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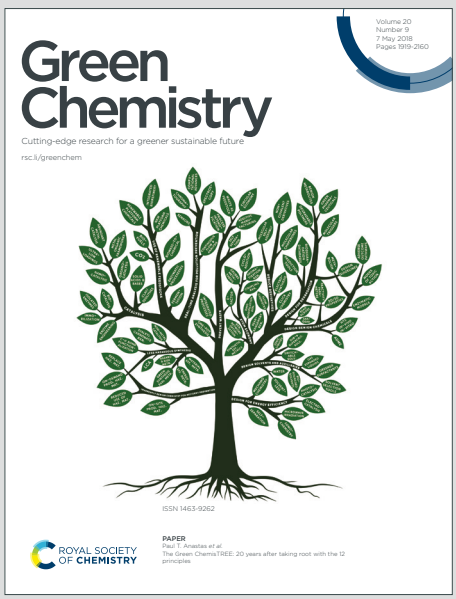

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Green Foundation

1. **Advancing the field of green chemistry:** we introduce a light-driven sequential route to Davis oxaziridines in which *m*CPBA is generated **on-demand** *in situ* and immediately consumed, avoiding bulk oxidant handling and preventing oxidant build-up, improving operational safety and sustainability.
2. **Green chemistry achievement:** kinetic profiling (SciPy modeling; five Ordinary Differential Equations ODEs) quantitatively verifies *m*CPBA forms only as needed with no detectable accumulation; the reaction runs at ambient temperature in non-halogenated solvents and **can use natural sunlight**/low-energy LEDs, enabling **safe gram-scale synthesis**.
3. **Further greening:** reduce solvent volume and enable solvent/byproduct (carboxylic acid) recycling.

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ARTICLE

Kinetically Guided On-Demand *m*CPBA Generation Enables Safe and Sustainable Light-Driven Synthesis of Davis Reagents

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Muthu Karuppasamy, [ab] Mohamed S. H. Salem, *[ac] Kwangkyun Jang, [ab] Mitsuhiro Arisawa, [b] Masayuki Kirihara [d] and Shinobu Takizawa *[a]

Oxaziridines, particularly Davis reagents, are widely used oxidants and valuable synthetic intermediates, yet their preparation typically requires handling bulk preformed oxidants such as *meta*-chloroperbenzoic acid (*m*CPBA), often in combination with halogenated solvents, creating safety and sustainability concerns. Herein, we report a safe, light-driven sequential strategy that generates *m*CPBA on demand from *meta*-chlorobenzaldehyde and molecular O_2 under natural sunlight or LED irradiation and immediately uses it to oxidize *N*-sulfonyl imines to Davis reagents. Kinetic analysis confirms *in situ m*CPBA formation without detectable accumulation, minimizing peracid-handling risk. The reaction proceeds at ambient temperature in non-halogenated solvents, offering a practical alternative to conventional protocols. A broad range of *N*-sulfonyl imines is converted in high yields, and the process is readily scalable. This operationally simple approach combines safety, efficiency, and green-chemistry principles for rapid access to Davis reagents.

Introduction

Oxaziridines are three-membered heterocycles comprising carbon, nitrogen, and oxygen atoms. The inherent ring strain and weak N–O bond enable them to be effective oxidizing and aminating agents in organic synthesis.[1] Among the various classes of oxaziridines, *N*-sulfonyloxaziridines (Davis reagents) are the most extensively utilized in organic synthesis.[2] Introduced by Davis and co-workers in 1977,[3] these reagents have become essential tools in the chemistry of oxidation, featuring stability, aprotic nature, and neutral character to render highly regio- and stereoselective oxidation of a wide variety of nucleophiles under mild conditions. Representative transformations include α-hydroxylation of carbonyl compounds, oxidation of sulfides to sulfoxides, phosphines to phosphine oxides, selenides to selenoxides, amines to amine oxides/nitrones, thiols to sulfenic and sulfinic acids, alkenes to epoxides, organolithium and Grignard reagents to alcohols, etc (Scheme 1a).[2]

Although Davis reagents are versatile, their synthesis often relies on conditions that compromise safety and sustainability (Scheme 1b). *N*-sulfonyl oxaziridines are commonly constructed by oxidizing *N*-sulfonyl imines using metal, organo, or phase-transfer catalysts with oxidants such as *m*CPBA or hydrogen peroxide, and many of these reactions afford the desired products in good yields and, when chiral catalysts are employed, with high enantiomeric excess (ee).[4] Since the 1980s, significant efforts have focused on the synthesis of these heterocycles using oxidants, including *m*CPBA, while avoiding metal, organo-, or phase-transfer catalysts.[5] However, handling stoichiometric amounts of *m*CPBA poses serious risks due to its energetic nature. Its weak O–O bond renders it susceptible to exothermic decomposition even at low temperatures. Scaling up further increases the risk of thermal runaway, inadequate heat dissipation, and pressure build-up that can lead to explosions. In addition, it is corrosive and tends to release toxic fumes upon decomposition. To overcome these issues, researchers have also explored alternative oxidizing systems. Hassine and co-workers developed trichloroacetonitrile–hydrogen peroxide oxidant system enabling the synthesis of Davis reagents in dichloromethane.[6] Kirihara et. al. later introduced pH-controlled aq. sodium hypochlorite oxidant system at reduced temperature.[7] Oxidation using buffered Oxone (ca. 6 equivalents) has also been documented.[8] Despite advancements, many of these methods still relied on biphasic water-organic solvent systems, which resulted in poor imine solubility and hydrolysis, leading to low yields, by-product formation, and a limited substrate scope. Most critically, the requirement for excess preformed oxidants and the hazards associated with handling bulk oxidant waste severely limit the safety, sustainability, and practical (particularly industrial) applicability of these protocols (Scheme 1b).

