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DMAP Mediated Efficient Construction of Functionalized Chromenes through One-Pot Reaction of *para*-Quinone Methides with Allenoates

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A novel DMAP-mediated Rauhut-Currier/*oxa*-Michael addition cascade reaction of hydroxylphenyl-substituted *para*-quinone methide with allenoate was reported for the first time. A series of functionalized chromenes were successfully obtained with moderate to good yields under this one-pot cascade reaction. The chromene products could be easily transformed into different derivatives and a plausible mechanism was proposed to elucidate this novel reaction.

Introduction

Chromenes and their derivative chromans are important motifs widely existing in numerous natural products as well as pharmaceutically relevant compounds.[1] Several examples of these bioactive molecules are illustrated in Figure 1. For example, ormeloxifene (**A**) is a readily available drug molecule which target estrogen receptor modulator.[2] Crolibulin (**B**) is known as anticancer compound and has remarkable properties as apoptosis inducer for anaplastic thyroid cancer treatment.[3] Compounds **C**–**D** exhibit potential anti-osteoporotic and anti-neoplastic activities.[4] Different strategies aiming at construction of such skeletons have been developed in recent years.[5] Nevertheless, as the increasing requirement of these type of molecules in medicine and pharmacy, a direct and valuable strategy for efficient synthesis of chromene framework, especially for the functionalized ones, is highly demanded.

para-Quinone methides (*p*-QMs) were considered as powerful precursor for construction of 1,1-diarylmethane compounds in recent years.[6] To be noticed, the installation of a suitable nucleophilic site on terminal aryl group could make the *p*-QMs into double functionalization substrates, and a set of seminal works based on these ortho-hydroxyphenyl-substituted *p*-QMs have been presented for the synthesis of chromene related structures with various catalytic strategies.[7] Enders and co-workers reported the first domino reaction of *ortho*-hydroxyphenyl-substituted *p*-QMs with isatin derived alkenes for construction of spiro-chromans.[8] Fan and co-workers realized the synthesis of dihydrocoumarin derivatives *via* phosphine-catalyzed intramolecular Rauhut-Currier reaction.[9] Two more examples for preparation of functional chroman derivatives were achieved with *ortho*-hydroxyphenyl-substituted *p*-QMs and azlactons by phosphoric acid catalysis.[10] Recently, two groups reported cycloaddition of *para*-quinone methide derivatives with activated alkynones or ynals, constructing functionalized chromenes independently (Scheme 1a).[11] Despite the reported impressive achievements, the development of novel strategies for construction of chromene is still challenging and meaningful. It was noticed that allenoates had been applied as useful synthons in catalytic Rauhut-Currier reaction with *p*-QMs enabled by base or Lewis base catalysts, where diarylmethane with allene motifs compounds were synthesized efficiently (Scheme 1b).[12] Encouraged by these pioneer works disclosed by Mohanan's, Chandra's and Tang's group independently, we hypothesis that it might undergoes a cascade reaction to form heterocyclic scaffold with an *orth*-nucleophilic site existence. As a part of our ongoing investigations on the organocatalyzed methodology of *p*-QMs,[13] hereby, we reported a Lewis base serving as effective catalyst in a one-pot domino reaction of the *ortho*-hydroxyphenyl-substituted *p*-QMs with allenoates, combining Rauhut-Currier reaction and hetero-Michael addition together to provide an effective synthetic protocol for the preparation of biologically interesting chromenes (Scheme 1c).

