

RESEARCH ARTICLE OPEN ACCESS

Visible Light-Induced Three-Component 1,2-Acylarylation of Quinoxalin-2(1*H*)-Ones with Styrenes and Oxime EstersSudip Sau¹ | Rahul Kumar Patel¹  | Shinobu Takizawa²  | Hun Young Kim³  | Kyungsoo Oh¹ ¹Center for Metareceptome Research, Graduate School of Pharmaceutical Sciences, Chung-Ang University, Seoul, Republic of Korea | ²SANKEN, The University of Osaka, Osaka, Japan | ³Department of Global Innovative Drugs, Chung-Ang University, Seoul, Republic of Korea**Correspondence:** Shinobu Takizawa (taki@sanken.osaka-u.ac.jp) | Hun Young Kim (hunykim@cau.ac.kr) | Kyungsoo Oh (kyungsooh@cau.ac.kr)**Received:** 13 April 2026 | **Revised:** 27 May 2026 | **Accepted:** 1 June 2026**Keywords:** oxime esters | quinoxalin-2(1*H*)-ones | styrenes | visible light

ABSTRACT

Visible light-driven transformations offer an attractive strategy for sustainable synthesis by simplifying reaction systems while maintaining high efficiency. Herein, we report an exogenous photocatalyst-free visible light-induced three-component 1,2-acylarylation of quinoxalin-2(1*H*)-ones with styrenes and acyl oxime esters under mild conditions. Exploiting the intrinsic photoactivity of quinoxalin-2(1*H*)-ones, direct excitation under purple LED irradiation enables single electron transfer (SET) to acyl oxime esters, generating acyl radicals without the need for external photocatalysts. The resulting radicals undergo a sequential addition to alkenes and quinoxalin-2(1*H*)-ones, affording structurally diverse C3-functionalized products in moderate to good yields. The current three-component coupling protocol exhibits broad substrate scope, tolerating a range of styrenes, quinoxalin-2(1*H*)-ones, and benzoyl oxime esters. Mechanistic studies, including radical trapping experiments, fluorescence quenching, and UV–Vis analysis, support a SET-driven radical relay pathway involving a photoexcited quinoxalin-2(1*H*)-one. This operationally simple and modular approach highlights the dual role of quinoxalin-2(1*H*)-ones as both substrate and photoactive species, expanding the scope of exogenous photocatalyst-free multicomponent radical transformations.

1 | Introduction

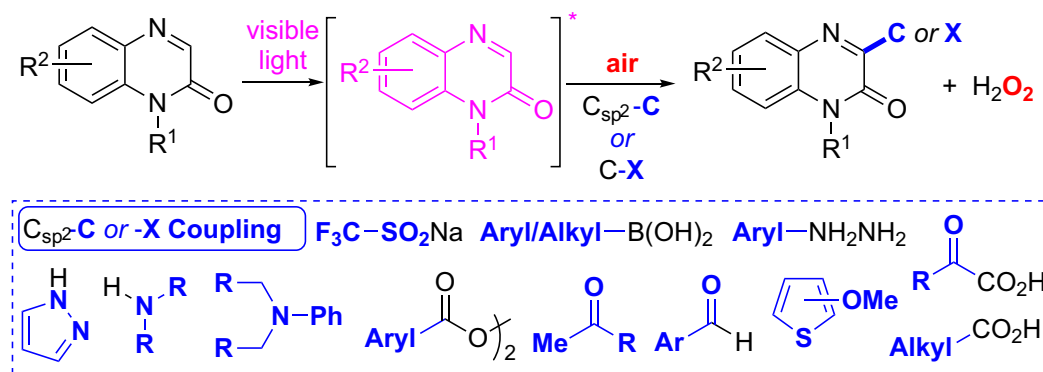
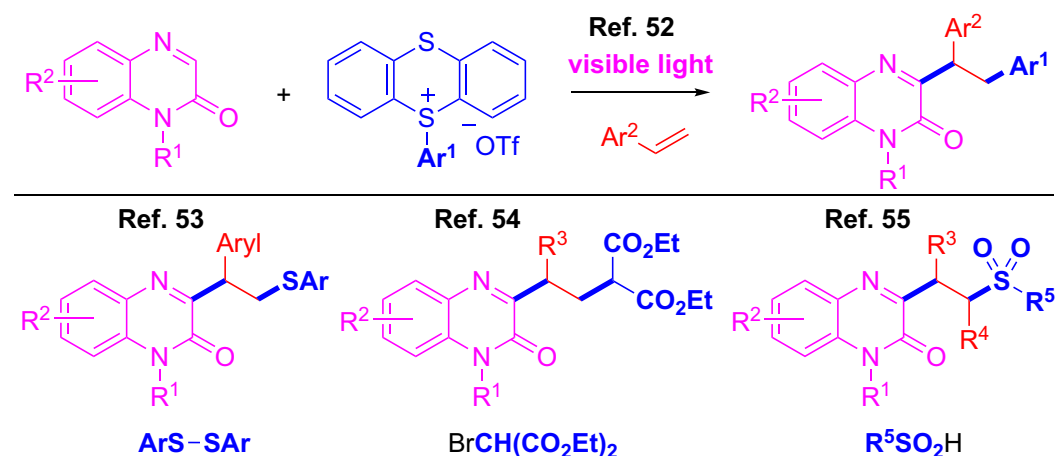
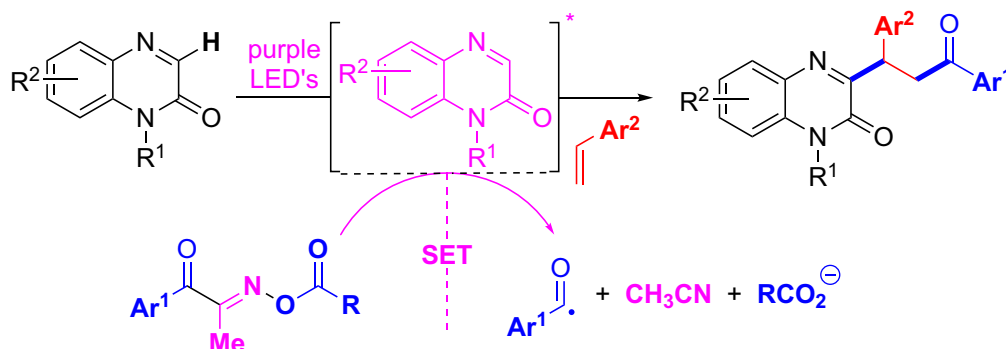
Visible light-promoted organic transformations have emerged as a powerful platform for sustainable chemical synthesis, enabling access to reactive open-shell intermediates under mild conditions without the need for stoichiometric redox reagents or harsh reaction conditions [1–5]. Although recent advances in visible light photoredox catalysis have substantially expanded precision-driven radical functionalization of both unsaturated and saturated molecules [6–12], exogenous photocatalyst-free visible light-driven reactions are particularly attractive, as they minimize reaction complexity, improve operational simplicity, and enhance functional group tolerance while aligning closely with the principles of green chemistry [12–15]. Among visible light photochromic heteroaromatic compounds [16–19], quinoxalin-2(1*H*)-ones have attracted growing interest in visible light

