DOI: 10.1002/adsc.200800452

Efficient Stereoselective Synthesis of Nitrocyclopropanes by the Oxidative Cyclization of Michael Adducts of Nitroolefins with Activated Methylene Compounds

Renhua Fan,[a,*] Yang Ye,[a] Weixun Li,[a] and Lingfei Wang[a]

[a] Department of Chemistry, Fudan University, 220 Handan Road, Shanghai 200433, People's Republic of China
Fax: (+86)-216-510-2412; e-mail: rhfan@fudan.edu.cn

Received: July 22, 2008; Published online: October 16, 2008

Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/adsc.200800452.

Abstract: An efficient oxidative cyclopropanation of the Michael adducts of nitroolefins with activated methylene compounds by the combination of iodobenzene diacetate and tetrabutylammonium iodide is reported. Highly functionalized nitrocyclopropanes are synthesized in moderate to good yields *via* the Michael addition and cyclopropanation with high diastereoselectivity and enantioselectivity under mild conditions.

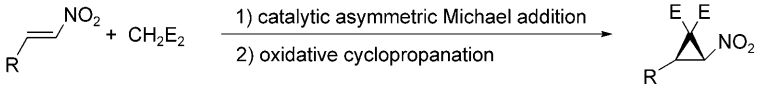
Keywords: cyclization; cyclopropanes; hypervalent compounds; Michael addition; oxidation; stereoselectivity

Nitrocyclopropanes are versatile intermediates in organic synthesis.[1] They not only appear as substructures in a variety of biologically active natural and unnatural compounds, for example, peptide-lactone hormaomycin[2] and nitropyrethroids,[3] but also can undergo transformations to other useful synthetic building blocks, e.g., the conformationally restricted aminocyclopropanecarboxylic acids.[4] Nitrocyclopropanes are most often prepared by the intermolecular addition of nitrodiazo esters,[5] ylides,[6] diazoalkanes,[7] or carbenoids[8] to unsaturated systems, or intramolecular cyclization of γ-nitroalkanols[9] or γ-halo nitro compounds.[10] However, compared to the extensive studies on asymmetric cyclopropanations,[11,12] the efficient catalytic asymmetric synthesis of functionalized nitrocyclopropanes is underdeveloped.

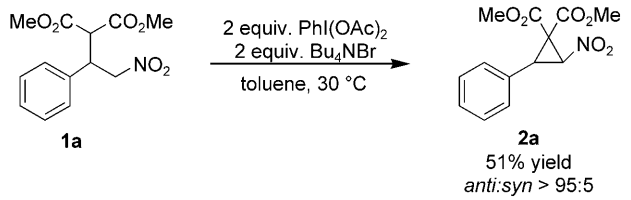
Catalytic asymmetric Michael additions of activated methylene compounds to nitroolefins to afford the Michael adducts in high enantioselectivity have been achieved.[13] The development of an expedient and efficient method for the synthesis of enantiomerically enriched functionalized nitrocyclopropanes by the direct cyclization of the corresponding Michael adducts, if applicable, will be desirable in many applications (Scheme 1).

Recently, Connon et al. reported the stereoselective synthesis of nitrocyclopropanes *via* a one-pot reaction involving the organocatalytic asymmetric Michael addition of dimethyl chloromalonate to nitroolefins, followed by cyclization in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) under carefully controlled reaction conditions.[14] However, the use of chloromalonate resulted the low enantioselectivity (7–47% *ee*), and highly toxic hexamethylphosphoramide (HMPA) was used as the solvent to provide efficient ring closure. On the other hand, although the asymmetric Michael addition of dimethyl malonate to nitroolefins gave the Michael adduct **1a** in high enantioselectivity,[13a] it could not be converted into the corresponding cyclopropane *via* bromination in the presence of a base.

