



Photocatalytic dicarboxylation of strained C–C bonds with CO₂ via consecutive visible-light-induced electron transfer

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ABSTRACT

Dicarboxylic acids have a wide range of applications in the polymer industry to construct valuable materials. Photocatalysis has recently emerged as an efficient and sustainable strategy to generate dicarboxylic acids. However, photocatalytic dicarboxylation with CO₂ is mainly limited to unsaturated bonds, and the dicarboxylation of C–C single bonds still remains a challenge. Herein, we report a photocatalytic dicarboxylation of C–C single bonds in strained rings with CO₂ units via consecutive photo-induced electron transfer (ConPET). It is also the first photocatalytic reductive ring-opening reaction of cyclobutanes. Notably, this transition-metal-free protocol exhibits good functional group tolerance, broad substrate scope, facile scalability, and easy product derivatizations. Moreover, diacids can easily be derivatized to main-chain liquid crystalline polyesters.

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As an inexpensive, nontoxic and recyclable one-carbon building block, carbon dioxide (CO₂) has been widely used for the synthesis of structurally diverse chemicals [1–7]. Carboxylic acids, which are widely found in many bioactive molecules and useful synthons, are some of the most valuable motifs among these chemicals synthesized from CO₂ [8–10]. Among them, the dicarboxylation reaction with CO₂ is considered a promising protocol to construct valuable diacids [11], which are important chemicals and intermediates with numerous applications in the food, pharmaceutical, and polymer industries (Scheme 1A) [12,13]. Therefore, developing efficient and elegant catalytic systems to synthesize diacids is of great relevance. However, it is difficult to synthesize diacids by inserting two CO₂ molecules due to the low reactivity of CO₂ and competitive side reactions. On the other hand, visible-light catalysis has emerged as an indispensable powerful tool in green organic synthesis, which utilizes light as the clean energy to promote carbon–carbon/heteroatom bond formation under eco-friendly and mild conditions [14–19]. Despite recent reports on photocatalytic dicarboxylation of unsaturated bonds with CO₂ [20–23], photocatalytic dicarboxylation of C–C single bonds, which are ubiquitous

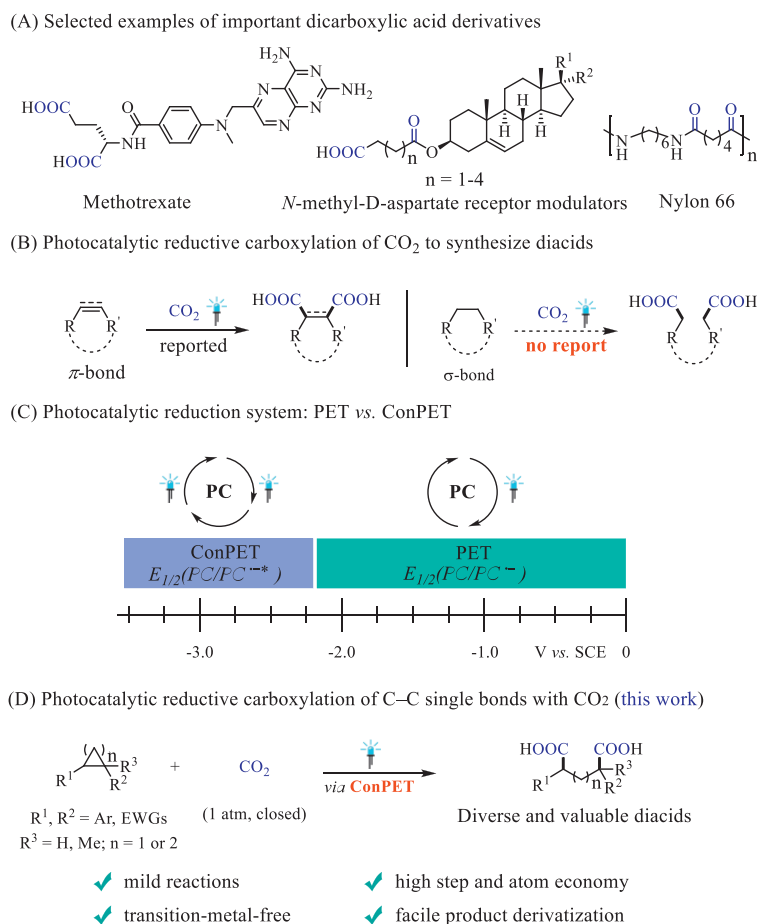
in organic molecules, has not been reported yet (Scheme 1B). Notably, compared with photocatalytic dicarboxylation of unsaturated bonds to provide succinic, photocatalytic dicarboxylation of C–C single bonds can obtain derivatives of glutaric acid and adipic acid, which also have important applications in the field of polymer synthesis [24,25].