Results and Discussion

Initial examinations use *ortho*-hydroxyphenyl-substituted *p*-QM **1a** and allenoate **2a** as model substrates in the presence of different Lewis base (PPh_3, DABCO and DMAP) to evaluate our hypothesis. Unfortunately, the desired product was not detected under the catalytic reaction conditions (Scheme 2, eq. 1), probably due to the affection of the hydroxyl moiety's acidity. To our delight, when TBS protected compound *p*-QM **1b** applied with DMAP as catalyst in toluene at room temper-

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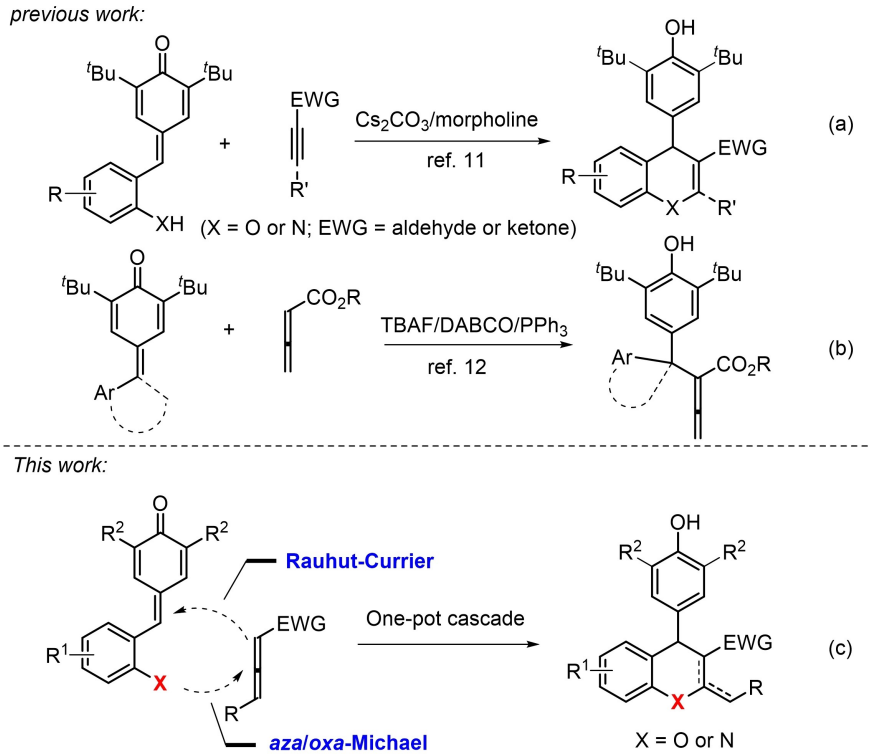


Scheme 1. Reaction design with Allenoate and *p*-QMs.

