

Asymmetric Michael reactions of α -substituted acetates with cyclic enones catalyzed by multifunctional chiral Ru amido complexes

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Abstract

Well-defined 16-electron chiral Ru amido complexes, Ru[(*R,R*)-diamine](η^6 -arene), efficiently catalyze asymmetric Michael additions of Michael donors to cyclic enones to give adducts in high yields and with excellent ees. β -Ketoesters or nitroacetate as Michael donors react with 2-cyclopentenone in toluene or *t*-butyl alcohol containing the Ru amido catalyst (S/C = 50) to afford the Michael adduct in 99% yield and with up to 92% ee. The outcome of the reaction was delicately influenced by the structures of the diamine and arene ligands as well as reaction conditions.

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Keywords: Multifunction; Ru amido complex; Michael reaction; Asymmetric; C–C bond formation

1. Introduction

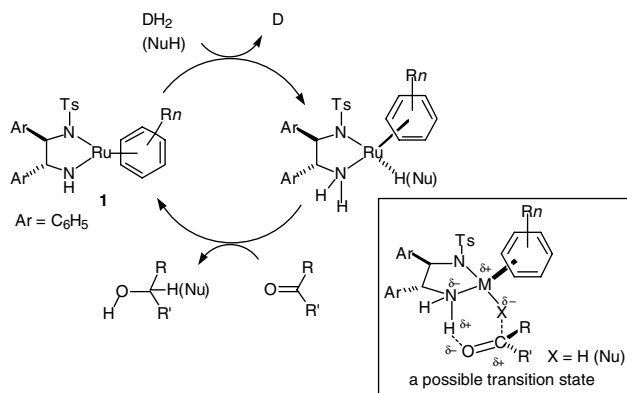
Enantioselective catalytic Michael additions are one of the most important types of C–C bond formations because of the versatility of the products as chiral building blocks. There are many reports on enantioselective Michael type reactions catalyzed by chiral metal catalyst systems [1]. They include copper [2], nickel [3], cobalt [4], rhodium [5], palladium [6], and heterobimetallic complexes [1a,7] as well as the chiral organic compounds as chiral catalysts [8]. We have recently reported that well-defined chiral Ru amido complexes, Ru[(*R,R*)-TsDPEN](η^6 -arene) (**1**) [9] and Ru[(*R,R*)-MsDPEN](η^6 -arene) (TsDPEN = *N*-(*p*-toluenesulfonyl)-1,2-diphenylethylenediamine, MsDPEN = *N*-methane-sulfonyl-1,2-diphenylethylenediamine), efficiently initiate the catalytic enantioselective Michael addition of malonates to cyclic enones to provide the corresponding Michael adducts in high yields with excellent ees [10b]. The reversible deprotonation of malonates with the Ru amido complex

bearing sufficient Brønsted basicity, leading to the corresponding malonate complex with a metal–C bond, was found to be a crucial step for the catalytic C–C bond formation [10]. We have expanded the scope of the Michael reaction catalyzed by the Ru amido complex and found that asymmetric Michael additions of α -substituted acetates including β -ketoesters, methyl cyanoacetate, and nitroacetates to cyclic enones proceeded smoothly to give the Michael adducts with good to excellent ees. We now describe the details of the reaction of acetates with 2-cyclopentenone. The conceptual metal–NH bifunctional effect, “Noyori effect”, of the chiral amido complex, which was originally developed in asymmetric transfer hydrogenation of ketones as shown in Scheme 1, has been successfully extended to enantioselective C–C bond formation.