Ring-opening via selective cleavage of C–C bond is known as a powerful strategy for the construction of complex molecules with diverse functional groups [26–30]. To our best knowledge, there are only a few reports on the photocatalytic reductive ring-opening of cyclopropanes [31], while photocatalytic reductive ring-opening reaction of cyclobutanes is elusive. We wondered whether we could develop an efficient reductive dicarboxylation of small rings with CO₂ via ring-opening difunctionalization. Notably, there are several challenges for such a transformation. Due to more negative potentials ($E_{\text{red}} = -3.3\text{V}$ vs. SCE for methyl 2-phenylcyclopropane-1-carboxylate) (see Supporting information for details), photocatalytic reduction of cyclopropane under mild conditions is extremely challenging. Moreover, side reactions such as protonation, monocarboxylation and homocoupling during the desired dicarboxylation process are possible to occur. Recent research has shown that consecutive photoinduced electron transfer (ConPET) can employ the combined energy of two visible-spectrum photons for a photocatalytic reduction [32]. The photocatalytic conversion of less reactive chemical bonds in organic synthesis is thus made

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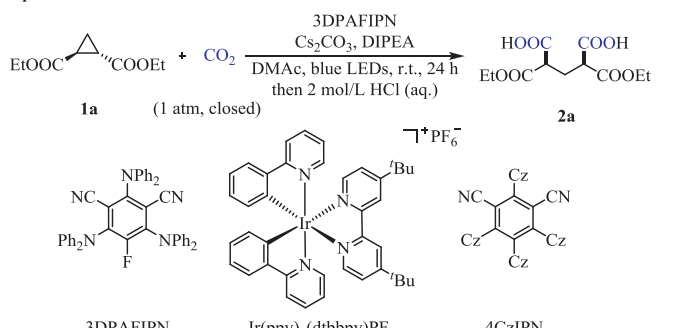
Scheme 1. (A) Importance of dicarboxylic acids and (B–D) photocatalytic reductive carboxylations with CO₂. PC = photocatalyst. EWGs = electron-withdrawing groups. r.t. = room temperature.

possible by ConPET (reaching 3.4 V against SCE) [33–41], which overcomes the present energy restriction ($E_{red} > -2.2$ V vs. SCE) of visible light photoredox catalysis (Scheme 1C) [42,43]. Encouraged by our recent success of using ConPET to realize ring-opening carboxylation of C–N bonds in cyclic amines with CO₂ [44], herein we report a novel photocatalytic ring-opening dicarboxylation of C–C single bonds in cyclopropanes and cyclobutanes with CO₂ to afford structurally diverse diacids that are otherwise difficult to access (Scheme 1D).

With such challenges in mind, we initiated our investigation of the dicarboxylation of small rings with CO₂ by testing reactions of diethyl *trans*-1,2-cyclopropanedicarboxylate **1a** with CO₂ under visible-light irradiation (Table 1). After systematic optimization of the reaction conditions, when we used 2,4,6-tris(diphenylamino)-5-fluoroisophthalonitrile (3DPAFIPN) as photocatalyst, Cs₂CO₃ as base, *N,N*-diisopropylethylamine (DIPEA) as electron donor, and *N,N*-Dimethylacetamide (DMAc) as solvent (Table 1, entry 1), the desired dicarboxylic acid **2a** was obtained in 72% isolated yield with moderate diastereomers selectivity (*dl:meso* = 4.8:1). Other photocatalysts, such as Ir-complex and 1,2,3,5-tetrakis(carbazol-9-yl)-4,6-dicyanobenzene (4CzIPN), gave lower yields (Table 1, entries 2 and 3). When Et₃N was used as a reductant or 3 equiv. of DIPEA was used, the yield dropped to 27% and 48%, respectively (Table 1, entries 4 and 5), which might ascribe to lower efficiency for reductive quenching of the excited photocatalyst. Other bases, such as K₂CO₃ and KO^tBu, provided a lower yield for the reaction (Table 1, entries 6 and 7). Moreover, the reaction could also be conducted in DMF to give the desired product in 66% yield (Table 1,