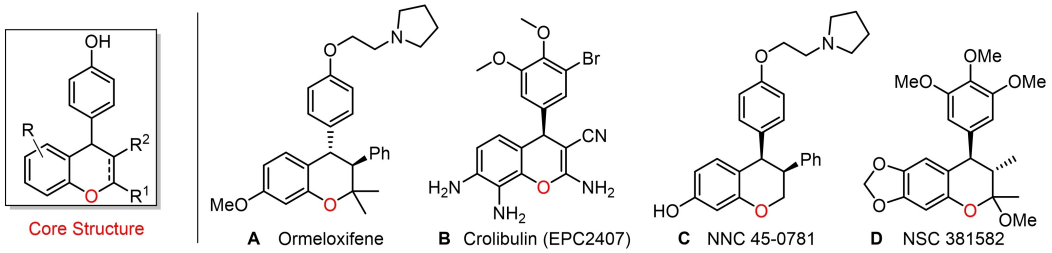
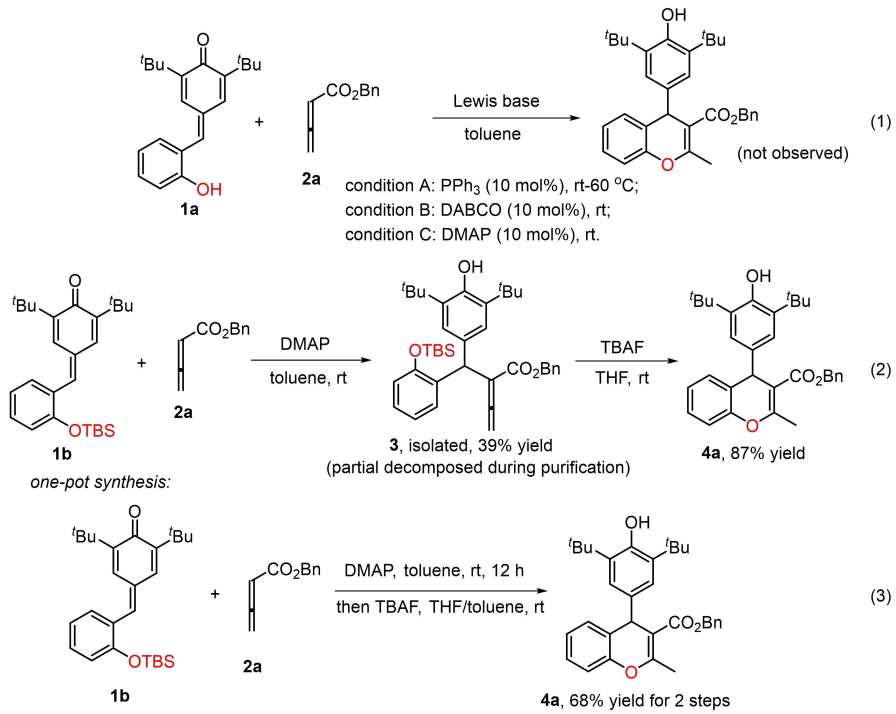


Figure 1. Natural products and pharmaceuticals containing chroman skeleton.

ature, the reaction proceeded well to afford desired product **3** in 39% yield (Scheme 2, eq. 2). A chromene product **4a** was successfully formed after treating with tetra-*n*-butyl aminonium fluoride (TBAF). Realized that compound **3** was not stable enough during the purification, we then tried to explore the reaction in a one-pot procedure without isolating the intermediate product to overcome this shortage. As expected, the yield of product **4a** was enhanced to 68% (Scheme 2, eq. 3).

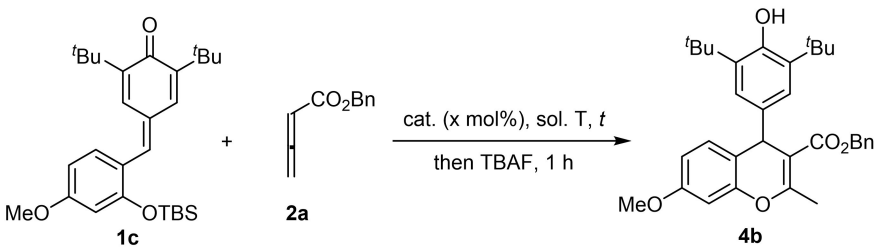
Encouraged by the initial results, we turned our attention to optimize the reaction conditions with *p*-QM **1c** and allenoate **2a** as model substrates, the results are summarized in Table 1. In the presence of different Lewis base catalysts, the corresponding product **4b** was generated in 63% yield under the DMAP catalysis, while the other catalysis options only gave trace product (Table 1, entries 1–3). We further optimized the reaction conditions by screening of different solvents (Table 1, entries 4–10). It was revealed that ethyl ether (Et_2O) was the best solvent in the reaction, giving the desired product **4b** in 74% yield (Table 1, entry 8). The other solvents such as toluene, *o*-xylene, DCM, THF, MTBE, 1,4-dioxane or CH_3CN failed to improve the efficiency (52-69%). A slight decreased yield was observed when using 30 mol% DMAP catalyst, but a 10 mol% catalyst loading in the one-pot reaction is still proceeded good to generate product **4b** in 75% yield (Table 1, entries 11–12). Lowering the reaction temperature to 0 °C, the yield decreased dramatically to 57% (Table 1, entry 13). However, it was also found the efficacy of the reaction could not be improved by further increasing the temperature (Table 1, entry 14). Moreover, the reaction could already proceed smoothly within 2 hours under standard catalytic conditions to generate product **4b** in 75% yield (Table 1, entry 15).

