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Enantioselective Michael Reactions of β,β-Disubstituted Nitroalkenes: A New Approach to $\beta^{2,2}$-Amino Acids with Hetero-Quaternary Stereocenters

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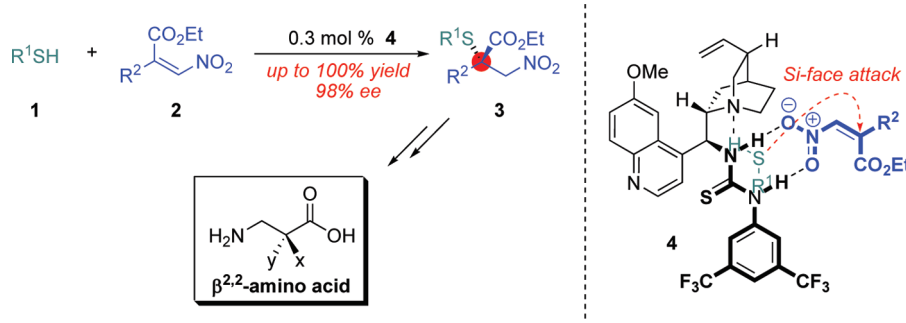
ABSTRACT



An atom-economic organocatalytic asymmetric Michael reaction of α,β,β-trisubstituted olefins has been successfully developed. The reaction exhibits excellent enantioselectivities under low loading of catalysts, and the conjugate addition products are valuable for the synthesis of novel $\beta^{2,2}$-amino acids and β-peptides.

Recent advances in β-peptides[1] and β-lactams[2] have significantly stimulated the development of catalytic asymmetric synthesis of β-amino acids. The introduction of a diverse array of functionalities to β-amino acids that is unprecedented in nature through chemical methods is an important endeavor.[3] In contrast to β^3-amino acids, which can be easily accessed by enantiospecific homologation of the natural amino acids, the β^2-analogues are much more difficult to obtain.[4] In particular, catalytic asymmetric synthesis of $\beta^{2,2}$-amino acids remains a challenge,[5] especially the de novo construction of the quaternary stereogenic center.[6]

The Michael addition has been established as a powerful tool to generate quaternary stereogenic centers (Scheme 1).

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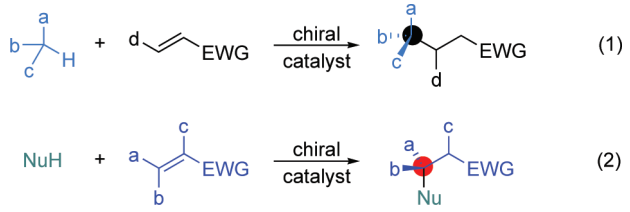
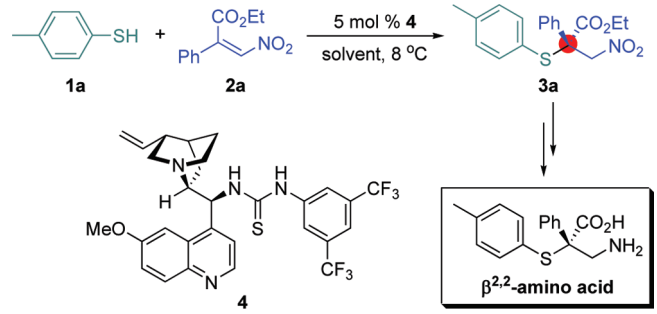
Scheme 1. General Strategies to Quaternary Stereocenters via Conjugate Additions



In this context, the conjugate addition of highly active trisubstituted carbon nucleophiles by metallic or organic catalysis has been well documented (Scheme 1, eq 1);[5a,7] however, an alternative strategy, the addition to β,β-disubstituted α,β-unsaturated systems (Scheme 1, eq 2), has been largely unexplored.[8] There are three main reasons that have, perhaps, precluded intensive research in this transformation, which include: (1) the intrinsic steric constraint and poor reactivity, (2) the difficulty in the stereocontrol, and (3) the reaction reversibility, especially in the case of heteronucleophiles.[9] Yet, such tri- or tetrasubstituted Michael acceptors constitute another kind of valuable synthon for the construction of both all-carbon and hetero-quaternary stereocenters. In this paper, we describe an organocatalytic Michael reaction of thiols with trisubstituted nitroacrylates to afford enantioenriched α-sulfenylated β-nitro esters, valuable precursors for the synthesis of α-thio-$\beta^{2,2}$-amino acids.[5b,c] Notably, this sulfa-Michael reaction[10] proceeds efficiently even in the presence of 0.3 mol % of catalyst conferring excellent enantioselectivities on the products (up to 98% ee).