entry 8). Increasing the amount of DMAc also led to a decrease in yield (Table 1, entry 9). Control experiments revealed that CO₂, visible light, photocatalyst, and reductant were all essential for this transformation (Table 1, entries 10–14).

With the optimized reaction conditions in hand, the scope of this approach has been explored (Scheme 2). The 1,2-diester-cyclopropanes (**1a–1c**) were first tested to give tetracid derivatives in moderate to good yields, which have potential applications in coordination chemistry [45]. Substrates bearing both aryl groups and ester groups (**1d–1h**) were also transformed smoothly into the corresponding desired products with moderate to good yields. Substrate containing an amide (**1i**) can also yield the target product with good yield. Some symmetric diaryl-substituted substrates bearing an electron-neutral group, an electron-donating group (EDG), or an electron-withdrawing group (EWG) at the meta position on the aryl ring (**1j–1m**), were also well compatible under these conditions. Compared to previous work, which was mainly limited to the synthesis of symmetrical diacids [46], our method can realize the synthesis of both symmetrical and unsymmetrical ones. Moreover, unlike ubiquitous ring-opening difunctionalization reactions that are limited to donor-acceptor cyclopropanes [30], our reaction was applicable to a broader substrate scope. Encouraged by these results, we further investigated other structurally unsymmetric cyclopropane derivatives. The substrates with unsymmetric diaryl substituents that contain different EDGs or EWGs (**1n–1v**) were all suitable for such reactions to deliver the desired products in moderate to good yields. Notably, functional groups, such as nitrile (**1n**, **1t**), boryl (**1p**), thio ether (**1r**), and ester (**1s**,

Table 1
Optimizations of reaction conditions.^a


Entry	Variation	Yield (%) ^b
1	None	72
2	Ir(ppy) ₂ (dtbbpy)PF ₆ instead of 3DPAFIPN	40
3	4CzIPN instead of 3DPAFIPN	56
4	Et ₃ N instead of DIPEA	27
5	<i>N,N</i> -Diisopropylethylamine (3.0 equiv.)	48
6	K ₂ CO ₃ instead of Cs ₂ CO ₃	14
7	KO ^t Bu instead of Cs ₂ CO ₃	53
8	<i>N,N</i> -Dimethylformamide instead of DMAc	66
9	DMAc (2 mL)	60
10	w/o CO ₂	n.d.
11	w/o light	n.d.
12	w/o 3DPAFIPN	n.d.
13	w/o DIPEA	n.d.
14	w/o Cs ₂ CO ₃	n.d.

^a Reaction conditions: **1a** (0.2 mmol), 3DPAFIPN (3 mol%, 0.006 mmol), Cs₂CO₃ (4.5 equiv., 0.9 mmol), DIPEA (4.0 equiv., 0.8 mmol), 1 atm of CO₂, DMAc (1 mL), 30 W blue LEDs, room temperature (r.t.), 24 h.

^b Isolated yield. DMAc = *N,N*-Dimethylacetamide. n.d. = not detected. w/o = without. Cz = Carbazole.

1u), were all tolerated under the reaction conditions. Moreover, the substrate **1w** containing a thiophene heterocycle was also suitable. The substrate with increased steric hindrance (**1x**) was also compatible in this reaction. To our delight, cyclobutane derivative (**1y**) can also be used in our method, representing the first example of a photocatalytic reductive ring-opening reaction of cyclobutanes. Remarkably, complex molecules of biological relevance, including derivatives from Citronellol (**1z**), Leaf alcohol (**1aa**) and Pregnenolone (**1ab**) all delivered the products in moderate yields.