photochemistry owing to their distinctive electronic properties and broad relevance in medicinal and materials chemistry [20]. The photochemical Minisci-type reactions of quinoxalin-2(1*H*)-ones have been predominantly investigated in the context of photoredox catalysis [21–25]. Alternatively, recent advances have revealed that quinoxalin-2(1*H*)-ones can enable diverse C–C and C–X bond-forming reactions by functioning as self-sensitized photoactive species, either through single electron transfer (SET) pathways [26–38] or through the formation of charge-transfer or electron donor–acceptor (EDA) complexes that become photoredox-active under visible-light irradiation (Scheme 1a) [39–45]. In these exogenous photocatalyst-free or self-catalyzed photochemical transformations, quinoxalin-2(1*H*)-ones or molecular oxygen commonly serve as the terminal oxidants. The intrinsic visible light-induced photoreactivity of quinoxalin-2(1*H*)-ones has thus opened new opportunities for

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(a) Visible Light-Excitable Quinoxalin-2(1*H*)-ones in **C-C** and **C-X** Bond Formations

(b) Visible Light Self-Catalysis of Quinoxalin-2(1*H*)-ones in Three-Component Reactions(c) Proposed Visible Light-Induced 1,2-Acylation of Quinoxalin-2(1*H*)-ones

SCHEME 1 | Visible light-excitable quinoxalin-2(1*H*)-ones and proposed three-component 1,2-acylarylation of quinoxalin-2(1*H*)-ones.

simplified reaction systems, while maintaining high reactivity and selectivity. This feature has likely contributed to recent advances in visible light-promoted multicomponent reactions involving quinoxalin-2(1*H*)-ones [46–49], enabling rapid molecular complexity generation through radical relay processes in a single operation [50, 51]. Consistent with the self-sensitizable character of quinoxalin-2(1*H*)-ones under visible light, our groups recently reported an exogenous photocatalyst-free visible light-induced three-component 1,2-di(hetero)arylation of styrenes using quinoxalin-2(1*H*)-ones and thianthrenium salts (Scheme 1b) [52]. Inspired by recent advances in the visible light self-catalysis of quinoxalin-2(1*H*)-ones in the three-component reactions explored by Roy [53], Jin [54], and Yang [55], combined

with our ongoing interest in exogenous photocatalyst-free photochemical processes [56–61], we envisioned that quinoxalin-2(1*H*)-ones could engage in an exogenous photocatalyst-free visible light-promoted three-component reaction with acyl oxime esters, using alkenes as a bridging component to relay radical reactivity (Scheme 1c). Acyl oxime esters serve as versatile acyl radical precursors in visible light photoredox catalysis, as pioneered by Wu and co-workers in 2019 through efficient visible light-induced single electron reduction [62, 63]. While acyl oxime esters have been widely adopted in photoredox-catalyzed transformations to enable diverse bond-forming reactions [64], their application in photocatalyst-free visible light systems remains to be explored. The strategy envisioned herein merges

