CHEMISTRY
A EUROPEAN JOURNAL

DOI: 10.1002/chem.201000059

Multiple-Organocatalyst-Promoted Cascade Reaction: A Fast and Efficient Entry into Fully Substituted Piperidines

Yao Wang,[a] De-Feng Yu,[a] Yao-Zong Liu,[a] Hao Wei,[a] Yong-Chun Luo,[a] Darren J. Dixon,[b] and Peng-Fei Xu*[a]

Piperidines have been used widely in the construction of natural products and pharmaceutical compounds.[1] Recently, the synthesis of polysubstituted piperidines has received much attention. For example, Hayashi et al. reported an enantioselective, formal aza-[3+3] cycloaddition reaction for their construction[2] and Chen et al. introduced two aza-Diels–Alder reactions for the formation of piperidines with high enantioenrichment.[3] New, non-asymmetric approaches to the formation of polysubstituted piperidines continue to be developed within the synthetic community.[4] Notably, one important class of polysubstituted piperidines, which are characterized by 3-nitro and 2,4-diaryl substituent groups, is bioactive.[5] Unfortunately, the synthetic route to these bioactive molecules requires resolution procedures to obtain enantioenriched products.[5] Current methods for the synthesis of fully substituted, optically active piperidines generally employ lengthy routes. Despite the potential utility of these valuable compounds, to the best of our knowledge, an enantioselective, one-pot catalytic cascade approach to optically active, fully substituted piperidines remains an elusive problem; this is, presumably, due to the challenge of constructing five contiguous stereocenters around the six-membered-ring heterocycle. In this context, the development of a simple-to-perform and single-operation catalysis cascade to form fully substituted piperidines that contain the core structure of some anti-cancer compounds would be a timely endeavour.

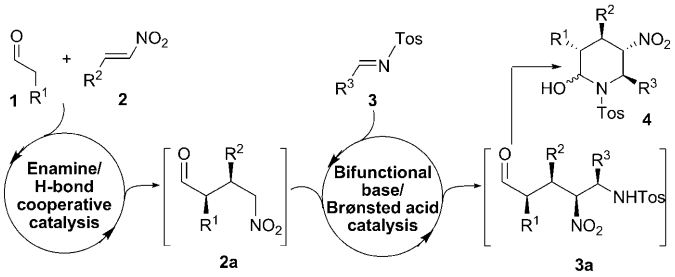
[a] Y. Wang, D.-F. Yu, Y.-Z. Liu, H. Wei, Y.-C. Luo, Prof. Dr. P.-F. Xu
State Key Laboratory of Applied Organic Chemistry
College of Chemistry and Chemical Engineering
Lanzhou University, Lanzhou 730000 (P. R. China)
Fax: (+86) 931-8915557
E-mail: xupf@lzu.edu.cn

[b] Prof. Dr. D. J. Dixon
Department of Chemistry, Chemistry Research Laboratory
University of Oxford, Mansfield Road
Oxford, OX1 3TA (UK)

Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/chem.201000059.

During the last few years, the development of asymmetric organocatalytic domino/cascade reactions has been hotly pursued.[6] Most of these elegant approaches involve two-step cascades and are based on covalent, or hydrogen-bonding catalysis.[6] However, many useful reactions cannot be effectively carried out by the use of a single organocatalyst. Thus, the combination of two organocatalysts has been employed in one-pot reactions with the controlled, sequential addition of reagents and/or catalysts throughout the course of the reaction.[7] The major challenge in the development of single-operation, catalysed cascade-reactions involving multiple organocatalysts is due to the incompatibility of reactants, intermediates, and catalyst-activated species with respect to the desired product outcome. Recently, a few such reactions have been successfully used in the synthesis of important chiral building blocks.[8] To make further advances in the field, our aim was 1) to develop a new, singly-operated, multiple-organocatalyst-promoted cascade reaction and 2) to combine covalent bond[9] and bifunctional-base/Brønsted acid catalysis[10] in a triple cascade reaction for the direct, asymmetric synthesis of fully substituted piperidines. Herein, we report our efforts towards meeting these challenges.

