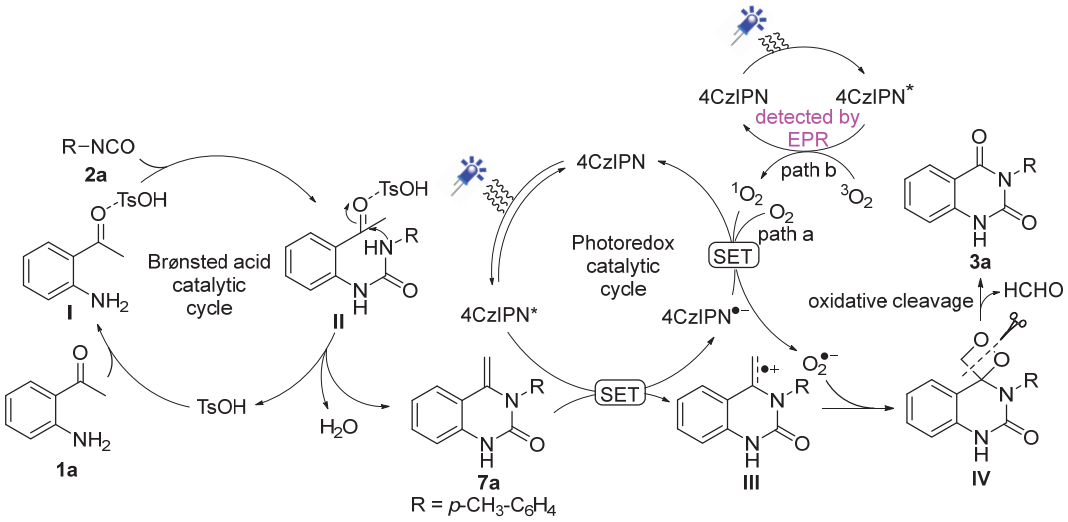
  

duce **3a** (Scheme 3, b～d). A similar result was achieved when the transformation was treated under $N_2$ atmosphere (Scheme 3, e).

On the basis of the control experiments and related reference,[15] a plausible mechanism for this reaction is depicted in Scheme 4. To begin with, 2-aminoacetophenone (**1a**) is coordinated with TsOH in the first Brønsted acid catalytic cycle to form the corresponding intermediate **I**, which acts as a nucleophile for an electrophilic addition of 4-tolyl isocyanate (**2a**). The resulting adduct **II** undergoes intramolecular cyclization to afford the 4-methylenequinazolinone **7a** and release the TsOH. Meanwhile, activation of the photocatalyst 4CzIPN by visible light produces a reactive species 4CzIPN* [$E_{1/2}^{red*}$ = +1.35 V (vs. SCE)][15a] which oxidizes **7** [$E_{ox}$ = +1.32 V (vs. SCE)] to radical cation **III** via single-electron oxidation (SET) and forms a 4CzIPN$^{\bullet-}$ intermediate.[15b] Meanwhile, the superoxide anion $O_2^{\bullet-}$ is produced via the SET or electron para-




**Scheme 3** Control experiments




**Scheme 4** Proposed mechanism

magnetic resonance (EPR) process. In the SET pathway (path a), the 4CzIPN$^{\bullet-}$ reduces $O_2$ to $O_2^{\bullet-}$ and regenerates the 4CzIPN.[15c] In the ET pathway (path b), $^1O_2$ was generated from $^3O_2$ with activated 4CzIPN* under light irradiation (detected by EPR).[15d,9] Subsequently, another SET process occurred between $^1O_2$ and 4CzIPN$^{\bullet-}$, giving $O_2^{\bullet-}$ and regenerating 4CzIPN for the next photoredox cycle.[15d] Finally, a cycloaddition between $O_2^{\bullet-}$ and radical cation intermediate **III** affords a dioxetane **IV**.[15b] The subsequent C—C bond cleavage of **IV** gives the desired product **3a**.[15e]

## 3 Conclusions

In conclusion, we have successfully developed a cascade process of Brønsted acid and visible-light relay catalysis for the [4+2] annulation/C—C bond cleavage reaction with air from readily available 2-aminoacetophenones and iso(thio)cyanates that can easily synthesize diverse quinazoline-2,4-diones and quinazoline-2-thione-4-ones in good yields. This new transformation is of great value from the viewpoint of green chemistry and organic synthesis as it features eco-friendly energy source and solvent, metal-free catalysts and air as green oxidant.