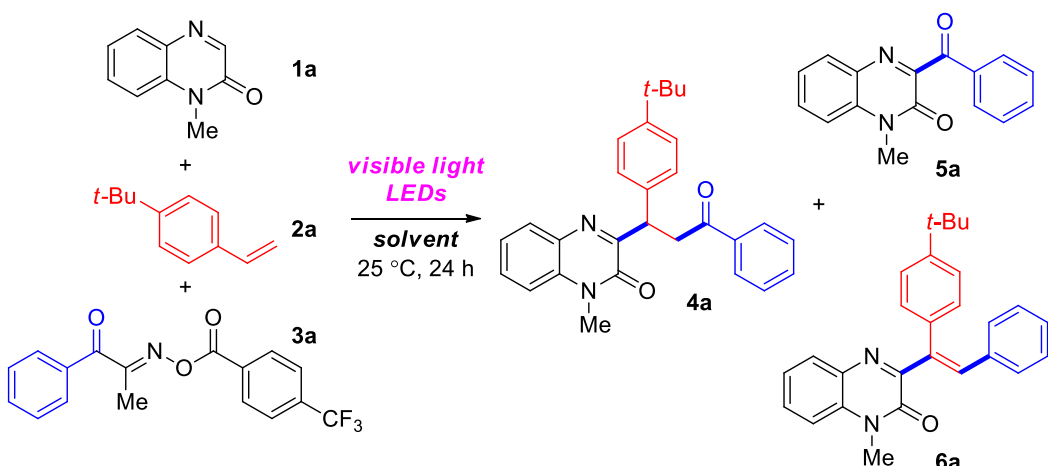
the intrinsic photoactivity of quinoxalin-2(1*H*)-ones with the radical-generating capacity of acyl oxime esters, enabling a modular and operationally simple exogenous photocatalyst-free three-component assembly of functionalized quinoxalin-2(1*H*)-one derivatives under mild conditions.

2 | Results and Discussion

Distinct from prior intramolecular acylative alkene addition approaches [65–71], the visible light-induced three-component

1,2-acylarylation of quinoxalin-2(1*H*)-ones was reacted with styrenes and acyl oxime esters under various irradiation conditions (Table 1). Initially, a 1:4:2 mixture of quinoxalin-2(1*H*)-one **1a**, 4-*tert*-butylstyrene **2a**, and benzoyl oxime ester **3a** was stirred in DMSO for 24 h in the absence of light (entry 1). All starting materials were recovered almost quantitatively, demonstrating their stability at ambient temperature. The reaction mixture also remained unchanged under green LED irradiation (entry 2). In contrast, blue LEDs promoted the formation of the desired three-component product **4a** in 22% yield, accompanied by the C3-acylated quinoxalin-2(1*H*)-one **5a** in 12% yield (entry 3). Although the overall reaction conversion under blue

TABLE 1 | Optimization of visible light-induced three-component 1,2-acylarylation.^a



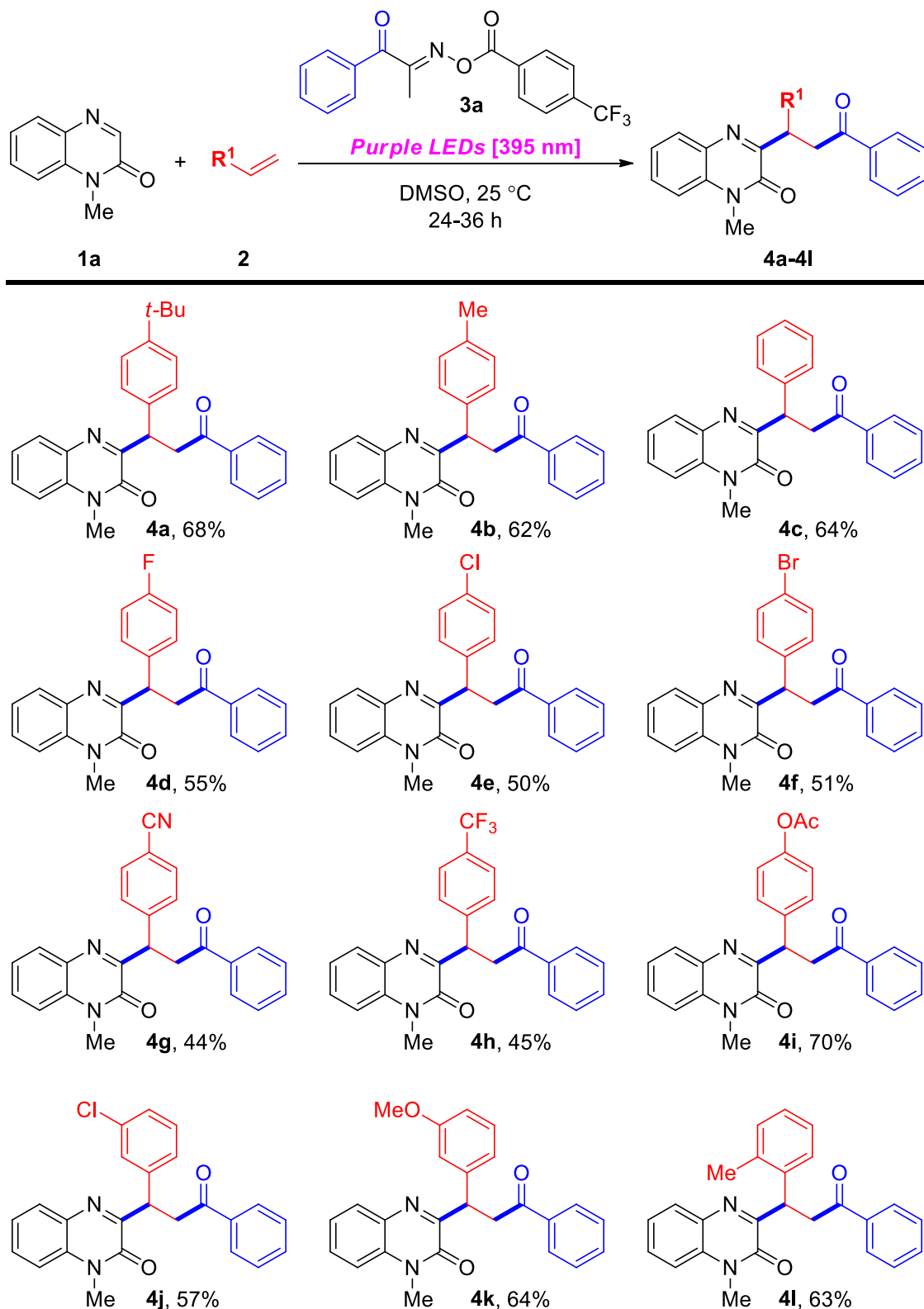
Entry	LEDs	Solvent	Deviations	Yield, % ^b		
				4a	5a	6a
1	—	DMSO	1a (>95%)	nr	nr	nr
2	Green	DMSO	1a (>95%)	nr	nr	nr
3	Blue	DMSO	1a (58%)	22	12	0
4	Purple	DMSO	—	63	8	0
5	White	DMSO	1a (65%)	14	9	0
6	Purple	Sulfolane, 1a (8%), 48 h		18	5	9
7	Purple	DMF, 1a (5%), 48 h		18	0	8
8	Purple	CH ₃ CN, 1a (3%), 48 h		20	4	30
9	Purple	DCE, 1a (5%), 48 h		27	10	18
10	Purple	THF, 1a (17%), 48 h		12	0	7
11	Purple	EtOAc, 48 h		24	0	16
12	Purple	HFIP, 1a (26%), 48 h		0	15	0
13	Purple	DMSO	2a (3.0 equiv)	53	12	0
14	Purple	DMSO	2a (5.0 equiv)	61	7	0
15	Purple	DMSO	3a (1.5 equiv)	51	8	0
16	Purple	DMSO	3a (2.5 equiv)	59	8	0
17	Purple	DMSO	0.07 M	54	12	0
18	Purple	DMSO	0.2 M	71 (68)	7	0
19	Purple	DMSO	Under air	16	3	0

