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Radical cascade cyclization for the green and simple synthesis of silylated indolo[2,1-*a*]isoquinoline derivatives *via* visible light-mediated Si–H bonds activation

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ABSTRACT

Photocatalytic and photoinduced silyl radicals cascade cyclization procedures for the green and simple preparation of fused tetracyclic skeleton silylated indolo[2,1-*a*]isoquinoline-6(5*H*)-ones from 2-aryl-*N*-acryloyl indoles with hydrosilanes are developed. The photocatalytic reaction is carried out with 9,10-dicyanoanthracene (DCA) as an organophotocatalyst and 3-acetoxyquinuclidine as hydrogen atom transfer (HAT) catalyst at room temperature under metal- and oxidant-free conditions. The keys to the success of photoredox-catalytic conversion include (1) the reductive quenching of DCA* [$E_{1/2}(^*P/P^-) = +1.97$ V *vs.* SCE in MeCN] by 3-acetoxyquinuclidine ($E_p = +1.22$ V *vs.* SCE in MeCN), and (2) the thermodynamic feasibility of hydrogen atom abstraction from hydridic Si–H bond by electrophilic $N^{+\bullet}$. Particularly, the simple photoinduced cascade cyclization using $(TMS)_3SiH$ with 2-aryl-*N*-acryloyl indoles was exploited *via* an electron–donor–acceptor (EDA) complex under visible light irradiation.

Organosilicon molecules are evoked remarkable interests and explored deeply by synthetic chemists, pharmacologists and material scientists because of their conspicuous chemical, physical, and biological properties (**I-V**, Fig. 1) [1–7]. Especially, silicon as isostere of carbon in biomolecules have become new drug-like candidates in drug discovery [8]. Classically, organosilicon derivatives were prepared by nucleophilic reactions of organometallic reagents with halosilanes [9–10], and transition-metal catalyzed cross-coupling of hydrocarbons or halogenated hydrocarbons with silylating reagents [11–20]. Recently, there were two effective methods for the synthesis of organosilicon compounds from hydrosilanes [21] or Si–X (X = Si [22], B [23], COOH [24]) reagents with alkenes, alkynes and arenes [25]. Among them, it was an atom-economical silylated approach *via* homolytic cleavage of Si–H bonds in hydrosilanes to generate silyl radicals. The archetypical way was thermo-promoted peroxide decomposition to trigger silyl radicals [26]. The second protocol was electron induced peroxide to initiate silyl radicals, the electron donors including transition-metal [27–29], TBAI [30] and photocatalyst [31]. The third way was that alkali initiated silyl radicals [32]. Although there have been significant advances, some methods suffered from harsh conditions or poor group compatibility, which would drive to find new strategies of triggering silyl radicals.

Photoredox catalysis [33–44] has appeared as a attractive protocol for silyl radicals generation via hydrogen atom transfer (HAT) of Si–H bonds [45–48]. Fagnoni *et al.* pioneered the tetrabutylammonium decatungstate (TBADT) as HAT photo-catalyst for trisubstituted silanes activation under phosphor-coated lamps irradiated by 310 nm [49]. Unfortunately, due to the comparably high bond dissociation energies (BDEs) of Si–H and α–Si–C–H bonds in alkyl-substituted silanes (*e.g.*, triethylsilane) [50], the HAT process initiated simultaneously the cleavage of Si–H and α–Si–C–H bonds. So the selectivity of HAT catalyst for Si-H bonds was poor. For achieving the desired HAT of Si–H bonds, it is necessary that using "aggressive" radicals to break the BDEs of Si–H bonds [51], hence the process is thermodynamically favorable. For instance, an electrophotocatalytic HAT process was developed for silyl radicals generation using MeOH as HAT reagent [52]. This

