ISSN 1070-4280, Russian Journal of Organic Chemistry, 2009, Vol. 45, No. 10, pp. 1509–1514. © Pleiades Publishing, Ltd., 2009.
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Asymmetric Sulfoxidation of Phenacyl Phenyl Sulfide Using Chiral Vanadium(IV) Complexes

E. V. Ashikhmina, S. A. Rubtsova, I. A. Dvornikova, and A. V. Kuchin

Institute of Chemistry, Komi Research Center, Ural Division, Russian Academy of Sciences, ul. Pervomaiskaya 48, Syktyvkar, 167982 Russia
e-mail: ashihmina-ev@chemi.komisc.ru

Received June 17, 2008

Abstract—Asymmetric sulfoxidation of phenacyl phenyl sulfide was performed in the presence of bis(acetylacetonanto)oxovanadium(IV) complexes with chiral Schiff bases, which were generated *in situ*. Chlorine dioxide and hydrogen peroxide as oxidants were compared.

DOI: 10.1134/S1070428009100145

Chiral sulfoxides are widely used as synthons in asymmetric synthesis [1, 2] and biologically active compounds in medicine [3–6], as well as ferroelectric liquid crystals [7]. Taking into account diversity of practically important properties of optically active sulfoxides, development of effective procedures for the preparation of these compounds via asymmetric sulfoxidation of the corresponding sulfides has become necessary.

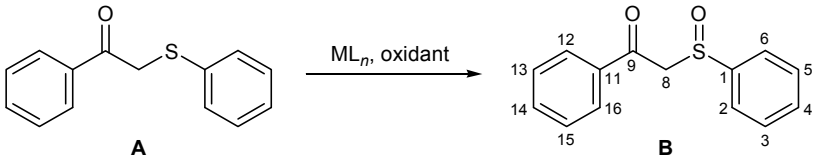
In this connection, the most interesting are catalytic systems based on transition metal complexes with chiral Schiff bases. The Bolm catalyst may be regarded as such system [8, 9]. Vanadium(IV) complexes are used in catalytic amounts (~1%), they require no special purification of solvents, reduced temperature, or inert atmosphere, and reaction products are readily separated from the catalyst. Several accessible Schiff bases have recently been described as ligands for vanadium(IV)-based catalytic systems. These ligands ensured oxidation of various aryl methyl sulfides to the corresponding sulfoxides in acceptable chemical and optical yields [10].

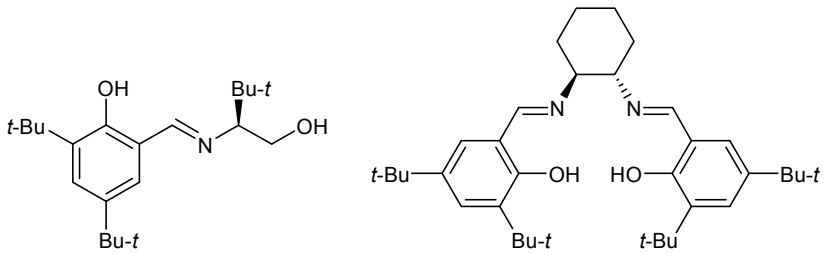
In the present work we used as oxidant both hydrogen peroxide and chlorine dioxide (ClO_2). The latter is one of the most interesting and accessible oxidants available on a large scale. There are published data [11–18] on the oxidation with chlorine dioxide of amines, phenols, olefins, and carbonyl compounds, while asymmetric oxidation with ClO_2 has not been reported. We previously showed that chlorine dioxide ensures chemoselective oxidation of sulfides to sulfoxides [11, 19, 20].

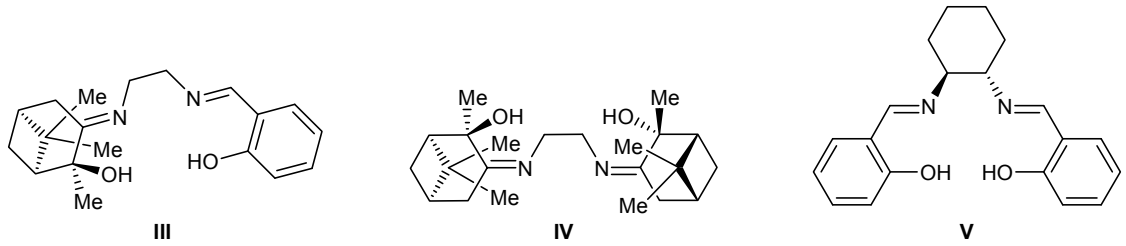
We examined asymmetric sulfoxidation of phenacyl phenyl sulfide [1-phenyl-2-(phenylsulfanyl)ethanone] **A** using as catalysts vanadium(IV) complexes with chiral ligands. Some chiral ligands were reported previously, while the others were synthesized by us for the first time. We also compared the results obtained with the use of hydrogen peroxide and chlorine dioxide as oxidants (Scheme 1, see table).