As a part of our development of the synthetic applications of hypervalent iodine compounds,[15] we were delighted to find that the oxidative cyclopropanation of **1a** occurred in the presence of $PhI(OAc)_2$ and Bu_4NBr, and afforded cyclopropane **2a** in 51% yield with high diastereoselectivity after 12 h (Scheme 2).



Scheme 1.



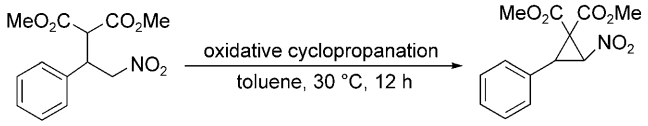
Scheme 2.

The presence of a strong base was not good for the cyclopropanation (Table 1, entry 1). As a strained-ring compound, cyclopropane **2a** was sensitive to the basic conditions, and it decomposed when the reaction was left for a longer time (Table 1, entry 2). The yield of **2a** increased to 78% using Bu_4NI (Table 1, entry 4), while no cyclopropane was detected in the presence of Bu_4NCl (Table 1, entry 3). The best ratio of **1a**, $PhI(OAc)_2$, and Bu_4NI was 1:1.5:1.5, with which the yield of **2a** increased to 93% (Table 1, entry 6). Catalytic amounts of Bu_4NI were not enough to finish the cyclopropanation (Table 1, entries 8 and 9). The reaction proceeded sluggishly at 0 °C (Table 1, entry 10). When the reaction mixture was warmed to 50 °C, the reaction time was shortened to 2 h, but a lower yield of **2a** was obtained (Table 1, entry 11). It was noteworthy that, when the reaction was carried out in an air-open system with untreated toluene as the solvent, the reaction still proceeded smoothly, and gave rise to cyclopropane **2a** in a slightly lower yield (Table 1, entry 12).

Several control experiments were conducted to investigate the possible reaction pathway. During the reaction, the formation of iodine was observed. However, no cyclopropanation was detected when I_2, the combination of K_2CO_3 with I_2, or $PhI(OAc)_2$ with I_2 was used (Table 1, entries 13–15). The combinations of H_2O_2 with Bu_4NI, and NIS with AcOH were also not effective to the cyclopropanation (Table 1, entries 16 and 17), while cyclopropane **2a** was obtained in 45% yield using *m*-CPBA and Bu_4NI under the same conditions (Table 1, entry 18).

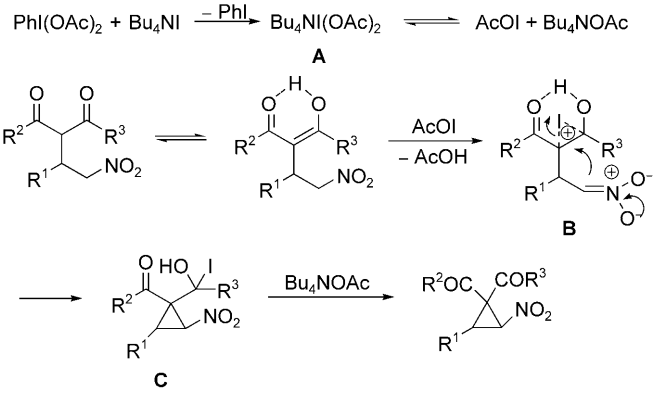
A proposed reaction pathway for the oxidative cyclopropanation is outlined in Scheme 3. The reaction

Table 1. Optimization of the cyclopropanation of **1a**.