## 4 Experimental section

### 4.1 General information

All light-promoted reactions were carried out in borosilicate glass tubes with Teflon-coated magnetic stirring bars, and placed 2 cm from a commercial LED strip (12 W, 1 M, $\lambda$ 460～470 nm) with a fan placed upside for cooling. All starting materials and reagents were purchased from commercial sources and used as received unless otherwise noted. Reactions were monitored using thin-layer chromatography (TLC) on commercial silica gel plates. Visualization of the developed plates was performed under UV light (254 nm). NMR spectra data were obtained on a Avance (III) HD 400 MHz instruments. $^1$H NMR and $^{13}$C NMR spectra were referenced to residual protic solvent peaks or TMS signal (0 ppm). $^{19}$F NMR chemical shifts were externally referenced to $CCl_3F$ ($\delta$ 0).

### 4.2 General synthesis of quinazoline-2,4-diones **3** and quinazoline-2-thione-4-ones **5**

A solution of 2-aminoacetophenones **1** (0.5 mmol), iso(thio)cyanates **2** (**4**) (0.55 mmol), TsOH (0.05 mmol) and 4CzIPN (0.01 mmol) were dissolved in EtOH (5 mL) under an ambient atmosphere. The reaction mixture was stirred under irradiation (12 W blue LEDs) at room temperature (30 ℃) in the presence of open air for 12 h. The reaction solution was concentrated under vacuum, diluted with brine (5 mL) and extracted with EtOAc (10 mL×3). The combined organic layers were dried over $Na_2SO_4$ and concentrated in vacuo. The resulting residue was purified by silica gel column chromatography with petroleum ether/ethyl acetate as eluent to afford the desired products **3** or **5**.

3-(*p*-Tolyl)quinazoline-2,4(1*H*,3*H*)-dione (**3a**): White solid, 84% yield, 211.7 mg. m.p. 263～264 ℃ (lit.[16a] 265～267 ℃); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 10.15 (s, 1H), 8.13 (d, $J$=7.6 Hz, 1H), 7.48 (t, $J$=7.6 Hz, 1H), 7.34 (d, $J$=8.0 Hz, 2H), 7.21 (t, $J$=7.2 Hz, 3H), 6.83 (d, $J$=8.2 Hz, 1H), 2.45 (s, 3H). The experimental data are in agreement with the literature report.[16a]

3-(4-Methoxyphenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3b**): White solid, 79% yield, 211.7 mg. m.p. 299～300 ℃ (lit.[16a] 298～300 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.51 (s, 1H), 7.93 (dd, $J$=8.2, 1.2 Hz, 1H), 7.73～7.69 (m, 1H), 7.21 (dd, $J$=8.0, 5.6 Hz, 4H), 7.02 (t, $J$=6.0 Hz, 2H), 3.80 (s, 3H). The experimental data are in agreement with the literature report.[16a]

3-(4-Fluorophenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3c**): White solid, 88% yield, 225.3 mg. m.p. 266～268 ℃ (lit.[16a] 265～267 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.57 (s, 1H), 7.94 (d, $J$=7.6 Hz, 1H), 7.70 (t, $J$=7.6 Hz, 1H), 7.39 (dd, $J$=8.8, 5.2 Hz, 2H), 7.31 (t, $J$=8.8 Hz, 2H), 7.26～7.19 (m, 2H). The experimental data are in agreement with the literature report.[16a]

3-(4-Chlorophenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3d**): White solid, 91% yield, 247.5 mg. m.p. 288～290 ℃ (lit.[16a] 290～292 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.72 (s, 1H), 7.96 (d, $J$=7.6 Hz, 1H), 7.80 (s, 1H), 7.73 (t, $J$=7.2 Hz, 1H), 7.51 (s, 2H), 7.44～7.39 (m, 1H), 7.27 (d, $J$=7.8 Hz, 2H). The experimental data are in agreement with the literature report.[16a]

