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# Organocatalytic enantioselective α-difluoromethylketone thiolation of β-keto esters using phthalimide-$SCF_2COAr$†

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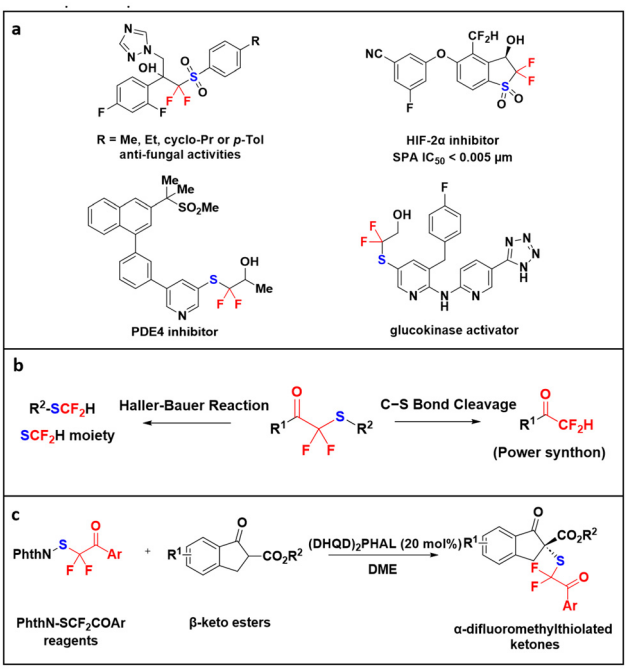
**The first asymmetric α-difluoromethylketone thiolation of diverse β-keto esters using an electrophilic phthalimide-$SCF_2COAr$ reagent (PhthN-$SCF_2COAr$) was reported. In the presence of cinchona-alkaloid-based catalyst $(DHQD)_2PHAL$, the reaction was achieved in moderate yields with a moderate to excellent enantioselectivity (up to 93% ee) under mild reaction conditions and exhibited functional group compatibility. An ammonium hydrogen-bonded induction model was proposed to rationalize the origin of enantioselectivity.**

The fluoroalkylthio groups, including trifluoromethylthio (–$SCF_3$), pentafluoroethylthio (–$SC_2F_5$), difluoromethylthio (–$SCF_2H$) and monofluoromethylthio (–$SCH_2F$), have attracted significant interest due to their tunable lipophilicity, metabolic stability and strong electron-withdrawing properties, as well as their promising applications in improving the pharmacokinetics of lead compounds for drug discovery.[1,2] In particular, one of these fluoroalkylthio groups, the α-difluorothiomethylated ketone group (–$SCF_2COAr$), is of great current interest.[3] The $SCF_2COAr$ groups and their derivatives not only serve as active scaffolds for agrochemical and pharmaceutical molecules[4] (Scheme 1a) but also act as effective precursors for the construction of diverse fluorine-containing compounds[5,6] (Scheme 1b). Therefore, developing robust methodologies for preparing α-difluorothiomethylated ketones has recently received attention.[7–9] To the best of our knowledge, the analogous catalytic asymmetric direct enantioselective introduction of the $SCF_2COAr$ group has never been reported. Thus, an efficient method for the highly enantioselective synthesis of α-difluorothiomethylated ketone derivatives is urgently needed.

We developed an electrophilic phthN-$SCF_2COAr$ reagent[9] (**1**), which can be stored for up to one year at room temperature without decomposition. These reagents are demonstrated to be efficient $SCF_2COAr$ transfer agents that react with various C-nucleophiles, including Grignard reagents, phenylboronic acids, β-keto esters, and anilines, to afford diverse α-difluorothiomethylated ketones under mild conditions. The enantioselective formation of a C–$SCF_2COAr$ bond, coupled with the simultaneous generation of a stereogenic carbon center, is a challenging task. This challenge prompted us to develop a catalytic asymmetric reaction utilizing electrophilic




**Scheme 1** (a) α-Difluorothiomethylated ketones derivatives in bioactive compounds. (b) Translation of difluorothiomethylated ketones. (c) The catalytic asymmetric synthesis of chiral α-difluoromethylthiolated ketones.