To demonstrate the utility of this method, we further conducted a gram-scale experiment and product derivatizations (Scheme 3). The gram-scale ring-opening dicarboxylation of **1a** with CO₂ proceeded smoothly to afford **2a** in 78% yield, demonstrating the potential utility of this method (Scheme 3A). The product **2j** can then be converted to corresponding ester with excellent yield (Scheme 3B). Diacid **2k** could be easily converted to dione **2k-1**, which has potential applications in chiral separation (Scheme 3C) [47]. Liquid crystals (LCs) have been extensively studied for both highly ordered nanostructures and external stimulus responses [48]. Although low-molecular-weight LC compounds can easily develop interesting nanostructures, their applications in devices and membranes are seriously limited by their poor mechanical properties [49]. On the other hand, polymers have characteristics such as high modulus, strong tensile strength, and high thermal stability. As a result, a liquid crystal polymer was developed, which combines the properties of liquid crystal and polymer and has been extensively researched in smart materials such as sensors [50] and actuators [51]. Therefore, we prepared CO₂-based main-chain liquid crystalline polymers **poly-1** and **poly-2** by solution polymerization of different diol monomers (**3-1** and **3-2**) and ring-opening dicarboxylation product **2j** (Schemes 3D and E). Pleas-

ingly, this polymerization method has produced two types of polyesters with molecular weights of 53,800 g/mol ($M_w/M_n = 1.57$) and 103,500 g/mol ($M_w/M_n = 1.97$), respectively. The glass transition temperatures (T_g) of **Poly-1** and **Poly-2** are 34.2 °C and 26.1 °C, respectively, and the temperature at 5% weight loss ($T_{5\%}$) are 349 °C and 384 °C, respectively, indicating the high thermal stability of **Poly-1** and **Poly-2**. The further application of this CO₂-based main chain liquid polyester is being studied in our laboratory.

To gain insights into the mechanism of this transformation, a series of control experiments were carried out (Scheme 4). When using 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO) as a radical scavenger, the dicarboxylation reaction was significantly inhibited, and the corresponding trapping adduct (**3a**) was detected by HRMS, indicating that this transformation involved a radical process (Scheme 4A). Next, we conducted deuterium-labeling experiments (Scheme 4B). When D₂O was added to the standard conditions, the deuterium incorporation ratio was up to 60%, demonstrating that a carbanion intermediate might exist in this reaction process. Moreover, we further carried out Stern–Volmer luminescence studies to confirm the electron donors in our catalytic system. The initial results showed that the luminescence of 3DPAFIPN was not effectively quenched by substrate **1a** while the DIPEA can efficiently quench excited-state photocatalysts with a slope of 403.5 (see Supporting information for details).

Cyclic voltammetry (CV) experiments on cyclopropane **1d** showed an obvious reductive peak at $E_p^{\text{red}} = -3.3$ V vs. SCE. However, the reduction potential of the photocatalyst 3DPAFIPN was found at $E_p^{\text{red}} = -1.8$ V, indicating that the reduced photocatalyst cannot reduce the substrate **1d** (see Supporting information for details). We speculated that this reaction might undergo the ConPET process to achieve a very low reduction potential. Therefore, we further investigated the behaviors of 3DPAFIPN and substrate **1v** via NMR tests (Fig. 1). First, we detected the characteristic ¹⁹F NMR signal of 3DPAFIPN with a mixture of 3DPAFIPN and DIPEA in DMSO-*d*₆ (step 1). After irradiating the solution with blue light, the characteristic signal of 3DPAFIPN disappeared due to the generation of unpaired electron species, which have a large effect on the NMR resonance transverse relaxation rates of adjacent fluorine atoms [38], indicating the generation of 3DPAFIPN^{•-} (step 2). Then, the addition of **1v** had no influence on 3DPAFIPN^{•-} in the dark (step 3). And there was no characteristic signal of the reductively protonated product **1v-1**, which meant 3DPAFIPN^{•-} might not directly transfer an electron to **1v**. Finally, when the mixture was irradiated by blue light, which enabled further excitation of 3DPAFIPN^{•-}, the characteristic signal of 3DPAFIPN reappeared, elu-