^aReaction conditions: **1a** (0.3 mmol), **2a** (1.2 mmol), and **3a** (0.6 mmol) in solvent (0.1 M) under visible light and argon atmosphere at 25 °C for 24 h.

^bYields determined by ¹H NMR using internal standard, CH₂Br₂ (isolated yield of **4a** in parentheses). LEDs: Green (520 nm); Blue (455 nm); White (400–700 nm); Purple (395 nm).

LEDs reached only 42%, switching to purple LEDs significantly improved the reaction efficiency, leading to complete consumption of the limiting reagent **1a** and providing **4a** in 63% yield (entry 4). White LEDs proved less effective, resulting in 35% consumption of **1a** and the formation of **4a** in 14% yield (entry 5). Subsequent solvent screening revealed the formation

of an additional by-product, the decarbonylated aryl addition product **6a**, in 7%–30% yield, which compromised the yield of **4a** (entries 6–11). Optimization of substrate stoichiometry established that 4 equivalents of 4-*tert*-butylstyrene **2a** (entries 13–14) and 2 equivalents of benzoyl oxime ester **3a** (entries 15–16) were optimal. Further adjustment of the DMSO volume

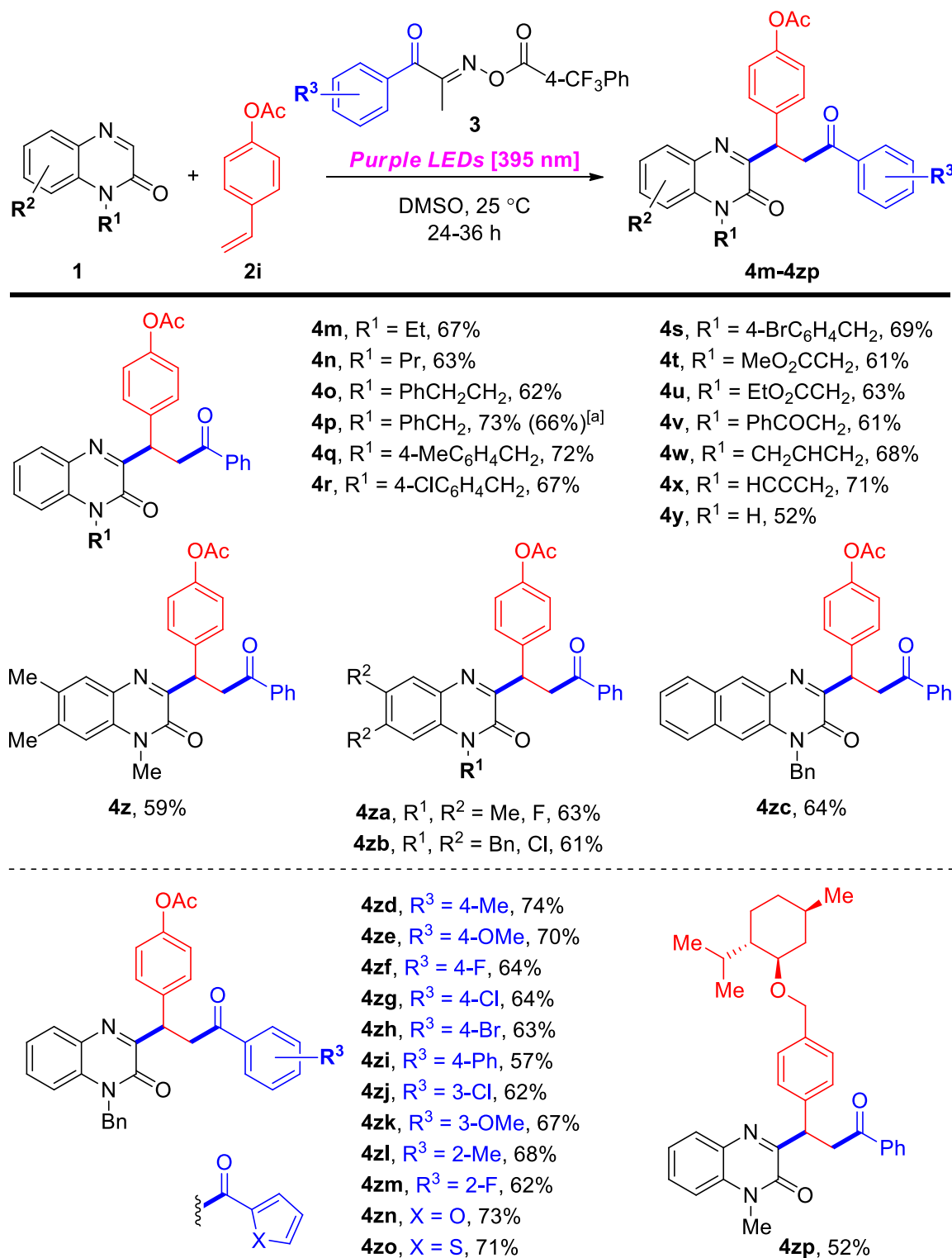


SCHEME 2 | Alkene substrate scope of visible light-induced three-component 1,2-acylarylation of quinoxalin-2(1H)-ones.

afforded the three-component product **4a** in 71% yield (entries 17–18). The reaction required an inert atmosphere; exposure to air led to rapid decomposition, providing **4a** in 16% yield (entry 19).

The optimized visible light-induced three-component 1,2-acylarylation of quinoxalin-2(1*H*)-ones was applied to a range of styrene

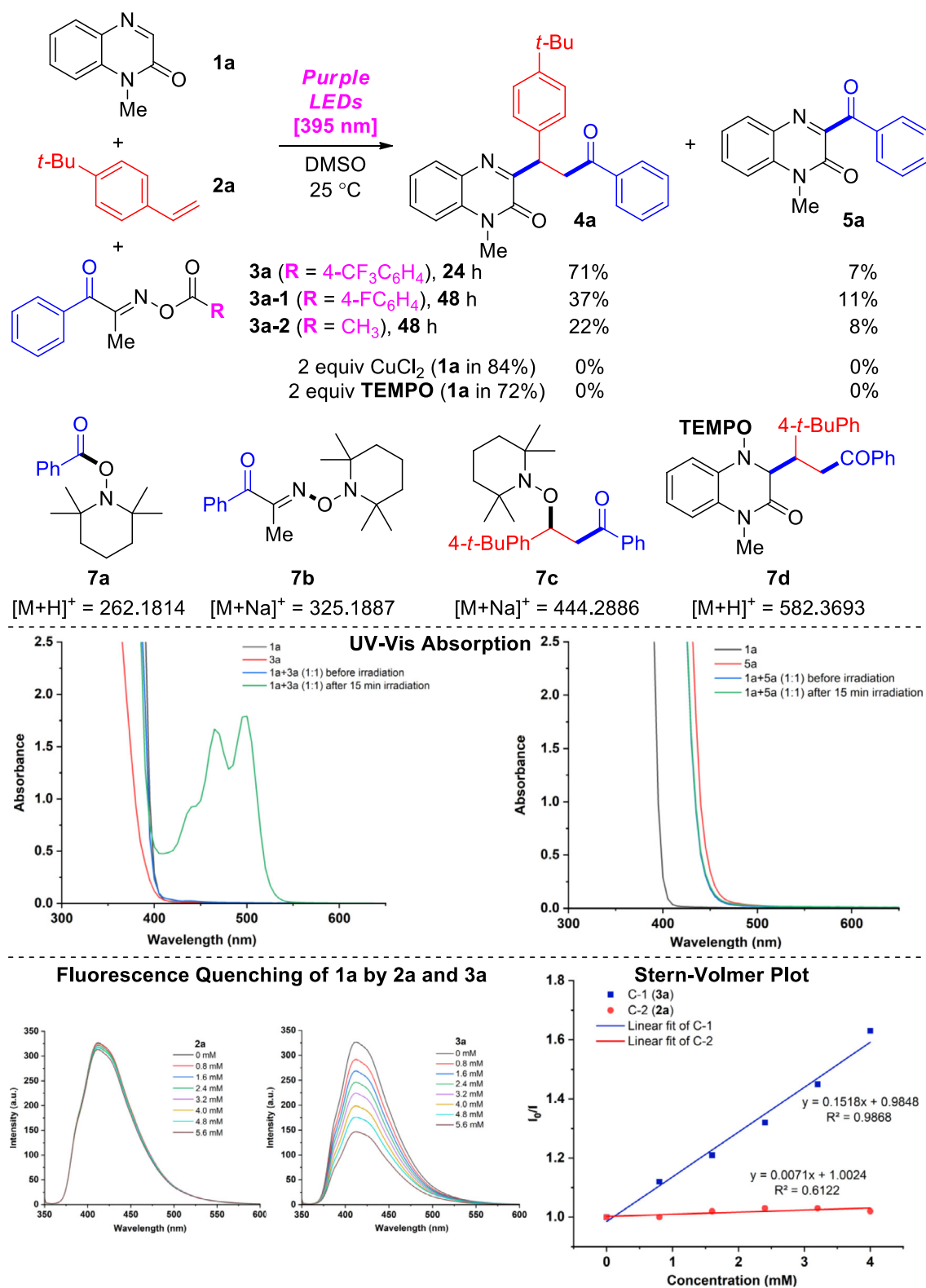
substrates (Scheme 2). Styrenes bearing electron-donating, electron-neutral, and electron-withdrawing groups were all well tolerated, affording the desired products **4a–4l** in 44%–70% yields. Notably, electron-withdrawing substituents generally led to a 10%–20% decrease in yield, as observed for **4d–4h**. Steric effects on the styrene component appeared minimal, as indicated by the comparable yields of products **4j–4l**. In contrast, non-styrenyl



