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Enantioselective Addition Reaction of Azlactones with Styrene Derivatives Catalyzed by Strong Chiral Brønsted Acids

*Jun Kikuchi and Masahiro Terada**

Abstract: *An enantioselective intermolecular addition reaction of azlactones, as carbon nucleophiles, with styrene derivatives, as simple olefins, was demonstrated using a newly developed chiral Brønsted acid catalyst, namely, F_{10}BINOL-derived N-triflyl phosphoramide. Addition products having vicinal tetrasubstituted carbon centers, one of which is an all-carbon quaternary stereogenic center, were formed in good yields with moderate to high stereoselectivities. Extremely high acidity of the new chiral Brønsted acid was confirmed by its calculated pK_a value based on DFT studies and is the key to accomplishing not only high catalytic activity but also efficient stereocontrol in the intermolecular addition.*

Olefins, one of the most common carbon resources having carbon–carbon double bond(s), are widely utilized in petrochemical production. Considering the efficient use of these rich carbon resources, the activation of the carbon–carbon double bond (C=C) by protonation is one of the classical yet vital approaches for generating a cationic intermediate for the manufacture of fine chemicals.[1] Indeed, a cationic intermediate is involved in a broad range of organic transformations and applicable for the construction of carbon frameworks. The Markovnikov reaction, which involves an addition to C=C, is also one of the representative reactions and a fundamental method for the formation of a multisubstituted carbon center. If the stereochemical outcome of a Markovnikov-type reaction, which is initiated by the protonation of C=C using a Brønsted acid catalyst, could be controlled in an enantioselective manner, the method would become much more valuable.

In this regard, a variety of chiral Brønsted acid catalysts have been developed to date.[2–4] In particular, binaphthol (BINOL)-derived phosphoric acids and derivatives have emerged as the most representative chiral Brønsted acid catalysts,[2,3] and a diverse range of enantioselective reactions have been accomplished with protonation as the initiation step. However, in most cases, the reactions are promoted by the activation of carbon–heteroatom double bonds, such as imine (C=N) and carbonyl (C=O) compounds, through the protonation of the heteroatom lone pair of electrons. In contrast, the activation of a nonpolarized carbon–carbon double bond (C=C) by a chiral Brønsted acid catalyst, namely, the protonation of the π-electrons of the nonpolarized double bond, is quite limited. Although several examples using electron-rich olefins,[5] such as alkenylcarbamates (or enamides)[5a–c] and alkenyl ethers,[5d,e] have been developed, in principle, these electron-rich double bonds are readily protonated by an acid catalyst because of the resonance stabilization of the generated cationic intermediate by the heteroatom. However, the corresponding addition reaction using a simple olefin has been scarcely reported presumably because of the following difficulties of the intended transformation (Figure 1): 1) To generate the carbocation intermediate **I**, the use of a strong Brønsted acid (H-*A**) is indispensable for the protonation of a simple olefin, even a styrene derivative, because **I** is less stable than the carbocation intermediate generated from the electron-rich double bond. 2) An efficient interaction between **I** and the conjugate base (*A**$^-$) of the acid catalyst is required to control the stereochemistry. However, it seems that the relative locations of these ionic species are difficult to control because the ordinary hydrogen bonds are not formed in the generated ion pair, although nonclassical hydrogen-bonding interactions,[6] such as C–H···O and C–H···π, as well as π-stacking interactions, would be expected. 3) It is predicted that the stereodetermining step, namely, the bond formation between **I** and the nucleophile (*Nu*-H), would proceed with a small activation energy barrier, because **I** is a less stabilized intermediate and hence, in general, it is highly reactive in comparison with a carbocation that is stabilized by a heteroatom through the formation of resonance structure. In principle, to realize a highly enantioselective transformation, a sufficient energy difference between the two enantiomeric pathways is requisite in the transition states. However, the differentiation of these two transition states seems difficult because of the small activation energy.