Inspired by our previous work on light-induced autoxidation of aldehydes, we envisioned the on-demand *in situ* generation of *m*CPBA to avoid the risks associated with handling preformed oxidants.[9] Herein, we introduce a safe and sustainable protocol

[a] SANKEN, The University of Osaka, 8-1 Mihogaoka, Ibaraki-shi, Osaka 567-0047, Japan.
Email: mohamedsalem43@sanken.osaka-u.ac.jp; taki@sanken.osaka-u.ac.jp
[b] Graduate School of Pharmaceutical Sciences, The University of Osaka, 1-6 Yamada-oka, Suita, Osaka 565-0871, Japan.
[c] Pharmaceutical Organic Chemistry Department, Faculty of Pharmacy, Suez Canal University, Ismailia 41522, Egypt.
[d] Department of Materials and Life Science, Shizuoka Institute of Science and Technology, 2200-2 Toyosawa, Fukuroi, Shizuoka 437-8555, Japan.
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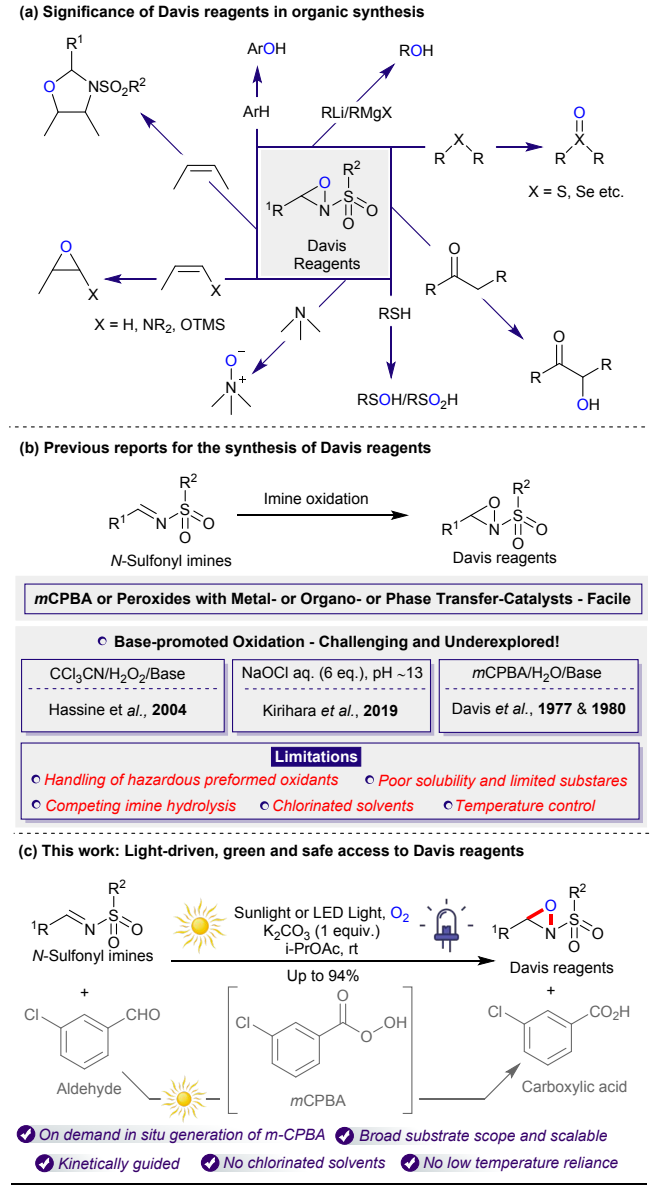
to access Davis reagents using sunlight or LED light. The method generates *m*CPBA *in situ*, enabling efficient oxidation of *N*-sulfonyl imines under ambient conditions while avoiding halogenated solvents. Kinetic studies further validate the on-demand nature of *m*CPBA generation without accumulation, ensuring process safety, practicality, and efficiency (Scheme 1c).



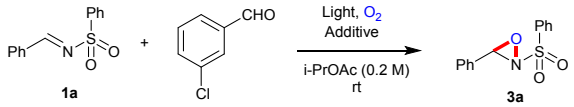
Scheme 1 Versatility and synthetic strategies of Davis reagents: Previous reports and this work