Having the optimal reaction conditions identified, the generality of the two steps in one-pot cascade reaction was examined using various *p*-QMs **1** and allenic esters **2**. The



Scheme 2. Hydroxyl-*p*-QM involved in Rauhut-Currier reactions.

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

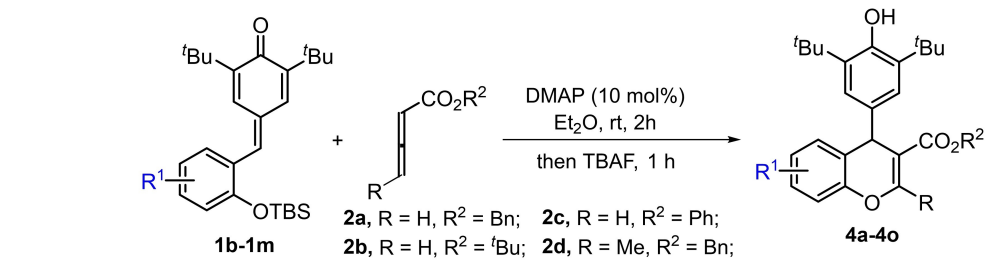


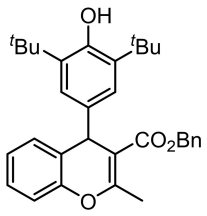
Entry	cat.	x (loading)	Solvent	T [°C]	*t* [h]	Yield [%][b]
1[c]	PPh_3	20	toluene	25	8	trace
2[c]	DABCO	20	toluene	25	8	trace
3	DMAP	20	toluene	25	12	63
4	DMAP	20	*o*-xylene	25	12	52
5	DMAP	20	DCM	25	12	61
6	DMAP	20	THF	25	12	56
7	DMAP	20	MTBE	25	12	62
8	DMAP	20	Et_2O	25	12	74
9	DMAP	20	1,4-dioxane	25	12	64
10	DMAP	20	CH_3CN	25	12	69
11	DMAP	30	Et_2O	25	12	72
12	DMAP	10	Et_2O	25	12	75
13	DMAP	10	Et_2O	0	12	57
14	DMAP	10	Et_2O	40	12	74
15	DMAP	10	Et_2O	25	2	75

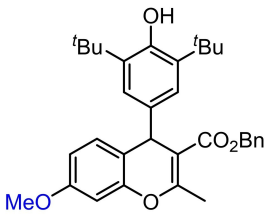
[a] The reactions were performed with **1c** (0.1 mmol), **2a** (0.12 mmol) and catalyst in solvent (1.0 mL); [b] Isolated yield; [c] Decomposed, only trace product.

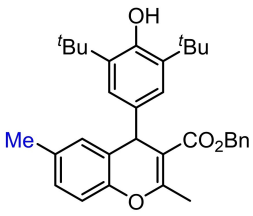
results are summarized in Table 2. All reactions proceeded smoothly to give the corresponding products **4** in moderate to good yields. In details, *p*-QMs **1b**–**1m** bearing electron-neutral (R^1 = H), electron-donating (R^1 = OMe, Me) or electron-withdrawing (R^1 = F, Cl, Br) groups on the aryl ring underwent this domino reaction process to afford chromene products **4a**–**4h**

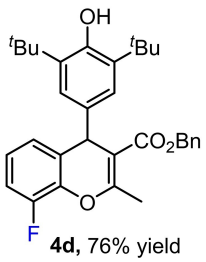
Table 2. Substrate scope of the one-pot reaction.[a]

