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SET-oxidative photocyclization reactions of *N*-(α-trimethylsilyl)-*N*-phenylglycinates with *N*-substituted maleimides: a role of silyl group in photocyclization

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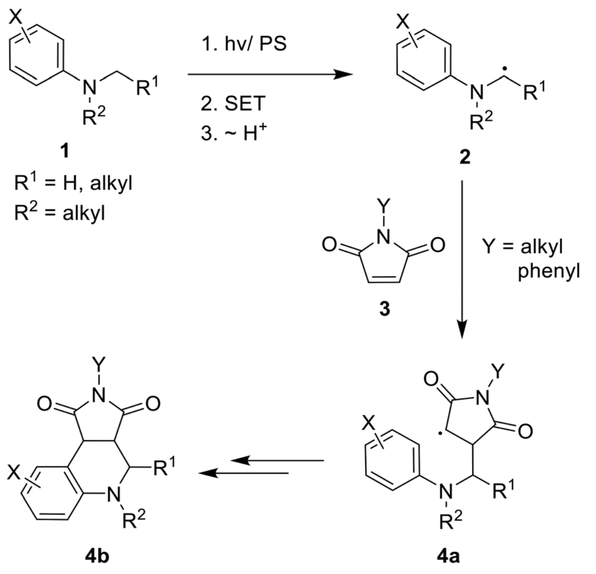
SET-promoted photocyclization reactions of *N*-(α-trimethylsilyl)-*N*-phenylglycinates with *N*-substituted maleimides were explored using methylene blue (MB) as a photosensitizer, and mechanistic studies were performed. In contrast to the non-silylated *N*-phenylglycinate substrates, which showed no reactivity with maleimides, the MB-photosensitized reactions of *N*-(α-trimethylsilyl)-*N*-phenylglycinates with maleimides led to the formation of tetrahydroquinoline derivatives efficiently. These observations reveal that the silyl group adjacent to the nitrogen atom plays a crucial role in enabling a successful SET-oxidative cyclization reaction. Overall, this reaction protocol can expand the range of amine substrates in the diverse SET-oxidative photochemical reactions.

Introduction

Since the recognition of photoinduced single electron transfer (SET) process as a central tool in synthetic photochemistry, numerous SET-promoted photochemical reactions using amine electron donors have been explored so far, mainly due to the low oxidation potentials (E_{ox} < 1 V *vs.* SCE) of aliphatic- and aromatic amines and unique chemical behaviors of *in situ* formed intermediates, amine radical cations.[1–8] For instance, when suitable electron acceptors (*i.e.*, either electron-accepting reactant or photosensitizer) with an excited-state reduction potential ($^*E_{red}$) higher than the oxidation potential of amines (E_{ox}) are used, photoirradiation can induce SET from amines to the acceptors, generating amine radical cations. These radical cations participate in diverse chemical pathways depending on the structure of amines and the reaction conditions.[3–9] The most common reaction pathways open to these intermediates are either α-C(sp^3)–H deprotonation to form α-amino radicals or a secondary SET process to form iminium ions.[1,10–12] A hydrogen-atom-transfer (HAT) process can result in the formation of iminium ions, providing an alternative pathway. Then, either α-amino radicals or iminium ions react with electrophilic or nucleophilic substrates to form amine moiety-containing molecules.

As one of the valuable synthetic applications of this chemistry, SET-oxidative photocyclization reactions between *N*,*N*-dialkylanilines and *N*-substituted maleimides to construct tetrahydroquinolines have drawn considerable attention due to a broad range of pharmacological and biological interest of the formed heterocycles.[9–15] In the presence of a photosensitizer—such as a transition metal complex,[13–15] metal oxide[16] or organic dyes—[17–20] irradiation with visible or UV light promotes the efficient conversion of *N*,*N*-dimethylanilines **1** to α-amino radicals **2** through a sequential SET- α-CH deprotonation (Scheme 1). These radicals were added to *N*-alkyl/aryl mal-



Scheme 1 SET-promoted photocyclization reactions of *N*,*N*-dialkylanilines with *N*-substituted maleimides.

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eimides **3** to form intermediates **4a**, which then underwent cyclization, followed by dehydrogenation, to form the tetrahydroquinolines **4b**. Recently, a diverse set of photocatalyst-free photochemical methods was also developed for the preparation of tetrahydroquinolines.[21–23] Alternatively, thermochemical reactions of *N*,*N*-dialkylanilines with *N*-substituted maleimides using either various oxidants or a combination of oxidant and metal ionic salts have also been explored to prepare tetrahydroquinolines.[24–32]