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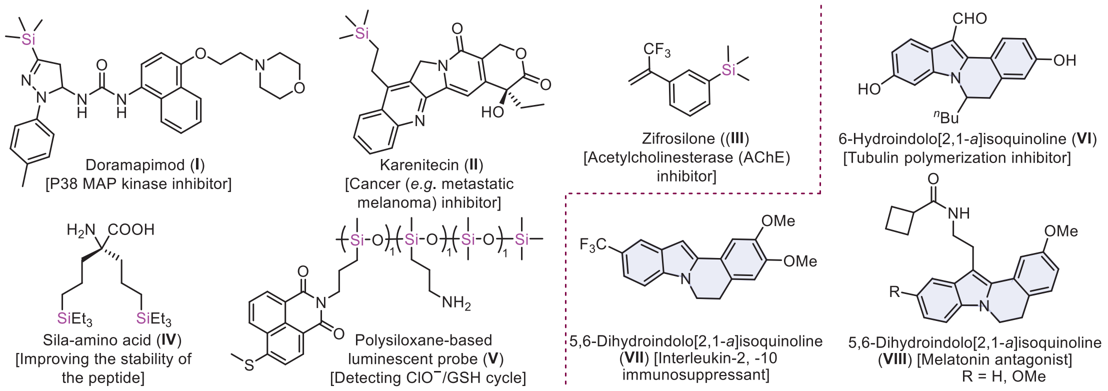


Fig. 1. Special examples of silicon-containing active molecules (**I-V**) and indolo[2,1-*a*]isoquinoline derivatives (**VI-VIII**).

work confirmed that hydrogen atom abstraction could be achieved by "aggressive" MeO$^•$ ($BDE_{O-H} = 105$ kcal/mol). Wu *et al.* developed an effective method for silyl radicals formation employing 3-acetoxyquinuclidine or triisopropyl-silanethiol as HAT reagent [53]. This also showed the feasibility of hydrogen atom abstraction by "aggressive" $N^{+•}$ ($BDE_{N^+-H} = 100$ kcal/mol) or S$^•$ ($BDE_{S-H} = 88.2$ kcal/mol). What is more, because hydrogen is more electronegative than silicon in hydrosilanes, according to the polarity-matched effect [54], the electrophilic radical (*e.g.*, O$^•$, $N^{+•}$, S$^•$) could be used to selectively abstract hydrogen of Si–H bonds in hydrosilanes rather than α–Si–C–H bonds.

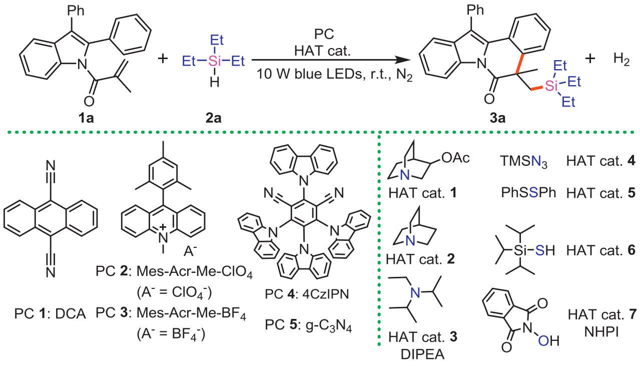
$(TMS)_3SiH$ was an ideal reagent in radical chemistry, which was used in many tris(trimethylsilyl)silylation or conversion processes. Because $(TMS)_3SiH$ has no α–Si–C–H bond and BDE_{Si-H} is relatively low, $(TMS)_3Si^•$ radical could be initiated *via* hydrogen atom abstraction by HAT reagent, single electron oxidation of $(TMS)_3SiH$ by PC* and then deprotonation [55], phosphor coating fluorescent lamp and UV light irradiation [49,56-58], *etc.* [59,60]. However, only two examples were reported that $(TMS)_3Si^•$ radical was produced *via* formation an electron–donor–acceptor (EDA) complex with alkyl or aryl halide to abstract halogen under visible light irradiation [61,62].

Indolo[2,1-*a*]isoquinolines containing the tetracyclic skeleton are widely found in bioactive and pharmaceutical molecules (**VI-VIII**, Fig. 1) [63–69]. Due to the potential of silicon incorporation in drug discovery, it is of great significance for the synthesis of silylated indolo[2,1-*a*]isoquinoline compounds. So far, few cases of synthesis have been reported, including $Cu(acac)_2$/TBPB-initiated triethylsilyl radical cascade cyclization (Scheme 1a) [70], cerium-electrophotocatalyzed methoxyl radical-mediated triethylsilyl radical cascade cyclization (Scheme 1b) [52], and palladium-catalyzed cascade cyclization with hexamethyldisilane [71] or $Me_3SiSiMe_2(O^nBu)$ (Scheme 1c) [72]. Despite significant advances, the fly in the ointment was that these examples were heating conditions, besides, there were one or more shortcomings, such as stoichiometric oxidant, poor atom economy, pre-activation of substrates and expensive transition-metal catalysts.