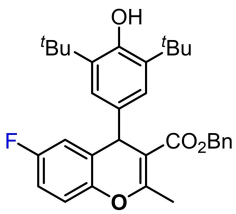


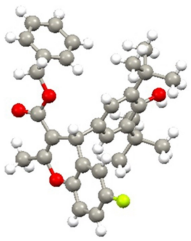

4a, 82% yield


4b, 75% yield


4c, 72% yield


4d, 76% yield


4e, 70% yield


X-ray of **4e**
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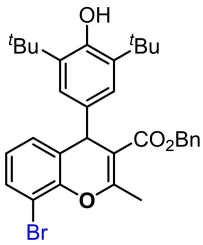
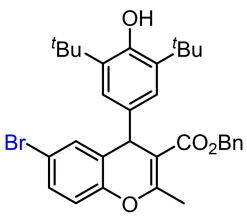
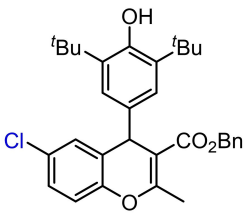
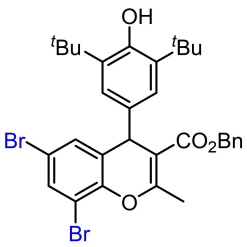
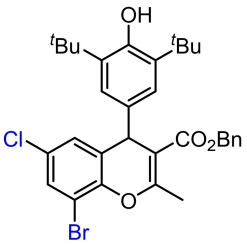
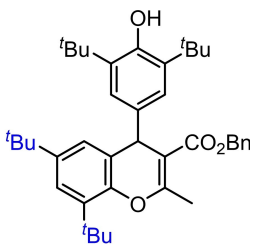