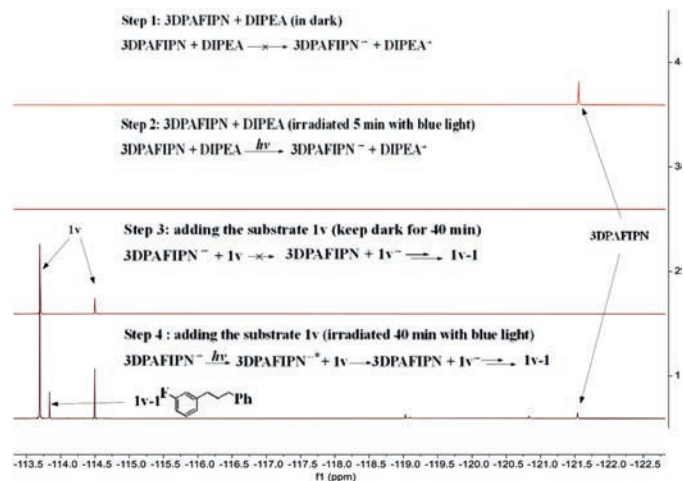
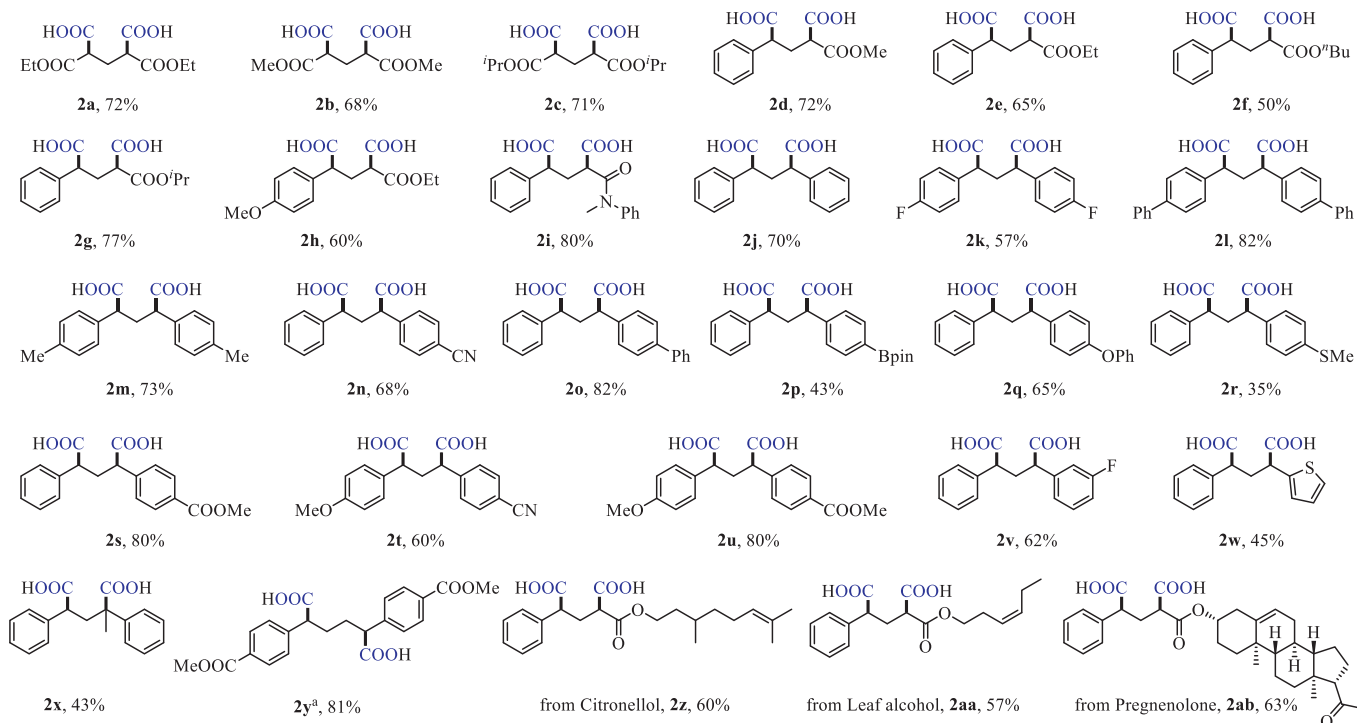
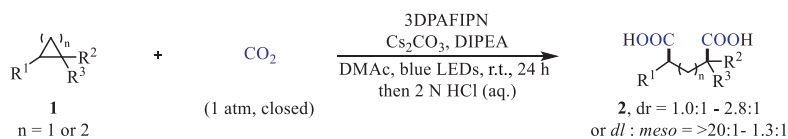
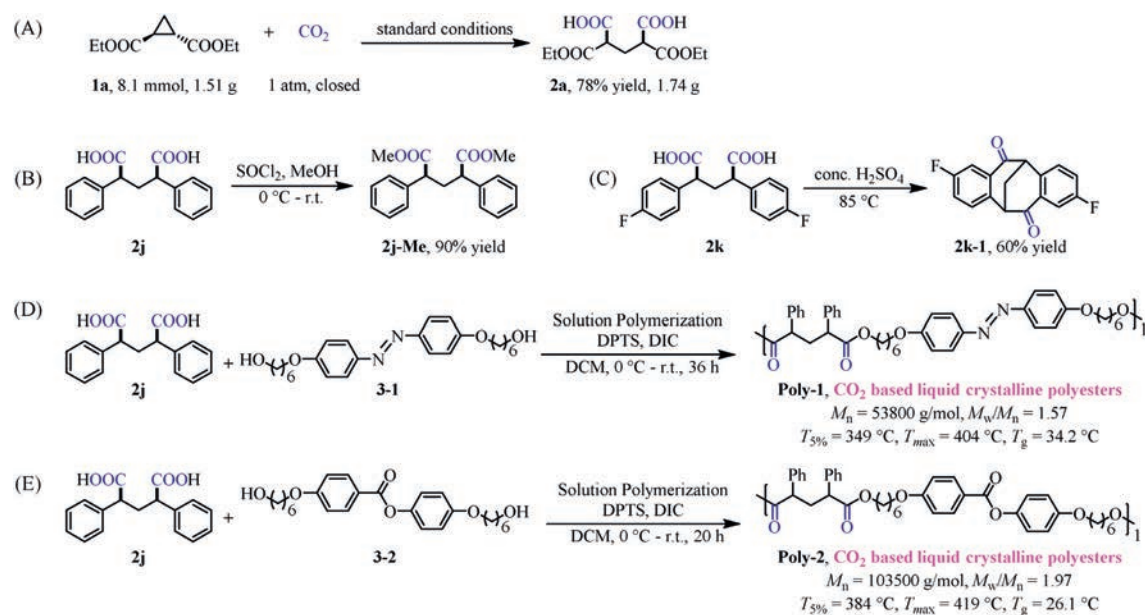