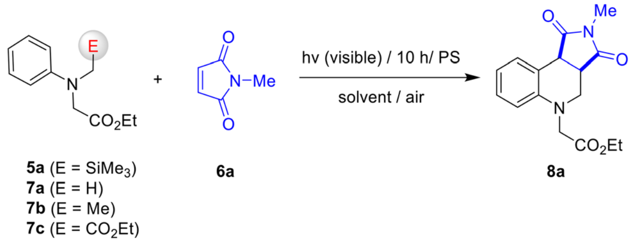
Despite the synthetic utility of these photo- and thermochemical methods, the substrate scope of amines remains largely restricted to *N*,*N*-dialkylanilines. In particular, Bian and coworkers showed that, unlike *N*,*N*-dialkylanilines, photocyclization reactions of anilines bearing electron-withdrawing groups, such as *N*-methyl-*N*-phenylglycinates (PhN(Me)CH_2CO_2Et), with *N*-phenylmaleimide proceeded inefficiently and produced unwanted byproducts, such as oxamates, competitively.[13] Thus, to overcome these limitations and expand substrate applicability, the development of more general synthetic protocols is required. Building on our recent success with silyl group-containing amine substrates (*i.e.*, *N*-α-silylamines) in SET-promoted reactions,[33–36] we envisioned that the silyl group within amine substrates could facilitate efficient α-amino radical formation, resulting in enhancing radical addition to maleimides and cyclization efficiency in the preparation of N-heterocyclic tetrahydroquinolines. Thus, as part of our continuing research efforts, we explored SET-oxidative photocyclization reactions of *N*-α-trimethylsilyl-*N*-phenylglycinates, bearing various substituents on the phenyl ring, with *N*-substituted maleimides. The results showed that SET-oxidative photocyclization reactions proceeded efficiently to form tetrahydroquinolines, and the silyl group played a crucial role in the cyclization. Detailed results are described and discussed below.

Results and discussion

To explore SET-oxidative photocyclization reactions of electron-withdrawing group tethered amines, *N*-α-trimethylsilyl-*N*-phenylglycinate **5a** and *N*-methylmaleimide **6a** were chosen as a model substrate. For the photoreaction, the solutions (15 mL) containing glycinate **5a** (0.38 mmol, 1 equiv.) and maleimide **6a** (0.75 mmol, 2 equiv.) in the presence of photosensitizer (3 or 5 mol%) were irradiated with a 20 W compact fluorescent lamp (CFL) under air at room temperature. To figure out the role of the silyl group within the glycinate substrate, the photoreaction of non-silylated **7a–7c**[37] with **6a** was also conducted under the same reaction conditions.

As shown in Table 1, the methylene blue (MB) -photosensitized photoreactions of **5a** with **6a** in MeOH solvent led to the efficient and exclusive formation of tetrahydroquinoline **8a** (entry 1). The stereochemistry of the formed tetrahydroquinoline **8a** was determined by comparison with the previously reported tetrahydroquinoline analogs.[38–44] In polar aprotic solvents, both conversion- and photoproduct yields dropped significantly (entries 2–5). These observations seem to result from silophilic MeOH-assisted fast desilylation (~$SiMe_3^+$) rather than α-CH deprotonation, leading to the generation of an α-amino radical intermediate.[7,8,33,34,36] Although other visible-light photosensitizers such as rose Bengal (RB), eosin-Y (EY), and $Ru(bpy)_3Cl_2$ could lead to the formation of **8a** as well, these sensitizers were less efficient than MB (entries 6–11). From a further screening of reaction conditions, we found that 5 mol% MB is the optimal amount for this SET-oxidative cyclization reaction (entry 12). Control experiments revealed that both photosensitizer and visible light were essential to these photochemical transformation reactions (entries 13, 14). Noticeably, the MB-photosensitized reaction of non-silylated glycinate **7a–7c** with maleimide **6a** did not afford any detectable photoproducts (entries 15, 16), even in the much longer visible-light irradiation condition (up to 30 h). (The glycinates **7a–7c** remained intact).