4f, 73% yield

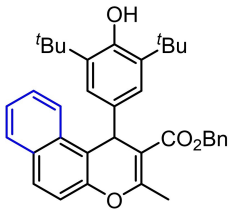

4g, 68% yield

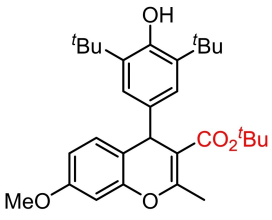

4h, 76% yield

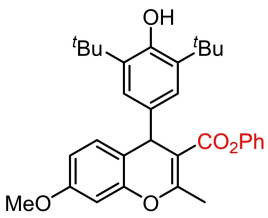

4i, 62% yield

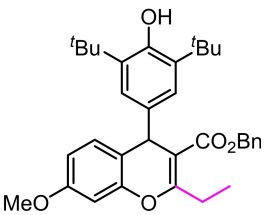

4j, 57% yield


4k, 51% yield


4l, 49% yield


4m, 58% yield


4n, 52% yield


4o, 38% yield

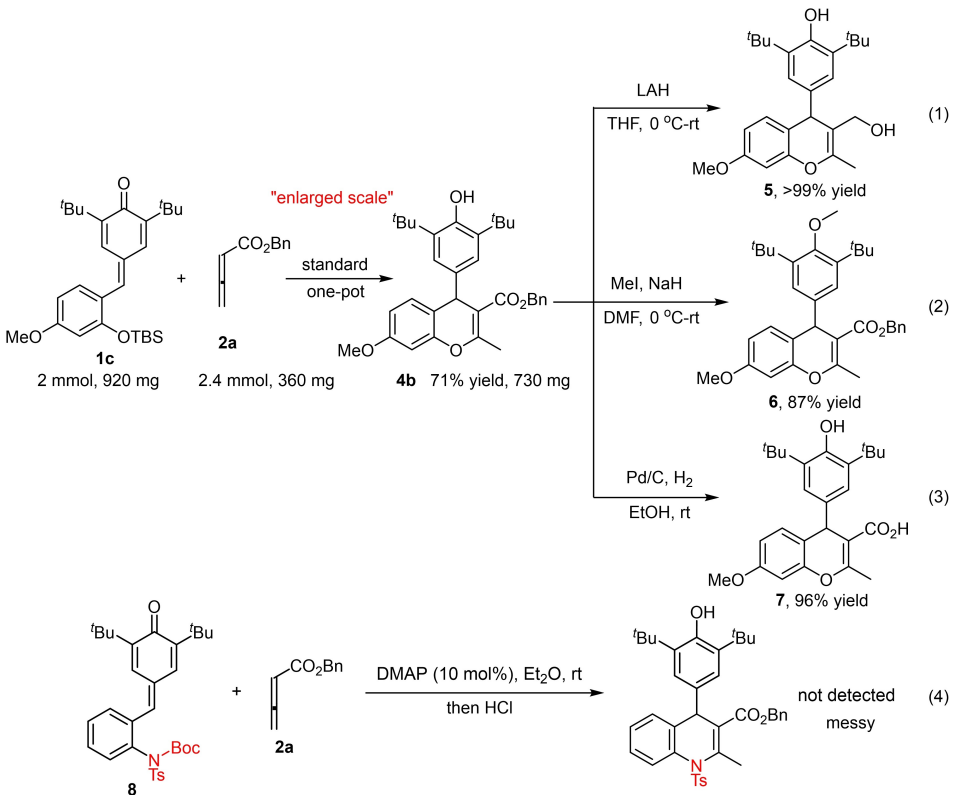
[a] All reaction carried out with **1** (0.1 mmol), **2** (0.12 mmol) and DMAP (0.01 mmol) in Et_2O (1.0 mL) at room temperature for 2 hours; then TBAF (1 M in THF, 0.1 mmol) for 1 hour.

in 68–82% yield. The structure of **4e** has been determined by X-ray diffraction and the ORTEP drawing is shown in Table 2.[14] Multi-substituted groups (even bulky group di-^{t}Bu$_2$) were tolerated in the reaction, the corresponding products **4i**–**4k** were produced in slightly decreased yields (51–62%). To be noticed, steric hindrance naphthyl is also suitable for the reaction, where product **4l** was obtained in 49% yield correspondingly. Moderate yields (52–58%) were obtained with tert-butyl buta-2,3-dienoate **2b** or phenyl buta-2,3-dienoate **2c** as substrates. Moreover, γ-substituted allenoate (**2d**) was also compatible in the reaction to generate the corresponding **4o** in 38% yield, the low efficiency is probably due to the proton shift from terminal methyl group of allenoate **2d**.[15]

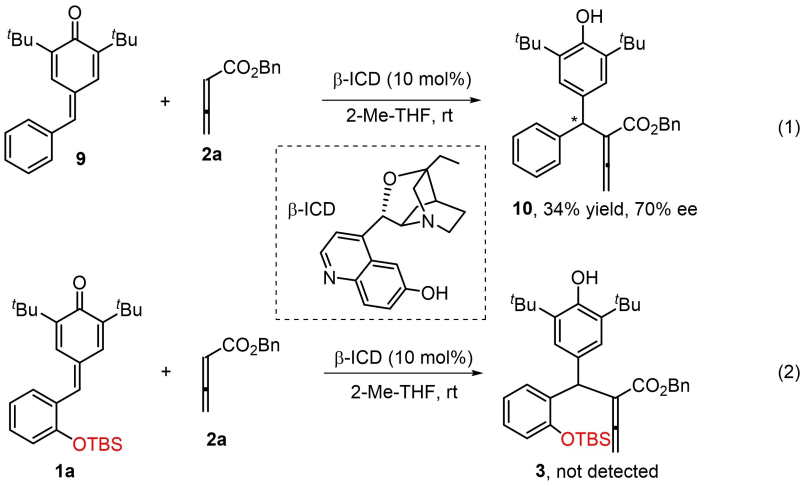
To evaluate the robustness and practical utility of this one-pot cascade reaction, an enlarge reaction of **1c** with **2a** was run under the optimal reaction conditions. As shown in Scheme 3, the desired chromene product **4b** was obtained in 71% yield. Moreover, several transformations and applications of **4b** have been explored to show the utility of this methodology (Scheme 3). The ester group was easily transfer to hydroxymethyl product **5** in high yield with the chromene skeleton maintenance. The hydroxyl of phenol was easily protected by methyl group to give product **6** in 87% yield under mild conditions. Moreover, the product **4b** could be transferred to a chromene acid **7** in 96% yield *via* heterogeneous Pd/C hydrogenation (Scheme 3, eq. 1–3). An amino-substituted *p*-QM **8** was also investigated in the reaction, but the reaction unfortunately became decomposed and no correspondent dihydroquinone product was detected (Scheme 3, eq. 4).