3-(4-(Trifluoromethyl)phenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3e**): White solid, 85% yield, 260.1 mg. m.p. 281～283 ℃ (lit.[16b] >250 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.66 (s, 1H), 7.95 (dd, $J$=8.0, 1.2 Hz, 1H), 7.88 (d, $J$=8.4 Hz, 2H), 7.75～7.67 (m, 1H), 7.62 (d, $J$=8.0 Hz, 2H), 7.29～7.20 (m, 2H). The experimental data are in agreement with the literature report.[16b]

3-(*m*-Tolyl)quinazoline-2,4(1*H*,3*H*)-dione (**3f**): White solid, 87% yield, 219.2 mg. m.p. 281～283 ℃ (lit.[16c] 278～280 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.56 (s, 1H), 7.93 (dd, $J$=8.2, 1.2 Hz, 1H), 7.78～7.64 (m, 1H), 7.36 (t, $J$=7.6 Hz, 1H), 7.29～7.20 (m, 3H), 7.11 (d, $J$=11.6 Hz, 2H). The experimental data are in agreement with the literature report.[16c]

3-(3-Fluorophenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3g**): Yellow solid, 89% yield, 227.8 mg. m.p. 261～263 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.62 (s, 1H), 7.94 (d, $J$=7.6 Hz, 1H), 7.71 (t, $J$=7.6 Hz, 1H), 7.53 (dd, $J$=14.8, 7.6 Hz, 1H), 7.30 (t, $J$=9.2 Hz, 2H), 7.23 (t, $J$=8.4 Hz, 3H). The experimental data are in agreement with the literature report.[16d]

3-(3-Chlorophenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3h**): White solid, 86% yield, 233.9 mg. m.p. 284～286 ℃ (lit.[16a] 283～285 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.61 (s, 1H), 7.93 (d, $J$=7.6 Hz, 1H), 7.71 (t, $J$=7.6 Hz, 1H), 7.50 (s, 3H), 7.33 (s, 1H), 7.23 (d, $J$=7.6 Hz, 2H). The experimental data are in agreement with the literature report.[16a]

3-(*o*-Tolyl)quinazoline-2,4(1*H*,3*H*)-dione (**3i**): White solid, 73% yield, 184.0 mg. m.p. 253～255 ℃ (lit.[16a] 253～255 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.62 (s,

1H), 7.95 (d, $J$=7.6 Hz, 1H), 7.72 (t, $J$=7.6 Hz, 1H), 7.22～7.36 (m, 6H), 2.04 (s, 3H). The experimental data are in agreement with the literature report.[16a]

3-(2-Methoxyphenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3j**): White solid, 70% yield. 187.6 mg. m.p. 291～293 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.56 (s, 1H), 7.93 (d, $J$=7.6 Hz, 1H), 7.71 (t, $J$=7.2 Hz, 1H), 7.42 (t, $J$=7.6 Hz, 1H), 7.24 (s, 3H), 7.16 (d, $J$=8.0 Hz, 1H), 7.04 (t, $J$=7.2 Hz, 1H), 3.70 (s, 3H). The experimental data are in agreement with the literature report.[16e]

3-(2-Fluorophenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3k**): White solid, 77% yield, 197.1 mg. m.p. 256～258 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.75 (s, 1H), 7.97 (dd, $J$=8.0, 1.2 Hz, 1H), 7.79～7.70 (m, 1H), 7.57～7.46 (m, 2H), 7.41 (t, $J$=8.8 Hz, 1H), 7.37～7.30 (m, 1H), 7.27 (dd, $J$=7.6, 5.6 Hz, 2H). The experimental data are in agreement with the literature report.[16d]

3-(2-Bromophenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3l**): White solid, 68% yield, 214.9 mg. m.p. 305～307 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.61 (s, 1H), 7.92 (d, $J$=7.6 Hz, 1H), 7.68 (t, $J$=7.2 Hz, 1H), 7.53 (d, $J$=8.0 Hz, 2H), 7.36 (d, $J$=8.0 Hz, 2H), 7.22 (t, $J$=7.6 Hz, 2H). The experimental data are in agreement with the literature report.[16d]