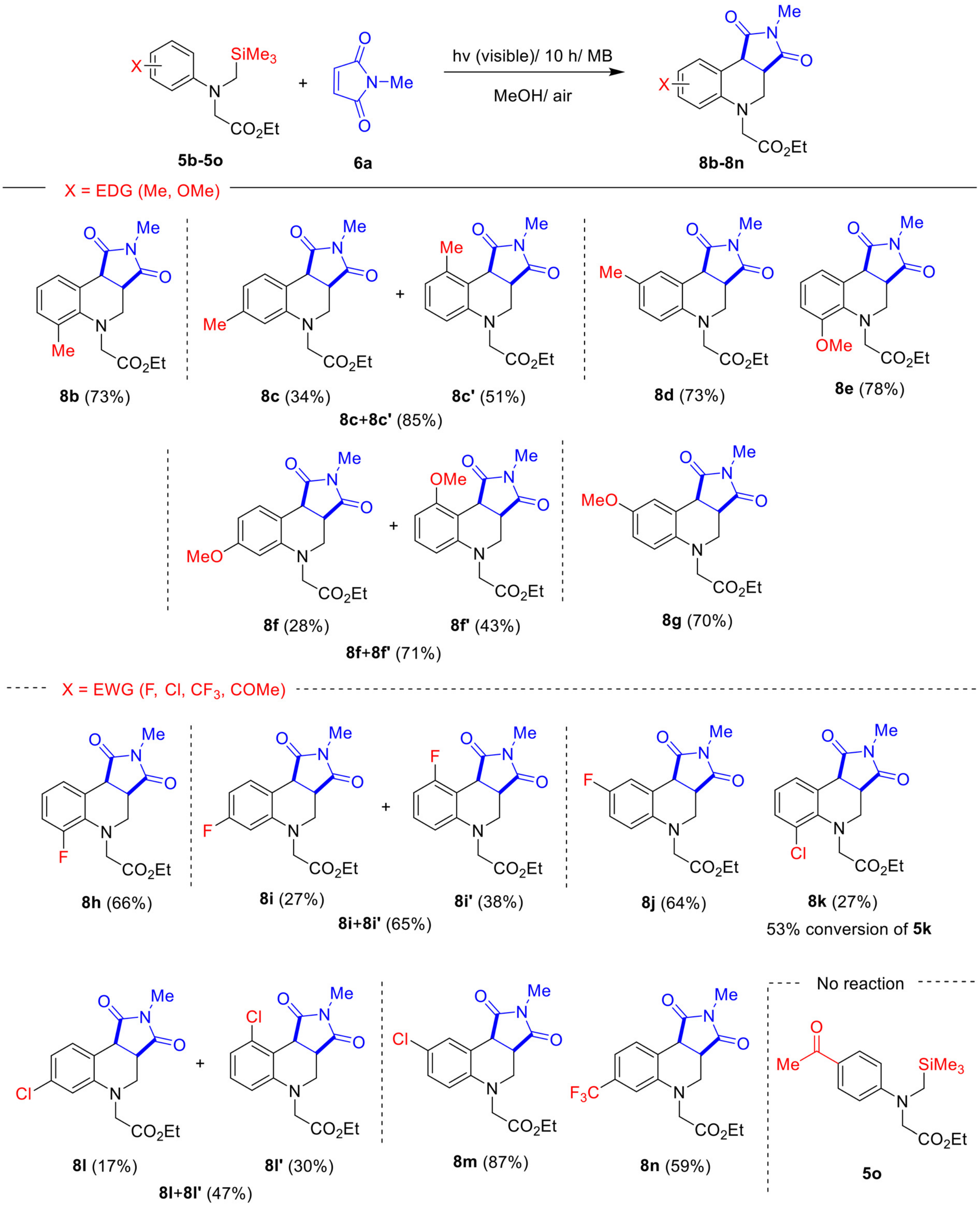
Table 1 Screening of SET-oxidative photocyclization of glycinate (**5a** and **7a–7c**) with *N*-methylmaleimide **6a**[a]



Entry	Glycinate	Reaction conditions	Conversion yield[b] (%)	Product yield[c] (%)
1	**5a**	MB[d]/MeOH	100	79
2	**5a**	MB[d]/MeCN	21	9
3	**5a**	MB[d]/DMF	65	34
4	**5a**	MB[d]/DMSO	49	20
5	**5a**	MB[d]/CH_2Cl_2	28	13
6	**5a**	RB[d]/MeCN	54	28
7	**5a**	RB[d]/MeOH	100	58
8	**5a**	EY[d]/MeCN	74	24
9	**5a**	EY[d]/MeOH	72	36
10	**5a**	$Ru(bpy)_3Cl_2$ [d]/MeCN	100	64
11	**5a**	$Ru(bpy)_3Cl_2$ [d]/MeOH	100	74
12	**5a**	MB[e]/MeOH	100	85
13[f]	**5a**	MB[e]/MeOH	0	n.d[g]
14	**5a**	No/MeOH	0	n.d[g]
15	**7a**	MB[e]/MeOH	0	n.d[g]
16	**7b**	MB[e]/MeOH	0	n.d[g]
17	**7c**	MB[e]/MeOH	0	n.d[g]

