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Asymmetric Synthesis of 4-Amino-4*H*-Chromenes by Organocatalytic Oxa-Michael/Aza-Baylis–Hillman Tandem Reactions

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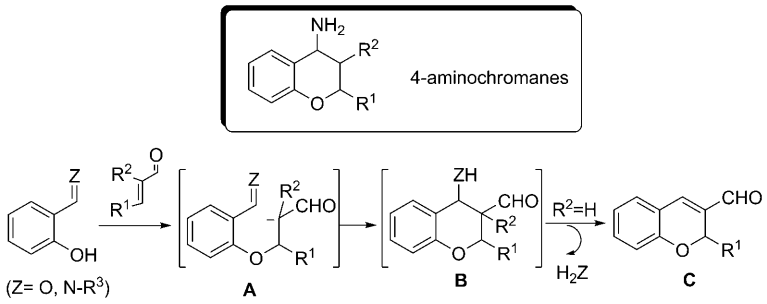
4-Aminochromanes (Scheme 1) are a class of structures that are integrated in hundreds of natural and bioactive compounds. Their importance is reflected in the existence of dozens of patents related to 4-aminochromanes bearing different aromatic rings at the NH_2 group.[1] Among these compounds, those with a CH_2OH group at C-3 (3-hydroxymethyl-4-aminochromanes)[2] exhibit interesting biological properties (e.g., antiobiotic),[2d] and take part in the tetrahydrochromanoquinolines core,[3] whereas 1,4*a*-5,10*b*-tetrahydro-4*H*-chromen[4,3b]pyridines are considered as aza analogs of Δ^1-transtetrahydrocannabinols.[3h–i] Moreover, some attractive alkaloids (e.g., martinelli[4]) with the 4-aminochromane structure have been described. Finally, the 4-aminochromanol moiety is also important and dozens of medicinal studies as well as the synthesis of related products have been reported.[5]



Scheme 1. Approach to the synthesis of 4-aminochromenes.

The most direct organocatalytic asymmetric method for obtaining these skeletons would involve the oxa-Michael reaction of α,β-unsaturated aldehydes to salicylaldehyde[6] or salicylimines,[7] followed by aldolic (or Mannich, $Z=NR^3$) reaction of the resulting intermediate **A** (Scheme 1). However, when R^2 is hydrogen, the intermediate **B** cannot be isolated, because H_2Z ($Z=O$, NR) is quickly eliminated, affording 2*H*-chromenes **C**,[7a–d] due to the large acidity of such hydrogen. When R^2 is not hydrogen, no reaction takes place, because the reactivity of **A** is strongly decreased.[8] At this point we reasoned that reactions of *N*-activated 2-hydroxy benzaldimines with alkynals would afford 4-amino-4*H*-chromenes **E** (Scheme 2c), which could retain the stereochemical information associated to the nitrogen function and could be used as precursors of **B** by reduction.[9] To the best of our knowledge, this transformation involving an oxa-Michael/aza-Baylis–Hillman (aza-BH)[10] tandem process[11] with alkynals has never been reported and prompted us to study it.

The classic aza-BH reaction[10a] consists of the reaction of a nucleophile (usually a catalyst) with a deactivated double bond and further addition of the resulting α-stabilized carbanion to a C=N bond, being finally the catalyst recovered after elimination (Scheme 2a). The use of deactivated triple bonds as electrophiles in aza-BH determines that the final elimination cannot take place (Scheme 2b). These reactions have hardly been explored,[12] which increased our interest for studying them. Since natural 4-aminochromanes are optically pure, we decided to study the oxa-Michael/aza-BH tandem reaction by using activation with silyl prolinol ethers