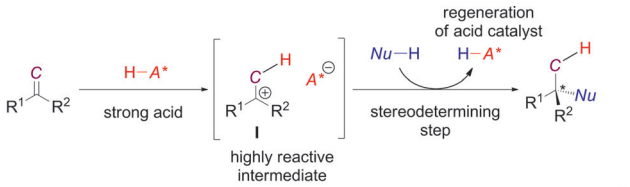

Figure 1. Difficulties in enantioselective transformation of a simple olefins initiated by protonation using a chiral Brønsted acid catalyst.

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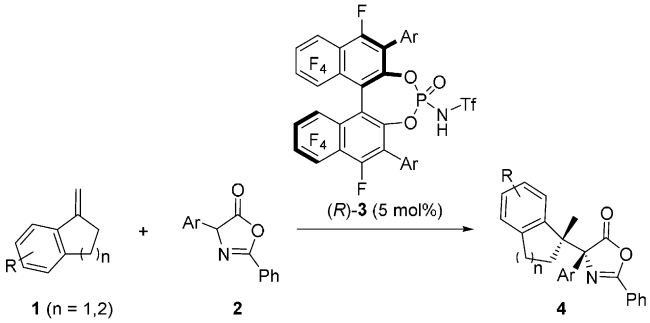
Recently, enantioselective transformations involving the protonation of C=C by a chiral Brønsted acid catalyst were reported in the hydroamination[7] and the hydroalkoxylation of simple olefins.[8] In all cases, to overcome the intrinsic difficulties mentioned above, an intramolecular system has been adopted, in which either a nitrogen or oxygen nucleophile is tethered to C=C, resulting in the restriction of conformational flexibility that is beneficial for stereocontrol. In contrast, no intermolecular variants have been reported, presumably because of the aforementioned difficulties, and in addition, a carbon nucleophile has never been utilized despite the fact that this type of carbon–carbon bond-forming (CCF) reaction would become a powerful method for the construction of carbon frameworks in an enantioselective manner. In this context, we envisioned the development of an intermolecular variant of the enantioselective CCF reaction of a carbon nucleophile, a reaction initiated through the protonation of a simple olefin by a chiral Brønsted acid catalyst. We herein report the enantioselective addition of the azlactones **2**, as the carbon nucleophile, to the styrene derivatives **1**, as the simple olefin, using the newly developed chiral Brønsted acid **3** (Scheme 1). The highly acidic catalyst **3** is essential for accelerating the reaction, in which **2** is regioselectively introduced at the multisubstituted side of the double bond, affording the products **4**, which have vicinal tetrasubstituted carbon centers, one of which is an all-carbon quaternary stereogenic center, in a stereoselective manner.



Scheme 1. Enantioselective addition reaction of azlactones with styrene derivatives catalyzed by a chiral Brønsted acid **3**.

To accomplish the unprecedented enantioselective intermolecular CCF reaction initiated by the activation of a simple olefin, the development of a chiral Brønsted acid catalyst that not only bears an efficient chiral environment for the enantioselective transformation but also has extremely high acidity is required. The most representative approach to enhance the acidity of a chiral Brønsted acid while maintaining an efficient chiral environment is the modification of the BINOL-derived phosphoric acid **5** (Ar = Ph, $pK_a = 3.33$[9a]; Figure 2a).[10] In particular, the BINOL-derived *N*-triflyl phosphoramide **6** reported by Yamamoto and co-workers[10a] has been widely utilized as a strong chiral Brønsted acid catalyst (Ar = Ph, $pK_a = -3.36$[9b]; Figure 2b). As an alternative approach, taking advantage of the strong electron-withdrawing property of the perfluoroaryl backbone, we recently developed the perfluorinated BINOL[11]

