Diastereo- and Enantioselective Conjugate Addition of α-Substituted Cyanoacetates to Maleimides Catalyzed by Binaphthyl-based Thiourea

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The Michael addition reaction is widely recognized as one of the most efficient and powerful methods for the C–C bonds formation in organic synthesis.[1] To date, various asymmetric Michael addition reactions have been developed.[2] Chiral α-cyanoacetates bearing a quaternary stereogenic center are an important class of substrates that serve as precursors of the highly functionalized chiral compounds such as amino acids and amino alcohols with simple functional group transformation.[3] Recently, organocatalytic enantioselective conjugate addition reactions of α-substituted cyanoacetates with various Michael acceptors have been reported.[4] Until now, there are a few reports for the catalytic conjugate addition of α-substituted cyanoacetates to maleimides using organocatalysts.[5] Although these methods are satisfyingly efficient in some extent, new organocatalytic conjugate addition of α-substituted cyanoacetates to maleimides is highly desired.

As part of our research program related to the development of synthetic methods for the enantioselective construction of stereogenic carbon centers,[6] we recently reported the asymmetric Michael addition reactions of active methines using chiral catalysts.[7] Herein, we wish to describe the conjugate addition of α-substituted cyanoacetates with maleimide promoted by binaphthyl-based organocatalysts.

To determine suitable reaction conditions for the catalytic enantioselective Michael addition of α-substituted cyanoacetates, we initially investigated the reactions with *N*-phenylmaleimide (**2a**) in the presence of 10 mol % binaphthyl-based organocatalysts **I–IV** bearing both central and axial chiral elements (Figure. 1). As shown in Table 1, binaphthyl-based (thio)urea catalysts **I–III** effectively promoted the reaction in toluene with high enantioselectivities (entries 1–3, Table 1). Replacement of urea function with squaramide (catalyst **IV**) slightly decreased the enantioselectivity (entry 4, -Table 1), and the highest enantioselectivities obtained with the binaphthyl-based thiourea catalyst **II**. For further improvement of selectivity, different solvents were then tested in the presence of 10 mol % of catalyst **II**. Aprotic solvents such as toluene, benzene, diethyl ether, THF, and dichloromethane were well tolerated in this Michael addition without a significant decrease of enantioselectivities (76–96% enantiomeric excesses [ee], entries 2 and 5–9, Table 1). Among the solvents probed, toluene provided the best results (79% yield, 9:1 dr, 96% ee, entry 2, Table 1). The present catalytic system tolerates catalyst loading down to 5 mol %, and both the yields and enantioselectivities were retained (entries 4 and 10–11, Table 1).

With the optimized conditions in hand, we proceeded to investigate the scope of the enantioselective conjugate addition of various α-substituted cyanoacetates **1** and *N*-phenylmaleimide (**2a**) in the presence of 5 mol % of binaphthyl-based thiourea-tertiary amine catalyst **II** in toluene at room temperature. The results of conjugate addition reaction are summarized in Table 2. A range of electron-donating and electron-withdrawing substitutions on the aryl ring of the α-aryl cyanoacetates **1** provided reaction products **3a–3e** in high yields (64–97%), high diastereoselectivities (7–9:1), and excellent enantioselectivities (84–98%, entries 2–5, Table 2). The α-heteroaryl cyanoacetate provided the products with high selectivity (87% ee, entry 6, Table 2). Moderate yield and selectivity were obtained when α-alkylsubstituted cyanoacetates were used as the substrate (entry 7, Table 2). An aryl substitution on the *N*-arylmaleimides **2** provided corresponding products **3h–3i** in high yields and high enantioselectivities (81–94%, entries 8–9, Table 2). Absolute configuration of products was determined by comparison either of the optical rotation or chiral HPLC data with those of the reported ones.[5]

Although the reason for the observed enantioselectivity is still unclear, we believe that a carbonyl group of the maleimide is activated by the thiourea moiety through hydrogen bonding, and the cyanoacetate moiety is activated by the basic nitrogen atom in tertiary amine (Figure 2). The attack from the *si*-face of the enol form of cyanoacetate to the *re*-face of the maleimide gives product **3**. Nevertheless, the precise catalytic mechanism still requires further investigation.