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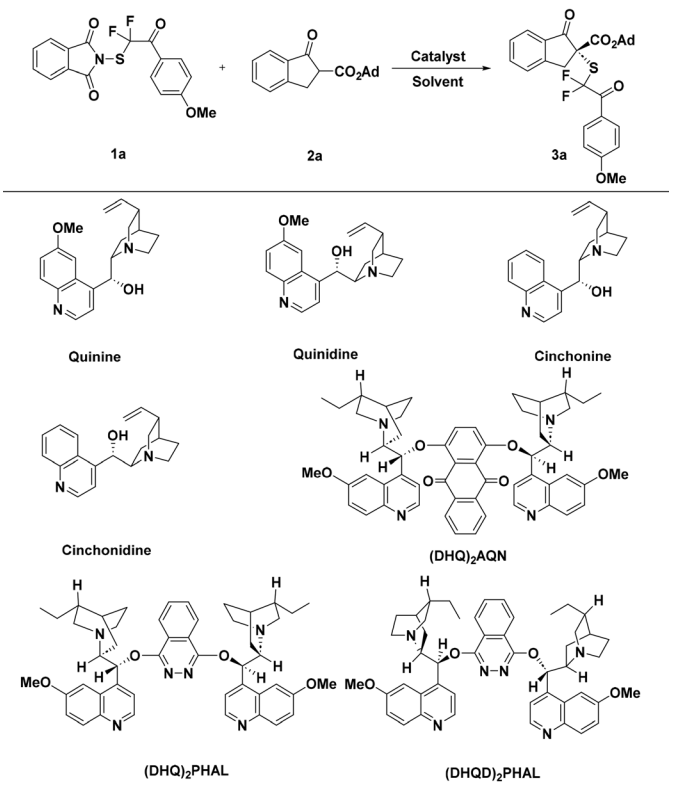
PhthN-$SCF_2COAr$ reagents. We envisaged that selecting a chiral organic Lewis base would be crucial to providing an ideal chiral environment for achieving enantiocontrol over the highly reactive nucleophilic species.

1-Indanones, particularly those bearing a stereogenic center at the C2 position, are prevalent structural motifs in biologically active natural products and pharmaceutical agents.[10] To address the synthetic challenges associated with these chiral architectures, various organocatalytic strategies have been developed for the asymmetric α-halogenation,[11,12] α-cyanation,[13] and α-azidation of β-ketoesters.[14] Herein, we report the asymmetric α-difluoromethylketone thiolation of β-keto esters with moderate to excellent enantioselectivity (up to 93% ee) using a cinchona alkaloid-derived catalyst, $(DHQD)_2PHAL$.

With the abundant and stable reagents in hand, the stereoselective incorporation of $SCF_2COAr$ groups into β-keto esters was subsequently explored. We initially selected the reaction of indanone-derived β-keto ester **2a** with reagent **1a** to optimize the reaction conditions. Because cinchona alkaloids can function as effective organic chiral Lewis bases and nucleophilic catalysts,[15,16]

**Table 1** Optimization of the reaction conditions[a]




| Entry | Catalyst | Solvent | Yield[b] (%) | ee[c] (%) |
|---|---|---|---|---|
| 1 | Cinchonine | DCM | 76 | −43 |
| 2 | Cinchonidine | DCM | 76 | 43 |
| 3 | Quinine | DCM | 81 | 23 |
| 4 | Quinidine | DCM | 76 | −57 |
| 5 | $(DHQ)_2AQN$ | DCM | 76 | −14 |
| 6 | $(DHQ)_2PHAL$ | DCM | 50 | −53 |
| 7 | $(DHQD)_2PHAL$ | DCM | 69 | 86 |
| 8 | $(DHQD)_2PHAL$ | THF | 56 | 87 |
| 9 | $(DHQD)_2PHAL$ | MeCN | 76 | 84 |
| 10 | $(DHQD)_2PHAL$ | DCE | 30 | 86 |
| 11 | $(DHQD)_2PHAL$ | DMF | 56 | 32 |
| 12 | $(DHQD)_2PHAL$ | PhMe | 20 | 90 |
| 13 | $(DHQD)_2PHAL$ | $CF_3Ph$ | 38 | 89 |
| 14 | $(DHQD)_2PHAL$ | DME | 61 | 93 |
| 15 | $(DHQD)_2PHAL$ | MTBE | 10 | 89 |
| 16 | $(DHQD)_2PHAL$ | 1,4-Dioxane | 48 | 89 |
| 17 | $(DHQD)_2PHAL$ | CPME | 4 | 84 |

[a] Reaction conditions: adamantyl 1-indanonyl-2-carboxylate **2a** (0.12 mmol), **1a** (0.1 mmol), catalyst (20 mol%), in solvent (1 mL) at 25 °C for 48 h. [b] Isolated yield. [c] The enantiomeric excess was determined by HPLC analysis.