Results and Discussion

Exploration of optimal reaction conditions

Motivated by our interest in green photoinduced synthesis[9,10] and efficient sequential processes,[11] we devised our strategy based on the light-induced *in situ* generation of *m*CPBA from *meta*-chlorobenzaldehyde **2a** and molecular O_2, thereby avoiding the use and handling of bulk preformed oxidant while directly oxidizing *N*-sulfonyl imines **1a** to the corresponding Davis reagents. To establish and optimize these conditions, we selected *N*-sulfonyl imine **1a** and *meta*-chlorobenzaldehyde **2a** as model substrates in isopropyl acetate under 405 nm LED irradiation and an O_2 atmosphere (Table 1, entry 1). Although *m*CPBA was detected, Davis reagent **3a** was not formed, and unexpectedly, *N*-sulfonyl imine **1a** was completely decomposed after 12 hours under light.[12] In an effort to trigger the formation of Davis reagent **3a**, we explored controlling the reactivity of *m*CPBA through various basic additives and water (entries 2–5). Among the additives studied, K_2CO_3 furnished **3a** in excellent yield of 92% within 3 hours (entry 3). Insights into how K_2CO_3 promotes the formation of **3a**, controls *m*CPBA generation, and suppresses **1a** degradation will be discussed in the next kinetic and mechanistic sections (Figure 1). Changing the K_2CO_3 loading (0.5–2.0 equiv.) did not result in any pronounced improvement (entries 6 and 7). Decreasing the loading of **2a** to 1.5 equiv. afforded Davis reagent **3a** in 89% yield (entry 8), while the reaction proceeded slightly faster when the amount of **2a** was increased to 3.0 equiv. (entry 9), but this did not translate into an improved yield. Subsequently, we explored the influence of different light energies in the presence of 1 equiv. of K_2CO_3 (entries 10-17). *N*-sulfonyl imine **1a** was unreactive under very high- and low-energy supplies (280, 521, and 631 nm; entries 10, 16, and 17), as *m*CPBA was not generated under these wavelength irradiations. Among the medium wavelength lights used, 395 nm LED light was very efficient and product **3a** was obtained in excellent yield of 94% (entries 11-15). Remarkably, performing the reaction under sunlight also delivered the Davis reagent **3a** in 94% yield (entry 18). This result underscores the sustainable and practical nature of this protocol, particularly its ease of scaling under natural sunlight without the need for specialized high-power LED sources. However, the use of pure O_2 in combination with organic compounds at atmospheric pressure poses a non-negligible safety risk, especially for large-scale applications. Therefore, the reaction was also examined under open-air conditions, affording the product in 69% yield after 5 hours. Notably, the yield increased to 90% when 3.0 equivalents of aldehyde **2a** were employed, albeit with slightly lower efficiency than that observed under O_2 balloon conditions (entries 19 and 20). Importantly, the compatibility of the reaction with open-air conditions further highlights the greenness and practicality of the protocol, offering a significantly safer alternative. Finally, the critical role of light was elucidated by the fact that no reaction proceeded without the irradiation (entry 21).

Table 1 Optimization of the reaction conditions[a]



Entry	LED (nm)	Additive (equiv.)	Time (h)	Yield of 3a[b] (%)	Recovery of 1a[c] (%)
1	405	-	12	3	2
2	405	Na_2CO_3 (1)	3	77	Traces

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3	405	K_2CO_3 (1)	3	92	0
4	405	KOH (1)	3	Traces	46%
5	405	H_2O (1)	3	No reaction of **1a**	
6	405	K_2CO_3 (0.5)	3	77	0
7	405	K_2CO_3 (2)	3	88	2
8	405	K_2CO_3 (1)[d]	3	89	0
9	405	K_2CO_3 (1)[e]	2	90	0
10	280	K_2CO_3 (1)	12	No reaction of **1a**	
11	310	K_2CO_3 (1)	3	84	0
12	340	K_2CO_3 (1)	3	82	0
13	365	K_2CO_3 (1)	3	78	Traces
14	385	K_2CO_3 (1)	3	81	3%
15[f]	**395**	**K_2CO_3 (1)**	3	**94**	**0**
16	521	K_2CO_3 (1)	12	No reaction of **1a**	
17	631	K_2CO_3 (1)	12	No reaction of **1a**	
18[f]	**Sunlight**	**K_2CO_3 (1)**	3	**94**	**0**
19[g]	Sunlight	K_2CO_3 (1)	5	69	0
20[g]	Sunlight	K_2CO_3 (1)[e]	2	90	0
21	No light	K_2CO_3 (1)	12	No reaction	

[a]Unless otherwise noted, all reactions were carried out with **1a** (0.2 mmol), **2a** (0.4 mmol) in 1 mL of isopropyl acetate at room temperature in the presence light and O_2. [b]NMR yield of **3a**. [c]NMR yield of **1a**. [d]1.5 equiv. of **2a** was used. [e]3.0 equiv. of **2a** was used. [f]Optimized reaction condition to access **3a**. [g]Reaction performed under open-air conditions

Mechanistic and kinetic investigations

To better understand the competing reaction pathways, a detailed kinetic analysis was conducted under various conditions as showcased in Figure 1. Production of the Davis reagent **3a** through *in situ*-generated *m*CPBA **4a** and *N*-sulfonyl imine **1a** involves multiple interconnected pathways, making the kinetic description of the system more complex (Figure 1A). The reaction begins with the autoxidation of aldehyde **2a** initiated by photoexcitation and subsequent reaction with molecular oxygen through a radical-mediated mechanism, generating peracid **4a** at a first-order rate constant, k_a. Peracid **4a** can then follow two distinct reaction pathways towards the formation of corresponding carboxylic acid **5a**: (i) A Baeyer-Villiger type oxidation,[13] where the peracid **4a** reacts with aldehyde **2a** to generate a Criegee intermediate, subsequently rearranging to yield carboxylic acid **5a** at a second-order rate constant k_b. Another pathway for the rearrangement of the Criegee intermediate into formate and carboxylic acid has been reported,[14] but this byproduct was not observed under our conditions for substrate **2a**. (ii) Direct decomposition of **4a** to yield carboxylic acid **5a** at a first-order rate constant, k_c. *N*-sulfonyl imine **1a**, another crucial species in our system, undergoes partial degradation to aldehyde and sulfonamide derivatives at a first-order rate constant, k_d. The kinetically dominant reaction is the conversion of **1a** to the target Davis reagent **3a** using the *in situ*-generated **4a** at a second-order k_e. These reactions can be mathematically described through the following system of ordinary differential equations (ODEs):