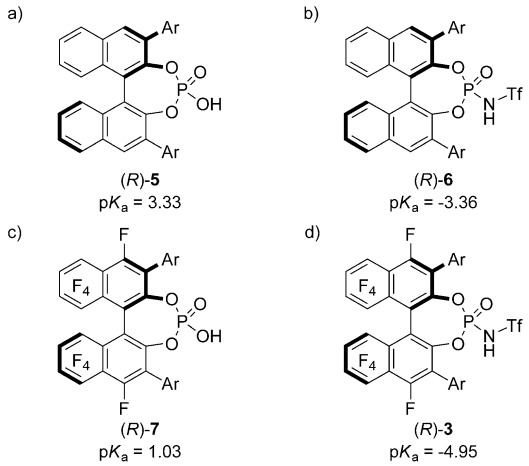
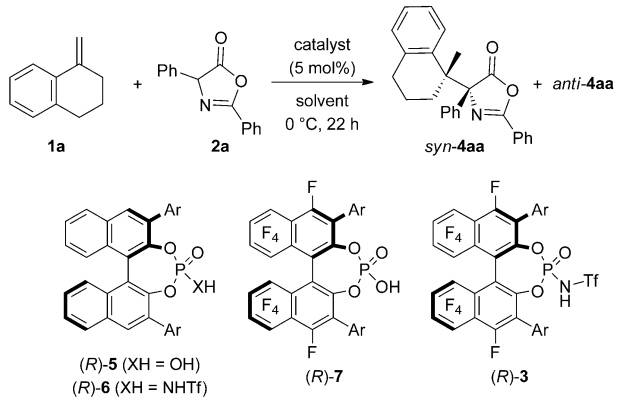


Figure 2. Chiral Brønsted acids (Ar = Ph) and their pK_a values in DMSO.

(F_{10}BINOL)-derived phosphoric acid catalyst **7** (Figure 2c).[12] Indeed, the enhancement of the acidity of F_{10}BINOL-derived phosphoric acid **7** (Ar = Ph, $pK_a = 1.03$[12c]) was estimated by DFT calculations of the acid catalyst and the corresponding conjugate base in accordance with Cheng's reported method.[9] To enhance the acidity further, we coupled a perfluorinated BINOL backbone with an *N*-triflyl phosphoramide moiety to give F_{10}BINOL-derived *N*-triflyl phosphoramide **3** (Figure 2d), as a novel strong acid

Table 1: Comparing the efficiency of the catalysts and optimization of the reaction conditions.[a]



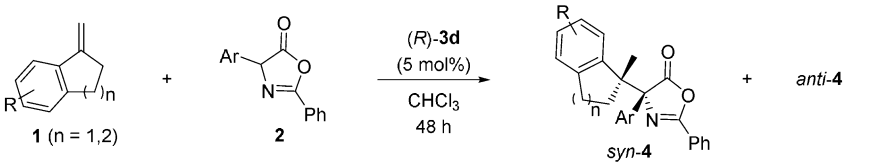
Entry	Catalyst (Ar)	Solvent	Yield [%][b]	*syn*/*anti*[c]	*ee* [%][d]
1	**5** (Ph)	CH_2Cl_2	4	68:32	–
2	**6** (Ph)	CH_2Cl_2	38	70:30	34
3	**7** (Ph)	CH_2Cl_2	54	66:34	29
4	**3a** (Ph)	CH_2Cl_2	75	63:37	36
5	**3a**	$CHCl_3$	84	70:30	41
6	**3b** (4-$MeOC_6H_4$)	$CHCl_3$	67	67:33	45
7	**3c** (9-phenanthryl)	$CHCl_3$	89	85:15	54
8	**3d** (1-pyrenyl)	$CHCl_3$	80	85:15	65
9[e]	**3d**	$CHCl_3$	82	89:11	72

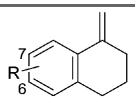
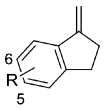
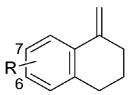
[a] Reaction conditions: **1a** (0.3 mmol), **2a** (0.1 mmol), catalyst (0.005 mmol), solvent (0.25 mL), 0 °C. [b] Yield of isolated product. [c] Diastereomeric ratio was determined by ^{1}H NMR analysis. [d] The *ee* value of major *syn*-**4aa** is shown and was determined by chiral stationary phase HPLC analysis. [e] At −20 °C.