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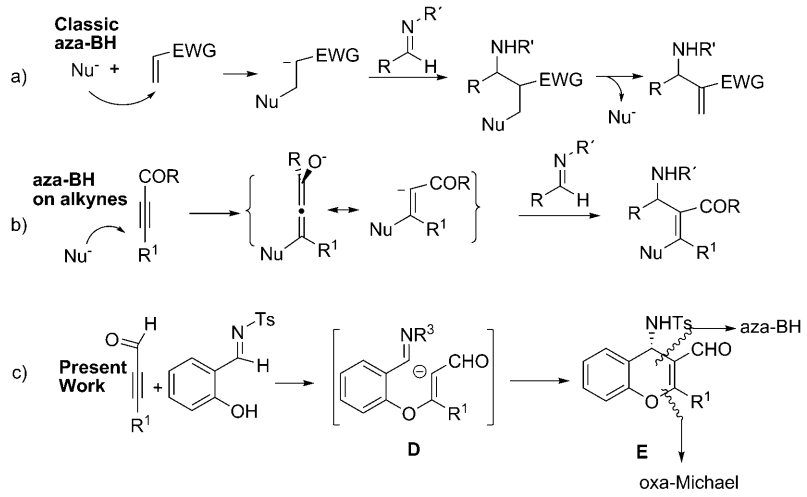


Scheme 2. Different approaches for the aza-BH reaction.

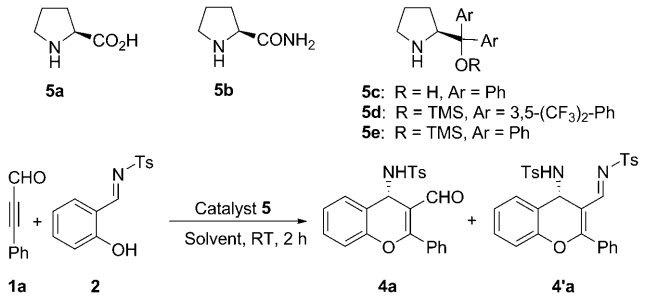
in an asymmetric version,[13] taking advantage of the efficient activation of alkynals[14] in their reactions with nitroalkenes has been recently reported.[14b]

In this work we present the first highly enantioselective organocatalytic[15] oxa-Michael/aza-BH tandem reaction between 2-alkynals and salicyl *N*-tosylimine, leading to optically active 4-amino-4*H*-chromenes by iminium activation (Scheme 2c).

The screening of salicyl *N*-tosylimine **2** with alkynal **1a** was used as the model reaction. Different secondary amines were used as catalyst and all reactions were stopped after 2 h (see Table 1). With proline **5a** and prolinol **5c**, none or very low conversion was observed (entries 1 and 3). In the rest of the cases, the reaction provided mixtures of two compounds, the expected 4-amino-4*H*-chromene **4a** and the **4′a**. With prolinamide **5b** full conversion was achieved in less than 2 h, but a 1:1 mixture of both compounds (**4a** and **4′a**) was obtained with rather low stereoselectivity (33% *ee* for **4a**, entry 2).[16] Better enantiomeric excesses were obtained with protected silyl prolinol ethers **5d** (95% conversion) and **5e** (77% conversion), but a significant amount of **4′a** was also formed (entries 4 and 5). The amount of the byproduct **4′a** could not be reduced by dilution in toluene or CH_2Cl_2 (entries 6 and 7), but it was increased when the ratio **1a/2** become smaller (entry 8). To our delight, the use of a 1.5:1 ratio of **1a/2** (entry 9), afforded a 80:20 mixture of **4a** and **4′a** and the result was even better by using an 2:1 ratio of **1a/2** (entry 10). Under these last conditions, we observed full conversion after two hours into a 95:5 mixture of **4a** and **4′a**, isolating **4a** with 80% yield. Both compounds **4a** and **4′a** were obtained with identical *ee* (entry 10). All mentioned reactions were performed by using 20 mol% of the catalysts. We studied the influence of the catalytic loading. Thus, good results were also obtained with 10 mol% (99% *ee* and 58% yield, entry 11) with only traces of **4′a** and also with 5 mol% of the catalyst (97% *ee*, 60% yield, entry 12); however, under these conditions the reaction time was slightly longer (8 h). The use of only 1 mol% of **5d** was not enough to activate the alkynal and no reaction was observed (entry 13).