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$$\frac{d[\mathbf{1a}]}{dt} = -k_d[\mathbf{1a}] - k_e[\mathbf{1a}][\mathbf{4a}] \quad (1)$$

$$\frac{d[\mathbf{2a}]}{dt} = -k_a[\mathbf{2a}] - k_b[\mathbf{2a}][\mathbf{4a}] + k_d[\mathbf{1a}] \quad (2)$$

$$\frac{d[\mathbf{3a}]}{dt} = k_e[\mathbf{1a}][\mathbf{4a}] \quad (3)$$

$$\frac{d[\mathbf{4a}]}{dt} = k_a[\mathbf{2a}] - k_c[\mathbf{4a}] - k_b[\mathbf{2a}][\mathbf{4a}] - k_e[\mathbf{1a}][\mathbf{4a}] \quad (4)$$

$$\frac{d[\mathbf{5a}]}{dt} = k_c[\mathbf{4a}] + 2\,k_b[\mathbf{2a}][\mathbf{4a}] \quad (5)$$

Experimental concentration-time profiles of **1a**, **2a**, **3a**, **4a**, and **5a** recorded under well-defined conditions were globally fitted to equations 1–5 using nonlinear regression (Python SciPy), enabling extraction of the kinetic parameters k_a–k_e (see ESI for details). To systematically address this kinetic complexity, individual rate constants were first determined under simplified setups for each step and k_e was then refined under full reaction conditions. The overall rate-determining step of this intricate network is the photoinduced oxidation of aldehyde **2a** to the corresponding peracid **4a** (k_a = 0.0074 min^{-1}) (Figure 1B, I). Once formed, the *in situ* generated **4a** is consumed through three competing pathways: (i) the desired oxidation of **1a** to the Davis reagent **3a** (k_e = 10.0 min^{-1}), (ii) a Baeyer–Villiger-type oxidation of **2a** to **5a** (k_b = 1.1466 min^{-1}), and (iii) direct decomposition to **5a** (k_c = 0.0086 min^{-1}). In the presence of K_2CO_3, our kinetic analysis revealed that the overall rate of **4a** consumption is approximately 1500-fold higher than its formation, such that the steady-state concentration of the oxidant remains effectively negligible throughout the reaction. Peracid **4a** is consumed essentially instantaneously, predominantly by reaction with **1a** (k_e) and, to a lesser extent, with **2a** (k_b), while unimolecular decay *via* k_c represents only a minor pathway (Figure 1B, II); under these quasi-steady-state conditions (**4a** ≈ 0 and $k_e \gg k_b \gg k_c, k_a$) (Figure 1B, III). As discussed in the optimization section, *N*-sulfonyl imine **1a** undergoes degradation (k_d) under photoinduced conditions, which is highly sensitive to irradiation wavelength, solvent, and pH (Figure 1B, IV). Shorter wavelengths significantly accelerate degradation, underscoring the strongly photochemical nature of this process, while i-PrOAc provides better stabilization than acetone or DCM. A weakly basic environment suppresses degradation, likely by stabilizing the imine or inhibiting proton-transfer pathways. Under our optimized conditions, the degradation rate of **1a** is relatively small (k_d = 0.0010 min^{-1}), and the 7.5-fold higher k_a, allowing the productive pathways to outcompete **1a** decomposition and enabling yields of **3a** up to 94%. The photochemical nature of the whole process was further confirmed by studying the effect of light intensity on the reaction rate; varying the LED power from 4.1 to 142 mW (calibrated *via* a thermal power sensor) resulted in a 13-fold increase in reaction rate (see ESI for details).

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The key role of K_2CO_3 is further highlighted by comparison with the reaction profile in its absence (Figure 1B, V & VI). Without base, the entire cascade is reshaped: the peracid-forming step k_a increases from 0.0074 to 0.0233 min^{-1} (≈3-fold), but the principal consumption pathways for **4a** slow dramatically—k_b decreases from 1.1466 to 0.036 min^{-1}, k_e becomes essentially zero, and k_c decreases from 0.0086 to 0.0041 min^{-1}—resulting in slower acid formation and noticeable accumulation of peracid **4a**.