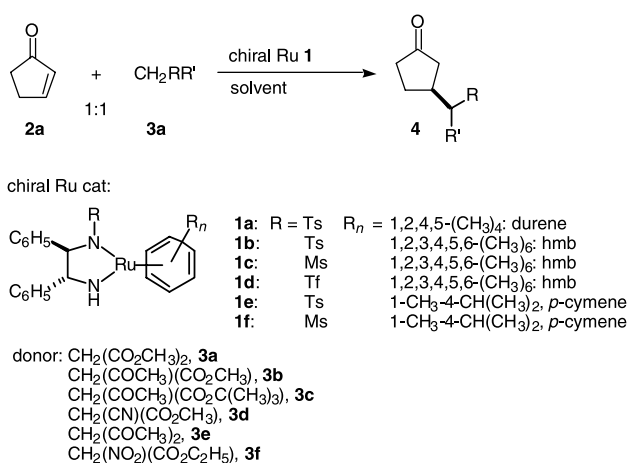
2. Results and discussion

A chiral Ru catalyst, Ru[(*R,R*)-diamine](η^6 -arene) (**1a–f**) has proven to efficiently effect enantioselective Michael addition of α -substituted acetates such as

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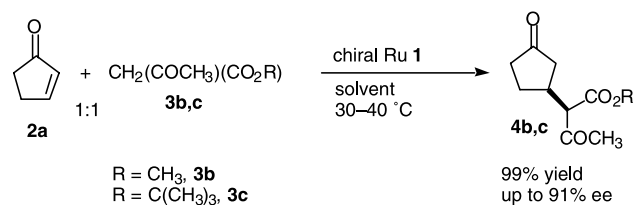


Scheme 1.



Scheme 2.

acetoacetates and ethyl nitroacetate to 2-cyclopentenone **2a**, giving the corresponding Michael adducts with good to excellent ees as summarized in Scheme 2. The reaction



Scheme 3.

of methyl acetoacetate **3b** with **2a** (acetate:enone:Ru = 50:50:1) in toluene containing Ru[(*R,R*)-TsDPEN](hmb) (HMB = hexamethylbenzene) provided the adduct **4b** in 99% yield and with 91% ee with respect to carbon atom on the cyclopentanone, although with a 1:1 mixture of two diastereomers with a single stereogenic center at the cyclopentanone ring (Scheme 3). The outcome of the reaction was significantly influenced by the reaction conditions and the structure of the arene and diamine ligands as observed in the reaction of malonates reported previously [10b]. As shown in Table 1, the durene complex **1a**, which is an excellent catalyst for the reaction of malonates, worked well but with relatively low enantioselectivity. The ee value of the product increases in the order of the durene-complex (**1a**) < HMB-complex (**1b**), possibly due to the steric reasons. The effect of the solvent on the catalyst performance is particularly significant in this reaction. Although *t*-butyl alcohol, toluene, and THF worked equally well for the reaction of malonates [10b], toluene was the best choice of the solvent in the reaction of β -ketoesters, as shown in Table 1. The reaction in toluene provided the adduct with up to 91% ee. The methanesulfonyl diamine (MsDPEN) complex **1c** gave the product quantitatively but with a slightly low ee.

Similarly, *t*-butyl acetoacetate **3c** reacted with **2a** to give the adduct **4c** in a reasonably high yield although

Table 1

Asymmetric Michael reactions of 2-cyclopentenone and dimethyl malonate **3a** and acetates **3b–d**, and acetylacetone **3e** catalyzed by chiral Ru amido complexes^a

Entry	Catalyst	Donor	Solvent	Temperature (°C)	Time (h)	Yield ^b (%)	ee ^c (%)	Configuration ^d
1	1a	3a	(CH ₃) ₃ COH	40	24	99	95	<i>S</i> [10b]
2	1b	3a	(CH ₃) ₃ COH	40	24	98	98	<i>S</i> [10b]
3	1a	3b	(CH ₃) ₃ COH	40	24	98	64	<i>S</i>
4	1b	3b	(CH ₃) ₃ COH	40	24	99	70	<i>S</i>
5	1b	3b	THF	40	24	99	84	<i>S</i>
6	1b	3b	Toluene	40	24	99	91	<i>S</i> [10b]
7	1c	3b	Toluene	40	24	99	85	<i>S</i>
8	1b	3c	Toluene	30	48	98	75 ^e	<i>S</i>
9	1c	3c	Toluene	30	48	88	72 ^e	<i>S</i>
10	1b	3d	THF	30	24	22	17	–
11	1b	3e	(CH ₃) ₃ COH	30	24	96	0	–