Taking into account the above aspects and our continuing interest in the preparation of heterocyclic molecules under visible light conditions [73–79], herein we report photocatalytic HAT selectively initiated silyl radicals cascade cyclization for the synthesis of silylated indolo[2,1-*a*]isoquinoline compounds. In addition, the simpler and greener cascade cyclization using $(TMS)_3SiH$ was exploited *via* novel EDA complex, the tris(trimethylsilyl)silylated indolo[2,1-*a*]isoquinolines can be obtained successfully under visible light irradiation even in the absence of photocatalyst and HAT catalyst (Scheme 1d).

Preliminary research was investigated by 1-(2,3-diphenyl-1*H*-indol-1-yl)-2-methylprop-2-en-1-one (**1a**) and triethyl-silane (**2a**) as model reaction substrates, and the outcomes were summarized in Table 1 and Tables S1-S5 (Supporting information). After screening these detailed conditions, it was found that the optimal choice including 0.1 mmol of **1a**, 10 equiv. of **2a**, 10 mol% of PC **1**, and 12.5 mol% of HAT cat. **1** in 2 mL dry MeCN under 10 W blue LEDs irradiation at room temperature for 30 h. And the target product **3a** was isolated with a yield of 70% under the optimal conditions.

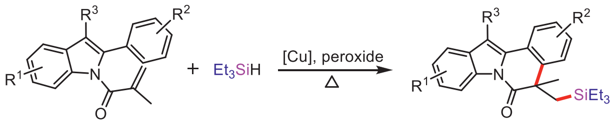
Table 1
Optimization of reaction conditions.[a]



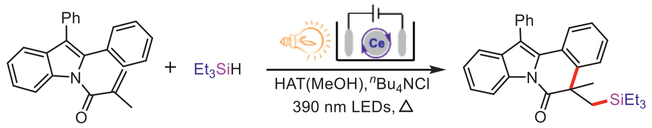
Entry	Variation from the reaction conditions	Yield (%)[b]
1	None	20
2 [c]	Without light	n.r.
3 [d]	Without PC **1** or without HAT cat. **1**	n.d.
4	Without N_2 atmosphere	6
5	Purple LEDs (390 nm) instead of blue LEDs	12
6	PC **2** instead of PC **1**	13
7	PC **3** instead of PC **1**	17
8	PC **4** instead of PC **1**	12
9 [e]	PC **5** instead of PC **1**	8
10	HAT cat. **2** instead of HAT cat. **1**	11
11	HAT cat. **3** instead of HAT cat. **1**	11
12	HAT cat. **4** instead of HAT cat. **1**	7
13 [d]	HAT cat. **5** instead of HAT cat. **1**	n.d.
14	HAT cat. **6** instead of HAT cat. **1**	9
15	HAT cat. **7** instead of HAT cat. **1**	7
16	1 mmol **2a**	54
17 [f]	30 h	64
18 [f,g]	12.5 mol% HAT cat. **1**	50
19 [f,g]	10 mol% PC **1**	57
20 [f,g]	10 mol% PC 1, 12.5 mol% HAT cat. 1	70

[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.2 mmol), PC **1** (5 mol%), HAT cat. **1** (10 mol%), dry MeCN (2 mL), 10 W blue LEDs, N_2, r.t., 20 h.
[b] Isolated yields.
[c] n.r. = no reaction.
[d] n.d. = no detected.
[e] PC **5** (8 mg).
[f] **2a** (1.0 mmol).
[g] 30 h.

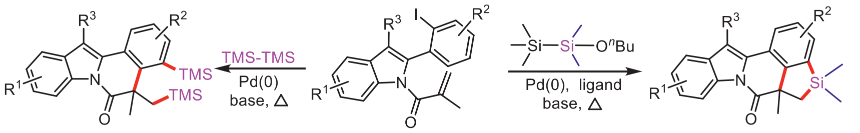
(a) Previous work: electron donors such as transition-metal induced peroxide to initiate triethylsilyl radical cascade cyclization (Yan *et al.* in 2022)