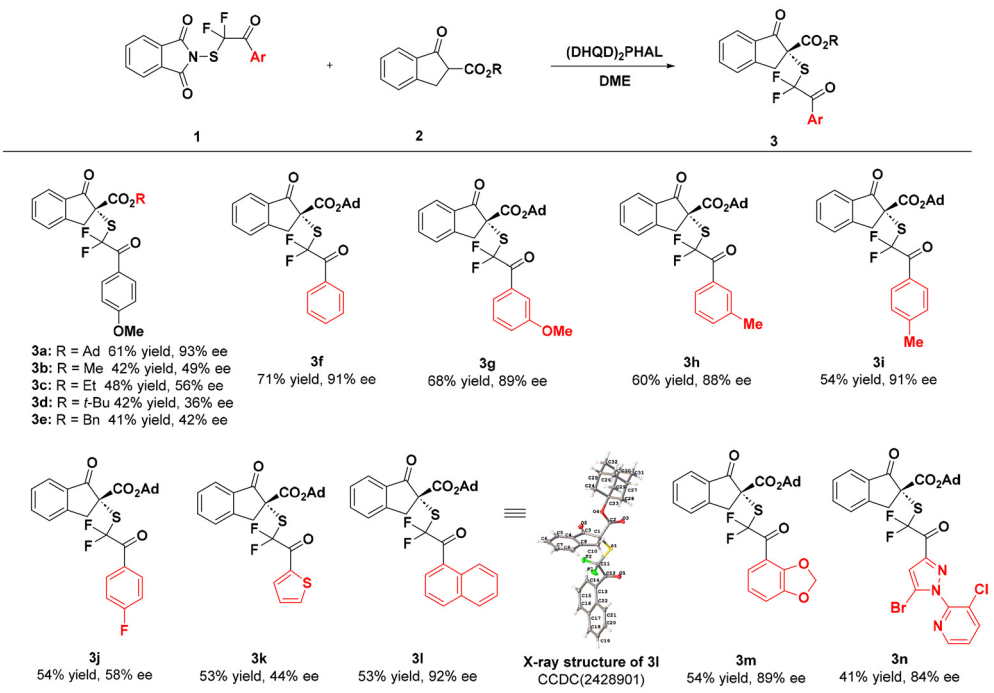
 

we first screened various cinchona alkaloid catalysts (Table 1, entries 1–7; for details, see Table S1 in the ESI†). While cinchonine, cinchonidine, quinine, and quinidine could efficiently initiate the reaction, lower enantioselectivity was observed (Table 1, entries 1–4). Three different bis(cinchona alkaloids) were further evaluated (Table 1, entries 5–7). In the case of $(DHQ)_2AQN$, the reaction gave the product in moderate yield but with significantly lower enantiomeric excess (Table 1, entry 5). When $(DHQ)_2PHAL$ was employed, the reaction efficiency slightly decreased, with moderate enantiomeric excess (Table 1, entry 6). Notably, $(DHQD)_2PHAL$ enabled the reaction to proceed in moderate yield with good enantiomeric excess (Table 1, entry 7).

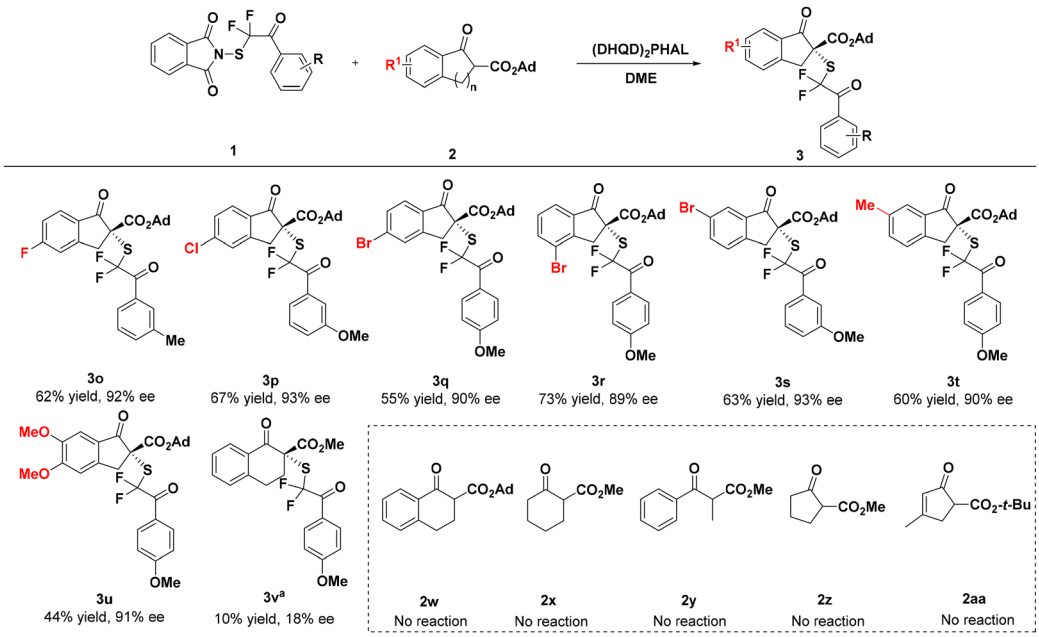
Subsequently, solvent screening was conducted under optimized conditions (Table 1, entries 8–17). Reactions in more polar solvents, such as $CH_3CN$ and THF, or in a less polar solvent, such as DCE, generated the corresponding products with comparable enantioselectivity (Table 1, entries 8–10). Toluene and trifluorotoluene exhibited excellent enantioselectivity but lower yields compared to other solvents. Among ethers such as dioxane, dimethoxyethane (DME), methyl *tert*-butyl ether (MTBE), and cyclopentyl methyl ether (CPME), similar enantioselectivity was observed, but the reactions were more efficient in DME than in other solvents (Table 1, entries 14–17). Interestingly, when DME was used as the solvent, the enantioselectivity of the reaction increased to 93% ee, albeit with a slight reduction in yield (Table 1, entry 14). Optimal results were achieved by performing the α-difluoromethylketone thiolation reaction in DME with $(DHQD)_2PHAL$ as the catalyst (Scheme 2). Ultimately, compound **3a** was obtained in 61% yield with 93% ee under these conditions (Table 1, entry 14).