SCHEME 3 | Quinoxalin-2(1*H*)-one and oxime ester substrate scope of visible light-induced three-component 1,2-acylarylation of quinoxalin-2(1*H*)-ones (^[a]reaction in 1 mmol).

alkenes, such as aliphatic terminal and internal alkenes as well as methyl acrylate, were incompatible under the current visible light-induced three-component alkylation conditions, as competitive direct C-3 acylation of quinoxalin-2(1*H*)-ones occurred without involvement of the alkene component (see the Supporting Information).

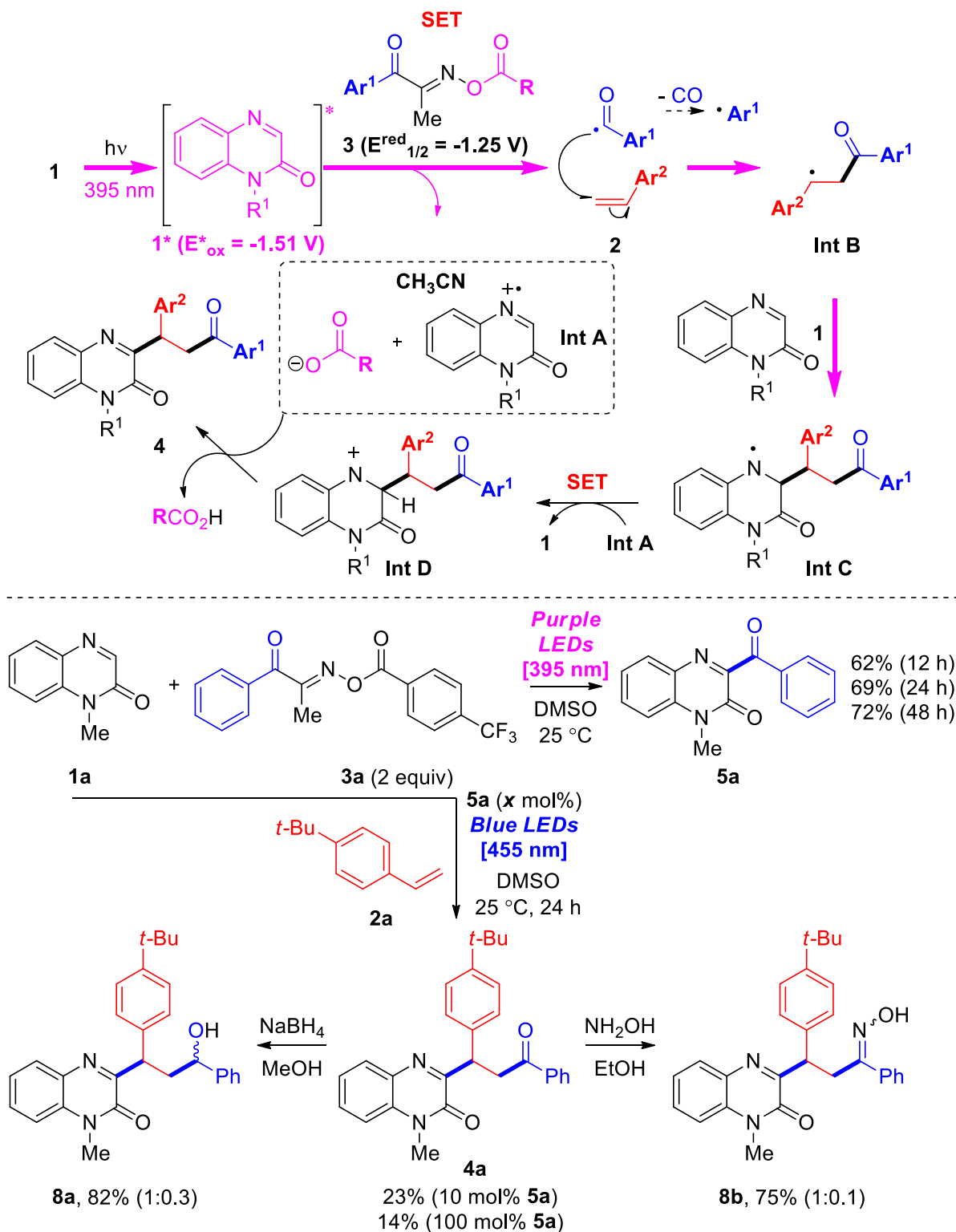
The substrate scope of quinoxalin-2(1*H*)-ones was initially examined with respect to the *N*-substituents (Scheme 3). A series of *N*-alkyl and benzyl quinoxalin-2(1*H*)-ones smoothly participated in the reaction to furnish products **4m–4x**, including those bearing diverse functional groups such as halogens (**4r–4s**), esters (**4t–4u**), and a ketone (**4v**). In addition, *N*-allyl and



SCHEME 4 | Control experiments and spectroscopic analysis.

