Copper(I) Chloride Mediated Cyclization of Optically Active 2,3-Allenoic Acids or 1:1 Salts of Optically Active 2,3-Allenoic Acids with Chiral Amines

Shengming Ma,* Zhanqian Yu

State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Lu, Shanghai 200032, P. R. of China
E-mail: masm@mail.sioc.ac.cn

Received 1 May 2006; revised 26 June 2006

Abstract: Copper(I) chloride mediated cyclization of optically active 2,3-allenoic acids or 1:1 salts of optically active 2,3-allenoic acids with chiral amines was developed. The reaction of optically active 2,3-allenoic acids in methanol requires only a catalytic amount of copper(I) chloride, while the reaction of the 1:1 salts of optically active 2,3-allenoic acids with chiral amines needs one equivalent of copper(I) chloride and should be carried out in dichloromethane to ensure high efficiency of chirality transfer.

Key words: chirality transfer, cyclization, copper(I) chloride, amines, 2,3-allenoic acids

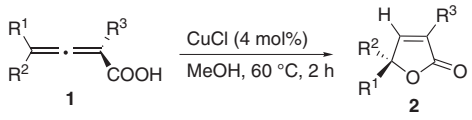
Butenolides make up a class of interesting compounds with potential biological activities,[1] and thus, much attention has been paid to the development of new synthetic methodologies to prepare these compounds.[2] Among these, cyclization involving 2,3-allenoic acids and their derivatives is one of the most efficient pathways.[3,4] In this paper, we wish to report the cycloisomerization of optically active 2,3-allenoic acids and 1:1 salts of optically active 2,3-allenoic acids with chiral amines.[5]

Under the catalysis of copper(I) chloride (4 mol%),[6] cycloisomerization of optically active 2,3-allenoic acids afforded the optically active butenolides **2a** and **2b** with very highly efficient chirality transfer. Four examples are listed in Table 1.

Since the optically active 2,3-allenoic acids were easily prepared by the resolution of racemic 2,3-allenoic acids with optically active amines,[5] we explored the possibility of using the 1:1 acid–chiral amine salts directly as the starting point. However, when the same reaction conditions were applied to the 1:1 salt of (*S*)-2-methyl-4-phenylbuta-2,3-dienoic acid with the naturally occurring cinchonidine (**3a**) (Table 2), product **2a** was obtained in 71% yield, but only 4.3% ee, although two equivalents copper(I) chloride was used (Table 2, entry 1). Poor results were also obtained in ethanol and ethyl acetate (Table 2, entries 2 and 3). Fortunately, when the reaction was conducted in tetrahydrofuran, the enantiopurity of (*R*)-(+)-**2a** was much higher (Table 2, entry 4). The best results were obtained when dichloromethane was used as the solvent: the reaction of **3a** at ambient temperature afforded (*R*)-(+)-**2a** without obvious loss of the chirality (Table 2, entries 5–9). On the basis of these studies, we concluded that the best results were obtained when the reaction was conducted at 25 °C with one equivalent of copper(I) chloride as the mediator and dichloromethane as the solvent (Table 2, entry 10). The requirement of one equivalent of copper(I) chloride may be due to the coordination ability of cinchonidine with copper(I).

With the established reaction conditions, differently substituted optically active butenolides **2** were prepared in moderate to excellent yields in high enantiopurity (Table 3). The stereochemical outcome of this transfor-

Table 1 Cycloisomerization of Optically Active 2,3-Allenoic Acids Catalyzed by Copper(I) Chloride

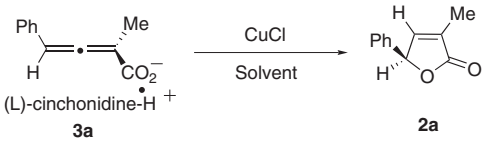


Entry	R^1	R^2	R^3	Starting material **1**	ee of **1** (%)	Product **2**	Yield (%)	ee of **2**[a] (%)
1	Ph	H	Me	(*S*)-(+)-**1b**	>97.3	(*R*)-(+)-**2a**	94	98.6
2	H	Ph	Me	(*R*)-(–)-**1b**	>94.3	(*S*)-(–)-**2a**	83	97.6
3	Ph	H	Pr	(*S*)-(+)-**1a**	>97.4	(*R*)-(+)-**2b**	85	99.8
4	H	Ph	Pr	(*R*)-(–)-**1a**	>97.2	(*S*)-(–)-**2b**	90	96.5

[a] Determined by HPLC analysis with chiral columns.