In summary, we have developed efficient catalytic enantioselective Michael addition reactions of α-substituted cyanoacetates **1** to maleimides **2** using binaphthyl-based thiourea catalyst. The desired Michael products were obtained in high yields and diastereoselectivities. Also, the excellent enantioselectivities were observed (up to 98% ee). Further studies on asymmetric reactions catalyzed by binaphthyl-based organocatalysts are underway in our laboratory.

Experimental

General. All commercial reagents and solvents were used without purification. TLC analyses were carried out on precoated silica gel plates with F_{254} indicator. Visualization

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was accomplished by UV light (254 nm), I_2, *p*-anisaldehyde, ninhydrin, and phosphomolybdic acid solution as an indicator. Purification of reaction products was carried out by flash chromatography using Merck silica gel 60 (Darmstadt, Germany) (230–400 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Jeol ECS 400 MHz NMR (Tokyo, Japan) (400 MHz for ^{1}H and 100 MHz for ^{13}C). Chemical shift values (δ) are reported in ppm relative to Me_4Si (δ 0.0 ppm). Optical rotations were measured on a JASCO-DIP-1000 digital polarimeter (Tokyo, Japan) with a sodium lamp. The ee were determined by HPLC. HPLC analysis was performed on Younglin M9100 Series, measured at 230 nm using the indicated chiral column.

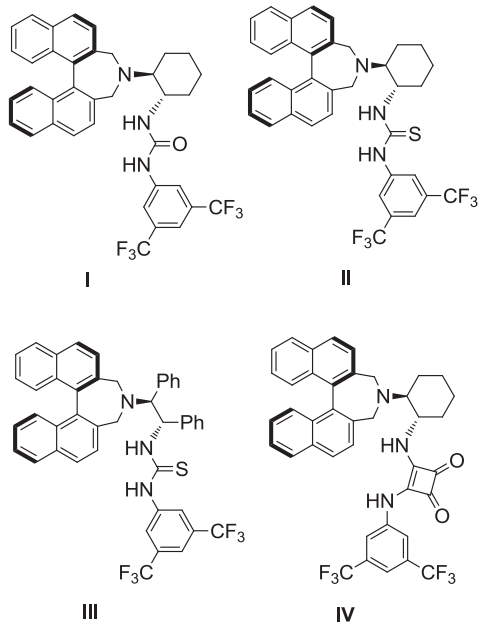
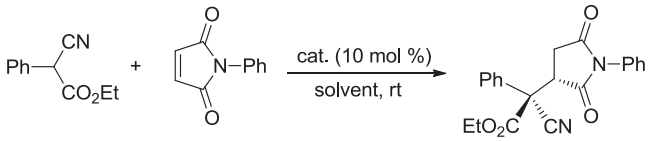


Figure 1. Structures of organocatalysts.

Typical Procedure for the Michael Addition of Ethyl 2-Cyanoacetate 1a to *N*-Phenylmaleimide 2a. To a stirred solution of *N*-phenylmaleimide (**2a**, 104 mg, 0.6 mmol, 1.2 equiv) and binaphthyl-modified thiourea **II** (1.7 mg, 2.5 μ mol, 5 mol %) in toluene (1.0 mL) was added ethyl 2-cyano-2-phenylacetate (**1a**, 95 mg, 0.5 mmol, 1.0 equiv) at room temperature. The reaction mixture was stirred at room temperature for 2 days. The reaction mixture was purified by column chromatography (EtOAc:hexane:dichlomethane = 1:8:6) to give the desired product (**3a**, 139 mg, 77%).