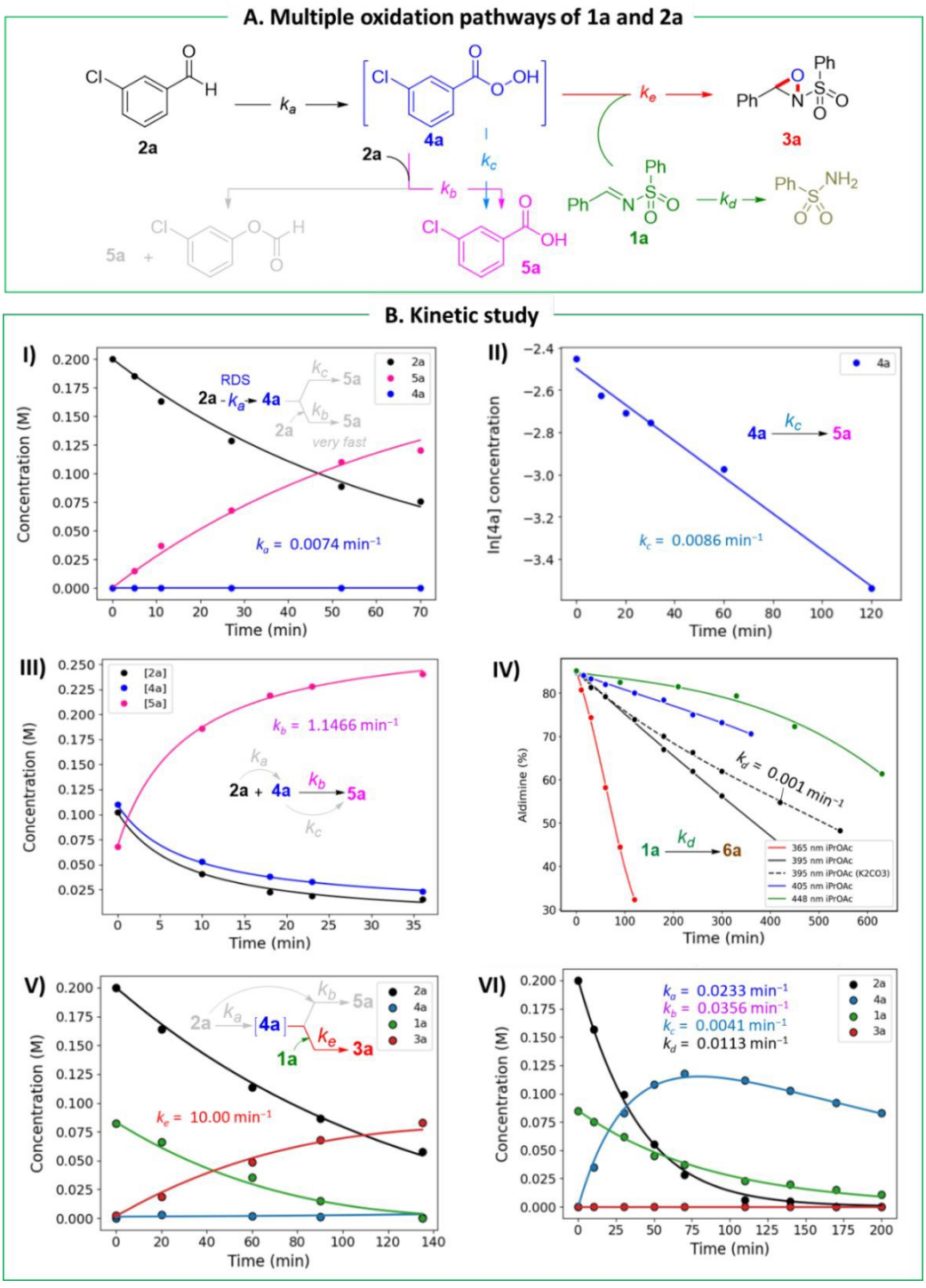

Figure 1 Reaction Kinetics: A) Multiple oxidation pathways of **1a** and **2a**. B) Kinetic study: (I) calculation of the k_a rate of aldehyde **2a** autoxidation; (II) calculation of the k_c rate of peracid **4a** decomposition; (III) calculation of the k_b rate of Baeyer–Villiger oxidation; (IV) calculation of the k_d rates of **1a** degradation at different conditions; (V) kinetic profile of the reaction in presence of K_2CO_3, and (VI) in the absence of K_2CO_3

At the same time, k_d increases by approximately an order of magnitude, effectively shutting down Davis reagent **3a**

