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Letter

$Ce(OTf)_3$/PyBox Catalyzed Enantioselective Hosomi-Sakurai Reactions of Aldehydes with Allyltrimethylsilane

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An efficient enantioselective Hosomi-Sakurai reactions catalyzed by chiral $Ce(OTf)_3$/PyBox complex have been explored. In the presence of 20 mol% of chiral catalytic complex prepared in situ from $Ce(OTf)_3$ and 2,6-bis[(S)-4-isopropyloxazolin-2-yl]pyridine {(*S*)-*i*-Pr-PyBox} or tetraphenyl substituted (*S*)-*i*-PrPyBox, the enantioselective allylation of aldehydes with allyltrimethylsilane proceeded smoothly at room temperature and provided the homoallylic alcohols in good yield (up to 95%) and excellent enantioselectivities (up to 99%).

Enantioselective allylation of carbonyl compounds are powerful and important processes for giving optically active homoallylic alcohols. Synthetic interests in homoallylic alcohols and analogues have increased very much over the past decade due to the notable biological activity and occurrence of this key structure in natural products and pharmaceutical lead compounds;[1] in addition, the alkene provides a functional handle for further transformations. Efficient and enantioselective methods to construct such functional group is strongly desired. Allylation reactions of allylsilanes, known as the Hosomi-Sakurai reaction[2] and as a useful method for carbon-carbon bond formation, provide an alternative and attractive way to synthesize homoallylic compounds instead of allylstannanes,[3] because allylsilanes are readily available, low toxicity and versatile reagents for organic synthesis. Therefore, much attention has been focused on the development of asymmetric Hosomi-Sakurai reaction in allylation transfers. Although valuable methods have been reported,[4] catalytic modification of the reaction is still a challenging target, especially for the exploitation chiral Lewis acid catalyzed addition of the less nucleophilic allyltrimethylsilanes to carbonyl functionality. The catalyst hardly regenerated from the produced homoallylic metaloxide will bring about the reaction sluggish or inferior stereoselectivity.

In recent years, metal complexes as efficient catalyst have gained widespread application in many important enantioselective organic synthetic transformations.[5] Chiral 2,6-bis(oxazolin-2-yl)pyridine (PyBox) metal complexes, in which tridentate pyridine bisoxazoline as ligands in combination with various Lewis acid metal (such as Ru, Sc, In, Cu, etc.) salts, have gained much attention. So far, chiral PyBox metal complexes have been used in catalysis of Diels-Alder reaction,[6] cyclopropanations,[7] Nitro-Michael addition,[8] Friedel-Crafts reaction,[9] cross-coupling reactions,[10] etherification,[11] hydrogenation,[12] allylation of carbonyl and imine groups with allylstannanes,[13] and so on. Further exploration of PyBox metal complexes and application of them in catalysis of organic reactions to afford high asymmetric induction is still a temptation in asymmetric field. Despite the development of catalytic asymmetric Hosomi-Sakurai reactions, less attention has been focused on cerium salts and their catalytic enantioselective reactivities are rarely explored.[14] Herein, we report allylation of aldehydes with allyltrimethylsilane based on Hosomi-Sakurai reaction. In the presence of $Ce(OTf)_3$/PyBox, TMSCl and molecular sieves (MS, 4 Å), the reaction proceeded smoothly and afforded the corresponding functionalized alcohols in good to excellent yields and enantiomeric excess (ee).

Our initial experiments were carried out in the reaction of benzaldehyde (**1a**) with allyltrimethylsilane at room temperature in the presence of a set of metal triflates with PyBox ligands (Figure 1) and molecular sieves (MS, 4 Å) in CH_2Cl_2, and the results are summarized in Table 1. For initial exploration, the effects of Lewis acids (20 mol%) were surveyed/screened. $In(OTf)_3$, $Cu(OTf)_2$, $Bi(OTf)_3$, $Ce(OTf)_3$ and $Zn(OTf)_2$ were screened respectively (entries 2-6). Among of them, $Ce(OTf)_3$ was found to be the most effective metal triflate under the same reaction conditions and application of the $Ce(OTf)_3$/PyBox-1 thereof got the product **2a** in 96% ee (entry 5). $Bi(OTf)_3$/PyBox-1 provided the racemic product **2a**, although in very high yield (entry 4). Furthermore, neither the ligand PyBox-1 in the absence of metal triflate nor the $Zn(OTf)_2$/PyBox-1 can catalyze the title reaction (entries 1 and 6). PyBox ligands with different structures were then screened in combination with $Ce(OTf)_3$ (entries 7-9). Interestingly, the reaction also proceeded smoothly by employing PyBox-3 as ligand and obtained the product **2a** with superior levels of asymmetric induction (>99%ee, entry 8). Both of