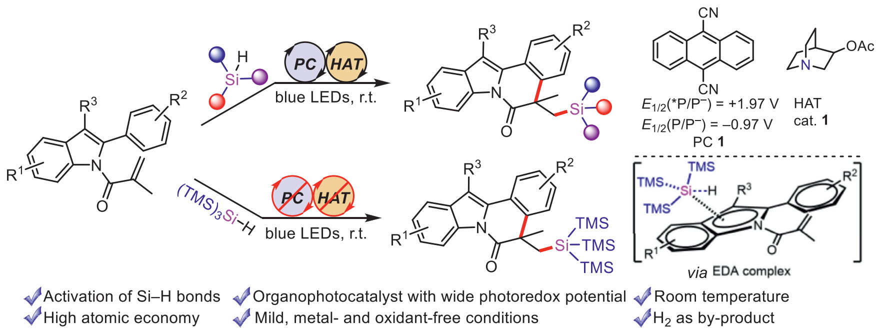
(b) Previous work: electrophotocatalytic HAT initiated triethylsilyl radical cascade cyclization (Zeng and Xu *et al.* in 2021)



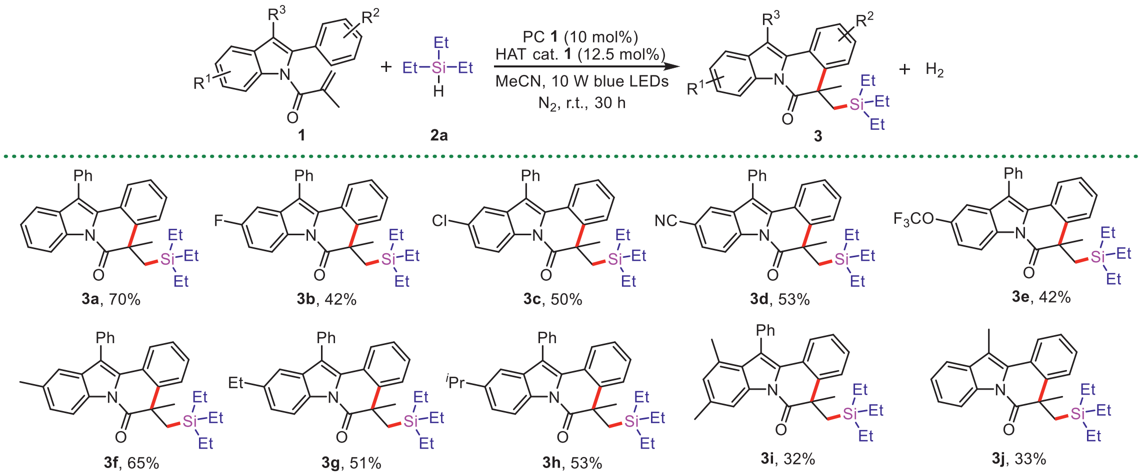
(c) Previous work: Pd-catalyzed cascade cyclization (Liang and Yang *et al.* in 2020, 2021)



(d) This work: visible light-mediated silyl radicals cascade cyclization *via* HAT catalysis and EDA complex initiation



Scheme 1. Different protocols for the synthesis of silylated indolo[2,1-*a*]isoquinoline-6(5*H*)-ones.