Initially, we examined the feasibility of the strategy by reaction of *p*-thiocresol **1a** with α-phenyl-β-nitroacrylate **2a** in the presence of various acid−base bifunctional organocatalysts.[11,12] We were gratified to observe that the reaction took place cleanly and effectively, affording the desired product **3a** in high yield (96%) and enantioselectivity (75% ee), using the 9-thiourea cinchona alkaloid catalyst **4** (Table 1, entry 1).[13] With this promising result in hand, we then undertook further optimization experiments, and the results are summarized in Table 1. As shown, most common solvents are compatible with this Michael reaction, and all reactions proceed effectively in high yields (83−98%). However, in terms of enantioselectivity, less polar solvents (71−80% ee, entries 1−7) are superior to polar solvents, which caused a dramatic drop in enantiomeric excess (0 and 11% ee, entries 8 and 9). It is noteworthy that decreasing the catalyst loading greatly improves the enantioselectivity

Table 1. Conjugated Addition of *p*-Thiocresol **1a** to α-Phenyl-β-nitroacrylate **2a** with Organocatalyst **4** under Various Conditions[a]



entry	solvent	time	yield[b] (%)	ee[c] (%)
1	toluene	<1 min	96	75
2	xylene	<1 min	94	77
3	benzene	<1 min	98	80
4	DCM	<1 min	96	74
5	DCE	<1 min	93	71
6	$CHCl_3$	<1 min	90	78
7	Et_2O	<1 min	95	78
8	CH_3CN	<1 min	87	11
9	DMF	1 h	83	0
10	benzene	<5 min	95	88[d]
11	benzene	20 min	95	89[e]
12	benzene	5 h	93	88[f]
13	benzene-Et_2O	4 h	98	92[e,g]

[a] Unless noted, reactions were carried out with **1a** (0.22 mmol), **2a** (0.20 mmol), and **4** (0.1−5 mol %) in 1.0 mL of solvent at 8 °C. [b] Isolated yield. [c] Determined by chiral HPLC. [d] 0.5 mol % of **4**. [e] 0.3 mol % of **4**. [f] 0.1 mol % of **4**. [g] Conducted at −25 °C.

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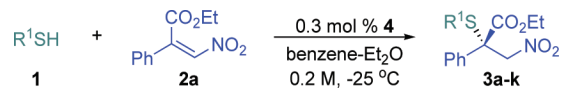
(entry 3 vs entries 10−12) with the best result obtained when 0.3 mol % of **4** was employed (89% ee, entry 11). Importantly, the reaction works efficiently even with 0.1 mol % of catalyst **4**, though a slightly longer reaction time is needed (88% ee, entry 12). Further improvement was achieved when the reaction was performed at a lower temperature using ether as the cosolvent (98% yield, 92% ee, entry 13).

Under optimal conditions, the Michael reactions of various thiols were then investigated, and the results are presented in Table 2. In the presence of 0.3 mol % of **4**, all arenethiols, regardless of their steric or electronic properties, underwent efficient reactions affording α-sulfenylated β-nitro esters containing quaternary stereocenters in excellent yields (89−98%) with high enantioselectivities (88−93% ee). For example, thiocresols bearing a methyl group at the *para*, *ortho*, and *meta* positions all provided the corresponding products in great yields and enantioselectivities (entries 1−3). Moreover, electronic variations of arenethiols also have minimal impact on the reaction. As revealed from entries 4−10, electron-rich (entries 4 and 5), electron-deficient (entries 6 and 7), and relatively electron-neutral (entry 8) thiols, including disubstituted and fused ones (entries 9 and 10), all reacted efficiently with excellent results. Less active alkanethiols are also promising substrates as long as a higher loading of catalyst is used. For instance, 4-methoxybenzylmercaptan **1k** reacted with α-phenyl ethyl β-nitroacrylate **2a** in the presence of 10 mol % of catalyst **4** to afford the corresponding product **3k** in 90% yield and 87% ee (entry 11).

