INTRODUCTION

Dicarbofunctionalization of alkenes has recently attracted increasing attention given the rapid generation of molecular complexity from readily available alkene starting materials. In regard to the booming radical chemistry, impressive advances have been achieved in the development of catalytic radical alkene difunctionalization initiated by intermolecular addition of carbon-centered radicals to alkenes catalyzed by transition-metal redox systems owing to the high innate reactivity and unique selectivity of radical intermediates. In contrast, catalytic radical asymmetric dicarbofunctionalization of olefins, especially for the intermolecular three-component version, is much less developed given the number of side reactions that highly reactive alkyl radical intermediates can undergo. In this aspect, Liu and co-workers recently adopted chiral metal species to trap reactive alkyl radical, a strategy pioneered by Fu and others (Scheme 1a), to elegantly develop state-of-the-art enantioselective intermolecular trifluoromethylarylation and cyanotri fluoromethylarylation (Scheme 1b). Although impressive results have been achieved with this approach, prefunctionalization of the carbon nucleophiles as boronic acid or trimethylsilane was mechanistically indispensable. On the other hand, there have been no general and practical solutions to allow for the efficient construction of chiral all-carbon quaternary stereocenters, presumably because of the inherently unfavorable steric hindrance. From the viewpoint of high step-economy as well as versatility, a mechanistically distinct and alternative approach for reactions capable of not only generating all-carbon quaternary stereocenters but also accommodating direct intermolecular C–H functionalization is highly desirable.

To this end, we wondered if our recently developed Cu(I)/chiral phosphoric acid (CPA) dual-catalysis for radical asymmetric intramolecular transformations could be translated into a general and practical solution toward this issue. It has been assumed that the in situ-generated electronically activated benzylic radical might readily undergo a single-electron oxidation process in the presence of CuII species to associate with chiral phosphate through electrostatic interactions, thereby resulting in a good chiral environment created by the hydrogen-bonding and ion-pair interactions between the chiral phosphoric acid catalyst and substrates, which leads to the enantioselective C–C bond formation.
Scheme 1. Asymmetric Three-Component Radical-Initiated 1,2-Dicarbofunctionalization of Alkenes

(a) Alkyl radical trapped with chiral metal species

(b) Intermolecular dicarbofunctionalization via trapping alkyl radical with copper

(c) Proposal for a mechanistically distinct strategy via carboxylation intermediates

(d) This work: CuCPA-catalyzed asymmetric intermolecular dicarbofunctionalization

those in previous works invoking chiral Cu species to trap alkyl radical as a key step.6,8 If achieved, this type of radical redox-relay cooperative catalytic strategy would be synthetically significant because the resulting chiral triarylmethanes bearing quaternary all-carbon stereocenters represent key structural elements of a large number of molecules in the dye industry, medicinal chemistry, and material science as well as organic synthesis (Figure 1),9 but their asymmetric construction remains a significant challenge and scarce.10 At the outset, we recognized that several challenges would need to be overcome to put this idea into practice, including securing the outset, we recognized that several challenges would need to be overcome to put this idea into practice, including securing

RESULTS AND DISCUSSION

Given the increasing importance of fluoroalkyl-containing molecules in the development of pharmaceuticals and agrochemicals as well as materials,16 we began our investigation using fluorine-containing carbon-centered radical precursors. To this end, we selected 1,1-diaryalkene 1aa bearing one OMe-substituted electron-rich arene as the pilot alkene substrate, indole derivative 2a as the carbon nucleophile, and perfluorobutanyl sulfonyl chloride 3a as the oxidative radical precursor (Table 1, entry 1). However, the three-component reaction in the presence of CuI and CPA (S)-A1 (10 mol %) with Ag2CO3 as an additive provided the desired product 4AA in poor yield and enantioselectivity, along with side monofunctionalization product 5AA in good yield through a β-hydride elimination process and direct hydroarylation product 6AA, respectively. Given this, we surmised that the installation of a possibly removable hydroxy or amino directing group on one of the two aryl groups might impart effective stereocontrol, considering the fact that a chiral phosphate anion is able to interact with such groups via hydrogen bonding.17 Subsequently, we chose the substrates 1ba, 1a, and 1ca bearing OH or NH2 groups at different positions (para or meta) of one aryl ring based on our initial hypothesis (Table 1, entries 2–4), and we were pleased to observe a significant increase in enantioselectivity with the hydroxy directing group, albeit with low product yield and along with other side reactions, including perfluoroalkylation and direct hydroarylation with indole.