(*R*)-Ethyl 2-cyano-2-((*S*)-2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-phenylacetate (3a): $[\alpha]_D^{25} = -44.3$ ($c = 1$, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.669–7.627 (m, 2H), 7.52–7.39 (m, 6H), 7.32–7.29 (m, 2H), 4.43–4.23 (m, 3H), 2.85–2.78 (dd, $J = 9.2$, 18.6 Hz, 1H), 2.58–2.51 (dd, $J = 6.4$, 18.8 Hz, 1H), 1.32–1.30 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 173.97, 172.88, 165.98, 131.21, 129.71, 129.67, 129.32, 129.09, 126.48, 115.86, 64.19, 55.44, 46.99, 31.65, 13.77; HPLC (70:30, *n*-hexane:*i*-PrOH, 220 nm, 1.0 mL/

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

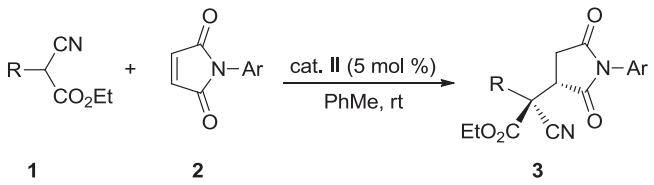


1a **2a** **3a**

Entry	Catalysts	Solvent	Time (h)	Yield[b] (%)	dr[c]	ee[d] (%)
1	**I**	PhMe	5	80	1.9:1	94
2	**II**	PhMe	5	79	9:1	96
3	**III**	PhMe	5	24	1.3:1	88
4	**IV**	PhMe	5	55	1.5:1	89
5	**II**	PhH	5	64	9:1	94
6	**II**	*p*-xylene	5	93	16:1	80
7	**II**	Et_2O	7	45	9:1	83
8	**II**	THF	7	70	9:1	76
9	**II**	CH_2Cl_2	9	55	3:1	69
10[e]	**II**	PhMe	48	77	9:1	98
11[f]	**II**	PhMe	72	35	9:1	90

[a] Reaction conditions: ethyl α-phenyl cyanoacetate (**1a**, 0.3 mmol), *N*-phenylmaleimide (**2a**, 0.36 mmol), catalyst (0.03 mmol), solvent (3.0 mL) at room temperature.
[b] Isolated yield.
[c] Determined ^{1}H NMR analysis of the crude mixture.
[d] Enantiopurity was determined by HPLC analysis using CHIRALPAK IA column.
[e] 5 mol % catalyst loading.
[f] 2.5 mol % catalyst loading.

Table 2. Substrate scope.[a]



1 2 3

Entry	**1**, R	**2**, Ar	Time (days)	Yield[b] (%)	dr[c]	ee[d] (%)
1	Ph	Ph	2	**3a**, 77	9:1	98
2	4-OMe–Ph	Ph	1	**3b**, 64	9:1	95
3	4-F–Ph	Ph	4	**3c**, 97	8:1	88
4	4-Cl–Ph	Ph	4	**3d**, 95	8:1	84
5	3-Cl–Ph	Ph	1	**3e**, 96	7:1	98
6	2-Thienyl	Ph	2	**3f**, 95	9:1	87
7	Bn	Ph	7	**3g**, 50	1.3:1	64
8	Ph	4-CN–Ph	1	**3h**, 93	9:1	94
9	Ph	4-OMe–Ph	5	**3i**, 86	20:1	81

[a] Reaction conditions: α-cyanoacetates **1** (0.3 mmol), maleimides **2** (0.36 mmol), catalyst (0.015 mmol), solvent (3.0 mL) at room temperature.
[b] Isolated yield.
[c] Determined ^{1}H NMR analysis of the crude mixture.
[d] Enantiopurity was determined by HPLC analysis using CHIRALPAK IA (**3a–3f**, **3h**,and **3i**) and AD-H (**3g**) columns.