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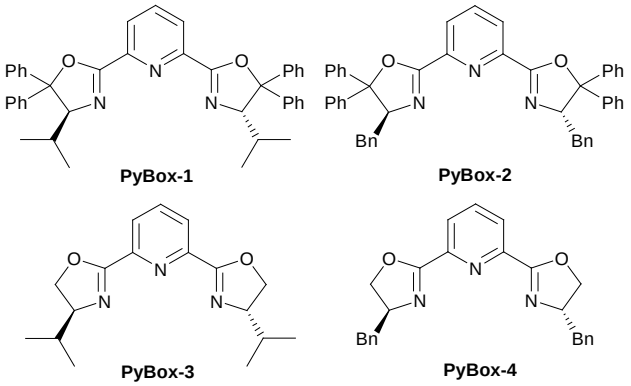
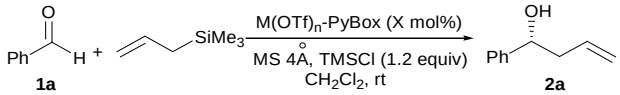


Figure 1 PyBox ligands tested in this study

Table 1. Optimization of $M(OTf)_n$/PyBox Catalyzed Hosomi-Sakurai Reaction[a]



Entry	$M(OTf)_n$	L (mol%)	Yield (%)[b]	ee (%)[c]
1	–	PyBox-1 (20)	–	n.d
2	$In(OTf)_3$	PyBox-1 (20)	87	78
3	$Cu(OTf)_2$	PyBox-1 (20)	72	5
4	$Bi(OTf)_3$	PyBox-1 (20)	90	<1
5	$Ce(OTf)_3$	PyBox-1 (20)	89	96
6	$Zn(OTf)_2$	PyBox-1 (20)	trace	–
7	$Ce(OTf)_3$	PyBox-2 (20)	89	72
8	$Ce(OTf)_3$	PyBox-3 (20)	89	>99
9	$Ce(OTf)_3$	PyBox-4 (20)	92	<1
10	$Ce(OTf)_3$	PyBox-1 (10)	76	92
11	$Ce(OTf)_3$	PyBox-1 (30)	91	94
12[d]	$Ce(OTf)_3$	PyBox-1 (20)	87	>99
13	$Ce(OTf)_3$	PyBox-3 (10)	trace	n.d
14	$Ce(OTf)_3$	PyBox-3 (30)	86	54
15[d]	$Ce(OTf)_3$	PyBox-3 (20)	81	42

[a] Unless otherwise specified, the reactions were performed with aldehyde (0.1 mmol), TMSCl (0.12 mmol) and allyltrimethylsilane (0.15 mmol), using metal triflates (19 mol%) and PyBox (20 mol%) in the presence of activated MS 4Å (80 mg) in CH_2Cl_2 (1 mL) with the reaction time of 30 h. [b] Isolated yield. [c] Determined by HPLC analysis with chiral OD-H. [d] allyltrimethylsilane (0.2 mmol).