Entry	Reagent (equivalents)	**2a** [%][a]	*dr*[f]
1	$PhI(OAc)_2$ (2), Bu_4NBr (2), *t*BuOK (1)	18	>95:5
2	$PhI(OAc)_2$ (2), Bu_4NBr (2), *t*BuOK (1)	0[b]	
3	$PhI(OAc)_2$ (2), Bu_4NCl (2)	0	
4	$PhI(OAc)_2$ (2), Bu_4NI (2)	78	>95:5
5	$PhI(OAc)_2$ (3), Bu_4NI (3)	65	>95:5
6	$PhI(OAc)_2$ (1.5), Bu_4NI (1.5)	93	>95:5
7	$PhI(OAc)_2$ (1), Bu_4NI (1)	67	>95:5
8	$PhI(OAc)_2$ (1), Bu_4NI (0.5)	45	>95:5
9	$PhI(OAc)_2$ (1.5), Bu_4NI (0.25)	21	>95:5
10	$PhI(OAc)_2$ (1.5), Bu_4NI (1.5)	<5[c]	
11	$PhI(OAc)_2$ (1.5), Bu_4NI (1.5)	74[d]	88:12
12	$PhI(OAc)_2$ (1.5), Bu_4NI (1.5)	88[e]	>95:5
13	I_2 (1.5)	0	
14	K_2CO_3 (1.5), I_2 (1.5)	0	
15	$PhI(OAc)_2$ (1.5), I_2 (1.5)	0	
16	H_2O_2 (1.5), Bu_4NI (1.5)	0	
17	NIS (1.5), AcOH (1.5)	0	
19	*m*-CPBA (1.5), Bu_4NI (1.5)	45	83:17

[a] Isolated yield based on **1a**.
[b] The reaction was stirred at 30 °C for 24 h.
[c] The reaction was carried out at 0 °C.
[d] The reaction was carried out at 50 °C.
[e] The reaction was carried out in an air-open system with untreated toluene as the solvent.
[f] The ratio was determined by ^1H NMR.

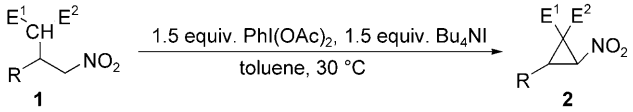


Scheme 3.

of $PhI(OAc)_2$ with Bu_4NI gives a tetrabutylammonium salt of diacetoxyiodine(I) **A**,[16] which can be regarded as an acetyl hypoiodite equivalent.[17] In the presence of intermediate **A** or acetyl hypoiodite, the enolate of the Michael adduct is converted into the intermediate **B**, which undergoes an intramolecular cyclization to afford the intermediate **C**. After the elimination of HI in the presence of Bu_4NOAc, the corresponding highly functionalized nitrocyclopropane is formed.

The generality of the present $PhI(OAc)_2/Bu_4NI$-induced cyclopropanation of Michael adducts was further explored under the optimized conditions (Table 2). The reactions of substrates with an aromatic or heteroaromatic ring proceeded smoothly, and gave the corresponding cyclopropanes in moderate to excellent yields (entries 1–12). Compounds with an

Table 2. $PhI(OAc)_2/Bu_4NI$-induced cyclopropanation of Michael adducts.



Entry	R	E^1	E^2	2	Yield [%][a]	*dr*[b]
1	C_6H_5	COOMe	COOMe	**2a**	93	>95:5
2	C_6H_5	COOEt	COOEt	**2b**	88	>95:5
3	4-CH_3O-C_6H_4	COOEt	COOEt	**2c**	64	>95:5
4	4-CH_3-C_6H_4	COOEt	COOEt	**2d**	78	>95:5
5	4-Cl-C_6H_4	COOEt	COOEt	**2e**	98	>95:5
6	4-F-C_6H_4	COOEt	COOEt	**2f**	80	>95:5
7	4-NO_2-C_6H_4	COOEt	COOEt	**2g**	91	>95:5
8	2-Br-C_6H_4	COOEt	COOEt	**2h**	88	>95:5
9	1-naphthyl	COOEt	COOEt	**2i**	87	>95:5
10	2-furanyl	COOEt	COOEt	**2j**	55	>95:5
11	2-thiophenyl	COOEt	COOEt	**2k**	47	>95:5
12	2-pyridinyl	COOEt	COOEt	**2l**	88	91:9
13	$CH_3(CH_2)_4$	COOEt	COOEt	**2m**	78	>95:5
14	$(CH_3)_2CHCH_2$	COOEt	COOEt	**2n**	88	>95:5
15	C_6H_5	COOBu-*t*	COOBu-*t*	**2o**	38	94:6
16	C_6H_5	$COCH_3$	COOEt	**2p**	52	>95:5
17	C_6H_5	$COCH_3$	$COCH_3$	**2q**	66	>95:5
18	C_6H_5	NO_2	COOEt	**2r**	0	
19	C_6H_5	CN	COOEt	**2s**	0	
20	C_6H_5	CN	CN	**2t**	0	