Our proposed cascade process is to couple three components, which comprise an aldehyde **1**, a nitroalkene **2**, and an imine **3**, to give product **4** in the presence of two commercially available, or readily prepared, organocatalysts, **5** and **6**.[11] The sequential transformation would involve three steps (Scheme 1). Initial activation of aldehyde **1**, by catalyst **5** (enamine activation), would facilitate selective addition to the hydrogen-bond-activated nitroalkene **2** in a Michael-type reaction.[12] In situ hydrolysis would liberate nitroalkane **2a**, which can then able to participate in the relay catalysis cycle. Catalyst **6** would promote the nitro-Mannich reaction of intermediate **2a** with imine **3** to generate the persubstituted *N*-Tos-protected aminoaldehyde **3a** (Tos = *p*-toluenesulfonyl), which can then undergo cyclization to form the final hemiaminal **4**. If successful, this sequence would constitute an atom economical,[13] selective,[14] and environ-



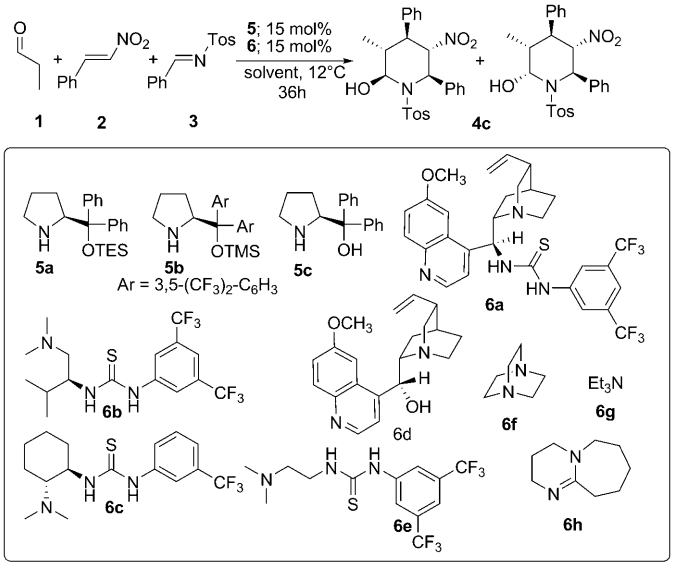
Scheme 1. Proposed reaction mechanism.

mentally friendly synthesis of fully substituted piperidines. Furthermore, it would be accomplished with readily available organocatalysts, from low cost, simple starting materials, and would exploit a rare nitro-Mannich reaction involving a hindered long-chain nitroalkane.[15]

To probe the feasibility of the proposed cascade process, preliminary experiments were performed using propionaldehyde, nitrostyrene and *N*-Tos-benzaldimine in the presence of catalysts **5a** and **6a** in dry toluene at 12 °C. Pleasingly, the cascade process proceeded smoothly to afford the desired product **4c** in 43 % yield and >99 % *ee* as, initially, a 1:1 mixture of α- and β-diastereomers that slowly equilibrated to a 2.5:1 mixture when left in [D_6]DMSO at room temperature for 18 h (Table 1, entry 1). This result led us to investigate the cascade reaction with various catalyst combinations and solvent conditions in order to improve the reaction efficiency (Table 1). The use of quinine **6d** and achiral compounds **6e–h** as catalysts resulted in lower yields (Table 1, entries 6–10). The best results were obtained using a combination of **5a** (15 mol %) and **6b** (15 mol %) in dry toluene at 12 °C. The product was then obtained in moderate yield and excellent stereoselectivity; it should be noted that under these conditions, aside from the diastereomers arising from the hemiaminal stereocenter, no other diastereomeric products were detectable in the crude reaction mixture (Table 1, entry 4; 56 % yield, >99:1 d.r., >99 % *ee*).