In order to check the scope of the reaction, we explored reactions of different aryl (**1a–g**), alkyl (**1h**) and alkenyl (**1i**) alkynals with **2** under the previously optimized conditions (entries 10–12, Table 1). Results are summarized in Table 2. Most reactions were completed in less than 2 h (8 h when the catalytic loading was 5 mol%, entries 1 and 2). All reactions were performed on a 0.2 mmol scale in 0.2 mL of toluene, except for entry 3 that was carried out in 2.0 mmol scale. Upon scaling up the reaction, **4a** was obtained in 76% isolated yield without decreasing the optical purity (98% *ee*). The incorporation of electron-donating groups (*p*-Me, *o*-MeO and *p*-MeO) at the alkynal's aromatic ring did not affect the stereoselectivity with *ee*'s ranging between 94 to

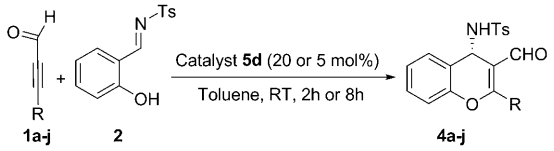
Table 1. Optimization of the reaction of aldehyde (**1a**) with salicyl *N*-tosylimine **2**.[a]



	Catalyst (mol%)	Solvent	**1a/2**	Conversion [%][c]	**4a/4′a**	*ee* [%][d]
1	**5a** (20)	toluene	1:1	nr	–	–
2	**5b** (20)	toluene	1:1	>98	50:50	33
3	**5c** (20)	toluene	1:1	nr	–	–
4	**5d** (20)	toluene	1:1	95	55:45	96
5	**5e** (20)	toluene	1:1	77	41:59	98
6	**5d** (20)	toluene[d]	1:1	85	40:60	nd
7	**5d** (20)	CH_2Cl_2[d]	1:1	60	70:30	96
8	**5d** (20)	toluene	1:1.5	>98	50:50	94
9	**5d** (20)	toluene	1.5:1	>98	80:20	96
10	**5d** (20)	toluene	2:1	>98 (80)[e]	95:5	98 (98)[f]
11	**5d** (10)	toluene	2:1	>98 (58)[e]	>98:2	99
12	**5d** (5)	toluene[g]	2:1	>98 (60)[e]	93:7	97
13	**5d** (1)	toluene[g]	2:1	nr	–	–

[a] All reactions were performed on a 0.2 mmol scale in 0.2 mL of solvent and stopped after 2 h. [b] Conversion was determined by ^{1}H NMR spectroscopy. [c] Enantiomeric ratio was determined by chiral HPLC; nr = no reaction. [d] Diluted up to 0.1 M. [e] Isolated yield after flash chromatography. [f] Enantiomeric ratio of the byproduct **4′a**. [g] These reactions were stopped after 8 h.

Table 2. Scope results for the reaction of salicyl *N*-tosylimine (**2**) with aldehydes **1a–j**.[a]