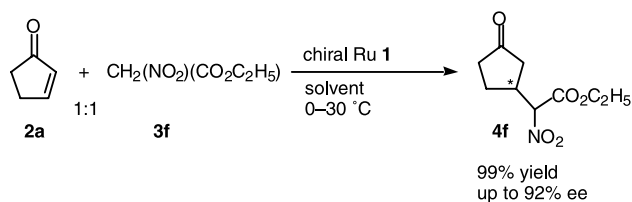
^a Unless otherwise noted, the reaction was carried out using 1.0 mmol of Michael acceptors and donors (1:1) in 1.0 ml of solvent. The molar ratio of acceptor:donor:Ru is 50:50:1 (*S/C* = 50).

^b Isolated yield after flash chromatography on the silica gel.

^c Determined by HPLC analysis.

^d Determined from the sign of rotation of the decarboxylation products.

^e Determined by ¹³C NMR of ketals of the decarboxylation product with (2*R*,3*R*)-butanediol (see Section 2.1).



Scheme 4.

with a lower ee value due to steric reasons. The reaction of ethyl cyanoacetate **3d** and **2a** gave a 1:1 adduct in 22% yield and with low ee in addition to the 2:1 adduct as a major product (52% yield) probably due to the strong interaction of the CN group with the Ru metal [11]. It should be noted that acetylacetonone **3e** was far less enantioselective although highly reactive, possibly because acetylacetonone has favored the chelating enolate formation, resulting in the replacement of the chiral diamine ligand. In fact, acetylacetonone favors the enolate form in the solution [12].

In a similar manner, ethyl nitroacetate **3f** readily reacts with **2a** to give almost quantitatively the corresponding Michael adduct with an excellent ee, although with a 1:1 diastereomer ratio (Scheme 4). The ee value of the product was determined by ^{13}C NMR spectroscopy of the ketal derived from the denitration product with (2*R*,3*R*)-butanediol. It should be noted that tuning of the chiral diamine and arene ligands as well as the reaction conditions significantly affected the enantiomeric excess of the reaction products as listed in Table 2. For example, complex **1b** bearing TsDPEN and HMB ligands, which was highly effective for the Michael reaction of dimethyl malonate **3a** and cyclic enone **2a**, was found to be far less effective for the reaction of nitroacetate **3f**, giving the product in 33% yield with 32% ee (entry 1). However, the use of the MsDPEN ligand (complex **1c**) caused a significant improvement in the reactivity and selectivity, leading to the Michael adduct **6a** with 82% ee in a quantitative yield. The complex **1d** bearing the more electron-withdrawing TfDPEN ligand gave the product with up to 91% ee, and the reaction in

t-amyl alcohol at the lower temperature of 0 °C, provided the product with up to 92% ee (entry 4). Noticeably, alcoholic solvents provided better catalyst performance in this reaction. The *p*-cymene complexes **1e,f** gave unsatisfactory results.

We have found that the 16-electron metal amido complex **1** with a M/NH bifunctional unit efficiently effected asymmetric catalytic Michael reactions of α -substituted acetates with cyclic enones leading to the corresponding adducts with good to excellent ees. The Brønsted basicity of the amido group in complex **1** is responsible for the excellent catalyst performance in this C–C bond formation. It has been reported that Michael addition promoted by metal complex catalysts proceeds through the nucleophilic attack of the O-bound metal enolate or the C-nucleophile attached to the metal to the olefin carbon in a 1,4-addition mode [2,7]. More recently, Hayashi reported highly efficient 1,4-addition of organoboronic acids to α,β -unsaturated ketones catalyzed by chiral Rh complexes, in which insertion of the olefinic part of the enone to the M–C bond was postulated [5e]. We have shown the possible mechanism of the addition of malonates to enones based on the single-crystal X-ray analysis of the Ru malonato complex and NMR investigation of the reaction mixture of malonate and the Ru amido complex [10b]. In comparison with the case of malonates, the reaction of α -substituted acetates with enones possibly proceeds via a similar transition state as postulated for the transfer hydrogenation and the C–C bond formation [10b] (Scheme 5). We are now working on further expansion of the scope of the reaction and studies aimed at clarifying the mechanism of the Michael reaction catalyzed by the Ru amido complexes.