At last, we turn our attention to explore the enantioselective control of this Lewis base-catalyzed reaction (Scheme 4). We first applied *p*-QM **9** and allenoate **2a** as model substrates to investigate the Rauhut-Currier reaction stereoselectivity control. After screening several different chiral phosphines and chiral tertiary amines which were widely used in asymmetric synthesis of allenoates transformation (see SI for details), it was found that cinchonine derived β-ICD[16] was the most efficient catalyst in the reaction, product **10** was obtained in 34% yield along with 70% ee (Scheme 4, eq. 1). Then, we tried to complete the one-pot chromene construction with β-ICD as catalyst. Unfortunately, product **3** was not detected under the optimal catalytic conditions because of the steric affection of substrate **1a** (Scheme 4, eq. 2).

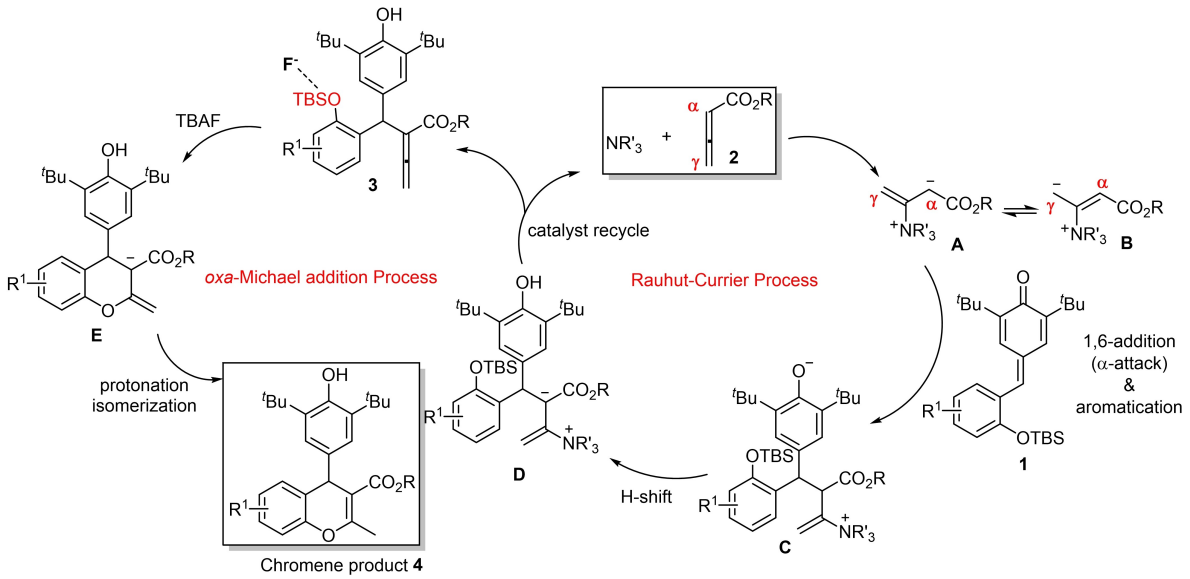
A plausible mechanism was illustrated on basis of our experiments and previous reports (Scheme 5).[17] The reaction was initiated by the nucleophilic attack of DMAP at the β-carbon of allenoate **2** to generate the zwitterionic intermediate **A**, which coexist with intermediate **B** via allyl equilibrium. An α-attack of **A** to substrate **1** followed by aromatication generate **C**. Sequentially, a proton shift and regenerated tertiary amine catalyst to give the product **3**. Then the de-protection of sillyl group by TBAF and the in-situ generated nucleophilic species undergo *oxa*-Michael addition to form intermediate **E**. Finally, intermediate **E** go through protonation and double bond isomerization to produce the desired chromene product **4**.