With the optimized conditions in hand, we first investigated the reaction of the phthN-$SCF_2COAr$ reagent with a variety of β-keto esters to explore the effect of ester group size on the enantioselectivity of the asymmetric α-difluoromethylketone thiolation. The results demonstrated that varying the ester group size (Me, Et, *t*-Bu, Bn, Ad) significantly influenced the enantioselectivity of the reaction. Specifically, the adamantyl ester of indanone (**3a**) provided the α-difluoromethylthiolated ketone in moderate yield with excellent enantioselectivity.

To demonstrate the universality of the reaction, we further investigated the influence of different phthN-$SCF_2COAr$ reagents. Various phthN-$SCF_2COAr$ reagents, including benzene ring (**3f**), electron-donating groups on the benzene ring (**3g**, **3h**, **3i**), naphthyl (**3l**), bisubstituted benzene ring (**3m**), and insecticidal activity fragments of chlorantraniliprole (**3n**) were well-tolerated, yielding products with good to excellent enantioselectivity (84–93% ee). Chlorantraniliprole[17] is the first commercialized anthranilic diamide pesticide modulating RyRs, promoted the development of diamide insecticides. Notably, the electron-withdrawing fluorine atom markedly reduced enantioselectivity, affording product **3j** in 54% yield and 58% ee. Additionally, the thiazolyl phthN-$SCF_2COAr$ reagent reacted with the adamantyl ester of indanone, but the product (**3k**) was obtained with only moderate efficiency and enantioselectivity. The absolute configuration of the stereo-




**Scheme 2** Construction of diverse α-difluoromethylthiolated ketones. Reaction conditions: **2** (0.12 mmol), **1** (0.1 mmol), catalyst (20 mol%) in 1 mL of DME at room temperature for 48 h. Yield of isolated product. The enantiomeric excess was determined by HPLC analysis.

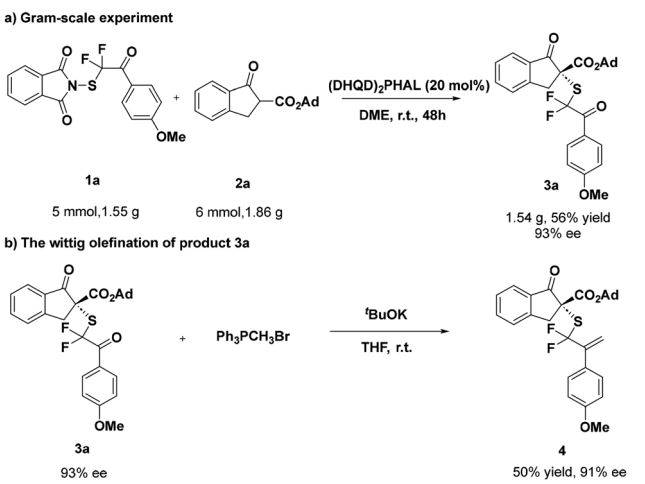



**Scheme 3** Scope for $(DHQD)_2PHAL$-catalyzed asymmetric α-difluoromethylketone thiolation of β-keto esters. Reaction conditions: **2** (0.12 mmol), **1** (0.1 mmol), catalyst (20 mol%) in 1 mL of DME at room temperature for 48 h. Yield of isolated product. The enantiomeric excess was determined by HPLC analysis. [a]Reactions (**3v**) were carried out at 60 °C for 7 days.

genic carbon center in the α-difluorothiomethylated ketone was determined to be (*R*) *via* X-ray crystallographic analysis of the optically active product **3l**.