This proof-of-principle result encouraged us to carry out further systematic optimizations of different reaction parameters with use of 1a as the model substrate (Table 2). To suppress the more common hydroarylation or perfluoroarylation clearly observed in this reaction and improve the enantioselectivity, we screened different copper salts and various organic solvents as well as reaction temperature. Unfortunately, either enantioselectivity or chemoselectivity could not be significantly improved under these reaction conditions (Table 2, entries 1–6). Considering the reported significant steric effect of CPAs in the direct C-alkylation reaction,10e we then screened various BINOL- and SPINOL-derived CPAs (Table 2, entries 7–12) and found that the use of a sterically bulky 6,6'-bis(2,4,6-triisopropylphenyl)-4,4'-dimethyl SPINOL-derived chiral phosphoric acid (S)-A418 dramatically inhibited the hydroarylation process, presumably because of its significant steric bulkiness hindering nucleophilic indole from approaching toward the alkene. It is worth noting that CPA (S)-A5 bearing sterically more bulky tricyclohexylphenyl groups at the 6,6'-positions totally abolished the perfluoroalkylation side reaction while significant inhibiting hydroarylation and efficiently providing 4A in superior yield (88%) and enantioselectivity (95% ee) with a reaction temperature at -3 °C (Table 2, entry 13).

With the optimal reaction conditions established, we next investigated the substrate scope of the asymmetric intermolecular perfluoroalkylarylation of alkenes (Table 3). Various diversely functionalized 1,1-diarylethenes, including those having aryl groups with electron-withdrawing (CF3, F, Cl, NO2, and CO2Me) or electron-donating groups (OMe and Me) at different positions (ortho, meta, or para) as well as a polyaromatic naphthalene ring were found to be suitable for this reaction.
substrates to afford the expected products $4A-4P$ in 56–97% yields with 91–98% ee. It is noteworthy that a substrate containing the other reactive and useful ethynyl group also afforded the corresponding product $4I$ with the additional triple bond intact. In addition, thiophene-substituted olefin was tolerated to give $4L$ in 95% yield and 97% ee. Furthermore, a range of substituted indoles all underwent the current perfluoroalkylation reaction smoothly to deliver the corresponding products $4Q-4V$ in excellent yields with excellent enantioselectivity. Encouraged by the above success, we thus switched our synthetic target to chiral difluoroacetetyl-triaryl methane, as the difluoromethyl group (CF₂R) has routinely served as a core pharmacophore in drug discovery in recent years. To our delight, the reaction of 1,1-diarylethylene substrate $1a$ with MeO₂CCF₂SO₂Cl ($3b$) under the otherwise identical reaction conditions delivered difluoroacetyl-containing product $5A$ in moderate yield with 80% ee.

The trihalomethyl unit has emerged as a key moiety presented in numerous bioactive natural products and

Table 1. Initial Exploration of Reaction Conditions

<table>
<thead>
<tr>
<th>entry</th>
<th>$1$</th>
<th>$R'$</th>
<th>$R''$</th>
<th>$4$</th>
<th>yield (%)</th>
<th>ee (%)</th>
<th>$5$</th>
<th>yield (%)</th>
<th>$6$</th>
<th>yield (%)</th>
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<tbody>
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<td>OMe</td>
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<td>$5Aa$</td>
<td>53</td>
<td>$6Aa$</td>
<td>24</td>
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<tr>
<td>2</td>
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<td>3-OH</td>
<td>OMe</td>
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<td>54</td>
<td>43</td>
<td>$5Ba$</td>
<td>44</td>
<td>$6Ba$</td>
<td>0</td>
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<tr>
<td>3</td>
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<td>4-OH</td>
<td>Me</td>
<td>$4A$</td>
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<td>$5Ca$</td>
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<td>$6Ca$</td>
<td>43</td>
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<tr>
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<td>68</td>
<td>$5Da$</td>
<td>68</td>
<td>$6Da$</td>
<td>0</td>
</tr>
</tbody>
</table>

*Reaction conditions: $1$ (0.1 mmol), $2a$ (0.12 mmol), n-C₄F₉SO₂Cl (0.12 mmol), CuI (10 mol %), Ag₂CO₃ (0.06 mmol), CPA (10 mol %), DCM (1.0 mL) under argon. *Isolated yield. *ee value on HPLC.