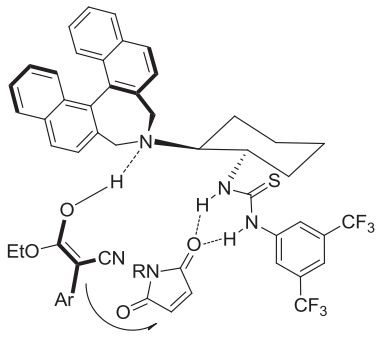


Figure 2. Proposed stereochemical model.

min) CHIRALPAK IA column, t_R = 17.1 min (major), t_R = 25.8 min (minor), 98% ee.

(*R*)-Ethyl 2-cyano-2-((*S*)-2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-(4-methoxyphenyl)acetate (3b): $[\alpha]_D^{24} = -68.1$ (c = 1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56–7.54 (d, J = 8.8 Hz, 2H), 7.49–7.41 (m, 3H), 7.32–7.29 (m, 2H), 6.98–6.96 (d, J = 8.8 Hz, 2H), 4.41–4.22 (m, 3H), 3.842 (s, 3H), 2.85–2.78 (dd, J = 9.6, 18.4 Hz, 1H), 2.58–2.52 (dd, J = 6.8, 18.6 Hz, 2H), 1.33–1.28 (t, J = 7.2 Hz, 3H); HPLC (70:30, *n*-hexane:*i*-PrOH, 220 nm, 1.0 mL/min) CHIRALPAK IA column, t_R = 23.6 min (major), t_R = 16.7 min (minor), 95% ee.

(*R*)-Ethyl 2-cyano-2-((*S*)-2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-(4-fluorophenyl)acetate (3c): $[\alpha]_D^{24} = -108.1$ (c = 1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.671–7.659 (m, 1H), 7.53–7.42 (m, 4H), 7.32–7.13 (m, 4H), 4.43–4.24 (m, 3H), 2.86–2.78 (dd, J = 9.6, 18.8 Hz, 1H), 2.56–2.50 (dd, J = 6.4, 18.4 Hz, 1H), 1.34–1.30 (t, J = 6.8 Hz, 3H); HPLC (70:30, *n*-hexane:*i*-PrOH, 220 nm, 1.0 mL/min) CHIRALPAK IA column, t_R = 14.0 min (major), t_R = 19.3 min (minor), 88% ee.

(*R*)-Ethyl 2-(4-chlorophenyl)-2-cyano-2-((*S*)-2,5-dioxo-1-phenylpyrrolidin-3-yl)acetate (4d): $[\alpha]_D^{21} = -15.5$ (c = 0.57, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61–7.58 (m, 2H), 7.48–7.42 (m, 5H), 7.32–7.26 (m, 2H), 4.42–4.24 (m, 3H), 2.86–2.79 (dd, J = 9.6, 18.4 Hz, 1H), 2.56–2.50 (dd, J = 6.4, 18.4 Hz, 1H), 1.34–1.30 (t, J = 7.2 Hz, 3H); HPLC (90:10, *n*-hexane:*i*-PrOH, 220 nm, 1.0 mL/min) CHIRALPAK IA column, t_R = 70.6 min (major), t_R = 41.6 min (minor), 84% ee.

(*R*)-Ethyl 2-(3-chlorophenyl)-2-cyano-2-((*S*)-2,5-dioxo-1-phenylpyrrolidin-3-yl)acetate (3e): $[\alpha]_D^{20} = -168.7$ (c = 0.17, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67–7.66 (m, 1H), 7.63–7.55 (m, 1H), 7.49–7.42 (m, 5H), 7.32–7.29 (m, 2H), 4.43–4.26 (m, 3H), 2.88–2.81 (dd, J = 9.6, 18.6 Hz, 1H), 2.57–2.51 (dd, J = 6.4, 18.0 Hz, 1H), 1.35–1.31 (t, J = 6.8 Hz, 3H); HPLC (70:30, *n*-hexane:*i*-PrOH, 220 nm, 1.0 mL/min) CHIRALPAK IA column, t_R = 17.5 min (major), t_R = 14.8 min (minor), 98% ee.

