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Catalytic Enantioselective Vinylogous Allylic Alkylation of Coumarins

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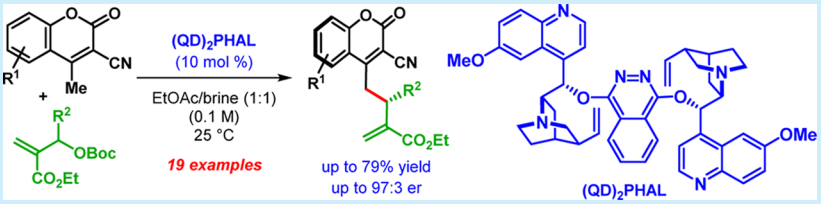
Supporting Information



ABSTRACT: An unprecedented, organocatalytic enantioselective vinylogous γ-allylic alkylation of 4-methylcoumarins has been developed. Using allylic carbonates as the allyl source, this reaction is catalyzed by Lewis basic dimeric *Cinchona* alkaloid $(QD)_2PHAL$ and proceeds exclusively in a γ- and branched-selective manner to produce densely functionalized coumarin derivatives generally in good yields with good to high enantioselectivities (up to 97:3 er).

The principle of vinylogy, as described by Fuson, accounts for the transmission of electronic effect of a given functional group through a conjugated π-system.[1] This concept allows for the formation of bonds away from the parent functional group, a phenomenon often termed as distant or remote functionalization.[2] In spite of its obvious advantages, the scope of vinylogous nucleophilic reactivity has been restricted to a handful of transformations[3] and calls for new development.

Coumarins are a class of structural motif distributed in over 1000 natural products and various biologically active synthetic targets (Figure 1).[4] In addition, the photophysical properties of

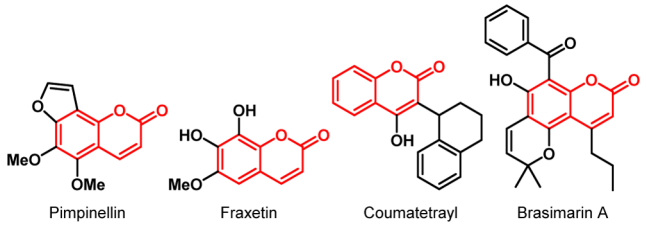


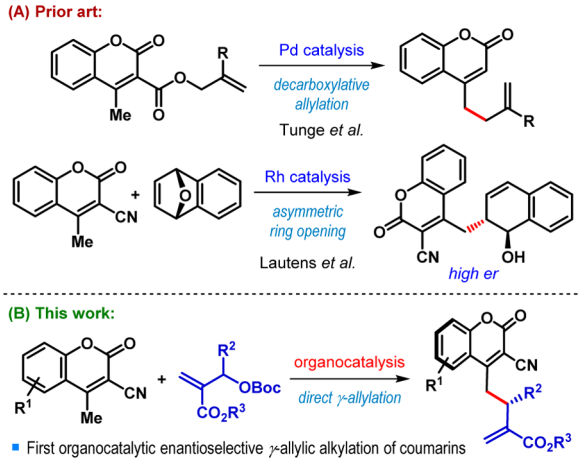
Figure 1. Bioactive compounds and natural products bearing coumarin scaffold.

coumarins make them attractive components in dye industries.[5] The popularity of coumarin-containing compounds as potential drugs as well as polymeric materials has created a demand for their enantioselective synthesis and functionalization.[6] The inherent electron-deficient nature of coumarins presents an opportunity for distant functionalization of appropriately substituted coumarins.

In this context, γ-functionalization of cyanocoumarins has received special attention during the past few years. In 2010, Xie et al. first disclosed the application of 3-cyano-4-methylcoumarins as vinylogous nucleophiles for enantioselective γ-functionalization.[7]

We became interested in advancing the concept of distant functionalization of coumarins to the domain of asymmetric allylic alkylation.[8] Introduction of an allyl group at the γ-position of 4-methylcoumarin was first reported by Tunge et al. in 2011 through a Pd-catalyzed migratory decarboxylative coupling (Scheme 1A).[9] However, enantioselective γ-allylation

Scheme 1. Catalytic Enantioselective Vinylogous Allylic Alkylation of Coumarins



of coumarins proved particularly challenging, possibly because of the difficulty associated with remote regio- and enantiocontrol in the bond-formation step. The only known example of enantioselective γ-allylation of cyanocoumarins is developed by Lautens and co-workers using a Rh-catalyzed ring-opening reaction of oxabicycles (Scheme 1A).[10]