Table 2. Screening of Reaction Conditions

<table>
<thead>
<tr>
<th>entry</th>
<th>[Cu]</th>
<th>CPA</th>
<th>solvent</th>
<th>$4A$</th>
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<th>$5Ca$</th>
<th>yield (%)</th>
<th>$6Ca$</th>
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<td>DCM</td>
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<td>CuI</td>
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<td>11</td>
<td>−95</td>
<td></td>
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</table>

*Reaction conditions: $1a$ (0.05 mmol), $2a$ (0.06 mmol), n-C₄F₉SO₂Cl (0.06 mmol), [Cu] (10 mol %), Ag₂CO₃ (0.03 mmol), CPA (10 mol %), dry solvent (0.5 mL), 0 °C, 60 h under argon. *Yield based on $^1$H NMR analysis of the crude product using CH₂Br₂ as an internal standard. *ee value on HPLC. *20 °C. *(S)-A5 (7.5 mol %) was used at −3 °C.
pharmaceutical drugs;¹⁹ thus, its preparation has spurred the development of many new reagents and strategies.²⁰ To this end, we successfully prepared trichloromethyl-containing products 6A−6D in good to excellent yields with 90−94% ee (Table 4) via reaction of 1,1-diarylethylene substrate 1 with trichloromethanesulfonyl chloride 3c under the almost identical conditions. As for chiral CF₃-containing triaryl-methanes, we identified the well-known Togni’s reagent (3d)²ᵈ but not trifluoromethanesulfonyl chloride (CF₃SO₂Cl)³⁰ as the appropriate CF₃ source to obtain 7A−7G in good to excellent yields with excellent ee. Meanwhile, it was striking to note that 2-methyl-1H-pyrrole as a nucleophile also underwent the current reaction to deliver 7H in 78% yield with promising enantioselectivity, which is currently under further optimization in our laboratory. The absolute configuration of 7G has been determined to be S by X-ray crystallographic analysis (Figure S2 in the Supporting Information for Experiments), and those of other perfluoroalkyl-, trichloromethyl-, difluoroacetyl-, and trifluoromethyl-containing triarylmethanes were inferred accordingly.

To further demonstrate the practicality of the current methodology in preparative organic synthesis, we carried out gram-scale synthesis of products 4C, 6E, and 7E. As displayed in Scheme 2, the asymmetric 1,2-dicarbofunctionalization of 1c with different radical precursors was performed on a 1.008 g scale under the standard reaction conditions, and high efficiency and enantioselectivity were still maintained all the time.

An apparent drawback of our current transformation is the requirement for a hydroxy directing group. In fact, this hydroxy group was readily removed by straightforward triflation and subsequent reduction to afford 7Bb with an unsubstituted phenyl ring (Scheme 3, eq 1). In addition, the triflated intermediate 7Ba also provided extra synthetic potentials for further derivatization by cross-coupling reaction, as demonstrated by synthesizing 7Bc with excellent efficiency (Scheme 3, eq 2). Besides, simple reduction or hydrolysis of the ester group in 5A smoothly generated the corresponding difluoro-containing alcohol 8A (Scheme 3, eq 3) and carboxylic acid 8B in good yields (Scheme 3, eq 4). An important aspect of the transformations discussed above is that no significant enantiopurity erosion has ever occurred, establishing the utility of this method in practical synthetic chemistry. It is interesting to note that the chiral dichloroolefin-containing product 9A was obtained in excellent yield and enantioselectivity through direct elimination of hydrogen chloride from corresponding trichloromethylated products in cases of electron-rich indole reactant (Scheme 3, eq 5). Such resultant dichlorooolefin can serve not only as a potential synthetic platform for facile access to other valuable chiral triarylmethanes but also as a key structural element for potent activity in numerous bioactive compounds.²¹

Table 3. Substrate Scope for Perfluoroalkylation or Difluoroacetylarylation of 1ᵃ,b,c