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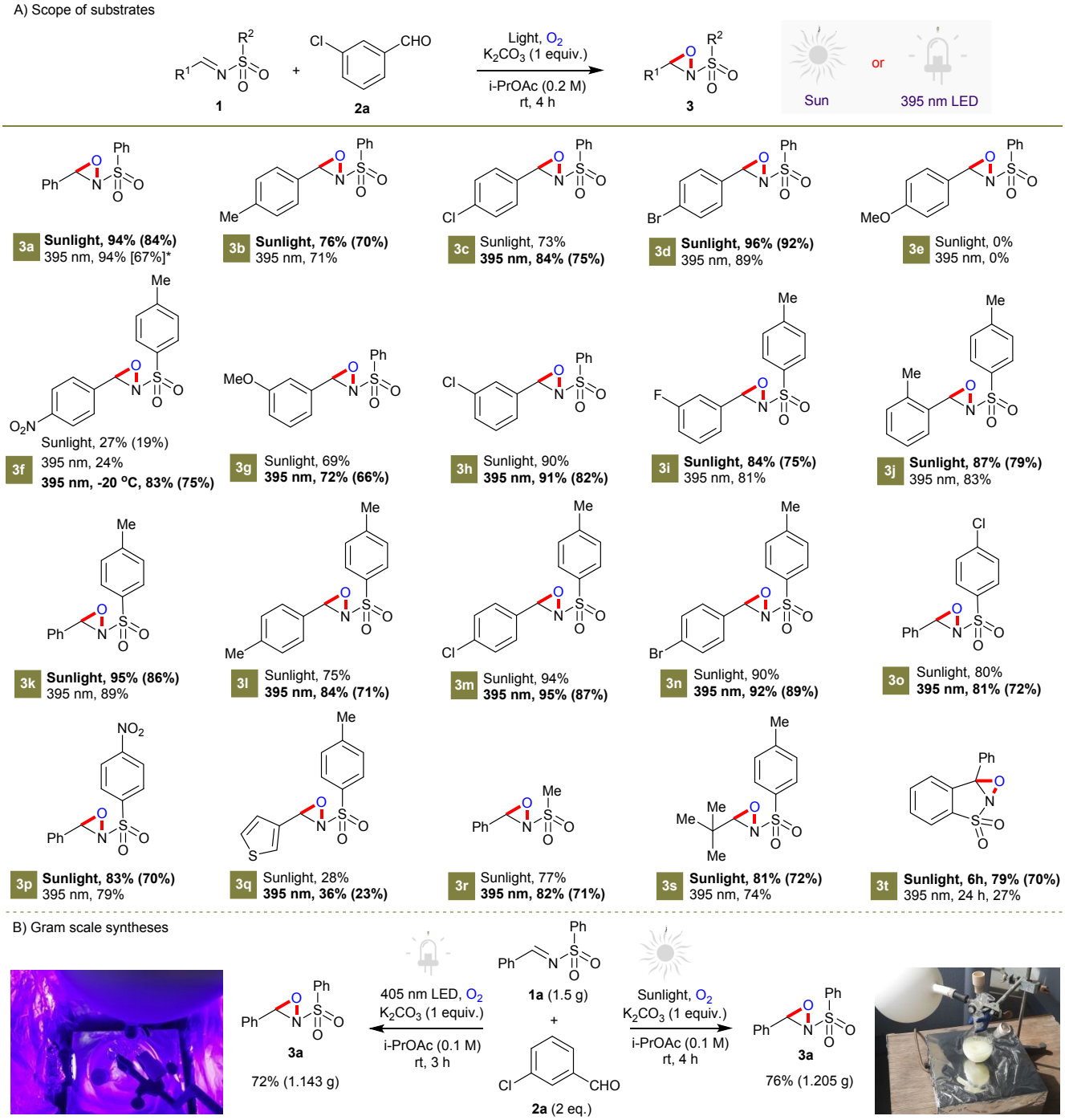
formation and leading instead to peracid **4a** build-up, its subsequent conversion to **5a**, and enhanced degradation of **1a** (Table 1, entry 1). Thus, K_2CO_3 plays a crucial role in modulating both reaction kinetics and pathway selectivity: it enables efficient cyclization to Davis reagent **3a**, prevents accumulation of dangerous peracid **4a** by promoting its rapid consumption in productive downstream steps, and suppresses photodegradation of sulfonyl imine **1a**. Overall, K_2CO_3 exerts a multifaceted influence that is essential for both the efficiency and practical applicability of the developed protocol.



Scheme 2 Substrate scope and scalability of light-driven synthesis of Davis reagents. A) Reaction conditions: **1** (0.4 mmol), **2a** (2 equiv.), K_2CO_3 (1 equiv.), i-PrOAc (2 mL), under an oxygen atmosphere at 25-34 °C. NMR yields are reported. Isolated yields are in parenthesis. *Isolated by trituration with hexane after workup. B) Gram scale synthesis of Davis reagent **3a** under sunlight and 405 nm LED irradiation: reaction conditions **1a** (6.11 mmol), **2a** (2 equiv.), K_2CO_3 (1 equiv.), i-PrOAc (0.1 M), under an oxygen atmosphere at 25-34 °C. The isolated yields are reported.

Exploring the substrate scope and scalability

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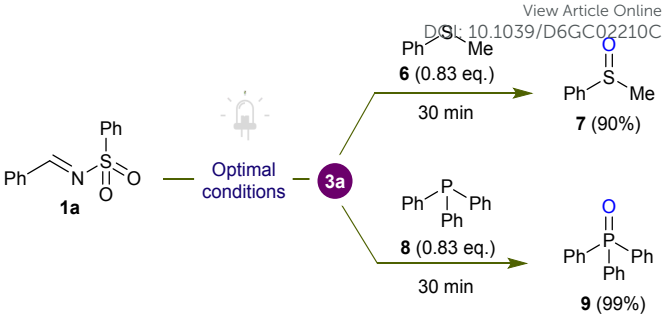
With the two high-yielding conditions in hand, the substrate scope of *N*-sulfonyl imines **1** was investigated under sunlight and 395 nm LED irradiation (Scheme 2A). Various electron-donating and electron-withdrawing substituents were introduced at the R^1 position. The *p*-Me-, *p*-Cl-, and *p*-Br-substituted *N*-sulfonyl imines furnished the corresponding Davis reagents **3b–3d** in good-to-excellent yields (76-96%). The *p*-NO_2-substituted imine **1f** afforded **3f** in 83% yield at −20 °C. Access to **3e** was unsuccessful due to the instability of **1e**, and no improvement was observed under 405 nm LED irradiation, or at lower temperature. The reaction also proceeded smoothly with *meta*-substituted substrates bearing *m*-OMe **1g**, *m*-Cl **1h**, and *m*-F **1i** groups, providing **3g–3i** in 72-91% yields. In addition, the *ortho*-substituted substrate **1j** was compatible, affording **3j** in 87% yield.