[a] Isolated yield based on **1**.
[b] The ratio was determined by ^1H NMR.

electron-withdrawing substituent were better substrates than those with an electron-donating substituent. Compounds derived from aliphatic nitroolefins were also good substrates (entries 13 and 14). The reaction of sterically hindered di-*tert*-butyl malonate derivative required a longer time, and gave a low yield (entry 15). Moreover, the reactions with ethyl acetoacetate and 2,4-pentanedione derivatives also gave the corresponding cyclopropanes in moderate yields (entries 16 and 17). All 1,1-dicarbonyl-2-nitrocyclopropanes were formed with high diastereoselectivity. The *anti* relative stereochemistry was confirmed by ^1H NMR and the comparison with known compounds.[14] No corresponding cyclopropanes were formed when ethyl 2-nitroacetate, ethyl 2-cyanoacetate, and malononitrile derivatives were employed (entries 18–20).

Enantiomerically enriched Michael adducts were prepared by the asymmetric addition of 1,3-dicarbonyl compounds to nitroolefins catalyzed by the bifunctional thiourea **3**,[13b] and then underwent the oxidative cyclopropanation under the optimized condition (Table 3). The corresponding functionalized nitrocyclopropanes were obtained in moderate to good yields over two steps with high diastereoselectivity and enantioselectivity. No racemization was observed during the cyclopropanation.

β-Aminocyclopropanecarboxylic acids are the conformationally most rigid cycloalkyl bridged β-alanine derivatives and are therefore of special interest.[18] The synthetically relevant reduction of 1,1-dicarbonyl-2-nitro-cyclopropane **2a** to afford the corresponding β-aminocyclopropane-1,1-dicarboxylate **4a** can be achieved (Scheme 4).[5b]

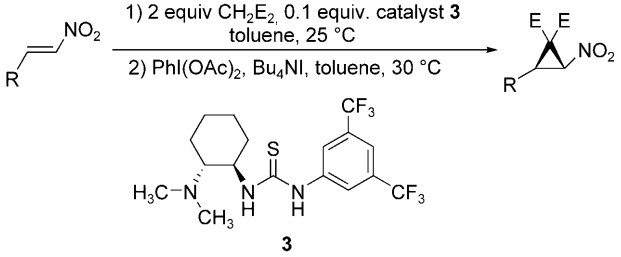
In summary, we report here an efficient oxidative cyclopropanation of the Michael adducts of nitroolefins with activated methylene compounds by the combination of iodobenzene diacetate and tetrabutylammonium iodide. Highly functionalized nitrocyclopropanes are synthesized in moderate to good yields with high diastereoselectivity and enantioselectivity. The potential of this reaction system can be evaluated by its mild conditions and simple process. Studies on the scope, mechanism, and synthetic application are ongoing and will be reported in due course.

Experimental Section

Typical Experimental Procedure

The mixture of **1a** (56 mg, 0.2 mmol) and $PhI(OAc)_2$ (97 mg, 0.3 mmol) in toluene (1 mL) was treated with Bu_4NI (111 mg, 0.3 mmol). The reaction was allowed to stir at

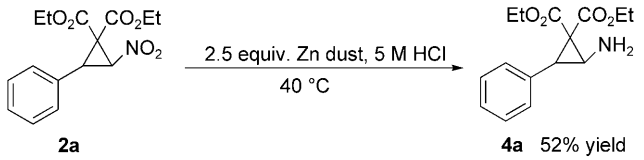
Table 3. Stereoselective synthesis of enantiomerically enriched functionalized nitrocyclopropanes.