SYNTHESIS 2006, No. 21, pp 3711–3714
Advanced online publication: 09.10.2006
DOI: 10.1055/s-2006-950293; Art ID: F06706SS

Table 2 Copper(I) Chloride Mediated Cyclization of the 1:1 Salt of (*S*)-2-Methyl-4-phenylbuta-2,3-dienoic Acid with L-(–)-Cinchonidine



3a **2a**

Entry	ee of **3a**[a] (%)	Solvent	CuCl (equiv)	Temp (°C)	Time (h)	Yield of **2a**[b] (%)	ee of **2a**[c] (%)
1	97.7	MeOH	2	70	3.6	71	4.3
2	97.7	EtOH	2	70	11	73	1
3	97.7	EtOAc	2	70	13.5	80	9
4	89.2	THF	2	60	12	82	81.9
5	89.2	CH_2Cl_2	2	reflux	4.5	82	87.5
6	89.2	CH_2Cl_2	2	30	4.5	80	90.1
7	89.2	CH_2Cl_2	2	0	5	67	88.5
8	89.2	CH_2Cl_2	1	0	10.5	62	88.6
9	89.2	CH_2Cl_2	10%	25	12	34	58.5
10	97.7	CH_2Cl_2	1	25	10	78	97.1

[a] Determined by HPLC analysis with chiral columns after converting **3a** into its ester.
[b] Isolated yield.
[c] Determined by HPLC analysis with chiral columns.

Table 3 Copper(I) Chloride Mediated Cyclization Reaction of 1:1 Salts of Optically Active 2,3-Allenoic Acids with Chiral Amines

Entry	**3**	R^1	R^2	R^3	Base[a]	ee of **3**[b] (%)	Time (h)	**2**	Yield (%)	ee of **2**[c] (%)
1	**3a**	Ph	H	Me	A	>97.7	10	(*R*)-(+)-**2a**	78	97.1
2	**3b**	H	Ph	Me	C	>94.3	10	(*S*)-(–)-**2a**	90	97.3
3	**3c**	Ph	H	Pr	B	>97.2	10	(*R*)-(+)-**2b**	94	99.9
4	**3d**	H	Ph	Pr	C	>97.4	10	(*S*)-(–)-**2b**	94	99.1
5	**3e**	Ph	H	allyl	B	>97.6	10	(*R*)-(+)-**2c**	96	96.9
6	**3f**	H	Ph	allyl	C	>97.8	10	(*S*)-(–)-**2c**	93	98.5
7	**3g**	1-Naph	H	Me	B	>98.3	12.5	(*R*)-(+)-**2d**	81	98.2
8	**3h**	H	1-Naph	Me	C	>96.7	10	(*S*)-(–)-**2d**	95	99.3
9	**3i**	4-BrC_6H_4	H	Pr	A	>99.0	10	(*R*)-(+)-**2e**	87	98.0

[a] A = L-(–)-cinchonidine, B = L-(–)-(1-phenylethyl)amine, C = D-(+)-α-(1-phenylethyl)amine.
[b] Determined by HPLC analysis with chiral columns after converting **3** into its ester.
[c] Determined by HPLC analysis with chiral columns.

mation was established by the X-ray diffraction study of (*R*)-(+)-**2e** (Figure 1).[7] By comparing the absolute configurations of (*R*)-(+)-**2e** with that of **3i** (Table 3, entry 9), it was concluded that the reaction proceeds via *anti*-oxymetalation.[8] All the absolute configurations of **2** are tentatively assigned on the basis of this study.

In conclusion, we have developed a copper(I) chloride mediated cycloisomerization of optically active 2,3-allenoic acids and cyclization of 1:1 salts of 2,3-allenoic acids with chiral amines. The reactions of optically active 2,3-allenoic acids in methanol require only a catalytic amount of copper(I) chloride, while the reactions of the optically active 1:1 salts of optically active 2,3-allenoic acids with chiral amines in dichloromethane need one equivalent of copper(I) chloride. Further studies in this area are being conducted in our laboratory.