A variety of *N*-sulfonyl imines bearing electron-donating and electron-withdrawing groups (Me, Cl, and NO_2) at the R^2 position smoothly underwent the transformation to furnish Davis reagents **3k–3p** in excellent yields (81-95%). The heteroarylated substrate **1q** also gave the corresponding **3q**, albeit in lower yield. Aliphatic R^1 and R^2 substituents (*t*-Bu and Me, respectively) were well tolerated, affording **3r** and **3s** in good yields. Furthermore, the reaction showed good compatibility with ketimines **1t**, delivering the ketimine-derived Davis reagent **3t** in 79% yield. In the case of compound **3a**, trituration with hexane after workup afforded the product in 67% yield, lower than that obtained by column chromatography (84%), yet serving as a complementary purification approach.

The scalability of this green procedure was validated on a gram scale. Thus, 1.50 g (6.11 mmol) of *N*-sulfonyl imine **1a** was transformed into **3a** under both natural sunlight and 405 nm LED irradiation, providing isolated yields of 76 % and 72 %, respectively (Scheme 2B). Notably, the use of ubiquitous sunlight as a renewable energy source, without any need for specialized equipment, underscores the simplicity and sustainability of the protocol. In addition, the procedure avoids the accumulation of *m*CPBA **4a**, ensuring a safe and operationally convenient process that is well suited for scale-up.

Application of Davis reagent in oxidation reactions

To demonstrate the practical efficacy of our approach, we explored its application in representative oxidation reactions. After generating Davis reagent **3a** under our optimized conditions, methyl(phenyl)sulfane **6** was added directly to the reaction mixture, excluding light and O_2; the corresponding sulfoxide **7** was obtained in an excellent yield of 90%. Similarly, oxidation of triphenylphosphine **8** offered triphenylphosphine oxide **9** in quantitative yield within 30 minutes. Although our kinetic investigation suggests that *m*CPBA does not accumulate under these conditions, we further eliminate any possibility for *m*CPBA contribution in these oxidation reactions by removing the light and O_2. The ability to accomplish such efficient oxidations in a single synthetic operation without intermediate purification underscores the versatility and practical significance of our system (Scheme 3).



Scheme 3 One-pot generation and subsequent reactions of Davis reagent **3a.** Reaction conditions: **1a** (0.4 mmol), **2a** (2 equiv.), K_2CO_3 (1 equiv.), i-PrOAc (2 mL), under 395 nm LED irradiation and O_2 at room temperature. LED light and O_2 were disconnected after the generation of Davis reagent **3a**, continued the stirring upon addition of 0.33 mmol of sulfide **6** or phosphine **8**. The isolated yields are reported.

Control experiments and Mechanistic insights

Next, we turned our attention to the mechanistic rationale. To probe the crucial role of light in the developed sequential process, a light-on/off experiment was performed using *N*-sulfonyl imine **1a**. The oxidation progressed only under continuous light irradiation; halting light promptly paused the reaction. In the absence of light, all key sequential steps (a) oxidation of aldehyde **2a** to *m*CPBA, (b) oxaziridization of imine **1a** to **3a** and (c) Baeyer–Villiger oxidation to carboxylic acid **4a** were completely stopped. This highlights the crucial role of light in driving the overall transformation and illustrates the immense advantage of on-demand generation of *m*CPBA (Figure 2).

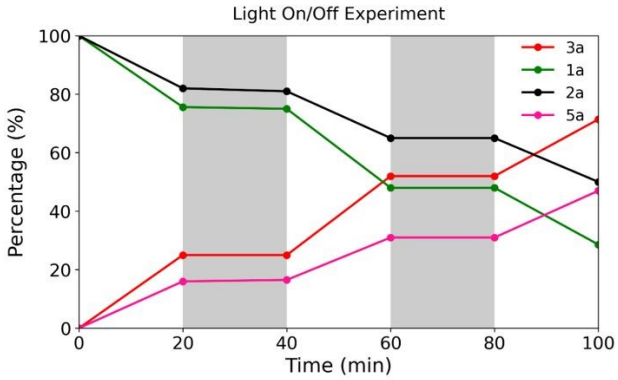


Figure 2 Light on/off switching study

In the presence of the radical scavenger tetramethylpiperidine-1-oxyl (TEMPO), the acyl-TEMPO adduct **10** was detected (HRMS [M+H$^+$ = 296.1412]), and the desired product **3a** was completely suppressed, supports the involvement of a radical pathway (Scheme 4A). Guided by insights from the control experiments, our kinetic analysis, and related reports,[15] the plausible mechanism can be proposed (Scheme 4B). Under light irradiation, aldehyde **1** is photoexcited to generate a highly