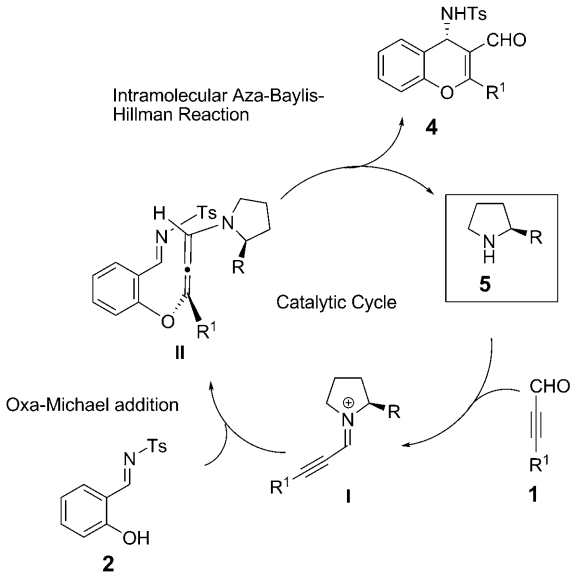
	R	Mol % catalyst	Product	Yield [%]	*ee* [%][b]
1	Ph (**1a**)	20	**4a**	80	98
2	Ph (**1a**)	5	**4a**	60	98
3	Ph (**1a**)	5	**4a**	76[c]	99
4	*p*-Me-C_6H_4 (**1b**)	20	**4b**	97	96
5	*o*-MeO-C_6H_4 (**1c**)	20	**4c**	80	96
6	*o*-MeO-C_6H_4 (**1c**)	5	**4c**	82	98
7	*p*-MeO-C_6H_4 (**1d**)	20	**4d**	94	94
8	3-5-CF_3-C_6H_4 (**1e**)	40	**4e**	55[d]	99
9	*t*Bu-C_6H_4 (**1f**)	20	**4f**	70	99
10	*t*Bu-C_6H_4 (**1f**)	10	**4f**	67	98
11	*p*-(C_5H_{11})-C_6H_4 (**1g**)	20	**4g**	68	98
12	*p*-(C_5H_{11})-C_6H_4 (**1g**)	5	**4g**	84[d]	96
13	*n*-pent (**1h**)	20	**4h**	79	98
14	1-cyclohexenyl (**1i**)	20	**4i**	77	99
15	1-cyclohexenyl (**1i**)	5	**4i**	78	99
16	TMS (**1j**)	20	**4j**	nr[e]	–

[a] All reactions were performed with salicyl *N*-tosylimine (0.2 mmol), alkyne (0.4 mmol), and the indicated amount of catalyst in solvent (0.2 mL) and stopped after 2 h. [b] Determined by chiral HPLC (see Supporting Information). [c] This reaction was carried out in a 2.0 mmol scale. [d] This reaction was stopped after 20 h. [e] No reaction.

98 % (Table 2, entries 4–7). No erosion of yield or stereoselectivity was observed by decreasing the catalytic loading to 5 mol % (entry 6). Interestingly, compound **1e**, bearing an electron-poor aromatic ring, also evolved with good enantioselectivity. However, the reactivity was substantially decreased and was necessary 40 mol % of the catalyst and 20 h for the consumption of the starting material (entry 8). Other alkyl groups at *para*-position, such as *n*-Pent and *t*Bu also produced excellent *ee*'s with both 20 mol % and 5 mol % of catalyst (entries 9–12). Finally, reactions of alkynals bearing alkyl or alkenyl chains, instead of aryl ones, produced good stereoselectivity and isolated yields (entries 13–15). Interestingly the reaction with the alkynal **1j** did not work at standard conditions (entry 16). The structure of **4a** and absolute configuration of compound **4′a** were unequivocally established by X-ray analysis (see the Supporting Information for more details).[17]

A plausible catalytic cycle for explaining the course of these reactions is depicted at Scheme 3. First, the catalyst **5** activates the alkynal **1**, forming an iminium intermediate **I**, that undergoes the oxa-Michael addition with the salicyl *N*-tosylimine **2**. Resulting alenamine intermediate **II**[18] reacts with the imine in an intramolecular fashion leading compounds **4**. The catalyst **5** is recovered in this last step.

In conclusion, herein we present the first highly enantioselective oxa-Michael/aza-Baylis–Hillman tandem reaction between 2-alkynals and tosylimines leading to optically active 4-amino-4*H*-chromenes. This reaction takes place in



Scheme 3. Proposed mechanism for the oxa-Michael/Baylis–Hillman tandem reaction.

less than 2 h with high yields and excellent enantioselectivities. The catalytic loading could be reduced to 5 mol % with slight increase in reaction times.

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Keywords: asymmetric synthesis · aza-Baylis–Hillman · diarylprolinol ethers · domino reactions · organocatalysis

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[17] CCDC-766985 (**4′a**) and 766986 (**4a**) contains the supplementary crystallographic data (see the Supporting Information for more details). These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

[18] The allenamine intermediate **II** has been previously proposed by Wang et al, see reference [14b].

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