<table>
<thead>
<tr>
<th>Reactions were conducted on 0.1 mmol scale.</th>
<th>Isolated yield based on 1 is given.</th>
<th>ee was determined by HPLC analysis.</th>
<th>Run at −3 °C.</th>
<th>Run at 0 °C for 72 h, then 25 °C for 42 h.</th>
<th>Is [5 mol % CuI].</th>
<th>Run at 0 °C for 72 h, then 29 °C for 42 h.</th>
<th>15 mol % CuI.</th>
<th>20 mol % CuI.</th>
<th>The reaction was conducted on 0.025 mmol scale with Ag₂CO₃ (0.5 equiv), (R)-3,3′-(3,5-(Ph)₂C₆H₃)₂-8H-BINOL-derived CPA at −30 °C for 72 h.</th>
</tr>
</thead>
</table>

Diagrams and images related to the chemical transformations are not provided in the text.
Table 4. Substrate Scope for Trichloromethylarylation and Trifluoromethylarylation of 1a,b

![Table 4. Substrate Scope for Trichloromethylarylation and Trifluoromethylarylation of 1a,b]

Using CO3(SO2Cl)2 as radical precursor

![Using CO3(SO2Cl)2 as radical precursor]

Using Togni’s reagent as radical precursor

![Using Togni’s reagent as radical precursor]

To gain some insight into the reaction mechanism, a series of control experiments were conducted. First, the present reaction was completely inhibited by the addition of 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO), and the radical trapping product n-C5F9−TEMPO was observed by 19F NMR and detected by GC-MS, suggesting that the n-C5F9 radical was likely generated in situ in the presence of Cu catalyst through a single electron-transfer process (Scheme 4, eq 1). In addition, no desired product 4A was obtained in the absence of any copper catalysts. Furthermore, rac-4A was obtained in lower yield in the absence of phosphoric acid. These observations, together with the above-mentioned significant effects of different CPAs in the reaction condition optimization study (Table 2), indicate that both the Cu(I) salt and CPA are essential for this reaction, and CPA might have played an important role in the activation of sulfonyl chloride.

In addition, either the N-protected indole 2i (Scheme 4, eq 2) or methyl-protected 1,1-diarylethylene 1aa (Table 1) is not an effective substrate to provide satisfactory enantioselectivity under similar reaction conditions. These facts indicate the important roles of the N–H and the O–H moieties in these substrates, presumably via formation of cooperative multiple hydrogen bonding with chiral phosphate. It is noteworthy that Sun and co-workers have elegantly developed direct C-alkylation reaction of indoles via formation of a tertiary carboxylation intermediate by protonation of electron-rich terminal 1,1-diarylethylene with CPA. In our reactions, a small amount of internal alkene product 5Ca was formed via radical alkylation of 1,1-diarylethylene in some cases, but its participation in our desired reaction via either protonation with CPA (path b in Figure 2b) or any other pathways was unlikely given that no reaction of 5Ca was observed either under standard conditions or in the presence of CPA only, respectively (Scheme 4, eq 3). Thus, the in situ generated carboxylation intermediate from benzylic radical might directly undergo attack by nucleophile (path a in Figure 2b), which would be mechanistically distinct from the previous work via the direct protonation of alkenes by CPA, i.e., path b. Owing to not only the facile oxidation of electronically activated radical to a carboxylation intermediate but also the straightforward installation of a diverse array of carbon-centered radicals to alkenes, the current synthetic protocol exhibits a number of clear advantages over the direct alkene protonation approach in terms of high reactivity, a broader substrate scope, and versatile functionalization of the products, thus constituting a more appealing alternative to the previous approach.