Bolm and Bienewald [8] reported on the oxidation of phenacyl phenyl sulfide **A** in the presence of bis(acetylacetonato)oxovanadium(IV) complex with chiral ligand **I**, which was generated *in situ*. As a result, phenacyl phenyl sulfoxide [**B**, 1-phenyl-2-(phenylsulfinyl)ethanone] was obtained in a chemical yield of 65% with 57% *ee*. We used these data as reference for our subsequent study on asymmetric sulfoxidation

Scheme 1.







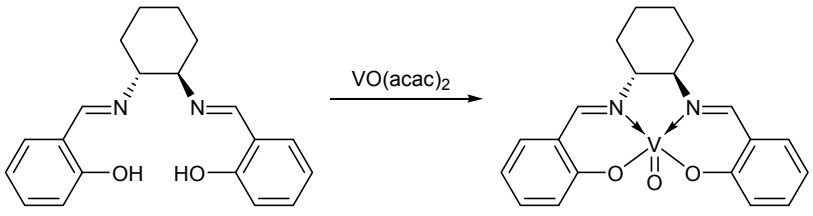
of the same substrate. Oxidation of phenacyl phenyl sulfide **A** was performed in the presence of bis(acetylacetonato)oxovanadium(IV) complexes with the following chiral Schiff bases: 2,4-di-*tert*-butyl-6-[(*E*)-{[(1*S*)-1-(hydroxymethyl)-2,2-dimethylpropyl]imino}-methyl]phenol (**I**), 2,4-di-*tert*-butyl-6-[(*E*)-{[(1*S*,2*S*)-2-{[(*E*)-(3,5-di-*tert*-butyl-2-hydroxyphenyl)methylidene]-amino}cyclohexyl]imino}methyl]phenol (**II**), (1*S*,2*S*,5*S*)-3-[{2-[(2-hydroxybenzylidene)amino]-ethyl}imino]-2,6,6-trimethylbicyclo[3.1.1]heptan-2-ol (**III**), 3-({2-[(2-hydroxy-2,6,6-trimethylbicyclo[3.1.1]-hept-3-ylidene)amino]ethyl}imino)-2,6,6-trimethylbicyclo[3.1.1]heptan-2-ol (**IV**), and 2-{(*E*)-[((1*R*,2*R*)-2-{[(*E*)-(2-hydroxyphenyl)methylidene]amino}cyclohexyl)imino]methyl}phenol (**V**). Schiff bases **I** and **II** are commercially available products, unsymmetrical diimine **III** and symmetrical diimine **IV** were synthesized by us for the first time from enantiomerically pure 2-hydroxypinan-3-one [21], and compound **V** was prepared according to known procedure [22]. Ligands **III** and **IV** were not used previously in analogous reactions.

The formation of VO^{2+}–**V** complex was reported in [23] (Scheme 2). We presumed that ligands **I**–**IV** with VO^{2+} form analogous complexes. We reproduced the oxidation of phenacyl phenyl sulfide **A** with hydrogen peroxide and bis(acetylacetonato)oxovanadium(IV) complex with chiral ligand **I** according to the procedure described in [8]. As a result, we isolated phenacyl phenyl sulfoxide **B** whose structure was consistent with the data given in [8], but the *ee* value was higher. The progress of the reaction was monitored by HPLC, and enantiomeric excess was determined by HPLC using a chiral stationary phase (see table).

As follows from the data in table, the use of hydrogen peroxide as oxidant in combination with ligand **IV** was low efficient: the corresponding sulfoxide was isolated in moderate yield and with poor optical purity. Unsatisfactory results were also obtained with compound **III** as chiral ligand and the same oxidant. The best yield of the target sulfoxide was attained in the presence of the catalytic system containing ligand **V**, and the optical purity was slightly improved as compared to published data using the catalytic system with ligand **I**.

Replacement of hydrogen peroxide by an aqueous solution of chlorine dioxide led to better *ee* value in the oxidation in the presence of $VO(acac)_2$ complex with terpene ligand **III**. The results obtained with the use of salen-type ligands were unsatisfactory. Presumably, the

Scheme 2.