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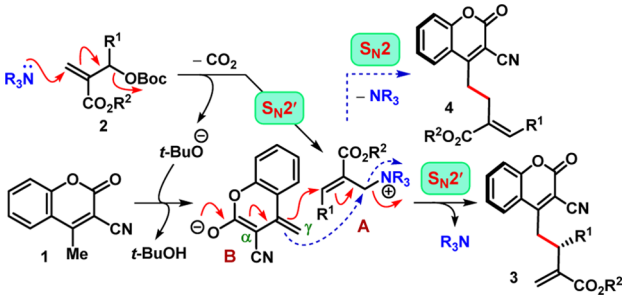
Despite these examples of transition-metal-catalyzed γ-allylic alkylation of coumarins[9−11] and other nucleophiles,[12] an organocatalytic enantioselective γ-allylation of coumarins is yet to be developed.[13] In fact, organocatalytic enantioselective γ-allylic alkylation reactions in general remain relatively unexplored.[14]

Herein, we present the first organocatalytic enantioselective vinylogous γ-allylic alkylation of coumarins.

Our strategy relies upon the use of allylic carbonates (**2**, Scheme 2) derived from Morita−Baylis−Hillman[15] adducts as

Scheme 2. Mechanistic Hypothesis of Catalytic Enantioselective Vinylogous γ-Functionalization Reaction

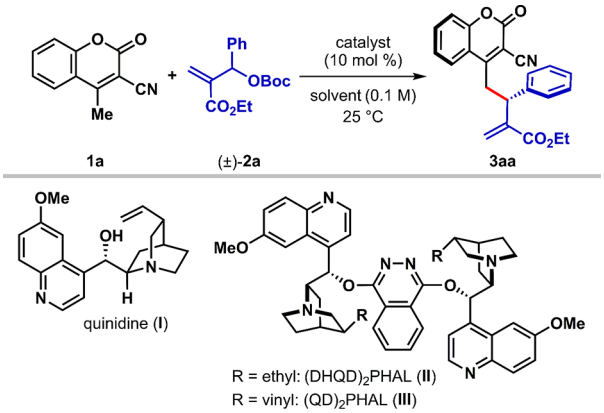


the allyl source.[16] The allylic carbonates of type **2** are known to undergo decarboxylation through S_N2' attack of Lewis basic tertiary amines (NR_3) to generate the active allylating agent **A**.[16] The resulting *tert*-butoxide is then expected to deprotonate cyanocoumarins (**1**) to produce the active vinylogous nucleophile **B**. Addition of **B** to **A** can then take place either in S_N2' fashion to furnish the desired branched γ-allylation product **3** or in S_N2 fashion to generate the linear (achiral) γ-allylation product **4**. Besides, **B** can also react directly through its α-position. Overcoming the divergent regiochemical possibilities on both nucleophile **B** (α- vs γ-) as well as on electrophile **A** (S_N2' vs S_N2) would be crucial to the success of this vinylogous γ-allylic alkylation reaction.

While the attack from nucleophile **B** is likely to favor the less sterically encumbered γ-position, we reasoned that the regioselectivity arising out of the addition to electrophile **A** to be catalyst-controlled.[17] Previous reports from Lu and co-workers[16i] as well as from our group[16c] demonstrated that bifunctional tertiary amino(thio)urea catalysts promote S_N2 addition on **A**. In contrast, S_N2' addition to **A** is facilitated under the influence of dimeric *Cinchona* alkaloids, as described by Chen and co-workers.[16j]

To put our design principles into practice, we initiated our studies with the reaction between cyanocoumarin **1a** and phenyl-substituted allylic carbonate **2a** in CH_2Cl_2 at 25 °C (Table 1). Although no conversion was detected in the presence of 10 mol % of unmodified quinidine (**I**) (entry 2), the related dimeric *Cinchona* alkaloids were indeed found to catalyze this vinylogous allylic alkylation reaction. Thus, with $(DHQD)_2PHAL$ (**II**) as the catalyst, the branched γ-allylated product **3aa** was obtained as the sole regioisomer with promising enantioselectivity (entry 3). As anticipated (vide supra), neither α-addition product nor any linear γ-allylation product (**4** in Scheme 2) could be detected. The use of $(QD)_2PHAL$ (**III**) as the catalyst led to enhanced enantioselectivity at the expense of reaction rate (entry 4). Addition of 4 Å MS improved both the reaction rate as well as the enantioselectivity (entry 5). Raising temperature to 50 °C