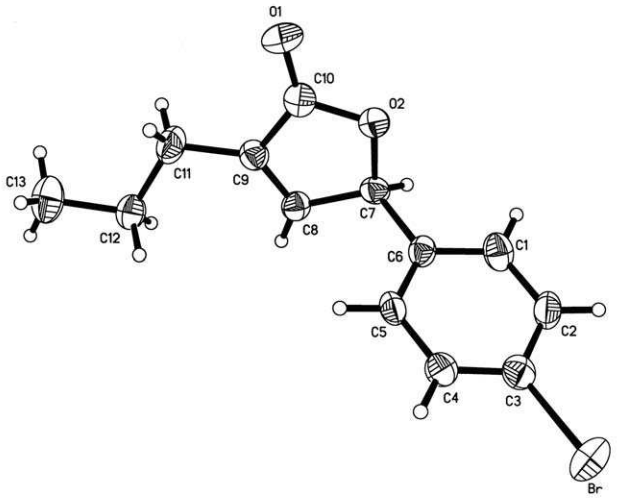


Figure 1 ORTEP representation of butenolide (*R*)-(+)-**2e**

NMR spectra were recorded on a Varian Mercury-300 or Bruker AM-300 spectrometer with chemical shifts reported in ppm relative to TMS as internal standard. IR spectra were measured by using an Avatar-360 spectrometer. EI mass spectra were determined with a HP 5989A mass spectrometer.

(*R*)-(+)-3-Methyl-5-phenylfuran-2(5*H*)-one [(*R*)-(+)-2a][5]
From (*S*)-(+)-**1b**: Under N_2, a soln of (*S*)-(+)-**1b** (69 mg, 0.4 mmol; >97.3% ee) and CuCl (1.6 mg, 0.016 mmol) in MeOH (4 mL) was stirred at 60 °C for 2 h. After evaporation, the residues were purified by flash chromatography (silica gel, PE–EtOAc, 10:1). Yield: 65 mg (94%); 98.6% ee.

From **3a**: Under N_2, a soln of the (*S*)-(+)-**1b**–L-(–)-cinchonidine salt (**3a**; 117 mg, 0.25 mmol, >97.7% ee) and CuCl (26 mg, 0.26 mmol) in CH_2Cl_2 (8 mL) was stirred at r.t. for 12 h. Yield: 34 mg (78%); 97.1% ee.

HPLC (AS column, rate = 0.7 mL/min, hexane–*i*-PrOH, 90:10); $[\alpha]_D^{20}$ 226.7 (*c* 1.025, EtOH, 98.6% ee); mp 57–58 °C (EtOAc, PE).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.40–7.25 (m, 5 H), 7.13 (d, *J* = 1.6 Hz, 1 H), 5.87 (s, 1 H), 1.99 (d, *J* = 1.5 Hz, 3 H).

(*S*)-(–)-3-Methyl-5-phenylfuran-2(5*H*)-one [(*S*)-(–)-2a][5]
From (*R*)-(–)-**1b**: Under N_2, a soln of (*R*)-(–)-**1b** (70 mg, 0.4 mmol; >94.3% ee) and CuCl (1.6 mg, 0.016 mmol) in MeOH (4 mL) was stirred at 60 °C for 2.5 h. After evaporation, the residues were purified by flash chromatography (silica gel, PE–EtOAc, 10:1).

Yield: 58 mg (83%); 97.6% ee; $[\alpha]_D^{20}$ –211.8 (*c* = 1.05, EtOH).

From **3b**: Under N_2, a soln of the (*R*)-(–)-**1b**–D-(+)-(1-phenylethyl)amine salt (**3b**; 60 mg, 0.2 mmol; >94.3% ee) and CuCl (21 mg, 0.21 mmol) in CH_2Cl_2 (2 mL) was stirred at r.t. for 10 h.

Yield: 32 mg (90%); 97.3% ee.

(*R*)-(+)-5-Phenyl-3-propylfuran-2(5*H*)-one [(*R*)-(+)-2b][5]
From (*S*)-(+)-**1a**: Under N_2, (*S*)-(+)-**1a** (82 mg, 0.4 mmol; >97.2% ee) and CuCl (1.6 mg, 0.016 mmol) in MeOH (4 mL) was stirred at 60 °C for 3 h. After evaporation, the residues were purified by flash chromatography (silica gel, PE–EtOAc, 15: 1).

Yield: 70 mg (85%); 99.8% ee.

From **3c**: Under N_2, a soln of the (*S*)-(+)-**1a**–L-(–)-(1-phenylethyl)amine salt (**3c**; 100 mg, 0.31 mmol; >97.2% ee) and CuCl (31 mg, 0.31 mmol) in CH_2Cl_2 (3 mL) was stirred at r.t. for 10 h.

Yield: 59 mg (94%); 99% ee.