3

Entry	R	E^1	E^2	**2**	Yield [%][a]	*ee* [%][b]	*dr*[c]
1	C_6H_5	COOEt	COOEt	**2b**	73	87	>95:5
2	4-CH_3O-C_6H_4	COOEt	COOEt	**2c**	52	89	>95:5
3	4-Cl-C_6H_4	COOEt	COOEt	**2e**	87	90	>95:5
4	4-F-C_6H_4	COOEt	COOEt	**2f**	71	93	>95:5
5	2-Br-C_6H_4	COOEt	COOEt	**2h**	81	94	>95:5
6	1-naphthyl	COOEt	COOEt	**2i**	80	93	>95:5
7	2-thiophenyl	COOEt	COOEt	**2k**	35	89	>95:5
8	C_6H_5	COOMe	COOMe	**2a**	81	92	>95:5
9	C_6H_5	$COCH_3$	$COCH_3$	**2q**	60	86	>95:5

[a] Isolated yield based on nitroolefins over two steps.
[b] Determined by HPLC (see Supporting Information).
[c] The ratio was determined by ^{1}H NMR.



2a **4a** 52% yield

Scheme 4.

30 °C for 12 h. Upon completion as shown by TLC, the reaction mixture was directly purified by flash column chromatography (5–10% ethyl acetate in hexanes) to provide the corresponding nitrocyclopropane **2a**; yield: 93%. The characterization data are available in the Supporting Information file.

Acknowledgements

Financial support from National Natural Science Foundation of China (20702006), the Shanghai Rising-Star program (07QA14007), and Fudan University are gratefully acknowledged.

References

[1] a) E. M. Budynina, O. A. Ivanova, E. B. Averina, T. S. Kuznetsova, N. S. Zefirov, *Tetrahedron Lett.* **2006**, *47*, 647–649; b) R. P. Wurz, A. B. Charette, *J. Org. Chem.* **2004**, *69*, 1262–1269; c) P. Rademacher, T. Coskun, K. Kowski, O. Larionov, A. de Meijere, *Chem. Eur. J.* **2003**, *9*, 2953–2962; d) O. V. Larionov, T. F. Savel′eva, K. A. Kochetkov, N. S. Ikonnokov, S. I. Kozhushkov, D. S. Yufit, J. A. K. Howard, V. N. Khrustalev, Y. N. Belokon, A. de Meijere, *Eur. J. Org. Chem.* **2003**, 869–877; e) J. Zindel, A. Zeeck, W. A. Koenig, A. de Meijere, *Tetrahedron Lett.* **1993**, *34*, 1917–1920; f) T. Vettiger, D. Seebach, *Liebigs Ann. Chem.* **1990**, 195–201; g) P. E. O'Bannon, W. P. Dailey, *J. Org. Chem.* **1990**, *55*, 353–355; h) P. E. O'Bannon, W. P. Dailey, *J. Am. Chem. Soc.* **1989**, *111*, 9244–9245; i) H. H. Baer, U. Williams, B. Radatus, *Carbohydr. Res.* **1988**, *174*, 291–303; j) P. A. Wade, W. P. Dailey, P. J. Carroll, *J. Am. Chem. Soc.* **1987**, *109*, 5452–5456; k) J. B. Lambert, J. J. Napoli, K. K. Johnson, K. N. Taba, *J. Org. Chem.* **1985**, *50*, 1291–1295; l) F. G. Bordwell, J. E. Bartmess, J. A. Hautala, *J. Org. Chem.* **1978**, *43*, 3113–3116; m) S. Ranganathan, C. S. Panda, *Tetrahedron Lett.* **1971**, *12*, 3841–3842; n) P. E. Iversen, *Chem. Ber.* **1971**, *104*, 2195–2198; o) E. P. Kohler, H. E. Williams, *J. Am. Chem. Soc.* **1919**, *41*, 1644–1655.