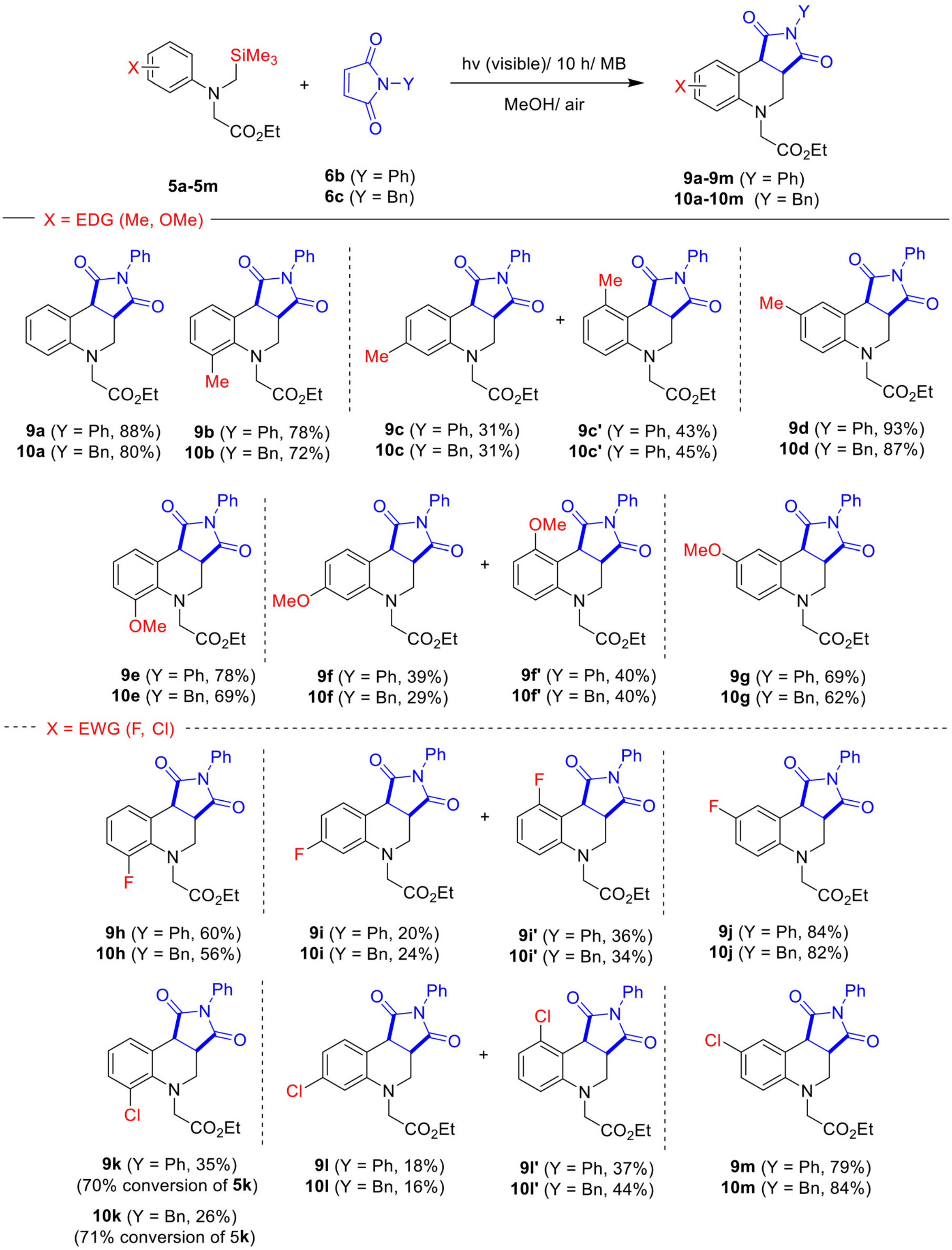
[a] Reaction conditions: the solutions (15 mL) of glycinate (**5a** or **7a–7b**, 0.38 mmol, 1 equiv.), *N*-methylmaleimide **6a** (0.75 mmol, 2 equiv.), and photosensitizer (3 or 5 mol%) were irradiated using a 20 W CFL for 10 h in the air. [b] Determined by recovered glycinate **5a** or **7a–7b**. [c] Isolation yields. [d] 3 mol% PS was used. [e] 5 mol% PS was used. [f] Reaction in the dark (no light). [g] Not detected. The starting glycinates remained intact.

With optimized reaction conditions in hand (entry 12 in Table 1), we explored the substrate scope for this photochemical reaction. Initially, we assessed the performances of *N*-α-trimethylsilyl-*N*-phenylglycinates **5b–5o**, bearing either various electron-donating (Me, OMe) or electron-withdrawing (F, Cl, CF_3, COMe) groups at *ortho*, *meta*, or *para* position of the phenyl ring. As depicted in Scheme 2, 10 h visible-light



Scheme 2 SET-oxidative photocyclization reactions of glycinate **5b–5o** with *N*-methylmaleimide **6a**. Reaction conditions: the MeOH solutions (15 mL) of glycinate (**5b–5o**, 0.38 mmol, 1 equiv.), *N*-methylmaleimide **6a** (0.75 mmol, 2 equiv.), and MB (5 mol%) were irradiated using a 20 W CFL for 10 h in the air. Otherwise noted, conversion yields of these photoreactions were 100%.