HPLC (AS column, rate = 0.7 mL/min, hexane–*i*-PrOH, 80:20); $[\alpha]_D^{20}$ +163.5 (*c* 0.955, EtOH; 99.8% ee).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.46–7.19 (m, 5 H), 7.09 (s, 1 H), 5.87 (s, 1 H), 2.4–2.28 (br, 2 H), 1.70–1.55 (br, 2 H), 1.08–0.91 (br, 3 H).

(*S*)-(–)-5-Phenyl-3-propylfuran-2-(5*H*)-one [(*S*)-(–)-2b][5]
From (*R*)-(–)-**1a**: Under N_2, a soln of (*R*)-(–)-**1a** (81 mg, 0.4 mmol; >97.4% ee) and CuCl (1.6 mg, 0.016 mmol) in MeOH (4 mL) was stirred at 60 °C for 2 h. After evaporation, the residue was purified by flash chromatography (silica gel, PE–EtOAc, 15:1).

Yield: 73 mg (90%); 96.5% ee; $[\alpha]_D^{20}$–162.4 (*c* 1.04, EtOH).

From **3d**: Under N_2, a soln of the (*R*)-(–)-**1a**–D-(+)-(1-phenylethyl)amine salt (**3d**; 100 mg, 0.31 mmol; >97.4% ee) and CuCl (31 mg, 0.31 mmol) in CH_2Cl_2 (3 mL) was stirred at r.t. for 10 h.

Yield: 59 mg (94%); 99.1% ee.

(*R*)-(+)-3-Allyl-5-phenylfuran-2(5*H*)-one [(*R*)-(+)-2c][5]
Under N_2, a soln of the (*S*)-2-allyl-4-phenylbuta-2,3-dienoic acid–L-(–)-(1-phenylethyl)amine salt (**3e**; 102 mg, 0.31 mmol; >97.6% ee) and CuCl (31 mg, 0.31 mmol) in CH_2Cl_2 (3 mL) was stirred at r.t. for 10 h.

Yield: 61 mg (96%); 96.9% ee.

HPLC (AS column, rate = 0.7 mL/min, hexane–*i*-PrOH, 75:25); $[\alpha]_D^{20}$ +126 (*c* 2.40, EtOH).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.45–7.22 (m, 5 H), 7.15–7.11 (m, 1 H), 5.82–6.00 (m, 2 H), 5.26–5.13 (m, 2 H), 3.11 (d, *J* = 5.4 Hz, 2 H).

(*S*)-(–)-3-Allyl-5-phenylfuran-2(5*H*)-one [(*S*)-(–)-2c]
Under N_2, a soln of the (*R*)-2-allyl-4-phenylbuta-2,3-dienoic acid–D-(+)-(1-phenylethyl)amine salt (**3f**; 103 mg, 0.32 mmol; >97.8% ee) and CuCl (31 mg, 0.31 mmol) in CH_2Cl_2 (3 mL) was stirred at r.t. for 10 h.

Yield: 60 mg (93%); 98.5% ee; $[\alpha]_D^{20}$ –128 (*c* 2.45, EtOH).[5]

(*R*)-(+)-3-Methyl-5-(1-naphthyl)furan-2(5*H*)-one [(*R*)-(+)-2d]
Under N_2, a soln of the (*S*)-2-methyl-4-naphthylbuta-2,3-dienoic acid–L-(–)-(1-phenylethyl)amine salt (**3g**; 87 mg, 0.252 mmol; >98.3% ee) and CuCl (27 mg, 0.27 mmol) in CH_2Cl_2 (3 mL) was stirred at r.t. for 10 h.

Yield: 46 mg (81%); 98.2% ee.

HPLC (AS column, rate = 0.7 mL/min, hexane–*i*-PrOH, 80:20); mp 95–97 °C; $[\alpha]_D^{20}$ +94 (*c* 0.975, EtOH).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.06 (d, *J* = 8.0 Hz, 1 H), 7.96–7.82 (m, 2 H), 7.68–7.50 (m, 2 H), 7.50–7.38 (m, 3 H), 6.65 (s, 1 H), 2.02 (s, 3 H).

(*S*)-(–)-3-Methyl-5-(1-naphthyl)furan-2(5*H*)-one [(*S*)-(–)-2d][5]
Under N_2, a soln of the (*R*)-2-methyl-4-naphthylbuta-2,3-dienoic acid–D-(+)-(1-phenylethyl)amine salt (**3h**; 140 mg, 0.406 mmol, >96.7% ee) and CuCl (41 mg, 0.41 mmol) in CH_2Cl_2 (4 mL) was stirred at r.t. for 10 h.