Perhaps most importantly, this Michael reaction has considerable versatility in the Michael acceptor component as well, as illustrated in Table 3. α-Aryl-, heteroaryl-, and alkyl-substituted β-nitroacrylates all reacted efficiently to provide the corresponding chiral α-sulfenylated β-nitro esters containing quaternary stereocenters in excellent yields (90−100%) with excellent enantioselectivities (90−98% ee)

Table 2. Asymmetric Addition of Thiols **1** to β-Phenyl Ethyl Nitroacrylate **2a** with Organocatalyst **4**[a]

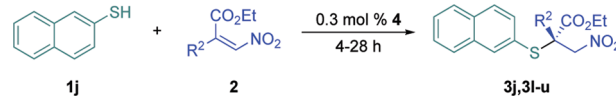


1 2a 3a-k

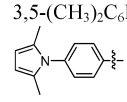
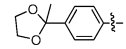
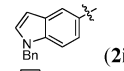
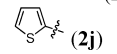
entry	R^1	time (h)	yield (%)[b]		ee (%)[c]
1	4-$CH_3C_6H_4$ (**1a**)	4	**3a**	98	92
2	2-$CH_3C_6H_4$ (**1b**)	2	**3b**	94	93[d]
3	3-$CH_3C_6H_4$ (**1c**)	2	**3c**	97	88
4	4-$CH_3OC_6H_4$ (**1d**)	2	**3d**	92	90
5	4-t-BuC_6H_4 (**1e**)	3	**3e**	93	92
6	4-FC_6H_4 (**1f**)	2	**3f**	92	90
7	4-ClC_6H_4 (**1g**)	2	**3g**	95	88
8	C_6H_5 (**1h**)	3	**3h**	94	92
9	3,5-$(CH_3)_2C_6H_3$ (**1i**)	4	**3i**	89	91
10	2-naphthyl (**1j**)	4	**3j**	97	93
11	4-CH_3OBn (**1k**)	96	**3k**	90	87[d,e]

[a] Unless noted, reactions were carried out with **1** (0.22 mmol), **2a** (0.20 mmol), and **4** (0.3 mol %) in 1.0 mL of benzene−Et_2O (v/v = 1/1) at −25 °C. [b] Isolated yield. [c] Determined by chiral HPLC. [d] Conducted in 1.0 mL of toluene at −45 °C. [e] 3.0 equiv of thiol and 10 mol % of **4** were used.

Table 3. Asymmetric Addition of 2-Thionaphthol **1j** to Various β-Nitroacrylates **2** with 0.3 mol % of Organocatalyst **4**[a]



1j 2 3j,3l-u

entry	R^2	method	yield (%)[b]		ee (%)[c]
1	C_6H_5 (**2a**)	A	**3j**	97	93
2	2-$CH_3C_6H_4$ (**2b**)	D	**3l**	99	92
3	3-$CH_3OC_6H_4$ (**2c**)	B	**3m**	93	90
4	4-$TBSOCH_2C_6H_4$ (**2d**)	C	**3n**	93	90
5	4-PhC_6H_4 (**2e**)	C	**3o**	97	94[d]
6	3,5-$(CH_3)_2C_6H_3$ (**2f**)	C	**3p**	100	92
7	 (**2g**)	C	**3q**	99	92
8	 (**2h**)	C	**3r**	95	98
9	 (**2i**)	C	**3s**	98	94
10	 (**2j**)	A	**3t**	93	92
11	c-hex (**2k**)	C	**3u**	90	95

[a] Method A: 1.0 mL of benzene−Et_2O (v/v = 1/1) at −25 °C. Method B: 1.0 mL of toluene at −25 °C. Method C: 1.0 mL of toluene at −45 °C. Method D: 1.0 mL of toluene at −60 °C. [b] Isolated yield. [c] Determined by chiral HPLC. [d] Absolute configuration of **3o** was assigned by X-ray analysis.

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in the presence of 0.3 mol % of catalyst **4**. Furthermore, the absolute configuration of the newly formed *S*-quaternary stereocenter in product **3o** was unambiguously determined to be *S* by X-ray crystallographic analysis.[14] Upon this and previous reported bifunctional aspects of Cinchona alkaloid based thiourea catalysts, we proposed a plausible working model (**TS1**) in which the synergistic cooperative activation of the nucleophilic thiol **1** and electrophilic nitroacrylate **2** should be essential for the observed high enantioselective discrimination in the present catalytic system (Figure 1).