2.1. Experimental

All experiments were performed in an atmosphere of dry argon using standard Schlenk tube techniques. Anhydrous toluene, acetone, THF, and CH_2Cl_2 were purchased from Kanto Chemical Co. Inc. ^1H NMR and ^{13}C NMR spectra were recorded on JEOL JNM-LA300

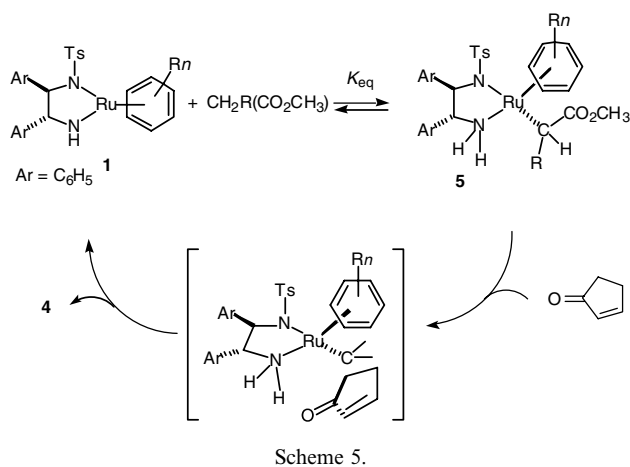
Table 2
Asymmetric Michael reactions of 2-cyclopentenone **2a** and ethyl nitroacetate **3f** catalyzed by chiral Ru amido complexes^a

Entry	Catalyst	Solvent	Temperature (°C)	Time (h)	Yield ^b (%)	ee ^c (%)
1	1b	(CH_3) ₃ COH	30	48	33	32
2	1c	(CH_3) ₃ COH	30	48	99	82
3	1d	(CH_3) ₃ COH	30	24	98	91
4	1d	(CH_3) ₂ CHCH ₂ OH	0	48	95	92
5	1d	Toluene	0	48	86	92
6	1e	(CH_3) ₃ COH	30	48	83	57
7	1f	Toluene	30	24	81	55

^a Unless otherwise noted, the reaction was carried out using 1.0 mmol of Michael acceptors and donors (1:1) in 1.0 ml of solvent. The molar ratio of acceptor:donor:Ru is 50:50:1 (S/C = 50).

^b Isolated yield after flash chromatography on the silica gel.

^c Determined by ^{13}C NMR of corresponding ketals derived from the denitration product and (2*R*,3*R*)-butanediol. The absolute configuration was not determined.



Scheme 5.

Fourier transform spectrometer with tetramethylsilane as an internal standard. High performance liquid chromatography (HPLC) analysis was performed on a JASCO PU-980 or JASCO PU1580 equipped with a UV-970 or UV-1510 detector. Optical rotation was measured with a JASCO DIP-370 digital polarimeter.

2.2. Typical experimental procedure for Michael reactions of cyclic enone with methyl acetoacetate catalyzed by *Ru*[(1*R*,2*R*)-*N*-(*p*-toluenesulfonyl)-1,2-diphenylethylenediamine](hexamethylbenzene)

Methyl acetoacetate (108 μl , 1.0 mmol), 2-cyclopentenone (84 μl , 1.0 mmol), and toluene (1.0 ml) were added to *Ru*[(1*R*,2*R*)-*N*-(*p*-toluenesulfonyl)-1,2-diphenylethylenediamine](hexamethylbenzene) (12.6 mg, 0.02 mmol) and the mixture was degassed by freeze–thaw cycles. The mixture was stirred at 40 °C for 24 h, then was evaporated with a vacuum pump and purified with flash column chromatography (silica gel, eluent:hexane/acetone = 9/1) to give 3-[(acetyl)(methoxycarbonyl)methyl]-1-cyclopentanone **4b** with 91% ee with a single stereogenic center at the cyclopentanone ring in 99% isolated yield.