(*R*)-Ethyl 2-cyano-2-((*S*)-2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-(thiophen-2-yl)acetate (3f): $[\alpha]_D^{22} = -27.4$ (c = 1.09, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50–7.37 (m, 5H), 7.31–7.24 (m, 2H), 7.07–7.04 (m, 1H), 4.46–4.25 (m, 3H), 2.94–2.87 (dd, J = 9.6, 18.6 Hz, 1H), 2.73–2.67 (dd, J = 6.0, 18.6 Hz, 1H), 1.38–1.34 (t, J = 7.2 Hz, 3H); HPLC (70:30, *n*-hexane:*i*-PrOH, 220 nm, 1.0 mL/min) CHIRALPAK IA column, t_R = 24.4 min (major), t_R = 20.3 min (minor), 87% ee.

(*S*)-Ethyl 2-cyano-2-((*S*)-2,5-dioxo-1-phenylpyrrolidin-3-yl)-3-phenylpropanoate (3g): $[\alpha]_D^{22} = -179.5$ (c = 0.22, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ (major + minor)

7.51–7.30 (m, 10H), 4.27–4.17 (m, 2H), 3.83–3.79 (d, J = 13.6 Hz, 0.5H), 3.56–3.51 (m, 2H), 3.40–3.37 (d, J = 13.2 Hz, 0.5H), 3.17–3.08 (m, 1H), 2.92–2.86 (dd, J = 6.0, 18.2 Hz, 0.5H), 2.79–2.73 (dd, J = 6.4, 18.4 Hz, 0.5H), 1.23–1.19 (m, 3H); HPLC (90:10, *n*-hexane:*i*-PrOH, 220 nm, 1.0 mL/min) CHIRALPAK AD-H column, t_R = 45.2 min (major), t_R = 99.8 min (minor), 64% ee.

(*R*)-Ethyl 2-cyano-2-((*S*)-1-(4-cyanophenyl)-2,5-dioxopyrrolidin-3-yl)-2-phenylacetate (3h): $[\alpha]_D^{23} = -57.4$ (c = 1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78–7.72 (m, 2H), 7.64–7.60 (m, 2H), 7.53–7.45 (m, 5H), 4.45–4.23 (m, 3H), 2.88–2.81 (dd, J = 9.2, 18.6 Hz, 1H), 2.59–2.53 (dd, J = 6.4, 18.8 Hz, 1H), 1.33–1.29 (t, J = 6.8 Hz, 3H); HPLC (70:30, *n*-hexane:*i*-PrOH, 220 nm, 1.0 mL/min) CHIRALPAK IA column, t_R = 18.5 min (major), t_R = 23.4 min (minor), 94% ee.

(*R*)-Ethyl 2-cyano-2-((*S*)-1-(4-methoxyphenyl)-2-oxopyrrolidin-3-yl)-2-phenylacetate (3i): $[\alpha]_D^{23} = -37.4$ (c = 1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65–7.63 (m, 2H), 7.49–7.43 (m, 3H), 7.22–7.14 (m, 2H), 6.99–6.93 (m, 2H), 4.41–4.23 (m, 3H), 3.81 (s, 3H), 2.82–2.75 (dd, J = 9.2, 18.6 Hz, 1H), 2.54–2.48 (dd, J = 6.4, 18.4 Hz, 1H), 1.32–1.29 (t, J = 6.8 Hz, 3H); HPLC (70:30, *n*-hexane:*i*-PrOH, 220 nm, 1.0 mL/min) CHIRALPAK IA column, t_R = 27.1 min (major), t_R = 20.6 min (minor), 81% ee.

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