Scheme 3. Scale-up and transformation experiments.



Scheme 4. Asymmetric allenylation.



Scheme 5. Proposed mechanism.

Conclusion

In summary, we have established a novel and efficient method to access functionalized chromene by using hydroxyl *p*-QMs and allenoates with moderate to good yields (up to 82%) in mild conditions. Different substituted groups were tolerated in this methodology and a set of transformations were furnished to exhibit the potential utility. A Rauhut-Currier and *oxa*-Michael cascade progress enabled by DMAP and TBAF organocatalysts in one-pot progress was proposed as the mechanism of the reaction. The methodology provides a new route to the construction of chromene related compounds. Further applications of this method for biologically active compounds and its asymmetric synthesis are ongoing in our lab.

Experimental Section

General Procedure: *p*-QMs **1** (1.0 eq.), DMAP (0.1 eq.) and anhydrous Et_2O (0.1 M) were added to an oven dried Schlenk tube, then allenotes **2** (1.2 eq.) was added and the reaction mixture were stirred at room temperature for 2 hours. Then, TBAF (1.0 M in THF, 1.0 equiv) were added directly in one-pot and the mixture were stirred for another 1 h. Then water (2 mL) was added to the reaction mixture and extracted with ethyl ether (3 mL*2). The organic layer was dried over with anhydrous Na_2SO_4 and filtered. The solvent was removed under reduced pressure and the residue were purified by column chromatography on silica gel (PE:EA = 20:1, R_f = 0.3–0.5), affording the corresponding products **4**.

Benzyl4-(3,5-di-*tert*-butyl-4-hydroxyphenyl)-2-methyl-4*H*-chromene-3-carboxylate (4a). Compound **4a** (33 mg, 68% yield) was obtained as a white solid following the general procedure from **1b** (0.1 mmol, 43 mg) and **2a** (0.12 mmol, 21 mg). R_f = 0.5 (PE:EA = 20:1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30–7.27 (m, 3H), 7.14–7.12 (m,

4H), 7.02–7.00 (m, 2H), 6.98 (s, 2H), 5.12 (d, $J=1.2$ Hz, 2H), 5.03 (s, 1H), 4.98 (s, 1H), 2.51 (s, 3H), 1.34 (s, 18H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 167.1, 161.0, 152.3, 149.5, 137.2, 136.4, 135.5, 129.0, 128.4, 127.7, 127.4, 127.2, 125.6, 124.5, 124.0, 116.0, 106.3, 65.8, 41.0, 34.2, 30.2, 19.5; **IR** ν 3635, 2958, 1712, 1643, 1596, 1493, 1209, 1062, 821, 696 cm^{-1}; **m.p.** 148–149 °C; **HRMS** calcd. for $C_{32}H_{36}O_4Na^+$ $[M+Na]^+$: 507.2511, found: 507.2506.

Supporting Information (see footnote on the first page of this article): Spectroscopic data of the compounds shown in Tables and Schemes, the detailed descriptions of experimental procedures and the crystal structure of **4e** (CCDC 2044635).

Acknowledgements

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Conflict of Interest

The authors declare no conflict of interest.

Keywords: Allenaotes · Chromenes · One-pot reaction · para-Quinone methides · Tertiary amine

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