[2] a) B. D. Zlatopolskiy, K. Loscha, P. Alvermann, S. I. Kozhushkov, S. V. Nikolaev, A. Zeeck, A. de Meijere, *Chem. Eur. J.* **2004**, *10*, 4708–4717; b) J. Zindel, A. de Meijere, *J. Org. Chem.* **1995**, *60*, 2968–2973.

[3] M. K. Chyan, S. J. Norton, *J. Agric. Food Chem.* **1995**, *43*, 2286–2290.

[4] a) F. Fülöp, *Chem. Rev.* **2001**, *101*, 2181–2204; b) J. Zindel, A. de Meijere, *Synthesis* **1993**, 190–194.

[5] a) B. Moreau, A. B. Charette, *J. Am. Chem. Soc.* **2005**, *127*, 18014–18015; b) R. P. Wurz, A. B. Charette, *J. Org. Chem.* **2004**, *69*, 1262–1269.

[6] a) H. T. Bonge, T. Hansen, *Synlett* **2007**, 55–58; b) J. Hübner, J. Liebscher, M. Pätzel, *Tetrahedron* **2002**, *58*, 10485–10500; c) V. K. Aggarwal, H. W. Smith, G. Hynd, R. V. H. Jones, R. Fieldhouse, S. E. Spey, *J. Chem. Soc. Perkin Trans. 1* **2000**, 3267–3276; d) G. Galley, J. Hübner, S. Anklam, P. G. Jones, M. Pätzel,

Tetrahedron Lett. **1996**, *37*, 6307–6310; e) G. Kumaran, G. H. Kulkarni, *Synthesis* **1995**, 1545–1548; f) C. R. Johnson, J. P. Lockard, E. R. Kennedy, *J. Org. Chem.* **1980**, *45*, 264–271; g) J. Asunskis, H. Shechter, *J. Org. Chem.* **1968**, *33*, 1164–1168.

[7] a) P. E. O'Bannon, W. P. Dailey, *Tetrahedron* **1990**, *46*, 7341–7358; b) P. E. O'Bannon, W. P. Dailey, *Tetrahedron Lett.* **1988**, *29*, 987–990; c) W. E. Parham, H. G. Braxton, C. Serres, *J. Org. Chem.* **1961**, *26*, 1831–1834.

[8] P. A. Wade, W. P. Dailey, P. J. Carroll, *J. Am. Chem. Soc.* **1987**, *109*, 5452–5456.

[9] a) J. Yu, J. R. Falck, *J. Org. Chem.* **1992**, *57*, 3757; b) B. Radatus, U. Williams, H. H. Baer, *Carbohydr. Res.* **1986**, *157*, 242–250.

[10] a) R. Ballini, D. Fiorini, A. Palmieri, *Synlett* **2003**, 1704–1706; b) G. A. Russell, M. Makosza, J. Hershberger, *J. Org. Chem.* **1979**, *44*, 1195–1199.