catalyst relative to the chiral phosphoric acid derivatives reported to date. In fact, the high acidity of **3** was also confirmed by DFT calculations (**3a**: Ar = Ph, pK_a = −4.95).[13]

The catalytic efficiency of the newly developed (*R*)-**3** was confirmed in the intended reaction through activation of α-methylenetetralin (**1a**), and compared with those of other reported acids (*R*)-**5**–(*R*)-**7**.[14] The reaction was performed using **1a**, the azlactone **2a**, and 5 mol% of the acid catalyst in CH_2Cl_2 at 0 °C for 22 hours. As shown in Table 1 (entries 1–4), the high performance of (*R*)-**3** is obvious in terms of its high catalytic activity. The reaction using the parent (*R*)-**5** (Ar = Ph) gave only a small amount of the addition product **4aa** in a diastereomeric mixture (entry 1). The more acidic (*R*)-**6** and (*R*)-**7** (Ar = Ph) afforded **4aa** in an enantioenriched form, albeit in moderate yields and stereoselectivities (entries 2 and 3). In contrast, (*R*)-**3a** gave **4aa** in good yield albeit with moderate stereoselectivity (entry 4). These results clearly indicate that the high acidity is crucial to accelerate the addition reaction smoothly. Further screening for the solvent exhibited that the use of chloroform slightly enhanced the yield and the stereoselectivity (entry 5). To improve the stereochemical outcome, the substituent effect of the 3- and 3′-positions of (*R*)-**3** was next investigated.[14] (*R*)-**3b** exhibited a slight increase in enantioselectivity, albeit with a reduction of the yield (entry 6). The use of (*R*)-**3c** afforded **4aa** in high yield with a marked improvement of diastereoselectivity (entry 7). Introduction of the more extended π-system, namely, 1-pyrenyl group, as substituent Ar led to further improvement of enantioselectivity (entry 8). Further optimization of the reaction conditions using (*R*)-**3d** was performed by reducing the temperature to −20 °C (entry 9). As expected, both diastereo- and enantioselectivity were improved to some extent while maintaining a high yield. Although the stereochemical outcome is unsatisfactory compared with those in previous reports of chiral Brønsted acid catalysis,[2–4] taking the aforementioned difficulties into consideration, the present achievement deserves high marks.

Table 2: Scope with respect to substrates.[a]



Entry	**1** (R)		**2** (Ar)	**4**	*T* [°C]	Yield [%][b]	*syn*/*anti*[c]	*ee* [%][d]
1		**1b** (7-MeO)	**2a** (Ph)	**4ba**	−20	72	93:7	63
2		**1c** (6-Cl)	**2a**	**4ca**	−10	80	93:7	77
3		**1d** (6-Br)	**2a**	**4da**	−10	53	93:7	76
4[e,f]		**1e** (H)	**2a**	**4ea**	−60	92	96:4	66
5[f]		**1f** (6-Br)	**2a**	**4fa**	−50	84	98:2	53
6[f]		**1g** (5-Br)	**2a**	**4ga**	−50	90	96:4	78
7		**1a** (H)	**2b** (4-BrC_6H_4)	**4ab**	−20	90	84:16	68
8		**1a**	**2c** (4-ClC_6H_4)	**4ac**	−20	82	84:16	62
9		**1a**	**2d** (4-MeC_6H_4)	**4ad**	−20	61	87:13	63
10		**1a**	**2e** (4-$MeOC_6H_4$)	**4ae**	−20	47	94:6	58
11		**1a**	**2f** (3-BrC_6H_4)	**4af**	−20	80	78:22	73
12		**1a**	**2g** (3-$CF_3C_6H_4$)	**4ag**	−20	87	72:28	58
13		**1a**	**2h** (3-$MeOC_6H_4$)	**4ah**	−20	64	89:11	75
14		**1a**	**2i** (2-naphthyl)	**4ai**	−20	81	90:10	87
15		**1d** (6-Br)	**2i**	**4di**	−20	74	95:5	93

[a] Reaction conditions: **1** (0.3 mmol), **2** (0.1 mmol), (*R*)-**3d** (0.005 mmol), $CHCl_3$ (0.25 mL), −20 °C. [b] Yield of isolated products. [c] Diastereomeric ratio was determined by ^{1}H NMR analysis. [d] The *ee* value of the major *syn*-**4** is shown and was determined by chiral stationary phase HPLC analysis. [e] For 16 h. [f] 0.2 mmol of **1**.