Pybox-1 and Pybox-3 were selected to study the effect of catalyst amount on the allylation enantioselectivity (Table 1, entries 10, 11, 13 and 14), it can be clearly seen that the less or more amount of PyBox-3 (10 mol% or 30 mol%) led to lower enantioselectivity (Table 1, entries 13 and 14). On the other hand, more stable enantioselectivity was observed when PyBox-1 was utilized in the catalytic allylation, 92% ee and 94% ee were obtained when 10 mol% and 30 mol% amount of catalyst were used respectively (Table 1, entries 10 and 11). The experimental results show the substituents on the oxazoline had indeed influence on their catalytic activity, the PyBox-2 and PyBox-4 deactivated the Hosomi-Sakurai reaction. The larger electrostatic and steric stabilization of PyBox will promote the homoallylic product with good enantiomeric excesses. The enantioselectivity of the product can also be reached more than 99% ee and 92% yield when 2.0 equiv of allyltrimethylsilane and ligand PyBox-1 were employed (Table 1, entry 12). As illustrated, PyBox-1 orPyBox-3 can catalyze the reaction in the presence of TMSCl. On the other hand, without TMSCl, the reaction either did not occur or only gave **2a** in lower yields. These observations suggest that the oxophilicity, i.e., the Lewis acidity of the cerium-PyBox promoted by TMSCl,[15] may play an important role in the Hosomi-Sakurai reaction. Next, a survey of appropriate solvents[16] for this reaction was undertaken in the presence of 20 mol % of PyBox-1 or PyBox-3. The use of solvents other than CH_2Cl_2 or $CHCl_3$ led to either decline in enantioselectivity (1,2-dichloroethane, 95% ee) or almost no reaction (Toluene or THF). The $Ce(OTf)_3$/PyBox-3 in $CHCl_3$ also provide the corresponding homoallylic alcohol with excellent enantioselectivity (>99% ee) and a pretty yield (87%). Remarkably, both CH_2Cl_2 and $CHCl_3$ proved to be the solvent of choice. $CHCl_3$ also as the selection in the catalytic Hosomi-Sakurai reaction. Finally, the amount of molecular sieves was also tested, the load of molecular sieves is 0.8 g/mmol **1a**.

With the optimal conditions established, different aldehydes on the applicability of the reaction were studied under the action of $Ce(OTf)_3$/PyBox-1 or $Ce(OTf)_3$/PyBox-3 as catalyst, TMSCl as promoter, and the reaction was conducted in the presence of activated MS 4Å in anhydrous CH_2Cl_2 or $CHCl_3$ at room temperature; the results are listed in Table 2. The reactions of aldehydes with allyltrimethylsilane gave the corresponding homoallylic alcohols with moderate to good yields (75-95%) and moderate to excellent enantioselectivities (up to 99% ee). Among which, benzaldehyde (**1a**) mediated allylation get the best enantioselectivity (entries 1 and 9). $Ce(OTf)_3$/PyBox-1 in CH_2Cl_2 was studied at first, the substituents of aromatic aldehydes can affect the yields and enantioselectivities of the products (entries 2-7). A marginal electronic influence was also observed with para-substituted phenyl aldehydes (**1c**, **1f** and **1g**; entries 3, 6 and 7), the corresponding homoallylic alcohol can be obtained 89% ee, 87% ee and 89% ee respectively (**2c**, **2f** and **2g**; entries 3, 6 and 7). However, the corresponding ortho (**2d**) or meta substitute (**2b** and **2e**) led to an unexpected lower enantioselectivity (entries 2, 4 and 5). It should be noted that 3-phenylpropanal (**1h**) gave the desired product in moderate yield but with higher enantioselectivity (86% ee, entry 8). $Ce(OTf)_3$/PyBox-3 in CH_2Cl_2 was then tested subsequently (entries 9-16). Except for comparable (entries 9-11 and 13) or slightly inferior (entry 14) enantioselectivity,

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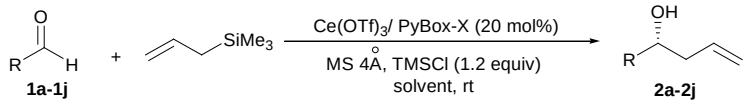
Table 2 Enantioselective Hosomi-sakurai of various aldehydes with allyltrimethylsilane[a]