Asymmetric oxidation of phenacyl phenyl sulfide **A**

Method[a]	Catalytic system (ratio)	Oxidant (solvent)	Yield,[b] %	Enantiomeric excess,[c] %	$[\alpha]_D$
a	$VO(acac)_2$–**I** (1 : 1.5)	H_2O_2[d]	67 (65 [8])	75.6 (57 [8])	–16.0
b	$VO(acac)_2$–**I** (1 : 1.5)	ClO_2 ($CHCl_3$)	82	8	–1.4
c	$VO(acac)_2$–**I** (1 : 1.5)	ClO_2 (H_2O)	76	9	–1.6
d	$VO(acac)_2$–**II** (1 : 1.5)	H_2O_2[d]	73	59	+12.0
e	$VO(acac)_2$–**II** (1 : 1.5)	ClO_2 ($CHCl_3$)	84	27	+5.3
f	$VO(acac)_2$–**II** (1 : 1.5)	ClO_2 (H_2O)	78	20	+3.9
g	$VO(acac)_2$–**III** (1 : 1.5)	H_2O_2[d]	67	3	+0.6
h	$VO(acac)_2$–**III** (1 : 1.5)	ClO_2 ($CHCl_3$)	78	32	+6.0
i	$VO(acac)_2$–**III** (1 : 1.5)	ClO_2 (H_2O)	69	32	+6.1
j	$VO(acac)_2$–**IV** (1 : 1.5)	H_2O_2[d]	65	8	–1.4
k	$VO(acac)_2$–**IV** (1 : 1.5)	ClO_2 ($CHCl_3$)	76	9	–1.6
l	$VO(acac)_2$–**IV** (1 : 1.5)	ClO_2 (H_2O)	69	15	–2.8
m	$VO(acac)_2$–**V** (1 : 1.5)	H_2O_2[d]	77	48	–10.0
n	$VO(acac)_2$–**V** (1 : 1.5)	ClO_2 ($CHCl_3$)	87	11	–2.2
o	$VO(acac)_2$–**V** (1 : 1.5)	ClO_2 (H_2O)	80	1.5	–0.3

[a] All experiments were carried out at 0°C with $VO(acac)_2$ in $CHCl_3$; reaction time 16 h; substrate–oxidant–catalyst ratio 1 : 1 : 0.02.
[b] The yield of sulfoxide **B** isolated by column chromatography on silica gel.
[c] Determined by HPLC using a chiral stationary phase (see Experimental).
[d] 33% aqueous solution.

corresponding complexes undergo decomposition in the presence of water or are unstable toward ClO_2, though the complex $VO(acac)_2$–**III** turned out to be more stable under the same conditions. This assumption was also confirmed by experiment with a solution of chlorine dioxide in chloroform, other conditions being equal; here, the presence of water was completely excluded. Solutions of ClO_2 in chloroform and in water gave similar *ee* values of the product in the system $VO(acac)_2$–unsymmetrical terpene ligand **III**.

Ligand **II** ensured better optical purity in the asymmetric oxidation of sulfide **A** with chlorine dioxide in the presence of $VO(acac)_2$ and salen-type ligands. A probable reason is steric hindrances to complex formation with fairly bulky ligand. Insignificant increase of *ee* value in the systems $VO(acac)_2$–salen-type ligand was observed upon replacement of aqueous chlorine dioxide by its solution in chloroform. No appreciable variation in the reaction efficiency and selectivity was observed in going to other solvents.

Increase in the chemical yield and decrease of enantiomeric purity of the product in the oxidation with solutions of chlorine dioxide may be rationalized assuming concurrent oxidation of oxo sulfide **A** with optically inactive oxodiperoxovanadium complex.

For comparison, we performed the reaction at a higher catalyst-to-substrate ratio (0.10 : 1 against 0.02 : 1). In this case, the chemical yield of the product did not change (77–78%), but its enantiomeric excess insignificantly increased (from 20 to 35.6%). We believe that increased concentration of the ligand and $VO(acac)_2$ favors formation of a larger amount of the complex promoting asymmetric oxidation of the substrate. Raising the temperature from 0°C to ambient had no appreciable effect on the optical and chemical yields of sulfoxide **B**.

Our results show that compounds **I**, **II**, and **V** are the most promising as ligands for asymmetric sulfoxidation and that hydrogen peroxide is the most effective as oxidant. Chlorine dioxide may be used in asymmetric oxidation in the presence of $VO(acac)_2$ and unsymmetrical terpene ligand **III**.