[11] a) H. Xie, L. Zu, H. Li, J. Wang, W. Wang, *J. Am. Chem. Soc.* **2007**, *129*, 10886–10894; b) H. Kakei, T. Sone, Y. Sohtome, S. Matsunaga, M. Shibasaki, *J. Am. Chem. Soc.* **2007**, *129*, 13410–13411; c) X. Deng, P. Cai, S. Ye, X. L. Sun, W. W. Liao, K. Li, Y. Tang, Y. D. Wu, L. X. Dai, *J. Am. Chem. Soc.* **2006**, *128*, 9730; d) V. K. Aggarwal, E. Grange, *Chem. Eur. J.* **2006**, *12*, 568; e) C. C. C. Johansson, N. Bremeyer, S. V. Ley, D. R. Owen, S. C. Smith, M. J. Gaunt, *Angew. Chem. Int. Ed.* **2006**, *45*, 6024; f) H. Jiang, X. Deng, X. L. Sun, Y. Tang, L. X. Dai, *J. Org. Chem.* **2005**, *70*, 15202; g) J. C. Zheng, W. W. Liao, Y. Tang, X. L. Sun, L. X. Dai, *J. Am. Chem. Soc.* **2005**, *127*, 12222; h) R. K. Kunz, D. W. C. MacMillan, *J. Am. Chem. Soc.* **2005**, *127*, 3240; i) N. Bremeyer, S. C. Smith, S. V. Ley, M. J. Gaunt, *Angew. Chem. Int. Ed.* **2004**, *43*, 2681; j) C. D. Papageorgiou, S. V. Ley, M. J. Gaunt, *Angew. Chem. Int. Ed.* **2003**, *42*, 828; k) V. K. Aggarwal, E. Alsono, G. Fang, M. Ferrara, G. Hynd, M. Porcelloni, *Angew. Chem. Int. Ed.* **2001**, *40*, 1433; l) V. K. Aggarwal, H. W. Smith, R. V. H. Jones, R. Fieldhouse, *Chem. Commun.* **1998**, 1785; m) A. H. Li, L. X. Dai, V. K. Aggarwal, *Chem. Rev.* **1997**, *97*, 2341; n) Y. Tang, Y. Z. Huang, L. X. Dai, J. Sun, W. Xia, *J. Org. Chem.* **1997**, *62*, 954.

[12] a) H. M. Hansen, D. A. Longbottom, S. V. Ley, *Chem. Commun.* **2006**, 4838; b) Y. Kazuta, A. Matsuda, S. Shuto, *J. Org. Chem.* **2002**, *67*, 1669; c) P. Langer, I. Freifeld, *Org. Lett.* **2001**, *3*, 3903; d) S. Arai, K. Nakayama, T. Ishida, T. Shioiri, *Tetrahedron Lett.* **1999**, *40*, 4215; e) D. Moye-Sherman, S. Jin, I. Ham, D. Lim, J. M. Scholtz, K. Burgess, *J. Am. Chem. Soc.* **1998**, *120*, 9435; f) S. Ramaswamy, K. Prasad, O. Repic, *J. Org. Chem.* **1992**, *57*, 6344; g) K. Burgess, K. K. Ho, *J. Org. Chem.* **1992**, *57*, 5931; h) M. C. Pirrung, S. E. Dunlap, U. P. Trinks, *Helv. Chim. Acta* **1989**, *72*, 1301; i) Y. Gao, K. B. Sharpless, *J. Am. Chem. Soc.* **1988**, *110*, 7538; j) G. Quinkert, U. Schwartz, H. Stark, W. D. Weber, F. Adam, H. Baier, G. Frank, G. Dürner, *Liebigs Ann. Chem.* **1982**, 1999; k) D. E. McClure, B. H. Arison, J. J. Baldwin, *J. Am. Chem. Soc.* **1979**, *101*, 3666.