Yield: 86 mg (95%); 99.3% ee.

(*R*)-(+)-5-(4-Bromophenyl)-3-propylfuran-2(5*H*)-one [(*R*)-(+)-2e]

Under N_2, a soln of the (*S*)-4-(4-bromophenyl)-2-propylbuta-2,3-dienoic acid–D-(–)-(1-phenylethyl)amine salt (**3i**; 115 mg, 0.2 mmol; >97.9% ee) and CuCl (20 mg, 0.2 mmol) in CH_2Cl_2 (2 mL) was stirred at r.t. for 10 h.

Yield: 49 mg (87%); 98.0% ee.

HPLC (AS column, rate = 0.7 mL/min, hexane–*i*-PrOH, 80:20); $[\alpha]_D^{20}$ 124 (*c* 0.795, EtOH); mp 79–80 °C (EtOAc, PE).

IR (KBr): 1741, 1649, 1590, 1489 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 7.49 (d, *J* = 7.4 Hz, 2 H), 7.12 (d, *J* = 7.4 Hz, 2 H), 7.06 (s, 1 H), 5.82 (t, *J* = 1.8 Hz, 1 H), 2.31 (t, *J* = 7.7 Hz, 2 H), 1.68–1.52 (m, 2 H), 0.96 (t, *J* = 7.4 Hz, 3 H).

^{13}C NMR (75.4 MHz, $CDCl_3$): δ = 13.6, 20.6, 27.1, 81.4, 123.0, 128.0, 132.0, 134.1, 134.2, 147.2, 173.7.

MS (EI, 70 eV): *m*/*z* (%) = 280 $[M(^{79}Br)]^+$, 66.84, 282 $[M(^{81}Br)]^+$, 65.80, 183 (100).

Anal. Calcd for $C_{13}H_{13}BrO_2$: C, 55.54; H, 4.66. Found: C, 55.65; H, 4.93.

Acknowledgment

Financial support from the National Natural Science Foundation of China (20121202 and 20332060) and the Shanghai Municipal Committee of Science and Technology is greatly appreciated.

References

(1) (a) Larock, R. D.; Riefling, B.; Fellows, C. A. *J. Org. Chem.* **1978**, *43*, 131; and references cited therein. (b) Brima, T. S. US 4968817, **1990**; *Chem. Abstr.* **1991**, *114*, 185246y. (c) Tanabe, A. Jpn. Kokai Tokyo Koho JP 63211276 [88211276], **1988**; *Chem. Abstr.* **1989**, *110*, 94978q. (d) Lee, G. C. M. Eur. Pat. Appl. EP 372940, **1990**; *Chem. Abstr.* **1990**, *113*, 191137j. (e) Ducharme, Y.; Gauthier, J. Y.; Prasit, P.; Leblanc, Y.; Wang, Z.; Leger, S.; Therien, M. PCT Int. Appl. WO 95 00501, **1995**; *Chem. Abstr.* **1996**, *124*, 55954y. (f) Lee Gary, C. M.; Garst, M. E. PCT Int. Appl. WO 91 16055, **1991**; *Chem. Abstr.* **1992**, *116*, 59197m.

(2) For some recent examples, see: (a) Chia, Y.; Chang, F.; Wu, Y. *Tetrahedron Lett.* **1999**, *40*, 7513. (b) Takahashi, S.; Maeda, K.; Hirota, S.; Nakata, T. *Org. Lett.* **1999**, *1*, 2025. (c) Siddiqui, B. S.; Afshan, F.; Ghiasuddin; Faizi, S.; Naqvi, S. N.-H.; Tariq, R. M. *J. Chem. Soc., Perkin Trans. 1* **1999**, 2367. (d) Cortez, D. A. G.; Fernandes, J. B.; Vieria, P. C.; Silva, M. F. G. F.; Ferreira, A. G.; Cass, Q. B.; Pirani, J. R. *Phytochemistry* **1998**, *49*, 2493. (e) Otsuka, H.; Kotani, K.; Bando, M.; Kido, M.; Takeda, Y. *Chem. Pharm. Bull.* **1998**, *46*, 1180. (f) Guo, S.; Wang, L.; Chen, D. *Indian J. Chem., Sect. B: Org. Chem. Incl. Med. Chem.* **1997**, *36*, 339. (g) Evidente, A.; Sparapano, L. *J. Nat. Prod.* **1994**, *57*, 1720. (h) Damtoft, S.; Jensen, S. R. *Phytochemistry* **1995**, *40*, 157. (i) Estevez-Reyes, R.; Estevez-Braun, A.; Gonzalez, A. G. *J. Nat. Prod.* **1993**, *56*, 1177. (j) Claydon, N.; Hanson, J. R.; Truneh, A.; Avent, A. G. *Phytochemistry* **1991**, *30*, 3802. (k) Seki, T.; Satake, M.; Mackenzie, L.; Kaspar, H. F.; Yasumoto, T. *Tetrahedron Lett.* **1995**, *36*, 7093. (l) Marshall, J. A.; Wolf, M. A.; Wallace, E. M. *J. Org. Chem.* **1997**, *62*, 367.