Entry	R	Aldehyde	L	Solvent	Product	Yield (%)[c]	Ee (%)[d]
1[b]	C_6H_5	**1a**	PyBox-1	CH_2Cl_2	**2a**	87	>99
2[b]	3-$CH_3C_6H_4$	**1b**	PyBox-1	CH_2Cl_2	**2b**	85	71
3[b]	4-$CH_3C_6H_4$	**1c**	PyBox-1	CH_2Cl_2	**2c**	86	89
4[b]	2-$OCH_3C_6H_4$	**1d**	PyBox-1	CH_2Cl_2	**2d**	75	46
5[b]	3-$OCH_3C_6H_4$	**1e**	PyBox-1	CH_2Cl_2	**2e**	94	55
6[b]	4-$OCH_3C_6H_4$	**1f**	PyBox-1	CH_2Cl_2	**2f**	92	87
7[b]	2,4-$(CH_3)_2C_6H_3$	**1g**	PyBox-1	CH_2Cl_2	**2g**	82	89
8[b]	$C_6H_5CH_2CH_2$	**1h**	PyBox-1	CH_2Cl_2	**2h**	79	86
9	C_6H_5	**1a**	PyBox-3	CH_2Cl_2	**2a**	89	>99
10	3-$CH_3C_6H_4$	**1b**	PyBox-3	CH_2Cl_2	**2b**	87	74
11	4-$CH_3C_6H_4$	**1c**	PyBox-3	CH_2Cl_2	**2c**	90	90
12	2-$OCH_3C_6H_4$	**1d**	PyBox-3	CH_2Cl_2	**2d**	91	92
13	4-$OCH_3C_6H_4$	**1f**	PyBox-3	CH_2Cl_2	**2f**	95	88
14	2,4-$(CH_3)_2C_6H_3$	**1g**	PyBox-3	CH_2Cl_2	**2g**	92	71
15	1- $C_{10}H_7$	**1i**	PyBox-3	CH_2Cl_2	**2i**	86	82
16	C_4H_3S	**1j**	PyBox-3	CH_2Cl_2	**2j**	90	96
17	3-$CH_3C_6H_4$	**1b**	PyBox-3	$CHCl_3$	**2b**	89	78
18	2-$OCH_3C_6H_4$	**1d**	PyBox-3	$CHCl_3$	**2d**	92	74
19	4-$OCH_3C_6H_4$	**1f**	PyBox-3	$CHCl_3$	**2f**	90	86
20	2,4-$(CH_3)_2C_6H_3$	**1g**	PyBox-3	$CHCl_3$	**2g**	89	90

[a] Unless otherwise specfied, the reaction was carried out with aldehyde (0.1 mmol), TMSCl (0.12 mmol), and allyltrimethylsilane (0.15 mmol) in the presence of the chiral cerium(III) catalyst prepared from $Ce(OTf)_3$ (19 mol%) and PyBox-1 (or PyBox-3, 20 mol%) in the presence of activated MS 4Å in anhydrous solvent (1 mL). The reaction mixture was kept for 30 h at room temperature. [b] allyltrimethylsilane (0.2 mmol) was used. [c] Isolated yield. [d] ee was determined by determined by HPLC analysis with chiral OD-H or AS-H. For further details see Supporting Information.

the 2-methoxylbenzaldehyde (**1d**) deliver the product with surprisingly good enantioselectivity (92% ee, entry 12), which is complimentary to that of $Ce(OTf)_3$/PyBox-1 (46% ee, entry 4). In addition, it was worthy to note that thiophene-2-carbaldehyde (**1j**) also gave the corresponding homoallylic alcohol (**2j**) with excellent enantioselectivity (96% ee). When the reaction was conducted in $CHCl_3$ and at the function of $Ce(OTf)_3$/PyBox-3 at the same time (entries 17-20), the enantioselectivity of compound **2g** can also be improved (90% ee, entry 20), which is comparable to that of $Ce(OTf)_3$/PyBox-1 in CH_2Cl_2 (89% ee, entry 7).

In conclusion, we have developed catalytic asymmetric allylation of aldehydes with allyltrimethylsilane at room temperature by utilizing chiral $Ce(OTf)_3$/PyBox catalyst with TMSCl as activator. The procedure can furnish a wide variety of homoallylic alcohols in moderate to good yields with good levels of enantioselectivities by combination of complementary ligand (PyBox-1 or PyBox-3) and solvent (CH_2Cl_2 or $CHCl_3$). The allylation transfer share characteristics of mild reaction condition, simple operation, environmentally friendly property, inexpensive and readily available reagents. It extends the applications of rare-earth metal compounds as catalysts in organic synthesis.

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Notes and references

‡ General methods for synthesis of homoallylic alcohols: Pybox-1 (0.044 mmol) was added to a mixture of $Ce(OTf)_3$ (0.04 mmol) and 4Å MS (80 mg) in CH_2Cl_2 (1 mL). A combined solution of aldehyde (0.2 mmol) and TMSCl (0.24 mmol) was then added in 2 hours later. Followed by the addition of allyltrimethylsilane (0.4 mmol) at 0 ^{o}C, the reaction was stirred at room temperature. The reaction mixture was purified with chromatography and afforded the pure homoallylic alcohol.

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