With optimal reaction conditions established, the scope of this triple cascade reaction was investigated. The fact that the residues R^1–R^3 of precursors **1**–**3**, respectively, can be varied (see Table 2) demonstrates the flexibility of our approach and allows the generation of a diverse range of reaction products. The moderate-to-good reaction yields and excellent enantioselectivities were independent of the electronic and structural characteristics of the substituents (electron-withdrawing, -donating, neutral, and heterocyclic). Likewise the substitution pattern (*ortho*-, *meta*-, and *para*-) of the substituents on aromatic rings had little-to-no adverse effect on the reaction efficiency and selectivity. The enantiomeric excesses of hemiaminal products **4k** and **4l** could not be determined directly by using chiral phase HPLC analysis. Accordingly, **4k** and **4l** were oxidized to their corresponding lactams and subsequently analysed to reveal that they were formed in excellent enantioselectivity (>99 % *ee*). Unfortunately, alkyl substrates did not produce good results under these catalytic conditions. However, we were pleased that,

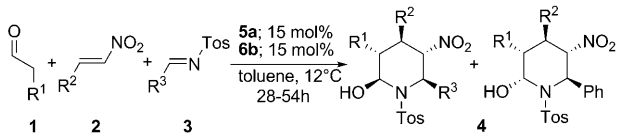
Table 1. Optimization of the cascade reaction.[a]



Entry	Catalysts	Solvent	Yield[b] [%]	*ee*[c] [%]
1	**5a + 6a**	toluene	43	>99
2	**5b + 6a**	toluene	15	>99
3	**5c + 6a**	toluene	8	>99
4	**5a + 6b**	toluene	56	>99
5	**5a + 6c**	toluene	32	>99
6	**5a + 6d**	toluene	12	>99
7	**5a + 6e**	toluene	29	>99
8	**5a + 6f**	toluene	<10	n.d.
9	**5a + 6g**	toluene	<10	n.d.
10	**5a + 6h**	toluene	<10	n.d.
11	**5a + 6b**	CH_2Cl_2	48	>99

[a] All the reactions were performed by using aldehyde **1** (0.4 mmol), nitroalkene **2** (0.4 mmol), imine **3** (0.2 mmol) and each organocatalyst (0.03 mmol) in dry solvent (0.4 mL) at 12 °C. [b] Yield of the isolated product. [c] Determined by chiral phase HPLC analysis.

Table 2. Survey of the reaction scope.[a]

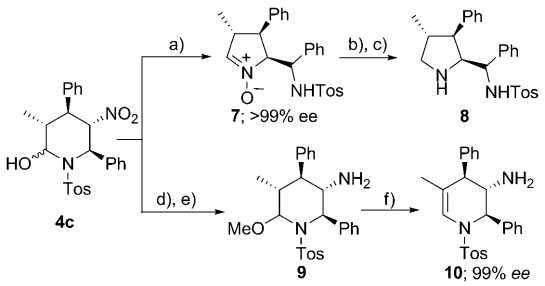


Entry	R^1	R^2	R^3	**4**	Yield[b] [%]	*ee*[c] [%]
1	nC_5H_{11}	Ph	Ph	**4a**	51	>99
2	Et	Ph	Ph	**4b**	65	>99
3	Me	Ph	Ph	**4c**	56	>99
4	Bn[d]	Ph	Ph	**4d**	48	98
5	Me	4-$CH_3C_6H_4$	Ph	**4e**	52	>99
6	Me	2-furyl	Ph	**4f**	65	>99
7	Me	3-ClC_6H_4	Ph	**4g**	61	>99
8	Me	Ph	3-FC_6H_4	**4h**	51	>99
9	Me	Ph	4-$CH_3C_6H_4$	**4i**	63	>99
10	Me	Ph	4-CNC_6H_4	**4j**	47	>99
11[e]	Me	2-BrC_6H_4	Ph	**4k**	54	>99
12[e]	Me	Ph	2-furyl	**4l**	71	>99

[a] Unless otherwise noted, all the reactions were performed by using aldehyde **1** (0.4 mmol), nitroalkene **2** (0.4 mmol), imine **3** (0.2 mmol), and each organocatalyst (0.03 mmol) in dry toluene (0.4 mL) at 12 °C. [b] Yield of the isolated product. [c] Determined by chiral phase HPLC analysis. [d] Bn = benzyl. [e] *ee* was determined by first oxidising to the corresponding lactam.