(3) (a) Basabe, P.; Delgado, S.; Marcos, I. S.; Diez, D.; Diego, A.; De Roman, M.; Urones, J. G. *J. Org. Chem.* **2005**, *70*, 9480. (b) Naito, S.; Escobar, M.; Kym, P. R.; Liras, S.; Martin, S. F. *J. Org. Chem.* **2002**, *67*, 4200. (c) Jiang, S.; Liu, Z.-H.; Sheng, G.; Zeng, B.-B.; Cheng, X.-G.; Wu, Y.-L.; Yao, Z.-J. *J. Org. Chem.* **2002**, *67*, 3404. (d) Busqué, F.; March, P.; Figueredo, M.; Font, J.; Sanfeliu, E. *Tetrahedron Lett.* **2002**, *43*, 5583. (e) Yoneda, E.; Kaneko, T.; Zhang, S.; Onitsuka, K.; Takahashi, S. *Org. Lett.* **2000**, *2*, 441. (f) Brown, S. P.; Goodwin, N. C.; MacMillan, D. W. C. *J. Am. Chem. Soc.* **2003**, *125*, 1192.

(4) (a) Ma, S.; Shi, Z.; Yu, Z. *Tetrahedron Lett.* **1999**, *40*, 2393. (b) Ma, S.; Shi, Z.; Yu, Z. *Tetrahedron* **1999**, *40*, 12137. (c) Fu, C.; Ma, S. *Eur. J. Org. Chem.* **2005**, 3942. (d) Ma, S.; Yu, Z. *Angew. Chem. Int. Ed.* **2002**, *41*, 1775. (e) Ma, S.; Yu, Z. *Chem. Eur. J.* **2004**, *10*, 2078. (f) Ma, S.; Yu, Z. *Org. Lett.* **2003**, *5*, 1507; corrigendum *Org. Lett.* **2003**, *5*, 2581. (g) Ma, S.; Yu, Z. *J. Org. Chem.* **2003**, *68*, 6149. (h) Ma, S.; Gu, Z. *J. Am. Chem. Soc.* **2005**, *127*, 6182. (i) Ma, S.; Yu, Z.; Gu, Z. *Chem. Eur. J.* **2005**, *11*, 2351.

(5) For copper(I) chloride catalyzed cycloisomerization of racemic 2,3-allenoic acids, see: Ma, S.; Yu, Z.; Wu, S. *Tetrahedron* **2001**, *57*, 1585.

(6) (a) Ma, S.; Shi, Z. *Chem. Commun.* **2002**, 540. (b) Ma, S.; Wu, S. *Chem. Commun.* **2001**, 441.

(7) Crystal data for compound (*R*)-(+)-**2e**: $C_{13}H_{13}BrO_2$, *M* = 281.14, monoclinic, space group *P*2 (1), Mo Kα, final *R* indices [$I > 2\sigma(I)$], *R*1 = 0.0657, *wR*2 = 0.1570, *a* = 4.3321 (19) Å, *b* = 10.916 (5) Å, *c* = 13.117 (6) Å, α = 90°, β = 92.942 (7)°, γ = 90°, *V* = 619.5 (5) $Å^3$, *T* = 293 (2) K, *Z* = 2, reflections collected/unique: 3518:2213 (R_{int} = 0.1593), no observation [$I > 2\sigma(I)$] 957, parameters 155; CCDC 602980 contains the data.

(8) (a) Ma, S.; Yu, Z. *J. Org. Chem.* **2003**, *68*, 6149. (b) Ma, S.; Yu, Z. *Angew. Chem. Int. Ed.* **2003**, *42*, 1955. (c) Ma, S.; Yu, Z.; Gu, Z. *Chem. Eur. J.* **2005**, *11*, 2351.