irradiation of MeOH solutions containing either methyl (Me)- or methoxy (OMe) group-containing glycinates **5b–5g** with maleimide **6a** furnished the corresponding tetrahydroquinolines **8b–8g** in moderate yields ranging from 70% to 85%. The MB-photosensitized reactions of fluoro- (**5h–5j**), chloro- (**5k–5m**), or trifluoromethyl (**5n**)-substituted glycinates provided the expected photoproducts **8h–8n**, but the yields obtained from the electron-withdrawing substituents **5h–5l** (except **5m**) were generally lower than those of their electron-donating analogues **5b–5g**. The photoreactions using acyl-substituted glycinate **5o** failed to afford the corresponding products, with the starting materials remaining unchanged. Notably, the photoreaction of *ortho*-chlorophenyl-substituted glycinate **5k** was sluggish, with only 53% conversion after 10 h of visible-light



Scheme 3 SET-oxidative photocyclization of glycinates **5a–5m** with *N*-phenyl or *N*-benzyl maleimide **6b–6c**. Reaction condition: the MeOH solutions (15 mL) of glycinate (**5a–5m**, 0.38 mmol, 1 equiv.), maleimide **6b–6c** (0.75 mmol, 2 equiv.), and MB (5 mol%) were irradiated using a 20 W CFL for 10 h in the air. Otherwise noted, conversion yields of all glycinates were 100%.

irradiation, yielding a low yield (27%) of photoproduct **8k**. In contrast, the photoreaction of *para*-chlorophenyl substituted glycinate **5m** with **6a** produced the highest yield (87%) of photoproduct **8m**. It is also noteworthy that the MB-photosensitized reactions of glycinates bearing *meta*-substituents (**5c**, **5f**, **5i**, **5l**) consistently afforded regioisomeric mixtures, in which one isomer (**8c′**, **8f′**, **8i′**, and **8l′**) predominated over the other (**8c**, **8f**, **8i**, and **8l**).[13,17,25,27]

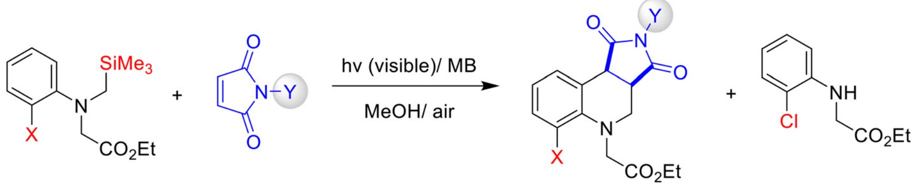
To examine the substrate scope of *N*-substituted maleimides, the MB-photosensitized reactions of *N*-α-trimethylsilyl-*N*-phenylglycinates **5a–5m** with *N*-phenyl- (**6b**) and *N*-benzylmaleimide (**6c**) were performed under the optimized conditions. As illustrated in Scheme 3, photoreactions between glycinates **5a–5m** and *N*-phenylmaleimide **6b** proceeded efficiently, yielding tetrahydroquinolines **9a–9m**. As with **5k** and **6a**, the photoreaction of glycinate **5k** (X = *o*-Cl) with **6b** was less reactive, affording only 70% conversion and 35% product yield under the optimized conditions. Likewise, similar trends were observed in the photochemical reactions of glycinates **5a–5m** with **6c**, where **5k** produced **10k** with 71% conversion and 26% product yield, underscoring its consistently lower reaction efficiency in the SET-oxidative photocyclization reactions (Scheme 3).

Because glycinate **5k** exhibited low reaction efficiency (*i.e.*, poor conversion and low product yields), we anticipated that extending the visible-light irradiation time for its reactions with **6a–6c** might enable complete consumption of **5k**, thereby improving product yields. However, even after extending the visible-light irradiation time to 25 h, no significant increase in the yields of **8k–10k** was observed. Instead, substantial formation of a secondary glycinate **11** was detected (Table 2). Notably, no analogous secondary glycinate was observed in the photoreactions of **5b** (X = *o*-Me) and **5h** (X = *o*-F) (entries). Although mechanistic details remain unclear at this stage, it is plausible that, unlike other glycinates, the radical cation of **5k**, generated *via* SET between **5k** and MB, undergoes competitive secondary photochemical pathways (*e.g.*, desilylation *vs.* H-atom abstraction followed by hydrolysis).