in the vast majority of cases (11 out of 12 examples in Table 2), complete enantiocontrol ($ee > 99\,\%$) was achieved. The absolute, and relative, configuration of the cascade-product hemiaminal **4k** was established by single-crystal X-ray crystallography and analysis of NMR data (see Supporting Information for details).[16]

To illustrate the value of this triple cascade reaction, we devised several useful transformations of product **4c** (Scheme 2). Partial reduction of piperidine **4c** with nickel



Scheme 2. Synthetic transformations of hemiaminal **4c**. a) $NiCl_2 \cdot 6H_2O$, $NaBH_4$, MeOH, 70 %; b) $NaBH_4$, EtOH; c) Fe, AcOH, 32 % over two steps; d) Ac_2O, pyridine, CH_2Cl_2, 94%; e) $NiCl_2 \cdot 6H_2O$, $NaBH_4$, MeOH, 56%; f) CF_3COOH, CH_2Cl_2, 73%

boride afforded the corresponding nitrone **7**, the structure of which was unambiguously established by single-crystal X-ray diffraction[16] (see Supporting Information for details), in good yield. Nitrones are reliable intermediates with a high value in a plethora of synthetic applications[17] and our approach provides rapid access to them. An two-step reduction of nitrone **7** then provided a route for the preparation of substituted pyrrolidine **8**. Pyrrolidines are important building blocks with attractive chemical and biological properties and this short route provides many opportunities. Importantly, these substituted pyrrolidines could be potential organocatalysts.[9c] Typical routes to single-enantiomer pyrrolidine organocatalysts rely on the manipulation of chiral-pool starting materials. After optimization, our route could provide a method for the discovery of new and effective pyrrolidine organocatalysts. In a further demonstration of the synthetic utility of the cascade products, the reduction of piperidine **4c** to the corresponding 3-amino derivative **9** was also achieved. Elimination of MeOH from **9** to form the valuable tetrahydropyridine derivative **10** was also facile. 3-Amino piperidine alkaloids are found extensively in nature and possess a wide range of biological activities;[1] thus, our route provides rapid access to a potentially diverse set of such compounds.

In conclusion, we have developed a new chemo-, diastereo-, and enantioselective three-component cascade reaction catalyzed by two organocatalysts to synthesize fully substituted piperidines. The merit of this cascade process is highlighted by its high efficiency in producing three new bonds and five new stereogenic centers in one operation, which is otherwise a major challenge. Notably, this cascade provides excellent enantiocontrol, atom economy, and the products are valuable for numerous synthetic applications. The possible anticancer activity of the synthesized piperidines, the catalytic activity of **8**, and the mechanistic course of the reaction are currently under investigation and will be reported in due course.

Experimental Section

General procedure for the triple cascade synthesis of piperidines: Freshly distilled aldehyde **1** (0.4 mmol) and imine **3** (0.2 mmol) were added to a solution of nitroalkene **2** (0.4 mmol) and organocatalysts **5a** (0.03 mmol, 11.0 mg, 15 mol %) and **6b** (0.03 mmol, 12.0 mg, 15 mol %) in dry toluene (0.4 mL) at 12 °C. After stirring for 28–54 h, the reaction was completed. A portion of the desired product, **4**, sometimes precipitated and Et_2O (0.5 mL) was added to the reaction mixture. Any precipitated product was then isolated by filtration, the filtrate concentrated and the residue purified by column chromatography to afford the remaining portion of the desired product, **4**. In cases in which no precipitation occurred, the reaction mixture was directly purified by flash column chromatography to afford the products.

Acknowledgements

We are grateful for the funding of the National Basic Research Program of China (No. 2010CB833200), the Foundation of NSFC (20772051, 20972058), and the "111" program from MOE of P. R. China. We would like to thank Syngenta for awarding a PhD studentship to Y.W.

Keywords: asymmetric catalysis · domino reactions · enantioselectivity · organocatalysis · piperidines

[1] For a review, see: M. Rubiralta, E. Giralt, A. Diez, *Piperidine: Structure, Preparation, Reactivity, and Synthetic Applications of Piperidine and Its Derivatives*, Elsevier, Amsterdam, **1991**.