N-propargyl quinoxalin-2(1*H*)-ones were well tolerated, delivering the corresponding products **4w–4x** in 68%–71% yields. The use of N-unsubstituted quinoxalin-2(1*H*)-one, as well as substituted quinoxalin-2(1*H*)-ones, furnished products **4y–4zc** in 52%–64% yields, further highlighting the structural versatility of this transformation with respect to the quinoxalin-2(1*H*)-one component. The structural variation of benzoyl oxime esters **3** exerted little influence on the efficiency of the three-component

alkylation, affording the desired products **4zd–4zp** in generally comparable yields. The reaction tolerated a broad range of substituents, including halogenated arenes (**4zf–4zh** and **4zj**), heteroaryl moieties (**4zn–4zo**), and a menthol-derived styrene (**4zp**). To date, the use of non-benzoyl oxime esters, such as acetyl and propionyl oxime esters, has not been successful due to the competitive decomposition of alkyl oxime esters.



SCHEME 5 | Plausible mechanism for visible light-induced 1,2-acylarylation of quinoxalin-2(1*H*)-ones and synthetic utilization.

To better understand the present 1,2-acylarylation, the substituent effect of oxime esters **3** was first investigated (Scheme 4). The 4-trifluoromethyl benzoate moiety proved to be a suitable substituent due to the enhanced stability of the 4-trifluoromethyl benzoate anion through the inductive effect exerted by the electron-withdrawing trifluoromethyl group on the phenyl ring, outperforming oxime esters bearing 4-fluorobenzoate or acetyl groups. The addition of an electron scavenger, anhydrous CuCl_2 , or a radical scavenger, TEMPO, completely suppressed the formation of the desired products, while slow consumption of **1a** and **3a** was still observed. However, the reaction mixture did not provide any isolable or identifiable products suitable for further structural characterization. Thus, HRMS analysis of the TEMPO-added reaction mixture revealed the formation of TEMPO-trapped radical intermediates **7a–7d**, supporting the involvement of radical species in the reaction pathway. The detected TEMPO-incorporated products **7a** and **7b** suggest that the acyl radical and iminyl radical species are generated from the oxime ester **3a**. In contrast, the benzyl radical and nitrogen-centered radical intermediates were captured by TEMPO from the respective two- and three-component coupled radical intermediates, leading to the formation of **7c** and **7d**, respectively. These observations are consistent with a SET process as the key mechanistic pathway. In particular, the electron scavenger CuCl_2 significantly inhibited the reaction, supporting the less likely involvement of an energy transfer pathway, which would not be expected to be strongly affected by electron scavengers. In addition, the rapid decomposition observed under an oxygen atmosphere (Table 1, entry 19), together with the requirement for continuous visible light irradiation for the three-component reaction to proceed efficiently, further supports the involvement of SET pathways in the present reaction mechanism (see the Supporting Information). Moreover, UV–Vis spectra of **1a**, **3a**, and **5a** exhibited absorption in the purple region without evidence of EDA complex formation. However, the 1:1 mixture of **1a** and **3a** exhibited new absorption bands at 465 and 495 nm after 15 min of irradiation with purple LEDs. Based on the previous studies on the photochemical behavior of quinoxalin-2(1H)-ones, [20–25] these new absorption bands were attributed to the formation of an EDA complex between the photo-excited state of **1a** and **3a** (see the Supporting Information for details). In addition, fluorescence quenching of **1a** by **3a** further supported the occurrence of a SET process between the photo-excited state of **1a*** and **3a**.

Based on the control experiments and the known photochemical properties of quinoxalin-2(1H)-ones, a plausible mechanism for the present visible light-induced 1,2-acylarylation is proposed (Scheme 5). Upon irradiation with purple visible light, quinoxalin-2(1H)-one **1** is excited to its photoactive state **1***, which initiates a SET process to oxime ester **3**. This event generates an acyl radical along with radical cation intermediate **Int A**, 4-trifluoromethyl benzoate anion, and neutral acetonitrile. The resulting acyl radical outcompetes decarbonylation and adds to alkene **2** to form a carbon-centered radical intermediate **Int B**, which subsequently undergoes radical addition to quinoxalin-2(1H)-one **1** to afford intermediate **Int C**. A subsequent SET process between **Int A** and **Int C** regenerates quinoxalin-2(1H)-one **1** and furnishes intermediate **Int D**. The formation of 4-trifluoromethyl benzoic acid then delivers the desired product **4**, completing the three-component