To conclude, we performed asymmetric sulfoxidation of phenacyl phenyl sulfide **A** with hydrogen peroxide and chlorine dioxide in the presence of bis(acetylacetonato)oxovanadium(IV) complexes with chiral Schiff bases, generated *in situ*. Enantioselective oxidation of oxo sulfide **A** with chlorine dioxide was accomplished for the first time. The use of an aqueous solution of ClO_2, as well as of a solution of ClO_2 in

chloroform, as oxidant in the system $VO(acac)_2$–salene ligand increases the chemical yield of the product, but reduces its optical purity as compared to hydrogen peroxide. The oxidation with a solution of ClO_2 in organic solvent provides better enantiomeric excess and chemical yield than in the oxidation with aqueous chlorine dioxide.

The opposite results are observed in the system $VO(acac)_2$–terpene ligand **III** or **IV**–ClO_2: enantiomeric excess increases, but the chemical yield decreases relative to the corresponding parameters obtained in the system $VO(acac)_2$–ligand **III** or **IV**–H_2O_2.

EXPERIMENTAL

The IR spectra (400–4000 cm^{-1}) were recorded on a Prestige-21 spectrometer from solutions in carbon tetrachloride (liquid substances, 0.2-mm cell) or KBr pellets (solids). The melting points were determined on a Gallenkamp–Sanyo melting point apparatus. The ^1H and ^{13}C NMR spectra were measured on a Bruker DRX-400 instrument at 400.13 and 100.62 MHz, respectively, using $CDCl_3$ as solvent and tetramethylsilane as internal reference.

Gaz chromatography was performed on a Chrom-5 chromatograph equipped with a flame-ionization detector and a 2-m×4-mm column packed with 5% of Carbowax-20 on Chromaton-N-AW-DMCS; carrier gas helium; oven temperature programming from 50 to 250°C at a rate of 6 deg/min. The enantiomeric composition was determined by HPLC on a Surveyor LC instrument; Chiralcel OV-H column; λ 254 nm; eluent hexane–propan-2-ol (50 : 50), flow rate 0.5 ml/min. The optical rotations were measured on a Kruss P3002RS automatic digital polarimeter. Sorbfil plates were used for thin-layer chromatography (eluent C_6H_{14}–Et_2O); spots were developed by treatment with a 5% solution of potassium permanganate. The elemental compositions were determined on an EA 1110 CHNS-O automatic analyzer.

Compounds **I** and **II** were commercial products (Alfa Aesar). Schiff bases **III** and **IV** were synthesized as described in [21], and ligand **V** was prepared according to the procedure reported in [22].

Initial 1-phenyl-2-(phenylsulfanyl)ethanone **A** was synthesized by reaction of benzenethiol with 2-bromo-1-phenylethanone [24]. Yield 98% (after recrystallization), mp 51–52°C. IR spectrum (KBr), ν, cm^{-1}: 1670 (C=O), 742 (C–S). ^1H NMR spectrum, δ, ppm: 4.28 s (2H, 8-H), 7.19–7.31 m (2H, H_{arom}), 7.39–7.59 m (5H, H_{arom}), 7.93–7.95 m (2H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 41.17 (CH_2), 127.04 (C^4), 128.70 (C^{12}, C^{16}), 128.70 (C^3, C^5), 129.11 (C^{13}, C^{15}), 133.49 (C^2, C^6), 134.96 (C^{11}), 135.44 (C^1), 194.05 (C^9). Found, %: C 73.70; H 5.31; S 14.06. $C_{14}H_{12}OS$. Calculated, %: C 73.68; H 5.26; S 14.03.