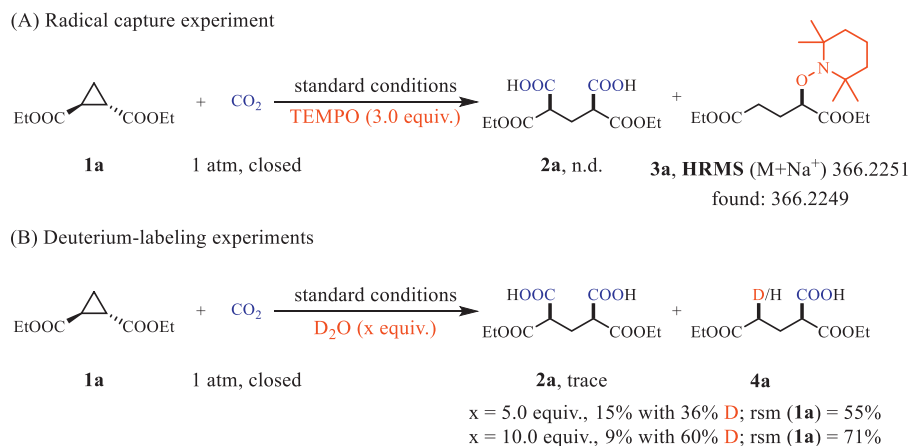
Fig. 1. ¹⁹F NMR spectroscopic evidence for ConPET process.