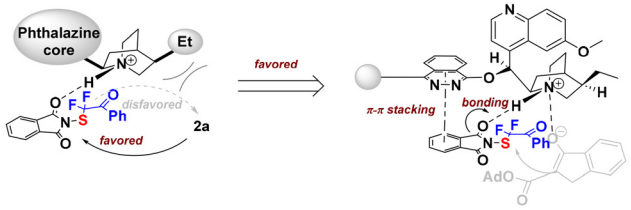
Under the optimized conditions for enantioselective α-difluoromethylketone thiolation, the reaction scope of various indanone-derived β-keto esters was further investigated (Scheme 3). A series of substrates bearing electron-donating and electron-withdrawing substituents on positions 4, 5, and 6 of the aromatic ring, such as halide (**3o–3s**), methyl (**3t**), dimethoxyl (**3u**) groups underwent smooth asymmetric α-difluoromethylketone thiolation. The corresponding products were obtained in moderate yields (55–72%) with excellent enantioselectivities (89–93% ee). Notably, substrates with electron-withdrawing groups exhibited enantioselectivity comparable to those with electron-donating groups. These results underscore the broad applicability of phthN-$SCF_2COAr$ reagents in this transformation. We also investigated the asymmetric α-difluoromethyl thiolation of a benzocyclohexanone-derived adamantyl β-ketoester (**2w**), however, the reaction failed to deliver the desired product. Suspecting that the poor reactivity was due to the substantial steric hindrance imposed by the adamantyl group, we next examined the corresponding methyl β-ketoester (**2v**). The target product **3v** was successfully obtained albeit with the low yield (10%) with limited enantioselectivity (18% ee). To further explore the influence of structural features, we conducted a systematic evaluation of alternative β-ketoester scaffolds, including a cyclohexanone derivative (**2x**), an acyclic analogue (**2y**), a cyclopentanone-derived methyl β-ketoester (**2z**), and a *tert*-butyl β-ketoester (**2aa**). All of these substrates showed negligible conversion under the standard reaction conditions. These findings underscore the pronounced differences in reactivity among various β-ketoester scaffolds and highlight the critical impact of structural factors on reaction efficiency and selectivity.

The scale-up experiment (Scheme 4) was conducted using the catalyst $(DHQD)_2PHAL$, which furnished product **3a** in 56% yield (1.54 g) without erosion enantioselectivity (93% ee). Wittig olefination of compound **3a** afforded alkene **4** in 50% yield with excellent enantiomeric excess (91% ee) (Scheme 4b). Based on previous studies,[18] a plausible transition state was proposed (Scheme 5). Two different types of interactions could occur: (1) An ion pair interaction between the ammonium center and the enolate of the β-keto esters. (2) Hydrogen




**Scheme 4** Scale-up reaction, and synthetic transformation.




Scheme 5 Proposed transition state.

bonding between the PhthN-$SCF_2COAr$ moiety and $(DHQD)_2PHAL$. Within this transition state, only the *Re* face of the enolate was accessible for the reaction.

## Conclusions

In summary, we have developed an enantioselective synthesis of α-difluorothiomethylated ketones from β-keto esters catalyzed by the cinchona alkaloid-based catalyst $(DHQD)_2PHAL$. This catalytic asymmetric method enables the construction of quaternary carbon centers tethered to the $SCF_2COAr$ group. Under mild conditions, substituted indanone derivatives were converted to the corresponding products with moderate to high enantioselectivities (up to 93% ee) and moderate yields. A variety of phthN-$SCF_2COAr$ reagents were demonstrated to act as effective electrophiles. Further investigations on expanding this enantioselective α-difluoromethylketone thiolation strategy to other bioactive substrates are currently in progress.

## Author contributions

W.-L. Y. and Z. L. conceived the projects. W. C., E. W. and J. L. performed the experiments. W. C., E. W. and W.-L. Y. prepared the ESI.† X. S., X. X., W.-L. Y. and Z. L. discussed the results and commented on the manuscript. W. C. and W.-L. Y. co-wrote the manuscript with the feedback of all other authors. All authors approved the final version of the manuscript for submission.

## Data availability

All experimental data and detailed procedures are available in the ESI.†

## Conflicts of interest

There are no conflicts to declare.

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