To assess the generality of these photochemical reactions, a range of amine derivatives bearing electron-withdrawing groups—such as benzyloxycarbonyl (CO_2Bn), cyano (CN), acetyl (COMe), and benzoyl (COPh) (**12a–12h**)—as well as chiral glycinates (**13a–13b**) were examined as substrates in MB-photosensitized reactions. As shown in Scheme 4a, silyl-free amine substrates **12e–12h** did not undergo any conversion during the photochemical reaction and remained unchanged. In contrast, amine substrates bearing both silyl- and electron-withdrawing groups—namely **12a** (EWG = CO_2Bn), **12b** (EWG = CN), **12c** (EWG = COMe), and **12d** (EWG = COPh)-reacted efficiently with **6a** to produce the corresponding tetrahydroquinoline **14a–14d** in good yields (65–83%). In the photoreactions using chiral glycinate derivatives **13a** and **13b**, two diastereomeric products (**15a**/**15a′** and **15b**/**15b′**) were formed, respectively, in approximately 1 : 1 ratios (Scheme 4b). Unfortunately, the stereochemistry of these isomeric photoproducts could not be determined due to their non-crystalline nature.

To verify the synthetic usefulness of this photochemical method, a gram-scale reaction using glycinate **5a** and several

Table 2 Extended visible-light irradiation time in SET-oxidative photocyclization of glycinate **5k** with *N*-substituted maleimide **6a–6c**[a]



5k (X = Cl)
5b (X = Me)
5h (X = F)

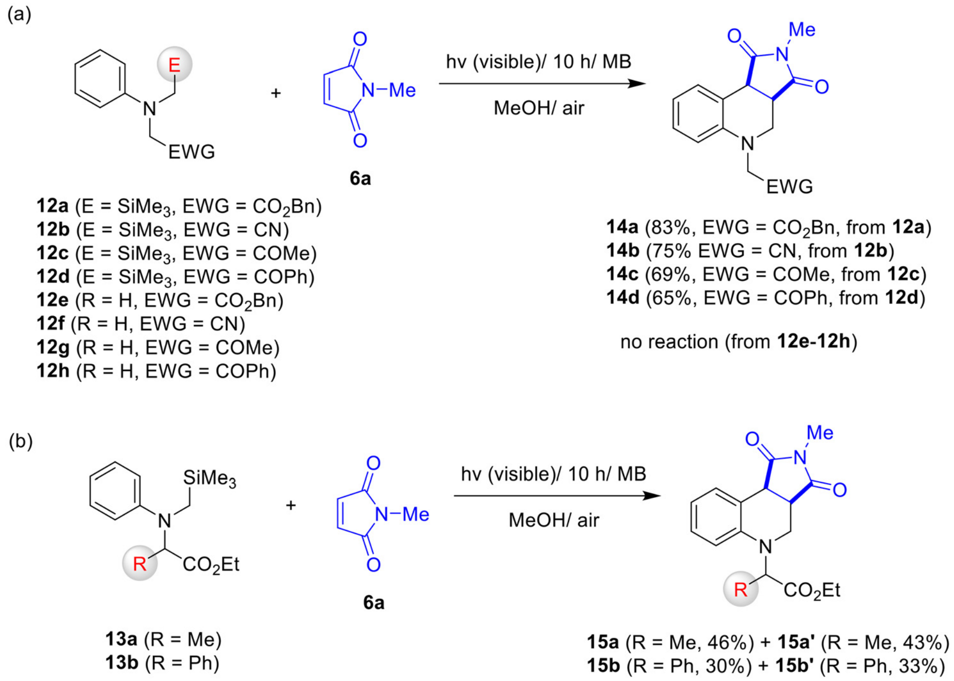
6a (Y = Me)
6b (Y = Ph)
6c (Y = Bn)

8k (X = Cl, Y = Me)
9k (X = Cl, Y = Ph)
10k (X = Cl, Y = Bn)
8b (X = Me, Y = Me)
8h (X = F, Y = Me)

11

Entry	Substrate	Irradiation time (h)	Conversion yield[b] (%)	Product yield[c] (%)
1	**5k** + **6a**	10	53	**8k** (27)
2	**5k** + **6a**	25	100	**8k** (35), **11** (23)
3	**5k** + **6b**	10	70	**9k** (35)
4	**5k** + **6b**	25	100	**8k** (41), **11** (22)
5	**5k** + **6c**	10	71	**10k** (26)
6	**5k** + **6c**	25	100	**10k** (40), **11** (33)
7	**5b** + **6a**	10	100	**8b** (73)
8	**5b** + **6a**	25	100	**8b** (71)
9	**5h** + **6a**	10	100	**8h** (66)
10	**5h** + **6a**	25	100	**8h** (65)