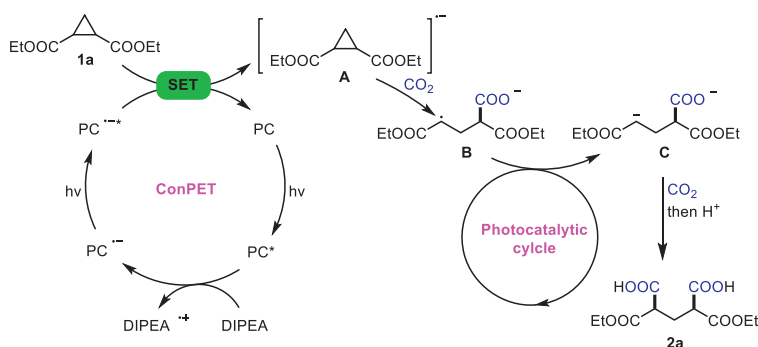
Scheme 2. Substrates scope. Conditions: **1a** (0.2 mmol), CO₂ (1 atm, closed), 3DPAFIPN (3 mol%, 0.006 mmol), Cs₂CO₃ (4.5 equiv., 0.9 mmol), DIPEA (4.0 equiv., 0.8 mmol), DMAc (1 mL), 30 W blue LEDs, room temperature (r.t.), 24 h. ^a 4DPAIPN (5 mol%, 0.01 mmol) instead of 3DPAFIPN, DMAc (0.5 mL), 48 h.



Scheme 3. Gram-scale experiment and product derivatization.



Scheme 4. Investigation of the mechanism.



Scheme 5. Possible mechanism.

indicating that **1v** could convert 3DPAFIPN^{•-} back to 3DPAFIPN via single electron transfer (SET). The emergence of the characteristic signal of **1v-1** further proved that photoexcitation was capable of reducing cyclopropane. In addition, the results of the ¹H NMR spectrum were consistent with the ¹⁹F NMR spectrum (see Supporting information for details), indicating that the ConPET process is engaged in this photocatalytic dicarboxylation of C–C bonds.

Based on these mechanistic studies and previous work [44], we proposed a possible reaction mechanism illustrated in Scheme 5. Excited photocatalyst PC* could likely be reductively quenched by DIPEA to generate DIPEA^{•+} and reduced photocatalyst PC^{•-}. PC^{•-} can further absorb a proton to form PC^{•-}, which reacts with **1a** via the SET process to produce PC and radical anion **A**. Then, intermediate **A** could react with CO₂ to obtain radical **B**, which undergoes SET to generate carbanion intermediate **C**. The final nucleophilic attack of intermediate **C** on CO₂ and acidification would give the diacid product **2a**.

In conclusion, we have developed a novel strategy to realize the dicarboxylation of C–C single bonds in strained rings that contain symmetrical or unsymmetrical structures with CO₂ via visible-light photoredox catalysis. Moreover, this method represents the first report of a photocatalytic reductive ring-opening reaction of cyclobutanes. This reaction shows high selectivity, high step and atom economy, mild reaction conditions (room temperature, 1 atm of CO₂), facile scalability and product derivatization, good functional group tolerance, and broad substrate scope, providing great potential for the synthesis of valuable but difficult-to-access dicarboxylic acids. The obtained diacid product could be converted into the main-chain liquid crystalline polymer through solution polymerization. Mechanistic studies indicate that the ConPET strategy might be the key to generating highly reactive photocatalysts, which en-

able the reductive activation of strained rings to generate carbon radicals and carbanions as the key intermediates. Further research on the ConPET strategy for CO₂ utilization is underway in our laboratory.

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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