3-(2,4-Difluorophenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3m**): White solid, 82% yield, 224.7 mg. m.p. 272～274 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.77 (s, 1H), 7.96 (dd, $J$=8.0, 1.2 Hz, 1H), 7.74 (dd, $J$=10.8, 4.7 Hz, 1H), 7.60 (dd, $J$=14.8, 8.4 Hz, 1H), 7.49 (t, $J$=9.6 Hz, 1H), 7.31～7.20 (m, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 163.7 (d, $J$=12 Hz), 162.1, 161.3 (d, $J$=12 Hz), 159.6 (d, $J$=13 Hz), 157.1 (d, $J$=13 Hz), 149.9, 140.3, 136.2, 133.1 (d, $J$=9 Hz), 128.1, 123.4, 120.0 (d, $J$=9 Hz), 116.0, 114.2, 112.4 (d, $J$=19 Hz), 105.1 ((dd, $J$=27, 24 Hz); HRMS (ESI-TOF) calcd for $C_{14}H_9F_2N_2O_2$ $[M+H]^+$ 275.0627, found 275.0632.

3-(3,4-Difluorophenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3n**): White solid, 84% yield, 230.2 mg. m.p. 268～270 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.62 (s, 1H), 7.94 (dd, $J$=8.0, 1.2 Hz, 1H), 7.79～7.67 (m, 1H), 7.63～7.49 (m, 2H), 7.33～7.18 (m, 3H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 162.6, 150.9 (d, $J$=13 Hz), 150.5, 148.4 (d, $J$=13 Hz), 140.3, 135.8, 132.8 (d, $J$=9 Hz), 128.0, 127.1 (d, $J$=3 Hz), 123.1, 119.4 (d, $J$=18 Hz), 117.8 (d, $J$=18 Hz), 115.7, 114.7; HRMS (ESI-TOF) calcd for $C_{14}H_9F_2N_2O_2$ $[M+H]^+$ 275.0627, found 275.0632.

3-(3,4-Dichlorophenyl)quinazoline-2,4(1*H*,3*H*)-dione (**3o**): White solid, 89% yield, 272.3 mg. m.p. 283～285 ℃ (lit.[16f] 282～283 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.66 (s, 1H), 7.94 (d, $J$=7.6 Hz, 1H), 7.78 (d, $J$=7.2 Hz, 2H), 7.72 (t, $J$=7.6 Hz, 1H), 7.42 (d, $J$=8.4 Hz, 1H), 7.24 (t, $J$=7.0 Hz, 2H). The experimental data are in agreement with the literature report.[16f]

3-Phenethylquinazoline-2,4(1*H*,3*H*)-dione (**3p**): White solid, 63% yield, 167.6 mg. m.p. 277～279 ℃; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 10.54 (s, 1H), 8.15 (d, $J$=8.0 Hz, 1H), 7.64 (t, $J$=7.6 Hz, 1H), 7.33 (q, $J$=7.6 Hz, 4H), 7.24 (dd, $J$=7.2, 4.4 Hz, 2H), 7.15 (d, $J$=8.0 Hz, 1H), 4.44～4.22 (m, 2H), 3.10～2.95 (m, 2H). The experimental data are in agreement with the literature report.[16b]

3-Butylquinazoline-2,4(1*H*,3*H*)-dione (**3q**): White solid, 61% yield, 133.0 mg. m.p. 152～154 ℃ (lit.[16g] 153～154 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.43 (s, 1H), 7.92 (d, $J$=7.6 Hz, 1H), 7.65 (t, $J$=7.2 Hz, 1H), 7.19 (dd, $J$=13.6, 7.6 Hz, 2H), 3.88 (t, $J$=7.2 Hz, 2H), 1.60～1.48 (m, 2H), 1.30 (dd, $J$=14.8, 7.2 Hz, 2H), 0.90 (t, $J$=7.2 Hz, 3H). The experimental data are in agreement with the literature report.[16g]