2.3. 3-[(acetyl)(methoxycarbonyl)methyl]-1-cyclopentanone **4b**

Obtained as a 1:1 mixture of two diastereomers with a single stereogenic center at the cyclopentanone ring. ^1H NMR (300 MHz, CDCl_3): δ 1.52–1.65 (m, 2H), 1.84 (dd, $J = 10.5$ Hz, 18.0 Hz, 1H), 1.96 (dd, $J = 11.0$ Hz, 17.9 Hz, 1H), 2.13–2.51 (m, 8H), 2.26 (s, 3H), 2.28 (s, 3H), 2.81–2.93 (m, 2H), 3.48 (d, $J = 5.1$ Hz, 1H), 3.52 (d, $J = 4.6$ Hz, 1H), 3.76 (s, 3H), 3.78 (s, 3H). ^{13}C NMR (75 MHz, CDCl_3): δ 27.11, 27.23, 29.07, 29.34, 35.54, 35.62, 37.73, 37.86, 42.43, 42.59, 52.34, 63.90, 64.19, 168.55, 168.65, 201.21, 201.37, 216.71, 216.72.

HPLC separation conditions: CHIRALPAK AS (4.6 mm i.d. \times 250 mm); eluent, hexane/IPA = 80/20; flow rate 1.0 ml/min; temperature 30 °C; detection UV 210

nm; retention time for the four stereoisomers: (major) 22.8 min (minor) 29.4 min (major) 35.8 min (minor) 41.8 min. $[\alpha]_{\text{D}}^{25} = 83.5$ (c 1.35 CHCl_3). MS (EI, 70 eV) 198 (M^+). Absolute configuration of cyclopentanone ring was determined by converting to a known diketone, (*R*)-3-(2-oxopropyl)cyclopentanone. $[\alpha]_{\text{D}}^{29} = 79.1$ (c 0.5 benzene) (lit. $[\alpha]_{\text{D}} = 70.6$ benzene), 92% ee (*R*) [13].

2.4. 3-[(acetyl)(*tert*-butoxycarbonyl)methyl]-1-cyclopentanone **4c**

Obtained as a 1:1 mixture of two diastereomers with a single stereogenic center at the cyclopentanone ring. ^1H NMR (300 MHz, CDCl_3): δ 1.47 (s, 9H), 1.49 (s, 9H), 1.63–2.02 (m, 2H), 2.24 (s, 3H), 2.26 (s, 3H), 2.16–2.34 (m, 8H), 2.45 (d, $J = 7.3$ Hz, 1H), 2.50 (d, $J = 7.3$ Hz, 1H), 2.81–2.87 (m, 2H), 3.32 (d, $J = 4.9$ Hz, 1H), 3.35 (d, $J = 4.9$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3): δ 27.13, 27.62, 27.76, 27.80, 28.95, 29.17, 35.63, 35.71, 37.95, 38.12, 42.55, 42.99, 65.76, 65.96, 82.49, 82.52, 167.44, 167.50, 201.64, 201.77, 217.30. $[\alpha]_{\text{D}}^{25} = 50.6$ (c 1.10 CHCl_3). MS (CI, 70 eV) 241($\text{M}+1$).

The enantiomeric excess was determined by the relative intensities of diastereomeric ketals obtained by the reaction of (*R*)-3-(2-oxopropyl)cyclopentanone, the decarboxylation adduct of 3-[(acetyl)(*tert*-butoxycarbonyl)methyl]-1-cyclopentanone, with (2*R*,3*R*)-butanediol catalyzed by *p*-TsOH in toluene [14]. Ketal of (*R*)-3-(2-oxopropyl)cyclopentanone with (2*R*,3*R*)-butanediol, ^{13}C NMR (75 MHz, CDCl_3): δ 16.47, 16.87, 17.04, 17.14, 26.08, 31.24, 33.02, 37.28, 45.56, 46.08, 77.82, 78.05, 78.20, 78.49, 117.03. Ketal of (*S*)-3-(2-oxopropyl)cyclopentanone with (2*R*,3*R*)-butanediol, ^{13}C NMR (75 MHz, CDCl_3): δ 16.46, 16.82, 17.06, 17.13, 25.98, 31.68, 33.47, 37.93, 45.87, 45.92, 77.80, 78.05, 78.07, 78.45, 116.94. Absolute configuration of cyclopentanone ring was determined by converting to a known diketone, (*R*)-3-(2-oxopropyl)cyclopentanone. $[\alpha]_{\text{D}}^{29} = 77.6$ (c 0.5 CHCl_3) (lit. $[\alpha]_{\text{D}} = 70.6$ benzene), 92% ee (*R*) [13].