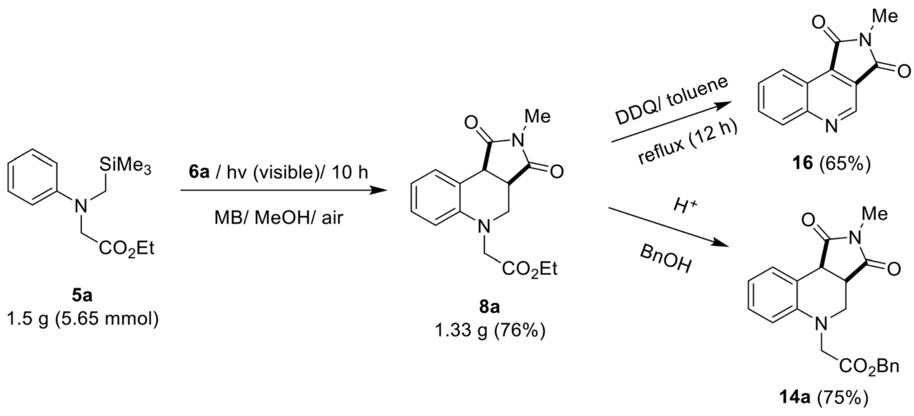
[a] Reaction condition: the MeOH solutions (15 mL) of glycinate (**5b**, **5h**, and **5k**, 0.38 mmol, 1 equiv.), maleimide (**6a–6c**, 0.75 mmol, 2 equiv.), and MB (5 mol%) were irradiated using a 20 W CFL for fixed irradiation time (10 and 25 h) in the air. [b] Determined by recovered glycinate **5b**, **5h**, and **5k**. [c] Isolation yields.



Scheme 4 SET-oxidative photocyclization reactions of amines (**12a–12h**) with *N*-methylmaleimide **6a**.

synthetic transformations was performed. MB-photosensitized reaction of **5a** (1.5 g, 5.65 mmol, 1 equiv.) with **6a** (2 equiv.) afforded **8a** in 76% yield (1.33 g). Reflux of **8a** at 120 °C in the presence of DDQ led to the formation of quinoline **16**,[29] and transesterification of **8a** with BnOH afforded **14a** in high yield (75%) (Scheme 5).

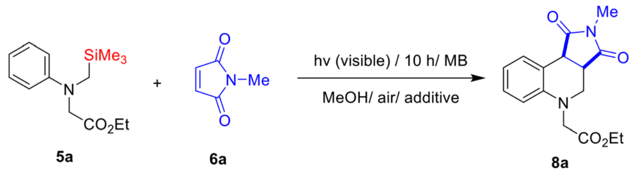
To gain a better understanding of the mechanistic pathways involved in tetrahydroquinoline formation, several experiments were carried out. To clarify the initial involvement of the SET process between glycinate substrates and MB, the photoluminescence quenching experiment of MB in the presence of varying concentrations of either silyl- (**5a**, **5d**, **5g**, **5j**) or silyl-free (**7**) glycinate substrates (0–3 mM) was monitored (Fig. S1 in SI). We envisioned that because the oxidation potentials of employed glycinate electron donors ($E_{\text{ox}} < 1$ V *vs.* SCE) are considerably lower than the reduction potential of triplet state of MB ($^3E_{\text{red}} = 1.6$ V *vs.* SCE), as a consequence, the SET process from glycinates to 3MB* would be thermodynamically favorable.[33,34,45–47] The results showed that the photoluminescence of MB gradually decreased with increasing concentration of the employed glycinates. Notably, although the photoluminescence of MB was quenched by non-silyl glycinate **7**, no reaction leading to photoproduct formation took place (Fig. S1 in SI). This suggests that the desilylation process ($\sim$SiMe$_3^+$) of the initially generated amine radical cations plays a crucial role in efficiently forming α-amino radicals, which serve as a key precursor in the formation of tetrahydroquinoline derivatives.



Scheme 5 Gram-scale reaction and synthetic application.

Furthermore, the results from the MB-sensitized photoreactions of **5a** with **6a** in the presence of various additives provided valuable mechanistic insight (Table 3). First, when 2 equiv. of DABCO, a known singlet oxygen ($^1O_2$) quencher, were added, no significant changes in either the conversion or product yields were observed. This indicates that $^1O_2$ is not involved in the reaction pathway, despite MB's ability to generate $^1O_2$ ($\Phi(^1O_2) = 0.52$). In contrast, the addition of TEMPO or BHT, well-known radical quenchers, led to a dramatic decrease in both conversion and product yields. These findings strongly suggest that radical intermediates are involved in the formation of the photoproducts.