6-Chloro-3-(*p*-tolyl)quinazoline-2,4(1*H*,3*H*)-dione (**3r**): White solid, 85% yield, 243.1 mg. m.p. 290～292 ℃ (lit.[16a] 291～293 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.69 (s, 1H), 7.86 (d, $J$=2.4 Hz, 1H), 7.75 (dd, $J$=8.8, 2.4 Hz, 1H), 7.27 (dd, $J$=13.6, 8.4 Hz, 3H), 7.19 (d, $J$=8.0 Hz, 2H), 2.37 (s, 3H). The experimental data are in agreement with the literature report.[16a]

6,7-Dimethoxy-3-(*p*-tolyl)quinazoline-2,4(1*H*,3*H*)-dione (**3s**): White solid, 93% yield, 290.2 mg. m.p. 312～314 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 11.34 (s, 1H), 7.26 (d, $J$=6.4 Hz, 3H), 7.15 (d, $J$=8.0 Hz, 2H), 6.71 (s, 1H), 3.85 (s, 3H), 3.79 (s, 3H), 2.37 (s, 3H). The experimental data are in agreement with the literature report.[16h]

3-(4-Fluorophenyl)-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one (**5a**): White solid, 81% yield, 220.3 mg. m.p. 206～208 ℃ (lit.[16i] 207～209 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 13.08 (s, 1H), 7.96 (d, $J$=7.6 Hz, 1H), 7.79 (t, $J$=7.6 Hz, 1H), 7.45 (d, $J$=8.0 Hz, 1H), 7.39～7.28 (m, 5H). The experimental data are in agreement with the literature report.[16i]

2-Thioxo-3-(4-(trifluoromethyl)phenyl)-2,3-dihydroquinazolin-4(1*H*)-one (**5b**): Yellow solid, 85% yield, 273.7 mg. m.p. 249～251 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 13.15 (s, 1H), 7.97 (d, $J$=7.6 Hz, 1H), 7.88 (d, $J$=8.0 Hz, 2H), 7.81 (t, $J$=7.6 Hz, 1H), 7.58 (d, $J$=8.0 Hz, 2H), 7.46 (d, $J$=8.0 Hz, 1H), 7.37 (t, $J$=7.6 Hz, 1H). The experimental data are in agreement with the literature report.[16e]

4-(4-Oxo-2-thioxo-1,4-dihydroquinazolin-3(2*H*)-yl)benzonitrile (**5c**): White solid, 77% yield, 214.8 mg. m.p. 267～269 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 13.17 (s, 1H), 7.97 (dd, $J$=12.8, 4.8 Hz, 3H), 7.83～7.77 (m, 1H), 7.58 (d, $J$=8.4 Hz, 2H), 7.46 (d, $J$=8.4 Hz, 1H), 7.37 (t, $J$=7.6 Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 175.9, 160.2, 144.2, 140.1, 136.2, 133.6, 131.2, 127.9, 125.0, 119.0, 116.7, 116.3, 111.5; HRMS (ESI-TOF) calcd for $C_{15}H_{10}N_3OS$ $[M+H]^+$ 280.0539, found 280.0533.

3-(3-Chlorophenyl)-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one (**5d**): White solid, 80% yield, 230.4 mg. m.p. 271～273 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 13.12 (s, 1H), 7.96 (d, $J$=8.0 Hz, 1H), 7.79 (t, $J$=7.6 Hz, 1H), 7.58～7.42 (m, 4H), 7.41～7.26 (m, 2H). The experimental data are in agreement with the literature report.[16e]

3-(2-Chlorophenyl)-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one (**5e**): White solid, 72% yield, 207.4 mg. m.p. 244～246 ℃ (lit.[16i] 246～247 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 13.22 (s, 1H), 7.99 (dd, $J$=8.0, 0.8 Hz, 1H),

7.86～7.78 (m, 1H), 7.62 (dt, *J*=6.8, 3.2 Hz, 1H), 7.52～7.46 (m, 4H), 7.39 (dd, *J*=11.2, 4.0 Hz, 1H). The experimental data are in agreement with the literature report.[16i]