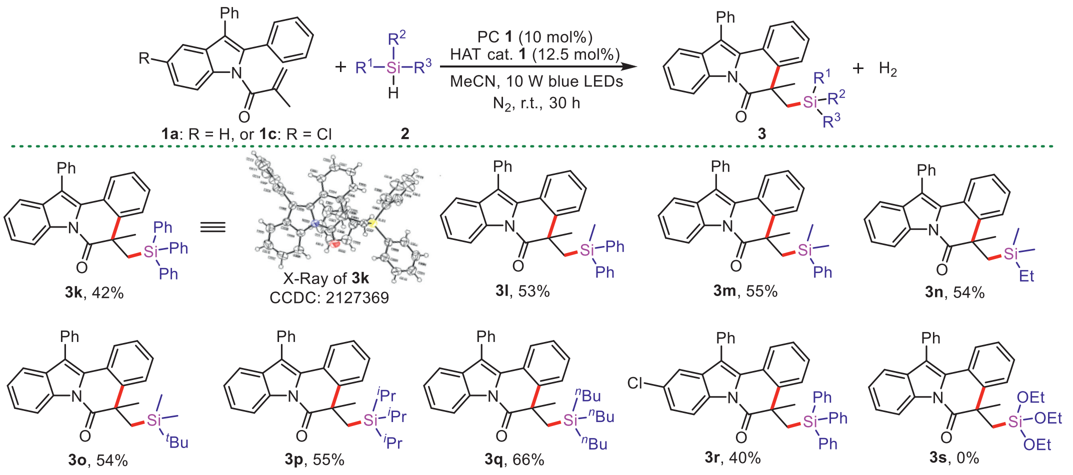


Scheme 2. Scope of 2-aryl-*N*-acryloyl indoles. Reaction conditions: **1** (0.1 mmol), **2a** (1 mmol), PC **1** (10 mol%), HAT cat. **1** (12.5 mol%), dry MeCN (2 mL), 10 W blue LEDs, N_2, r.t., 30 h. Isolated yields.

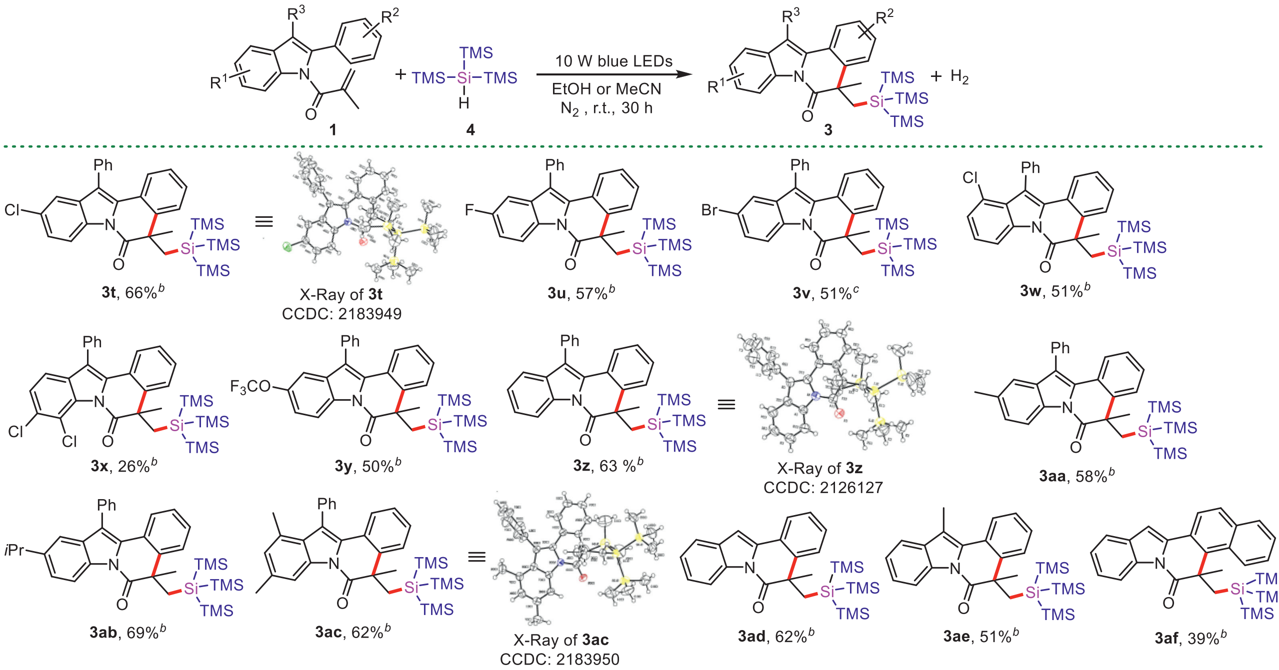
Having confirmed the optimal reaction conditions, we next evaluated the scope of 2-aryl-*N*-acryloyl indoles (Scheme 2). For example, substrates containing electron-withdrawing groups (F-, Cl-, CN-, and CF_3O-) at C5-position of indole ring could yield the desired products **3b**-**3e** in 42%-53% yields. Electron-donating groups (Me-, Et-, and ^{i}Pr-) were also good compatibility, giving the expected products **3f**-**3h** with yields of 53%-65%.