Table 1. Evaluation of Catalyst and Reaction Conditions for Vinylogous Allylic Alkylation[a]



entry	cat.	solvent	additive	time[b]	er[c]
1	none	CH_2Cl_2	–	48 h[d]	–
2	**I**	CH_2Cl_2	–	48 h[d]	–
3	**II**	CH_2Cl_2	–	12 h	89:11
4	**III**	CH_2Cl_2	–	72 h	93:7
5	**III**	CH_2Cl_2	4 Å MS[e]	48 h	94:6
6	**III**[f]	CH_2Cl_2	–	18 h	90:10
7	**III**	CH_3CN	4 Å MS[e]	24 h	94:6
8	**III**	EtOAc	4 Å MS[e]	6 d	95.5:4.5
9	**III**	EtOAc/H_2O (1:1)	–	5 d	96:4
10	**III**	EtOAc/brine (1:1)	–	5 d	96.5:3.5

[a]Reactions were performed using 1.0 equiv of **1a** and 1.1 equiv of **2a** on a 0.05 mmol scale. [b]Time required for complete consumption of **1a**. [c]Enantiomeric ratio (er) as determined by HPLC analysis using a column with a chiral stationary phase. [d]No conversion after 48 h. [e]MS = molecular sieves. [f]Reaction performed at 50 °C.

offered noticeable enhancement of the reaction rate but with reduced enantioselectivity (entry 6). A survey of reaction media, at this stage, revealed EtOAc to be the best with respect to enantioselectivity of the reaction, albeit with significantly slow conversion (entry 8). To our surprise, even better er was observed in a heterogeneous mixture (1:1) of EtOAc and water (entry 9). Following the same trend, 1:1 mixture of EtOAc and brine turned out to be the optimum reaction medium, affording the product with 96.5:3.5 er (entry 10). Tweaking various other reaction parameters, including the ester substituent on the allylic carbonate, neither improved the yield nor enantioselectivity.[18] Similarly, the use of allylic acetate instead of allylic carbonate in the presence of stoichiometric amount of an external base (Na_2CO_3) failed to generate the desired product, even after 5 days.

The scope and limitations of this vinylogous allylic alkylation reaction of cyanocoumarins were then evaluated under the optimized catalyst and reaction conditions (Table 1, entry 10). A variety of β-aryl-substituted allylic carbonates with diverse steric and electronic demand on the aryl ring (**2a−n**) were well tolerated, and the desired products (**3aa−an**) were obtained as a single regioisomer in moderate to good yields and with good to high enantioselectivities (Table 2A). Irrespective of the position of the substituents on the aryl ring (*o*-, *m*-, or *p*-), the products were generally formed with similar range of yields and enantioselectivities. The same level of yield and er was also observed for 2-naphthyl-substituted allylic carbonate **2o**. β-Heteroaryl-substituted allyl carbonate could also be used as the substrate as shown for 2-thienyl-substituted allyl carbonate **2p**:

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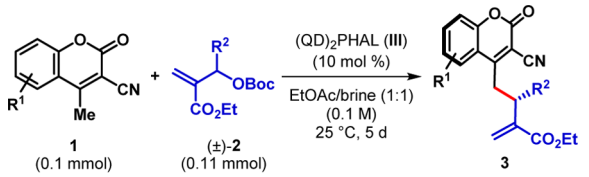
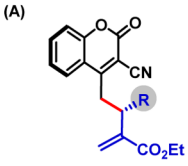
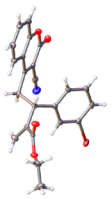
Table 2. Substrate Scope of Vinylogous Allylic Alkylation[a]



(A)

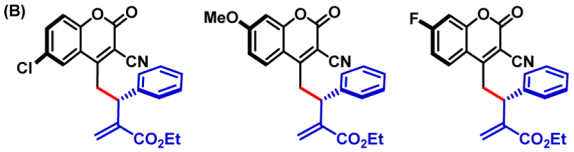


R	yield (%)	er
Ph (**3aa**)	67	96:4
2-MeC$_6$H$_4$ (**3ab**)	62	95:5
2-FC$_6$H$_4$ (**3ac**)	66	95:5
2-ClC$_6$H$_4$ (**3ad**)	66	95:5
2-BrC$_6$H$_4$ (**3ae**)	64	94.5:5.5
3-FC$_6$H$_4$ (**3af**)	64	95:5
3-BrC$_6$H$_4$ (**3ag**)	66	93.5:6.5 (97:3)[b]
4-FC$_6$H$_4$ (**3ah**)	77	94.5:5.5
4-ClC$_6$H$_4$ (**3ai**)	69	95.5:4.5
4-BrC$_6$H$_4$ (**3aj**)	66	97:3
4-MeC$_6$H$_4$ (**3ak**)	72	96:4
4-(*i*-Pr)C$_6$H$_4$ (**3al**)	72	97:3
4-CF$_3$C$_6$H$_4$ (**3am**)	68	95:5
4-OMeC$_6$H$_4$ (**3an**)	74	96.5:3.5
2-naphth (**3ao**)	71	95.5:4.5
2-thienyl (**3ap**)	79	92:8