[2] Y. Hayashi, H. Gotoh, R. Masui, H. Ishikawa, *Angew. Chem.* **2008**, *120*, 4076–4079; *Angew. Chem. Int. Ed.* **2008**, *47*, 4012–4015.

[3] a) B. Han, J.-L. Li, C. Ma, S.-J. Zhang, Y.-C. Chen, *Angew. Chem.* **2008**, *120*, 10119–10122; *Angew. Chem. Int. Ed.* **2008**, *47*, 9971–9974; b) B. Han, Z.-Q. He, J.-L. Li, R. Li, K. Jiang, T.-Y. Liu, Y.-C. Chen, *Angew. Chem.* **2009**, *121*, 5582–5585; *Angew. Chem. Int. Ed.* **2009**, *48*, 5474–5477.

[4] For selected recent examples, see: a) N. Sarkar, A. Banerjee, S. G. Nelson, *J. Am. Chem. Soc.* **2008**, *130*, 9222–9223; b) W. Zhu, M. Mena, E. Jnoff, N. Sun, P. Pasau, L. Ghosez, *Angew. Chem.* **2009**, *121*, 5994–5997; *Angew. Chem. Int. Ed.* **2009**, *48*, 5880–5883.

[5] S. Nara, R. Tanaka, J. Eishima, M. Hara, Y. Takahashi, S. Otaki, R. J. Foglesong, P. F. Hughes, S. Turkington, Y. Kanda, *J. Med. Chem.* **2003**, *46*, 2467–2473.

[6] For reviews, see: a) D. Enders, C. Grondal, M. R. M. Hüttl, *Angew. Chem.* **2007**, *119*, 1590–1601; *Angew. Chem. Int. Ed.* **2007**, *46*, 1570–1581; b) D. W. C. MacMillan, A. M. Walji, *Synlett* **2007**, 1477–1489; for selected recent examples of organocatalytic domino reactions, see: c) D. B. Ramachary, N. S. Chowdari, C. F. Barbas III, *Angew. Chem.* **2003**, *115*, 4365–4369; *Angew. Chem. Int. Ed.* **2003**, *42*, 4233–4237; d) N. Halland, P. S. Aburel, K.-A. Jørgensen, *Angew. Chem.* **2004**, *116*, 1292–1297; *Angew. Chem. Int. Ed.* **2004**, *43*, 1272–1277; e) J. Zhou, B. List, *J. Am. Chem. Soc.* **2005**, *127*, 15036–15037; f) M. Marigo, T. Schulte, J. Fränzen, K.-A. Jørgensen, *J. Am. Chem. Soc.* **2005**, *127*, 15710–15711; g) R. K. Kunz, D. W. C. MacMillan, *J. Am. Chem. Soc.* **2005**, *127*, 3240–3241; h) J. Casas, M. Engqvist, I. Ibra-