To further elaborate the mechanistic details, we studied the relationship between product enantioselectivity and catalyst enantipurity. The observed linear relationship indicates the involvement of one CPA catalyst in the enantioselectivity-determining transition states (Figure 2a). On the basis of above observations and previous reports, we have proposed a working mechanism, as shown in Figure 2b. Cu(I) first reacts with CPA-activated RS(O)Cl by hydrogen bonding via single-electron transfer, giving the crucial chiral Cu(II) phosphate complex A accompanied by the generation of carbon-centered radical and a stoichiometric amount of sulfur dioxide and hydrogen chloride (HCl). The additive Ag2CO3 acts as a HCl scavenger via the formation of insoluble AgCl in organic solution. Subsequently, the addition of carbon-centered radical to alkene 1 gives alkyl radical B, which subsequently undergoes single-electron oxidation with the Cu(II) complex A to form the corresponding carbocation intermediate. This carboxylation intermediate is next attacked by electron-rich heteroaromatics through intermediate C or its p-quinone methide resonance structure C* by direct hydrogen-bonding interactions and ion-pair interactions in C or only hydrogen bonding interactions in C* to realize excellent stereoccontrol. In the case of the meta-phenol substrate 1Ba (4Ba up to 53% ee, Scheme S1), only intermediate C is reasonably involved because no quinone methide resonance structure can be invoked.

Our hypothesized enantioselective C–C bond formation through intermediate C is supported by density functional theory (DFT) calculations. Using (S)-AS as the model CPA...
catalyst and 1a and indole 2b as the model substrates, we were able to locate the C–C bond formation transition states TS10-S and TS10-R that lead to enantiomeric product formation (Figure 3). Various complexation types between CPA catalyst and substrates, as well as careful conformational searches, are considered to ensure that the most favorable transition states are located (see Figures S1–S4 in the Supporting Information for Computations). Both transition states involve the proposed hydrogen bonding and ion-pair interactions between the CPA catalyst and substrates, which create the chiral environment for enantiodiscrimination.\(^{23}\) TS10-S is 5.0 kcal/mol more favorable than TS10-R in terms of free energy (Figure 3), which is consistent with the observed enantioselectivity (Table 4). Calculations with additional functionals were performed to further validate the energy difference between the two transition states (Table S1 in Supporting Information for Computations). The leading factor that differentiates the two competing transition states is the CH-π interaction between the cyclohexyl group of CPA catalyst and the para-methylphenyl group of alkene.\(^{24}\) This favorable CH-π interaction stabilizes TS10-S by 3.9 kcal/mol based on the calculations of interacting fragments (Figure S5 in Supporting Information for Computations), while TS10-R does not possess this interaction because of the stereogenic configuration of forming the C–C bond. The proposed CH-π interaction is also proved by IGM analysis;\(^{25}\) the green oval
represents the favorable interaction between the highlighted fragments (Figure 3). Therefore, our calculations indicate the importance of hydrogen bonding and ion-pair interactions in the chiral induction, as well as the noncovalent CH-π interaction that differentiates the enantiomeric C−C bond formations.

**CONCLUSION**

In summary, we have utilized copper(I) and chiral phosphoric acid cooperative catalysis to develop an asymmetric intermolecular three-component radical-initiated dicarbocationalization of 1,1-diarylalkenes with a diverse array of carbon-centered radical precursors and electron-rich heteroaromatics, encompassing a direct intermolecular arene C(sp^2^)−H functionalization. It provides straightforward access to chiral triarylmethanes bearing quaternary all-carbon stereocenters with high efficiency as well as excellent chemoo- and enantioselectivity. Incorporating a removable/convertible hydroxy group as the directing group and introducing a sterically demanding chiral phosphoric acid jointly favor the desired radical difunctionalization over the otherwise remarkable side reactions. This method represents a mechanistically distinct approach to enable the rapid asymmetric difunctionalization of olefins via the intermediacy of a carbbocation species through single-electron oxidation. The obtained products can serve as practical synths toward valuable chiral molecular entities in the fields of pharmaceuticals, agrochemicals, and materials. Mechanistic investigations by combined experiments and computations elucidated the reaction mechanism and origins of enantioselectivity. The key enantioselective C−C bond formation process between a heteroaromatic compound and a carbocation intermediate occurs in a chiral environment, which is created by the hydrogen-bonding and ion-pair interactions with CPA catalyst. The controlling factor of enantioselectivity is the CH-π interaction between the cyclohexyl group of CPA catalyst and one of the aryl rings on the alkenesubstrates.

**ASSOCIATED CONTENT**

* Supporting Information
The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/jacs.8b11736.

Theory (DFT) calculations and characterization data (PDF)
Figures S1 and S2 and experimental procedures (PDF)
Crystallographic data for 7G (CIF)

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Author Contributions

Notes
The authors declare no competing financial interest.

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