With the efficient catalyst (*R*)-**3d** in hand, the substrate scope of the addition reaction was investigated (Table 2).[15] The reaction of α-methylenetetraline derivatives **1** having a variety of substituents, R, afforded **4** with high diastereoselectivities and fairly good enantioselectivities (entries 1–3). 1-Methyleneindane and its derivatives **1e**–**g** exhibited higher reactivities than α-methylenetetraline derivatives, thus leading to the formation of **4** in high yields, even at low temperatures (entries 4–6).[16] Although the enantioselectivities were markedly dependent on the substituent position and ranged from 53 to 78% *ee*, extremely high diastereoselectivities were observed regardless of the substituent pattern. Further investigations of the substrate scope with respect to **2**, having a series of aromatic substituents (Ar), were carried out (entries 7–15). The reaction of azlactones having an electron-withdrawing group at the *para*-position, **2b** and **2c**, proceeded smoothly (entries 7 and 8). In contrast, the introduction of an aryl moiety having an electron-donating group at the *para*-position, **2d** and **2e**, decelerated the reaction (entries 9 and 10), although the enantioselectivities were moderate irrespective of the electronic nature of the aryl moieties (entries 7–10). Azlactones having a *meta*-substituted aryl moiety, **2f**–**h**, also underwent the addition reaction with moderate stereoselectivities (entries 11–13). To our delight, the use of azlactone having a 2-naphthyl substituent **2i** resulted in the formation of a product with high enantioselectivities (entries 14 and 15). In particular, excellent diastereo- and enantioselectivity (95% *syn*, 93% *ee*) were achieved in the addition reaction of **2i** with **1d** (entry 15). In the present addition reaction, a highly stereoselective transformation was established, even though a specific substrate was used. These achievements clearly indicate that the use of the newly developed chiral Brønsted acid, namely, F_{10}BINOL-derived *N*-triflyl phosphoramide **3**, sucessfully addressed the difficulties of an enantioselective intermolecular addition to a simple olefin.

In conclusion, we have developed an enantioselective intermolecular addition reaction of azlactones with styrene derivatives, and it is catalyzed by a newly developed chiral Brønsted acid, thereby realizing the construction of a carbon framework from a simple olefin. The reaction that was initiated through the activation of the nonpolar carbon–carbon double bond afforded an addition product having vicinal tetrasubstituted stereogenic centers, one of which is an all-carbon quaternary center, in good yields with moderate to high stereoselectivities. A new chiral Brønsted acid catalyst, namely, F_{10}BINOL-derived *N*-triflyl phosphoramide, which features extremely high acidity as confirmed by the calculated pK_a values from DFT studies, is the key to accomplishing not only high catalytic activity but also efficient stereocontrol in the intermolecular addition reaction with a simple olefin. Further studies of the development of other enantioselective reactions using the novel highly acidic chiral Brønsted acid catalyst will be conducted in due course.

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Conflict of interest

The authors declare no conflict of interest.

Keywords: acidity · alkenes · asymmetric catalysis · Brønsted acid · organocatalysis

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[13] The pK_a value of (*R*)-**3a** in DMSO was calculated with the SMD/M06-2x/6–311++G(2df,2p)//B3LYP/6–31+G(d) method. The direct method was applied to estimate the pK_a value. See the Supporting Information for details.

[14] A series of catalyst screening are summarized in the Supporting Information (see Table S1).

[15] Although α-methylstyrene was applicable to the addition reaction of **2a**, yield and enantioselectivity (60% yield, 36%

ee) were lower than those of the addition reaction to **1a**. See the Supporting Information for details.

[16] CCDC 1890467 (**4ga**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre. See the Supporting Information for details.

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