hem, B. Kaynak, A. Córdova, *Angew. Chem.* **2005**, *117*, 1367–1369; *Angew. Chem. Int. Ed.* **2005**, *44*, 1343–1345; i) D. Enders, M. R. M. Hüttl, C. Grondal, G. Raabe, *Nature* **2006**, *441*, 861–863; j) M. Rueping, A. P. Antonchick, T. Theissmann, *Angew. Chem.* **2006**, *118*, 3765–3768; *Angew. Chem. Int. Ed.* **2006**, *45*, 3683–3686; k) M. Marigo, S. Bertelsen, A. Landa, K.-A. Jørgensen, *J. Am. Chem. Soc.* **2006**, *128*, 5475–5479; l) W. Wang, H. Li, J. Wang, L. Zu, *J. Am. Chem. Soc.* **2006**, *128*, 10354–10355; m) D. Enders, M. R. M. Hüttl, J. Runsink, G. Raabe, B. Wendt, *Angew. Chem.* **2007**, *119*, 471–473; *Angew. Chem. Int. Ed.* **2007**, *46*, 467–469; n) C. E. Aroyan, S. J. Miller, *J. Am. Chem. Soc.* **2007**, *129*, 256–257; o) J. Wang, H. Li, H. Xie, L. Zu, X. Shen, W. Wang, *Angew. Chem.* **2007**, *119*, 9208–9211; *Angew. Chem. Int. Ed.* **2007**, *46*, 9050–9053; p) A. Carlone, S. Cabrera, M. Marigo, K.-A. Jørgensen, *Angew. Chem.* **2007**, *119*, 1119–1122; *Angew. Chem. Int. Ed.* **2007**, *46*, 1101–1104; q) Y. Hayashi, T. Okano, S. Aratake, D. Hazelard, *Angew. Chem.* **2007**, *119*, 5010–5013; *Angew. Chem. Int. Ed.* **2007**, *46*, 4922–4925; r) E. Reyes, H. Jiang, A. Milelli, P. Elsner, R. G. Hazell, K.-A. Jørgensen, *Angew. Chem.* **2007**, *119*, 9362–9365; *Angew. Chem. Int. Ed.* **2007**, *46*, 9202–9205; s) J. Zhou, B. List, *J. Am. Chem. Soc.* **2007**, *129*, 7498–7499; t) J. L. Vicario, S. Reboredo, D. Badía, L. Carrillo, *Angew. Chem.* **2007**, *119*, 5260–5262; *Angew. Chem. Int. Ed.* **2007**, *46*, 5168–5170; u) S. Cabrera, J. Aleman, P. Bolze, S. Bertelsen, K.-A. Jørgensen, *Angew. Chem.* **2008**, *120*, 127–131; *Angew. Chem. Int. Ed.* **2008**, *47*, 121–125; v) O. Penon, A. Carlone, A. Mazzanti, M. Locatelli, L. Sambri, G. Bartoli, P. Melchiorre, *Chem. Eur. J.* **2008**, *14*, 4788–4791; w) D. Enders, C. Wang, J. W. Bats, *Angew. Chem.* **2008**, *120*, 7649–7653; *Angew. Chem. Int. Ed.* **2008**, *47*, 7539–7542; x) M. Lu, D. Zhu, Y. P. Lu, Y. X. Hou, B. Tan, G. F. Zhong, *Angew. Chem.* **2008**, *120*, 10341–10345; *Angew. Chem. Int. Ed.* **2008**, *47*, 10187–10191; y) R.-G. Han, Y. Wang, Y.-Y. Li, P.-F. Xu, *Adv. Synth. Catal.* **2008**, *350*, 1474–1478; z) M. Rueping, A. Kuenkel, F. Tato, J. W. Bats, *Angew. Chem.* **2009**, *121*, 3754–3757; *Angew. Chem. Int. Ed.* **2009**, *48*, 3699–3702.

[7] For a timely review, see: a) J. Zhou, *Chem. Asian J.* **2010**, *5*, 422–434; for examples, see: b) Y. Huang, A. M. Walji, C. H. Larsen, D. W. C. MacMillan, *J. Am. Chem. Soc.* **2005**, *127*, 15051–15053; c) J. Vesely, I. Ibrahem, R. Rios, G.-L. Zhao, Y. Xu, A. Córdova, *Tetrahedron Lett.* **2007**, *48*, 2193–2198; d) Y. Chi, S. T. Scroggins, J. M. J. Fréchet, *J. Am. Chem. Soc.* **2008**, *130*, 6322–6323; e) L. Albrecht, B. Richter, C. Vila, H. Krawczyk, K.-A. Jørgensen, *Chem. Eur. J.* **2009**, *15*, 3093–3102; T. Akiyama, T. Katoh, K. Mori, *Angew. Chem.* **2009**, *121*, 4290–4292; *Angew. Chem. Int. Ed.* **2009**, *48*, 4226–4228; f) H. Jiang, P. Elsner, K. L. Jensen, A. Falcicchio, V. Marcos, K.-A. Jørgensen, *Angew. Chem.* **2009**, *121*, 6976–6980; *Angew. Chem. Int. Ed.* **2009**, *48*, 6844–6848; g) B. Simmons, A. M. Walji, D. W. C. MacMillan, *Angew. Chem.* **2009**, *121*, 4413–4417; *Angew. Chem. Int. Ed.* **2009**, *48*, 4349–4353.