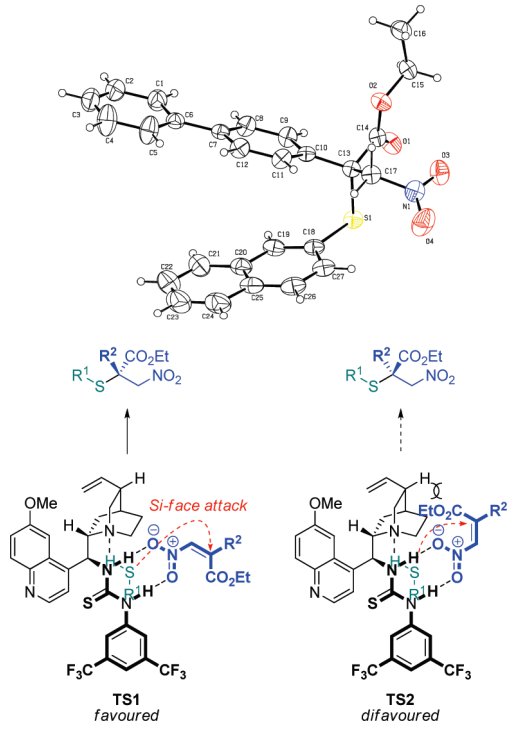
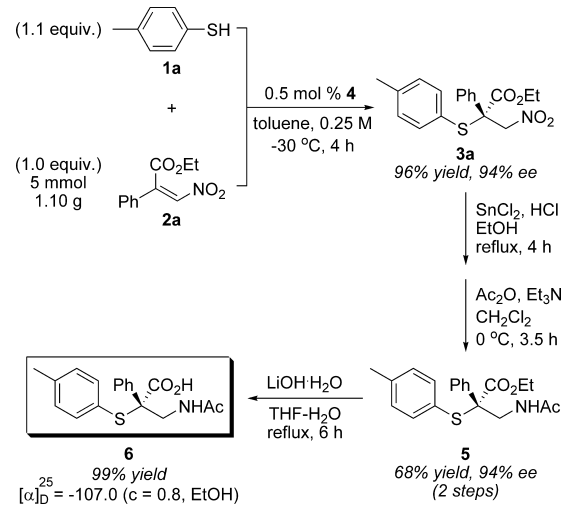


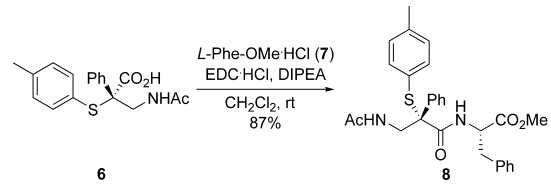
Figure 1. X-ray structure of **3o** and proposed transition state (**TS**) of the Michael reaction. Key: C, gray; H, white; N, blue; O, red; S, yellow.

The significance of the current protocol was then demonstrated in the expedient synthesis of α-thio-$\beta^{2,2}$-amino acids from the conjugate addition products. As a representative example, the (*S*)-3-acetamido-2-phenyl-2-(*p*-tolylthio)propanoic acid **6** was obtained in an overall yield of 65% with retention of optical purity after a straightforward three-step sequence (nitro-reduction/amine acylation/ester hydrolysis) from the Michael adduct **3a**, which was conveniently prepared in a gram-scale reaction of *p*-thiocresol **1a** and α-phenyl-β-nitroacrylate **2a** in 96% yield with 94% ee (Scheme 2).

Scheme 2. Synthesis of α-Thiol-$\beta^{2,2}$-amino Acid **5**



In addition, the $\beta^{2,2}$-amino acids would be valuable precursors for the synthesis of novel β or mixed β/α-peptides as illustrated in the synthesis of dipeptide **8** (eq 3). In the presence of peptide-coupling reagent 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDC·HCl) and *N*,*N*-diisopropylethylamine (DIPEA), α-thio-$\beta^{2,2}$-amino acid **6** reacted with L-Phe-OMe·HCl **7** cleanly to provide dipeptide **8** in 87% yield after 12 h.



(3)

In conclusion, we have developed a highly efficient and enantioselective organocatalytic Michael reaction with α,β,β-disubstituted Michael acceptors, which opened a new way to construction of a heteroquaternary stereocenter, and synthesis of $\beta^{2,2}$-amino acid and β-peptides. The reaction itself features simple experimental procedures under benign conditions and is completely atom-economic in character.

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Supporting Information Available: Experimental procedures and compound characterization data including X-ray crystal data (CIF) for **3o**. This material is available free of charge via the Internet at http://pubs.acs.org.

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(14) Crystal data of **3o**: $C_{27}H_{23}NO_4S$, M_r = 457.52, monoclinic, space group *P*2(1), a = 12.7746(9) Å, b = 5.8697(4) Å, c = 16.0370(11) Å, α = 90°, β = 102.412(4)°, γ = 90°, V = 1174.40(14) Å^3, D_{calc} = 1.294 Mg/m^3, T = 298(2) K, Z = 2, 12749 reflections collected, 4770 independent (R_{int} = 0.0239), final R indices [$I > 2\sigma(I)$]: R_1 = 0.0473, wR_2 = 0.1188, R indices (all data): R_1 = 0.0563, wR_2 = 0.1243, absolute structure parameter: 0.02(8), GOF(F_2) = 1.051. CCDC 735278 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.