We next inspected the scope of hydrosilanes (Scheme 3). Under optimal conditions, arylsubstituted silanes such as triphenylsilane, diphenylmethylsilane and phenyldimethylsilane could be gave the desired products **3k** (proved by X-ray crystallography), **3l** and **3m** in 42%-55% yields. Trialkylsilanes showed good selectivity to afford the desired products **3n**-**3q** in 54%-66% yields, whereas the competing reaction of the C–H adjacent to silicon was not observed. Moreover, 2-aryl-*N*-acryloyl indoles and hydrosilanes could also combine freely to make new products, such as substrates **1c** and triphenylsilane worked smoothly. But the synthesis of triethoxysilylated product **3s** was failed, presumably because the BDE of Si-H bond in triethoxysilane is high.

We further found that 2-aryl-*N*-acryloyl indoles could successfully react with $(TMS)_3SiH$ under 10 W blue LEDs irradiation (Scheme 4). The reaction conditions were optimized and displayed



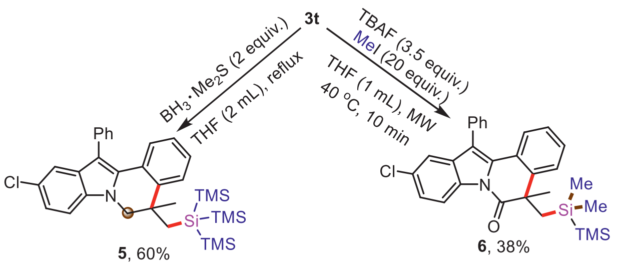
Scheme 3. Scope of hydrosilanes. Reaction conditions: **1a** or **1c** (0.1 mmol), **2** (1 mmol), PC (DCA, 10 mol%), HAT cat. **1** (12.5 mol%), dry MeCN (2 mL), 10 W blue LEDs, N_2, r.t., 30 h. Isolated yields.



Scheme 4. Scope of reaction between 2-aryl-*N*-acryloyl indoles and $(TMS)_3SiH$. Reaction conditions: **1** (0.1 mmol), **2** (1 mmol), dry EtOH[b] or MeCN[c] (1 mL), 10 W blue LEDs, N_2, r.t., 30 h. Isolated yields.

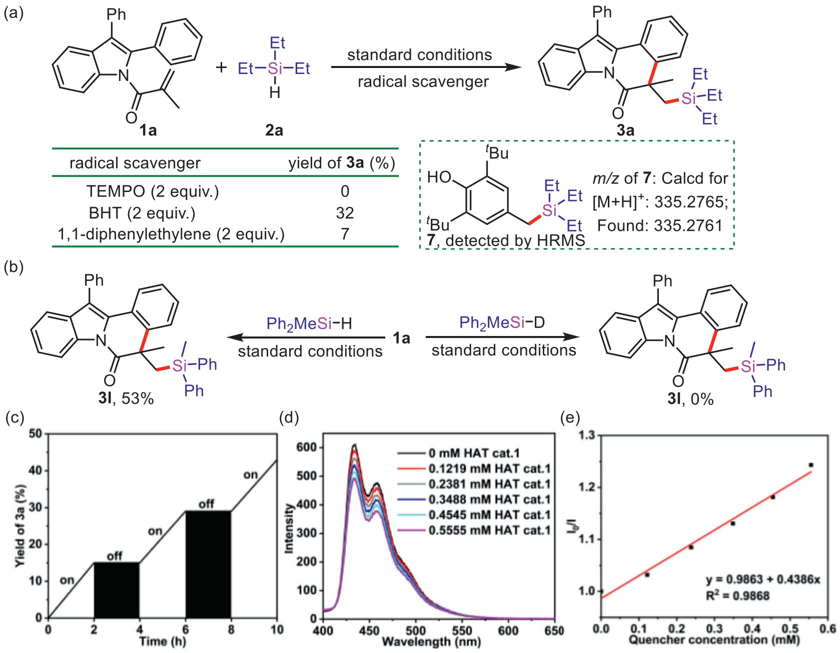
in Tables S6-S9 (Supporting information). Under the optimized reaction conditions, expected products **3t** (confirmed by X-ray crystallography), **3u**-**3w, 3y** from F-, Cl-, Br-, and CF_3O- groups located at C4- or C5-position of indole ring could be gained with moderate yields of 50%-66%. The 6,7-dichloro substituted product was also obtained, although the product **3x** with a low yield. Electron-donating groups (5-methyl, 5-isopropyl, 4,6-dimethyl) were tolerated, giving the desired products (**3aa**, **3ab**, **3ac**) in 58%-69% yields.