[8] a) Y. Wang, R.-G. Han, Y.-L. Zhao, S. Yang, P.-F. Xu, D. J. Dixon, *Angew. Chem.* **2009**, *121*, 10018–10022; *Angew. Chem. Int. Ed.* **2009**, *48*, 9834–9838; b) S. P. Lathrop, T. Rovis, *J. Am. Chem. Soc.* **2009**, *131*, 13628–13630; c) S. T. Scroggins, Y. Chi, J. M. J. Fréchet, *Angew. Chem.* **2009**, DOI: 10.1002/ange.200902945; *Angew. Chem. Int. Ed.* **2009**, DOI: 10.1002/anie.200902945.

[9] For selected reviews, see: a) P. I. Dalko, *Enantioselective Organoctalysis*, Wiley-VCH, Weinheim, **2007**; b) A. Dondoni, A. Massi, *Angew. Chem.* **2008**, *120*, 4716–4739; *Angew. Chem. Int. Ed.* **2008**, *47*, 4638–4660; c) S. Mukherjee, J. W. Yang, S. Hoffmann, B. List, *Chem. Rev.* **2007**, *107*, 5471–5569; d) A. Erkkilä, I. Majander, P. M. Pihko, *Chem. Rev.* **2007**, *107*, 5416–5470.

[10] For selected reviews, see: a) M. S. Taylor, E. N. Jacobsen, *Angew. Chem.* **2006**, *118*, 1550–1573; *Angew. Chem. Int. Ed.* **2006**, *45*, 1520–1543; b) A. G. Doyle, E. N. Jacobsen, *Chem. Rev.* **2007**, *107*, 5713–5743; c) T. Akiyama, *Chem. Rev.* **2007**, *107*, 5744–5758.

[11] a) S. H. McCooey, S. J. Connon, *Angew. Chem.* **2005**, *117*, 6525–6528; *Angew. Chem. Int. Ed.* **2005**, *44*, 6367–6370; b) J. Ye, D. J. Dixon, P. S. Hynes, *Chem. Commun.* **2005**, 4481–4482; c) T. Okino, Y. Hoashi, T. Furukawa, X. Xu, Y. Takemoto, *J. Am. Chem. Soc.* **2005**, *127*, 119–125; d) P. S. Hynes, P. A. Stupple, D. J. Dixon, *Org. Lett.* **2008**, *10*, 1389–1391; e) J. M. Andrés, R. Manzano, R. Pedrosa, *Chem. Eur. J.* **2008**, *14*, 5116–5119.

[12] a) For the pioneering work of direct catalytic asymmetric Michael additions of aldehydes to nitroolefins, see: J. M. Betancort, C. F. Barbas III, *Org. Lett.* **2001**, *3*, 3737–3740; b) for diphenylprolinol silyl ether catalyzed Michael additions of aldehydes to nitroolefins, see: Y. Hayashi, H. Gotoh, T. Hayashi, M. Shoji, *Angew. Chem.* **2005**, *117*, 4284–4287; *Angew. Chem. Int. Ed.* **2005**, *44*, 4212–4215.

[13] B. M. Trost, *Science* **1991**, *254*, 1471–1477.

[14] B. M. Trost, *Science* **1983**, *219*, 245–250.

[15] For reviews, see: a) B. Westermann, *Angew. Chem.* **2003**, *115*, 161–163; *Angew. Chem. Int. Ed.* **2003**, *42*, 151–153; b) E. Marqués-López, P. Merino, T. Tejero, R. P. Herrera, *Eur. J. Org. Chem.* **2009**, 2401–2420.

[16] CCDC-719367 (**4k**) and 719368 (**7**) contain 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.

[17] P. Merino in *Science of Synthesis, Vol. 27* (Ed.: A. Padwa), Thieme, Stuttgart, **2004**, p. 511.

Received: January 26, 2010
Published online: March 5, 2010