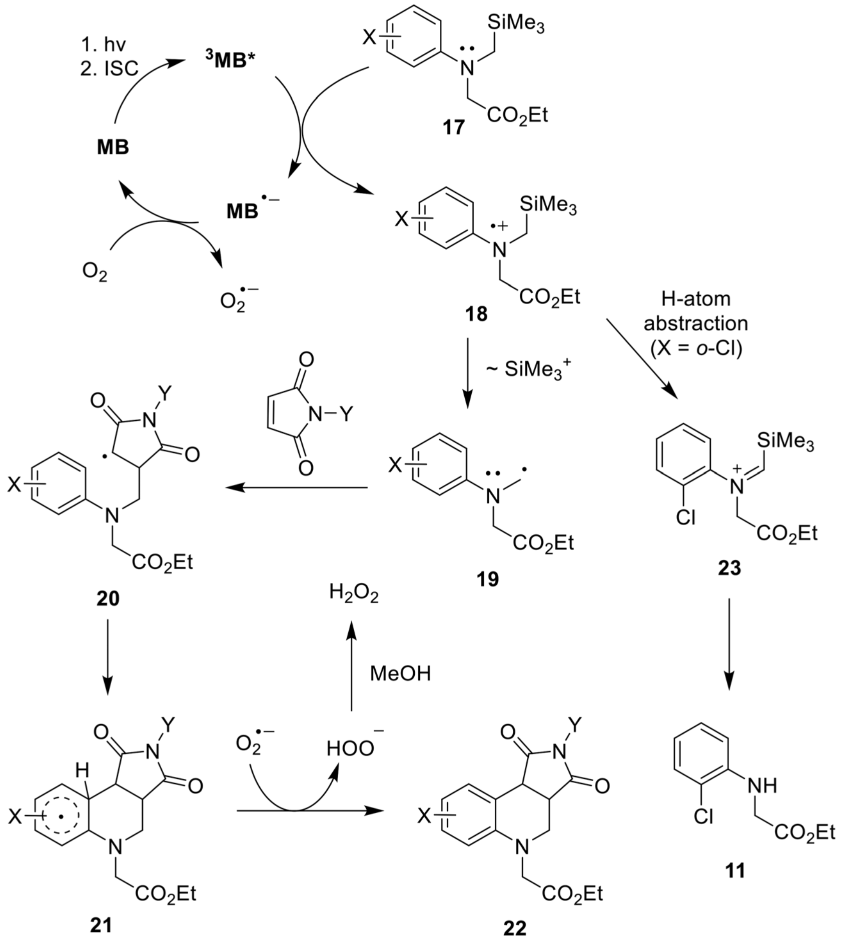
Table 3 SET-oxidative photocyclization reactions of glycinate **5a** with **6a** in the presence of an additive[a]



Additive	Conversion yield[b] (%)	Yield[c] (%)
No	100	85
DABCO (2 equiv. for **5a**)	100	83
TEMPO (2 equiv. for **5a**)	52	38
BHT (2 equiv. for **5a**)	43	26

[a] Reaction conditions: the solutions (15 mL) of **5a** (0.38 mmol, 1 equiv.), **6a** (0.75 mmol, 2 equiv.), and MB (5 mol%) were irradiated using a 20 W CFL for 10 h in the air. [b] Determined by recovered glycinate **5a**. [c] Isolation yields.

Based on the above observations and the previous reports,[17–20,48–51] a feasible mechanistic pathway responsible for the MB-sensitized photocyclization reactions was suggested (Scheme 6). Through visible-light irradiation, SET occurs from glycinates **17** to the photochemically formed triplet state of MB (^{3}MB*) to produce glycinate radical cations and radical anions of MB ($MB^{\bullet-}$). While $MB^{\bullet-}$ ions subsequently react with O_2 to regenerate the ground-state MB and $O_2^{\bullet-}$, the generated glycinate radical cations **18** undergo solvent-assisted desilylation to produce α-amino radicals **19**. These intermediates then add to the ene moiety of the maleimides to produce radical intermediates **20**, which undergo intramolecular cyclization reactions, followed by dehydrogenative oxidation, to form tetrahydroquinolines **22**. When the *ortho*-chlorophenyl



Scheme 6 Reaction mechanism.

substituted glycinate **17** (X = *o*-Cl) is employed, its radical cation **18** (X = *o*-Cl) can also undergo competitive H-atom abstraction to form iminium ion **23**, which is subsequently hydrolyzed to yield secondary amine **11**.

Conclusion

We investigated SET-oxidative photocyclization reactions of electron-deficient *N*-(α-trimethylsilyl)-*N*-phenylglycinates with *N*-substituted maleimides under visible light using the organic photosensitizer methylene blue (MB). The results revealed that, unlike their non-silylated *N*-alkyl-*N*-phenylglycinate counterparts, *N*-(α-trimethylsilyl)-*N*-phenylglycinates underwent efficient formation of α-amino radicals *via* sequential SET-desilylation processes, in which these radicals subsequently reacted with maleimides to afford tetrahydroquinoline products. Notably, the presence of the silyl group at the α-position to the nitrogen atom proved critical for the success of cyclization reactions. Overall, this photochemical strategy offers a valuable approach for expanding the scope of amine substrates in the diverse SET-promoted photochemical reactions.

Conflicts of interest

There are no conflicts to declare.

Data availability

The data supporting this article are included in the supplementary information (SI). Supplementary information: experimental section, ^{1}H and ^{13}C NMR spectroscopic data for compounds, and MB photoluminescence quenching data by glycinates. See DOI: https://doi.org/10.1039/d5ob01839k.

Acknowledgements

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