The synthetic application of compound **3t** was presented *via* further transformations (Scheme 5). Reduction of **3t** was investigated, the carbonyl group could be reduced to obtain compound **5** in 60% yield. Desilylation of the tri(trimethylsilyl)silyl group in **3t** was performed with Bu_4NF (TBAF) to give a disilane product **6** in 38% yield under microwave irradiation.



Scheme 5. Synthetic transformations of **3t**.

To explore the reaction mechanism, control experiments were implemented. The photocatalytic reaction was completely restrained while adding 2 equiv. of TEMPO under the standard conditions. Similarly, the reaction was conducted in the presence of BHT or 1,1-diphenylethane, the yield of **3a** was significantly reduced (Fig. 2a), which demonstrated that a radical process might be involved. Furthermore, the BHT-trapped product **7** was detected by HRMS. Besides, no reaction happened while employing deuterated diphenylmethyl silane (Ph_2MeSiD) (Fig. 2b). Such a significant kinetic isotope effect (KIE) suggested that the rate-determining step



radical scavenger	yield of **3a** (%)
TEMPO (2 equiv.)	0
BHT (2 equiv.)	32
1,1-diphenylethylene (2 equiv.)	7

Fig. 2. Mechanistic investigations. (a) Radical trapping experiment; (b) Investigation of the KIE; (c) ON/off experiments; (d) Fluorescence quenching experiment (DCA (1 μmol/L in MeCN) with different concentration of HAT cat. **1** were irradiated by 365 nm); (e) Stern-Volmer plot of HAT cat. **1**.

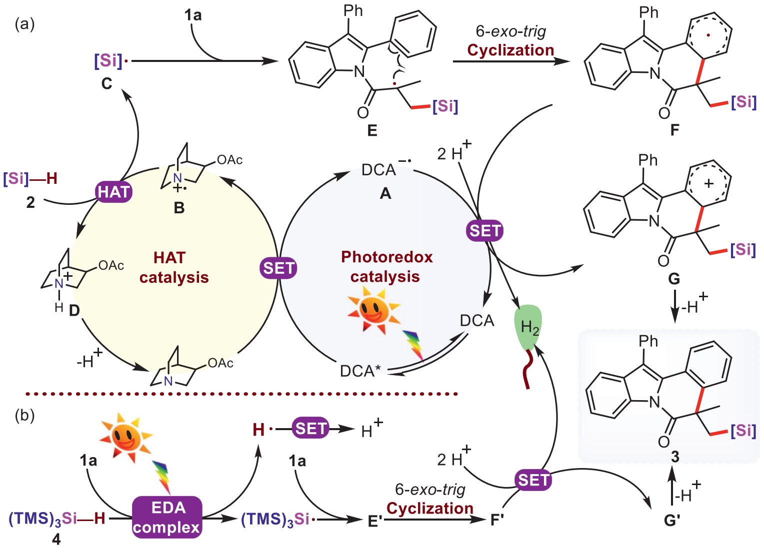


Fig. 3. Proposed reaction mechanism for the formation of silylated indolo[2,1-*a*]isoquinoline-6(5*H*)-ones.

involved the cleavage of the Si–H bonds. The H_2 was monitored by H_2 detector and GC under standard conditions (Fig. S4 in Supporting information). "On/off" experiments indicated that visible light played an important role (Fig. 2c). In addition, Stern-Volmer fluorescence quenching experiments demonstrated that the excited-state DCA* was quenched by HAT cat. **1** (Figs. 2d and e) through single electron transfer (SET) process.