1,2-acylarylation sequence. The estimated photoexcited-state oxidation potential (E^*_{ox}) of **1a** in CH_3CN was -1.51 V versus SCE, whereas the reduction potential ($E^{\text{red}}_{1/2}$) of **3a** was -1.25 V versus SCE. These results indicate that SET from the photoexcited state of **1a** ($E^*_{\text{ox}} = -1.51$ V vs. SCE) to the ground state of **3a** ($E^{\text{red}}_{1/2} = -1.25$ V vs. SCE) is thermodynamically feasible, supporting the proposed mechanism (see the Supporting Information for details). Because C3-acyl quinoxalin-2(1H)-one **5a** exhibits absorption in the blue visible light region (>450 nm), its possible involvement under the reaction conditions was also examined. However, the addition of varying amounts of **5a** under blue LED irradiation produced only small amounts of **4a**, effectively ruling out a self-photocatalytic pathway mediated by **5a**. Previously, the Niwetmarin group reported a DBU-promoted C3-acylation of quinoxalin-2(1H)-ones through EDA complexation with oxime esters in a 1:1 mixture of DMSO and CH_2Cl_2 under blue LEDs [72]. In contrast, our SET-driven C3-acylation of quinoxalin-2(1H)-ones under purple LEDs proceeded in 72% yield, further supporting the proposed SET process between photo-excited quinoxalin-2(1H)-one **1*** and oxime ester **3**. Finally, the ketone moiety in product **4a** could be readily transformed into the corresponding alcohol **8a** and oxime **8b**, highlighting the synthetic utility of the 1,2-acylarylation products.

3 | Conclusion

In summary, we have developed an exogenous photocatalyst-free visible light-induced three-component 1,2-acylarylation of quinoxalin-2(1H)-ones with styrenes and acyl oxime esters under mild conditions. Harnessing the intrinsic photoactivity of quinoxalin-2(1H)-ones, purple light irradiation enabled direct excitation of quinoxalin-2(1H)-one to its photoactive state, which underwent SET to acyl oxime esters, generating acyl radicals that participate in a tandem radical addition sequence with styrenes and quinoxalin-2(1H)-ones. This exogenous photocatalyst-free strategy merges the self-sensitizable character of quinoxalin-2(1H)-ones with the radical-generating capacity of oxime esters, providing a modular and operationally simple approach to structurally diverse C3-functionalized quinoxalin-2(1H)-one derivatives. By expanding the repertoire of exogenous photocatalyst-free visible light transformations of quinoxalin-2(1H)-ones, this work highlights the potential of photo-excitable heteroaromatic scaffolds as both substrates and redox-active species in multicomponent radical assembly. Ongoing studies in our laboratory are directed toward further exploration of the visible light photoredox reactivity of quinoxalin-2(1H)-one derivatives, and these results will be reported in due course.

4 | Experimental Section

General Procedure for Visible Light-Induced Three-Component Radical Reactions (For the standard 0.3 mmol scale procedure, see the Supporting Information): A 20 mL borosilicate scintillation vial equipped with a magnetic stir bar was charged with 1-benzylquinoxalin-2(1H)-one (**1e**, 236 mg, 1.0 mmol, 1.0 equiv) and **3a** (670 mg, 2.0 mmol, 2.0 equiv) with anhydrous DMSO (5.0 mL, 0.2 M) at ambient temperature. After the addition of

4-acetoxystyrene (**2i**, 0.61 mL, 4.0 mmol, 4.0 equiv) to the reaction mixture, the reaction system was thoroughly degassed with argon and then stirred under the irradiation of purple LEDs (395 nm, 180 LED stripes (3 m), 3.5W/meter, distance app. 5 cm) for 12 h. After the reaction completion was confirmed by TLC, the reaction mixture was extracted with ethyl acetate (15 mL $\times$ 3). The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude mixture was purified by silica gel column chromatography using 6% ethyl acetate in hexanes to provide the desired product **4p** in 66% yield (0.33 g).

4-(1-(4-Benzyl-3-oxo-3,4-dihydroquinoxalin-2-yl)-3-oxo-3-phenyl propyl)phenyl acetate (4p): White solid; m.p. = 158 °C–160 °C. ¹H NMR (600 MHz, CDCl₃): δ 8.03 (dd, J = 8.4, 1.8 Hz, 2H), 7.73 (dd, J = 7.2, 1.2 Hz, 1H), 7.58–7.56 (m, 3H), 7.47 (t, J = 7.2 Hz, 2H), 7.35–7.33 (m, 1H), 7.29 (t, J = 7.2 Hz, 2H), 7.24–7.18 (m, 5H), 7.05 (d, J = 8.4 Hz, 2H), 5.53 (d, J = 15.6 Hz, 1H), 5.50 (dd, J = 10.2, 4.2 Hz, 1H), 5.37 (d, J = 15.6 Hz, 1H), 4.47 (dd, J = 18.0, 10.2 Hz, 1H), 3.50 (dd, J = 18.0, 4.2 Hz, 1H), 2.28 (s, 3H); ¹³C NMR (150 MHz, CDCl₃): δ 198.1, 169.6, 160.3, 154.6, 149.7, 138.2, 137.1, 135.4, 133.1, 132.8, 132.6, 130.1, 129.8, 129.7, 129.0, 128.6, 128.2, 127.6, 126.9, 123.4, 121.7, 114.4, 46.1, 43.8, 42.4, 21.3; IR (neat): 3053, 2923, 1757, 1655, 1597, 1201 cm⁻¹; HRMS (ESI): m/z calculated for C₃₂H₂₇N₂O₄ [M + H]⁺ 503.1965; Found 503.1976.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data underlying this study are available in the published article and its online supplementary material.

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Supporting Information

Additional supporting information can be found online in the Supporting Information section.