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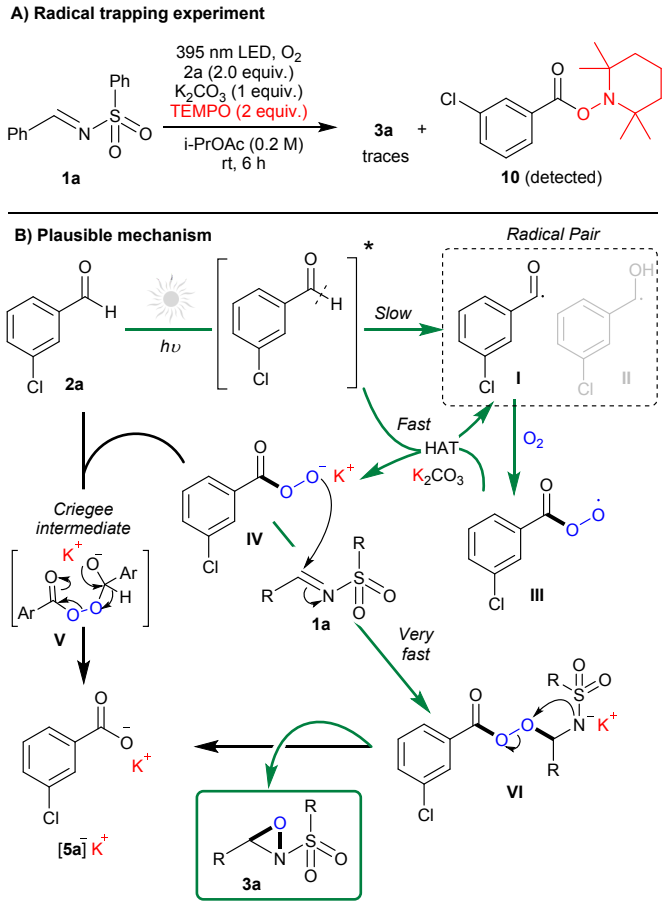
reactive triplet radical pair (**I** and **II**), which undergoes a radical-mediated pathway with molecular oxygen to generate the corresponding peroxy radical **III**. Subsequent hydrogen atom transfer (HAT) generates *m*CPBA which is further deprotonated by potassium carbonate into more reactive peroxycarboxylate salt **IV**. As supported by our kinetic studies, imine **1** instantly traps **IV** as soon as its generation to form intermediate **VI**. The final oxaziridization involving the nucleophilic attack to the electrophilic oxygen atom by nitrogen anion furnishes Davis reagents **3**. In a parallel, *meta*-chlorobenzoate (potassium salt of **5a**) is formed by the nucleophilic addition of peroxycarboxylate **IV** to aldehyde **2a** through a pathway similar to Baeyer-Villiger oxidation (Scheme 4B).[13]



Scheme 4 Control experiment and proposed mechanism.

Conclusions

In conclusion, we developed a safe, light-driven sequential strategy that provides rapid access to Davis reagents in high yields using natural sunlight or LEDs. Under irradiation, a broad range of *N*-sulfonyl imines undergo efficient oxidation by *m*CPBA generated *in situ* from *meta*-chlorobenzaldehyde and O_2, delivering a scalable and operationally simple protocol. A central strength of this method is the on-demand formation of *m*CPBA: kinetic analysis confirms that only the required amount is produced, with no detectable accumulation during the reaction. This avoids the storage and direct handling of bulk peracid reagents, thereby reducing the active oxidant inventory while minimizing latent energy risk and the complexity of peracid handling. Compared with previous protocols, which typically rely on oxidants such as *m*CPBA in combination with halogenated solvents, this procedure eliminates the use of halogenated solvents and proceeds at ambient temperature, thereby improving practicality and reducing environmental impact. Ongoing studies are focused on elucidating the role of K_2CO_3 in intermediate stabilization and extending the platform to asymmetric variants.

Experimental Section

Gram-Scale Synthesis of Davis Reagent 3a

To a solution of *N*-sulfonyl imine **1a** (6.11 mmol) and i-PrOAc (60 mL) in a dry 100 mL two-neck round-bottom flask, *meta*-chlorobenzaldehyde **2a** (12.22 mmol) and K_2CO_3 (6.11 mmol) were added. The reaction mixture was stirred under sunlight at 25–33 °C for 4 hours (11:00 AM–3:00 PM; the temperature remained relatively constant during this period, showing minimal fluctuation) in an oxygen atmosphere using an O_2 balloon at atmospheric pressure with no additional pressurization. The reaction vessel was evacuated using a Schlenk line prior to O_2 introduction; this step is not mandatory but is recommended for higher yields and reproducibility. After completion, as indicated by TLC, the white suspension was diluted with water, and extracted with EtOAc. The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by flash column chromatography using *n*-hexane/ethyl acetate (99:1, v/v) as the eluent to afford the desired Davis reagent **3a** in 76% yield. The isopropyl acetate reaction mixture/water system is already biphasic in nature, and isopropyl acetate can also be used for efficient extraction.

Additional reaction information: place: (Osaka, Japan); date (19/06/2025); weather (mostly sunny); outdoor temperature (25–33 °C); time (11:00 AM–15:00 PM); humidity (55–43%); wind (9.6–12.9 km/h).

Author contributions

M. K.: Conceptualization, Designing and performing the experiments, analysing data, and writing the first draft of manuscript. **M. S. H. S.:** Conceptualization, designing experiments, performing kinetic analysis, data curation, and editing the final version of the manuscript. **K. J.:** Performing the experiments and analysing data. **M. A.** and **M. K.:** supervising the project and editing the final version of the manuscript. **S. T.:** Conceptualization, Fund acquisition, project administration, and editing the final version of the manuscript.

Conflicts of interest

There are no conflicts to declare.

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Data availability

The data supporting this article have been included as part of the supplementary information (SI), including additional experimental procedures, characterization data for synthesized compounds, NMR spectra, and photographs of the experimental setup. See DOI: https://doi.org/..

Code availability

Codes of the kinetic study were deposited in a DOI-minting repository (Zenodo) under the title of 'Kinetically guided on-demand *m*CPBA generation enables safe and sustainable light-driven synthesis of Davis reagents' See the following DOI: https://doi.org/10.5281/zenodo.17657673

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