3ag
(CCDC 1566910)

(B)



3ba
52% yield, 97:3 er

3ca
62% yield, 96:4 er

3da
46% yield, 95:5 er

[a]Yields correspond to the isolated yield. Enantiomeric ratios (er) were determined by HPLC analysis using a chiral stationary phase. [b]Values in the parentheses indicate er of the product after a single recrystallization.

While the resulting product **3ap** was obtained in good yield, enantioselectivity remain rather modest.

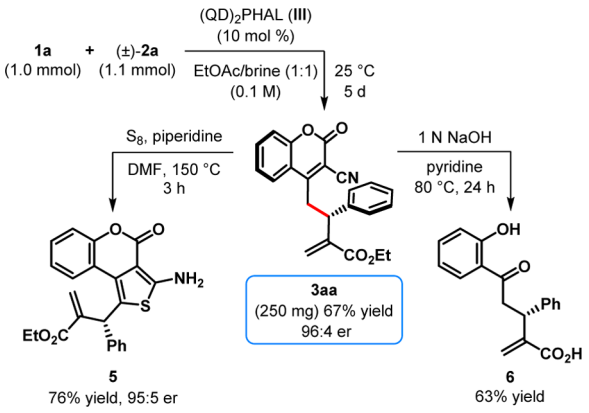
Our protocol was found to be equally efficient for substituted cyanocoumarins (**1b−d**), and the desired products (**3ba−da**) were obtained in moderate yields with good to high enantioselectivities (Table 2B). However, β-alkyl-substituted allylic carbonates were found to be completely unreactive under the standard reaction conditions, which marks a prominent limitation of our protocol.[18] Similarly, removal or replacement of the cyanide group in **1** with ester or amide group led to complete attenuation of its reactivity.[18]

The absolute configuration of **3ag** was established by the X-ray diffraction analysis of its single crystals obtained from petroleum ether/EtOAc mixture and found to be *R* (Table 2A).[19] The absolute configurations of the other allylation products were assigned by analogy as the same.

To confirm the scalability of our protocol, the reaction was performed on a scale 10 times higher than that used for evaluation of substrate scope (Table 2). Thus, a reaction between **1a** and **2a** on a 1.0 mmol scale under otherwise standard reaction conditions furnished the desired product **3aa** in 67% yield with the same level of enantioselectivity (96:4 er) as obtained in the smaller scale reaction (Scheme 3).

The products of this allylic alkylation reaction are densely functionalized and could be converted to useful building blocks. For example, exposure of **3aa** to sulfur under basic conditions[20] led to the formation of a tricyclic aminothiophenocoumarin **5** with 76% yield and 95:5 er (Scheme 3). This structural motif is

Scheme 3. Reaction on a 1.0 mmol Scale and Functionalization of the Product



known as antifungal agent and has also been used as an intermediate in dye industries.[20,21] Base-mediated retro-Knoevenagel reaction followed by hydrolysis resulted in the formation of acyclic compound **6** with 63% yield. Compound **6** may be considered as the α-allylic alkylation product of 2′-hydroxyacetophenone.

In conclusion, a catalytic enantioselective vinylogous allylic alkylation of 4-methylcoumarins has been developed using ester-substituted allylic carbonates as the allyl source. Catalyzed by a dimeric *Cinchona* alkaloid (QD)$_2$PHAL, this reaction led to the γ-allylic alkylation of cyanocoumarins in exclusively branched-selective fashion to furnish densely functionalized products in good yields with good to high enantioselectivities. This report represents the first organocatalytic enantioselective vinylogous γ-allylic alkylation of coumarins. Single-step conversion of the product to useful motifs and building blocks has also been demonstrated.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b02421.

Experimental details, characterization data, and crystallographic data (PDF)
NMR spectra and HPLC data (PDF)
Crystallographic data for **3ag** (CIF)

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Notes

The authors declare no competing financial interest.

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(18) See the Supporting Information for details.
(19) CCDC 1566910 contains the crystallographic data for **3ag**. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.
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