2.5. 3-(diacetylmethyl)-1-cyclopentanone **4e**

^1H NMR (300 MHz, CDCl_3): δ 1.30–1.41 (m, 1 H), 1.56–1.65 (m, 1H), 2.04 (s, 3H), 2.05 (s, 3H), 1.95–2.22 (m, 4H), 2.70–2.78 (m, 1H), 3.51 (d, $J = 10.5$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3): δ 27.02, 29.12, 29.39, 35.79, 37.51, 42.19, 74.00, 202.27, 202.48, 216.28.

2.6. Typical experimental procedure for the reaction of 2-cyclopentenone with ethyl nitroacetate catalyzed by *Ru*[(1*R*,2*R*)-*N*-trifluoromethanesulfonyl-1,2-diphenylethylenediamine](hexamethylbenzene)

Ethyl nitroacetate (111 μl , 1.0 mmol), 2-cyclopentenone (84 μl , 1.0 mmol), and *t*-butyl alcohol (1.0 ml) were added to *Ru*[(1*R*,2*R*)-*N*-trifluoromethanesulfonyl-1,2-

diphenylethylenediamine)(hexamethylbenzene) (12.0 mg, 0.02 mmol) and the mixture was degassed by freeze-thaw cycles. The mixture was stirred at 30 °C for 24 h, then evaporated with a vacuum pump and purified with flash column chromatography (silica gel, eluent:hexane/ethyl acetate = 5/1) to give 3-[(ethoxycarbonyl)(nitro)methyl]-1-cyclopentanone **4f** with 91% ee as 1:1 mixture of two diastereomers with a single stereogenic center at the cyclopentanone ring in 98% isolated yield.

2.7. 3-[(ethoxycarbonyl)(nitro)methyl]-1-cyclopentanone **4f**

Obtained as a 1:1 mixture of two diastereomers with a single stereogenic center at the cyclopentanone ring. ¹H NMR (300 MHz, CDCl₃): δ 1.32 (t, *J* = 7.1 Hz, 3H), 1.34 (t, *J* = 7.1 Hz, 3H), 1.76–1.88(m, 2H), 2.07–2.61 (m, 10H), 3.15–3.18 (m, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 4.34 (q, *J* = 7.1 Hz, 2H), 5.13 (d, *J* = 2.7 Hz, 1H), 5.16 (d, *J* = 2.2 Hz, 1H). ¹³C NMR (75 MHz, CDCl₃): δ 13.64, 13.67, 25.41, 26.17, 37.12, 37.36, 37.70, 40.87, 41.39, 63.07, 63.10, 90.68, 90.89, 163.13, 163.20, 214.66, 214.85. [α]_D²⁴ – 78.5(c 1.5 CHCl₃). MS (CI, 70 eV) 216 (M+1).

The enantiomeric excess was determined by the relative intensities of diastereomeric ketals obtained by the reaction of the denitration product with (2*R*,3*R*)-butanediol catalyzed by *p*-TsOH in toluene. Major ketal compound: ¹³C NMR (75 MHz, CDCl₃): δ 14.16, 16.76, 17.03, 30.11, 34.08, 37.68, 40.17, 44.26, 60.06, 78.18, 116.64, 172.68. Minor ketal compound: ¹³C NMR (75 MHz, CDCl₃): δ 14.16, 16.86, 17.03, 29.71, 33.65, 37.29, 40.35, 43.93, 60.06, 78.30, 116.64, 171.92.

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