[13] a) S. H. McCooey, S. J. Connon, *Angew. Chem. Int. Ed.* **2005**, *44*, 6367–6370; b) T. Okino, Y. Hoashi, T. Furukawa, X. Xu, Y. Takemoto, *J. Am. Chem. Soc.* **2005**, *127*, 119–125; c) M. Watanabe, A. Ikagawa, H. Wang, K. Murata, T. Ikariya, *J. Am. Chem. Soc.* **2004**, *126*, 11148–11149; d) W. Notz, F. Tanaka, C. F. Barbas III, *Acc. Chem. Res.* **2004**, *37*, 580–591; e) H. Li, Y. Wang, L. Tang, L. Deng, *J. Am. Chem. Soc.* **2004**, *126*, 9906–9907; f) T. Okino, Y. Hoashi, Y. Takemoto, *J. Am. Chem. Soc.* **2003**, *125*, 12672–12673; g) D. M. Barnes, J. Ji, M. G. Fickes, M. A. Fitzgerald, S. A. King, H. E. Morton, F. A. Plagge, M. Preskill, S. H. Wagaw, S. J. Wittenberger, J. Zhang, *J. Am. Chem. Soc.* **2002**, *124*, 13097–13105; h) O. M. Berner, L. Tedeschi, D. Enders, *Eur. J. Org. Chem.* **2002**, 1877–1894; i) N. Krause, A. Hoffmann-Röder, *Synthesis* **2001**, 171–196; j) M. P. Sibi, S. Manyem, *Tetrahedron* **2000**, *56*, 8033–8061; k) J. Ji, D. M. Barnes, J. Zhang, S. A. King, S. J. Wittenberger, H. E. Morton, *J. Am. Chem. Soc.* **1999**, *121*, 10215–10216.

[14] S. H. McCooey, T. McCabe, S. J. Connon, *J. Org. Chem.* **2006**, *71*, 7494–7497.

[15] a) R. Fan, Y. Ye, *Adv. Synth. Catal.* **2008**, *350*, 1526–1530; b) R. Fan, W. Li, Y. Ye, L. Wang, *Adv. Synth. Catal.* **2008**, *350*,1531–1536; c) R. Fan, D. Pu, J. Gan, B. Wang, *Tetrahedron Lett.* **2008**, *49*, 4925–4928; d) R. Fan, D. Pu, F. Wen, *J. Org. Chem.* **2007**, *72*, 8994–8997; e) R. Fan, W. Wang, D. Pu, J. Wu, *J. Org. Chem.* **2007**, *72*, 5905–5907; f) R. Fan, F. Wen, L. Qin, D. Pu, B. Wang, *Tetrahedron Lett.* **2007**, *48*, 7444–7447.

[16] a) A. Kirschning, C. Plumeier, L. Rose, *Chem. Commun.* **1998**, 33–34; b) G. Doleschall, G. Tóth, *Tetrahedron* **1980**, *36*, 1649–1665; c) C. Szántay, G. Blaskó, M. Bárczai-Beke, P. Péchy, G. Dörnyei, *Tetrahedron Lett.* **1980**, *21*, 3509–3512.

[17] For reviews of hypervalent iodine, see: a) R. M. Moriarty, *J. Org. Chem.* **2005**, *70*, 2893; b) P. J. Stang, *J. Org. Chem.* **2003**, *68*, 2997; c) V. V. Zhdankin, P. J. Stang, *Chem. Rev.* **2002**, *102*, 2523; d) P. J. Stang, *Chem. Rev.* **1996**, *96*, 1123; e) R. M. Moriarty, R. K. Vaid, *Synthesis* **1990**, 431.

[18] a) S. Yamazaki, T. Inoue, T. Hamada, T. Takada, K. Yamamoto, *J. Org. Chem.* **1999**, *64*, 282; b) A. A. Tishkov, A. V. Kozintsev, I. M. Lyapkalo, S. L. Ioffe, V. V. Kachala, Y. A. Strelenko, V. A. Tartakovsky, *Tetrahedron Lett.* **1999**, *40*, 5075; c) J. Voigt, M. Noltemeyer, O. Reiser, *Synlett* **1997**, 202; d) R. Csuk, Y. Scholz, *Tetrahedron* **1996**, *52*, 6383; e) R. Csuk, Y. Scholz, *Tetrahedron* **1995**, *51*, 7193; f) J. G. Cannon, J. E. Garst, *J. Org. Chem.* **1975**, *40*, 182; g) C. C. Shroff, W. S. Stewart, S. J. Uhm, J. W. Wheeler, *J. Org. Chem.* **1971**, *36*, 3356.