3-(2-Methoxyphenyl)-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one (**5f**): Yellow solid, 64% yield, 181.8 mg. m.p. 247～249 ℃ (lit.[16i] 245～247 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 13.07 (s, 1H), 8.00～7.89 (m, 1H), 7.84～7.75 (m, 1H), 7.49～7.33 (m, 3H), 7.24 (dd, *J*=7.6, 1.2 Hz, 1H), 7.15 (d, *J*=8.0 Hz, 1H), 7.05 (dd, *J*=11.2, 4.0 Hz, 1H), 3.72 (s, 3H). The experimental data are in agreement with the literature report.[16i]

3-(3,5-Bis(trifluoromethyl)phenyl)-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one (**5g**): Yellow solid, 89% yield, 347.1 mg. m.p. 255～257 ℃; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 13.26 (s, 1H), 8.23 (d, *J*=6.4 Hz, 3H), 8.02～7.95 (m, 1H), 7.86～7.78 (m, 1H), 7.48 (d, *J*=8.4 Hz, 1H), 7.39 (t, *J*=7.6 Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$: 176.1, 160.4, 141.9, 140.1, 136.3, 131.6, 131.2, 127.8, 125.0, 124.9, 122.7, 122.2, 116.8, 116.3. HRMS (ESI-TOF) calcd for $C_{16}H_9F_6N_2OS$ $[M+H]^+$ 391.0334, found 391.0336.

3-Benzyl-2-thioxo-2,3-dihydroquinazolin-4(1*H*)-one (**5h**): White solid, 68% yield, 182.2 mg. m.p. 293～295 ℃ (lit.[16i] 291～293 ℃); $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$: 13.10 (s, 1H), 7.96 (dd, *J*=8.0, 0.8 Hz, 1H), 7.82～7.72 (m, 1H), 7.43 (d, *J*=8.0 Hz, 1H), 7.38～7.19 (m, 6H), 5.67 (s, 2H). The experimental data are in agreement with the literature report.[16i]

### 4.3 Synthesis of alkaloid **6**

A mixture of 1-(2-aminophenyl)ethan-1-one (**1a**, 1.35 g, 10 mmol), (2-isocyanatoethyl)benzene (**2p**, 1.62 g, 11 mmol), TsOH (172 mg, 1.0 mmol) and 4CzIPN (158 mg, 0.2 mmol) was dissolved in EtOH (100 mL) under an ambient atmosphere. The reaction mixture was stirred under irradiation (12 W blue LEDs) at room temperature (30 ℃) in the presence of open air for 12 h. The reaction solution was concentrated under vacuum and then diluted with brine (100 mL) and extracted with EtOAc (50 mL×3). The combined organic layers were dried over $Na_2SO_4$ and concentrated in vacuo. The resulting residue was purified by silica gel column chromatography with petroleum ether/ethyl acetate as eluent to afford the **3p** (1.68 g, 63% yield) as white solid.

The reaction in a sealed tube was carried out with **3p** (1.68 g, 6.3 mmol), MeI (228 mg, 9.5 mmol) and NaH (158 mg, 11.3 mmol) in dry DMSO (20 mL) at 80 ℃ for 16 h. The reaction mixture was diluted with brine (100 mL) and extracted with EtOAc (30 mL×3). The combined organic layers were dried over $Na_2SO_4$ and concentrated in vacuo. The resulting residue was purified by silica gel column chromatography with petroleum ether/ethyl acetate as eluent to afford the desired natural product alkaloid (1.66 g, 94% yield) as a yellow solid.

1-Methyl-3-phenethylquinazoline-2,4(1*H*,3*H*)-dione (**6**): Yellow solid, 59% yield, 1.65 g. m.p. 100～102 ℃ (lit.[16j] 99～101 ℃); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$: 8.25 (d, *J*=7.6 Hz, 1H), 7.70 (t, *J*=7.6 Hz, 1H), 7.32 (dt, *J*=14.8, 7.2 Hz, 5H), 7.23 (t, *J*=8.0 Hz, 2H), 4.37～4.25 (m, 2H), 3.62 (s, 3H), 3.02～2.94 (m, 2H). The experimental data are in agreement with the literature report.[16j]

**Supporting Information** Cyclic voltammetry (CV) measurements, determination of electron paramagnetic resonance (EPR) and NMR spectra. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn.

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