On the basis of the above mechanistic investigations, a plausible mechanism was proposed, as depicted in Fig. 3a. Initially, DCA was excited to produce the long-life photoexcited-state DCA* ($t = 14.9$ ns) [80]. The reductive quenching of DCA* [$E_{1/2(^*P/P^-)} = +1.97$ V *vs.* SCE in MeCN] [81] by 3-acetoxyquinuclidine ($E_p = +1.22$ V *vs.* SCE in MeCN) [82] leaded to the radical anion **A** and a radical cation intermediate **B**. Due to its high electrophilicity, quinuclidinium radical cation **B** selectively abstracted the hydrogen atom from the more hydridic Si-H bonds of hydrosilanes to produce the corresponding silyl radical **C**, as well as quinuclidinium cation **D**. This abstraction event should be thermodynamically favorable because the BDE_{Si-H} of hydrosilanes was up to 94.6 kcal/mol and the BDE_{N^+-H} in quinuclidinium cation **D** was 100 kcal/mol. Subsequently, a carbon-centered radical intermediate **E** was generated by the addition of the silylic radical **C** to the C=C bond of the indole substrate **1a**, then the radical intermediate **E** was cyclized *via* 6-*exo-trig* pathway to afford the radical intermediate **F**. Afterwards, the single-electron oxidation process of **F** and **A** [$E_{1/2(P/P^-)} = -0.97$ V *vs.* SCE in MeCN] [81] occurred simultaneously to give the cation

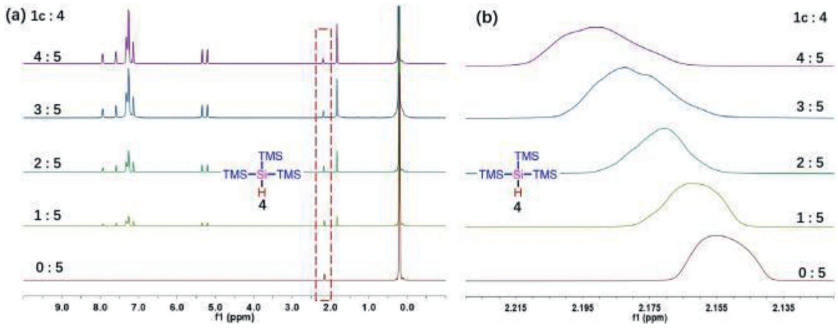


Fig. 4. ^{1}H NMR experiments between **1c** and **4**.

G and regenerate the DCA. In this process, there was sufficient driving forces to yield H_2 through the reduction of two protons [83,84]. Finally, the deprotonation of the cation **G** gave the silylated product **3**.

Particularly, controlled experiments were performed to study the mechanism of the reaction that **1c** react with $(TMS)_3SiH$ (**4**) under the photoinduced conditions in Scheme 4. The product **3t** was fully inhibited when 2 equiv. of TEMPO was added. Meanwhile, the BHT or 1,1-diphenylethane was added, the yield of **3t** was decreased (Section 4.1 in Supporting information), which demonstrated a radical pathway might also be involved. With the reaction proceeded, the color of the solution became yellow gradually, the H_2 was observed clearly and the concentration increased gradually (Fig. S5 in Supporting information). Subsequently, "on/off" LED irradiation experiments also showed that visible light played a key role in the reaction (Fig. S6b in Supporting information). We tested optical absorption of the EtOH solution of **1c** and **4**, it was not observed the red-shift or new absorption peak in the UV-vis absorption spectra (Fig. S8 in Supporting information). Next, we conducted ^{1}H NMR experiments, and the chemical shift of **4** shifted downfield with increasing amounts of **1c** (Fig. 4). These experimental results showed the formation of EDA complexes from 2-aryl-*N*-acryloyl indoles **1** with **4**. So the $(TMS)_3Si^\bullet$ radical could be formed by excited EDA complexes and an energy transfer under blue LEDs irradiation (Fig. 3b). The remaining mechanism including $(TMS)_3Si^\bullet$ radical addition and cascade cyclization was the same as described in Fig. 3a. It was worth noting that the single electron oxidation of **F'** was accompanied by the reduction of protons.

In summary, we developed green and simple photocatalytic and photoinduced silyl radicals cascade cyclization protocols for the synthesis of silylated indolo[2,1-*a*]-isoquinoline-6(5*H*)-ones. The photocatalytic procedure was conducted in the presence of DCA as photocatalyst and 3-acetoxyquinuclidine as HAT catalyst. To implement the desired silylation, the reductive quenching of DCA* by HAT catalyst, the abstraction hydrogen using "aggressive" radical were necessary, and the polarity-matched effect was also the key factor in success. The simple photoinduced method achieved the straightforward preparation of tris(trimethylsilyl)silylated indolo[2,1-*a*]-isoquinoline-6(5*H*)-ones *via* EDA complex. These two facile and greener procedures have the advantages including high atomic economy, H_2 as by-product, metal-free, oxidant-free, easy operation, and mild reaction conditions.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2023.108633.

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