1-Phenyl-2-(phenylsulfanyl)ethanone B. *a*. A mixture of 1 mg (4 μmol) of $VO(acac)_2$ and 2 mg (6 μmol) of ligand **I** in 4 ml of chloroform was stirred for 1 h. The originally blue solution turned brown. The mixture was cooled to 0°C, 228 mg (1 mmol) of sulfide **A** was added, the mixture was stirred for 10 min, 93 μl (1 mmol) of 33% hydrogen peroxide was added dropwise, the mixture was kept for 16 h, and the reaction was terminated by adding 10 ml of distilled water. The organic phase was separated, the aqueous phase was extracted with chloroform, and the extract was combined with the organic phase, washed with a solution of sodium carbonate (25 ml) and a saturated aqueous solution of sodium chloride (25 ml), dried over magnesium sulfate, and evaporated under reduced pressure. The residue was subjected to column chromatography to isolate 0.16 g (67%) of compound **B**, $[\alpha]_D = -16.0°$ (*c* = 1.0, EtOH), *ee* = 75.6%. IR spectrum (KBr), ν, cm^{-1}: 1678 (C=O), 1047 (S=O). ^1H NMR spectrum, δ, ppm: 4.27 d and 4.50 d (1H each, 9-H, J = 14.4 Hz), 7.33–7.41 m (2H, H_{arom}), 7.48–7.53 m (5H, H_{arom}), 7.6–7.66 m (2H, H_{arom}). ^{13}C NMR spectrum, δ_C, ppm: 65.95 (CH_2), 124.24 (C^2, C^6), 128.73 (C^{13}, C^{17}), 128.73 (C^3, C^5), 129.29 (C^{14}, C^{16}), 134.05 (C^4), 134.00 (C^{15}), 136.00 (C^{12}), 143.40 (C^1), 191.40 (C^{11}). Found, %: C 68.87; H 4.95; S 13.14. $C_{14}H_{12}SO_2$. Calculated, %: C 68.85; H 4.92; S 13.11.

b. Oxidation of 228 mg (1 mmol) of sulfide **A** with 1.9 ml (1 mmol) of ClO_2 in chloroform (concentration 0.52 M) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 2 mg (6 μmol) of ligand **I** (reaction time 16 h) gave 0.20 g (82%) of compound **B** with $[\alpha]_D = -1.4°$ (*c* = 1.0, EtOH), *ee* = 8.0%.

c. Oxidation of 228 mg (1 mmol) of sulfide **A** with 10 ml of a 0.1 M aqueous solution of ClO_2. (1 mmol) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 2 mg (6 μmol) of ligand **I** (reaction time 16 h) gave 0.185 g (76%) of sulfoxide **B** with $[\alpha]_D = -1.6°$ (*c* = 1.0, EtOH), *ee* = 8.9%.

d. Oxidation of 228 mg (1 mmol) of sulfide **A** with 93 μl (1 mmol) of 33% hydrogen peroxide in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 3.1 mg (9.4 μmol) of ligand **II** (reaction time 16 h) gave 0.18 g (73%) of sulfoxide **B** with $[\alpha]_D = +12.0$ (*c* = 1.0, EtOH), *ee* = 59.1%.

e. Oxidation of 228 mg (1 mmol) of sulfide **A** with 1.9 ml (1 mmol) of ClO_2 in chloroform (c = 0.52 M) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 3.1 mg (9.4 μmol) of ligand **II** (reaction time 16 h) gave 0.20 g (84%) of compound **B** with $[\alpha]_D$ = +5.3° (c = 1.0, EtOH), *ee* = 26.7%.

f. Oxidation of 228 mg (1 mmol) of sulfide **A** with 10 ml of a 0.1 M aqueous solution of ClO_2 (1 mmol) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 3.1 mg (9.4 μmol) of ligand **II** (reaction time 16 h) gave 0.19 g (78%) of compound **B** with $[\alpha]_D$ = +3.9° (c = 1.0, EtOH), *ee* = 19.8%.

g. Oxidation of 228 mg (1 mmol) of sulfide **A** with 93 μl (1 mmol) of 33% hydrogen peroxide in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 1.9 mg (5.7 μmol) of ligand **III** (reaction time 16 h) gave 0.16 g (67%) of compound **B** with $[\alpha]_D$ = +0.6° (c = 1.0, EtOH), *ee* = 3.2%.

h. Oxidation of 228 mg (1 mmol) of sulfide **A** with 5 ml (1 mmol) of ClO_2 in chloroform (c = 0.20 M) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 1.9 mg (5.7 μmol) of ligand **III** (reaction time 16 h) gave 0.19 g (78%) of compound **B** with $[\alpha]_D$ = +6.0° (c = 1.0, EtOH), *ee* = 32.1%.

i. Oxidation of 228 mg (1 mmol) of sulfide **A** with 6.7 ml (1 mmol) of an aqueous solution of ClO_2 (c = 0.148 M) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 1.9 mg (5.7 μmol) of ligand **III** (reaction time 16 h) gave 0.17 g (69%) of compound **B** with $[\alpha]_D$ = +6.1° (c = 1.0, EtOH), *ee* = 32.2%.

j. Oxidation of 228 mg (1 mmol) of sulfide **A** with 93 μl (1 mmol) of 33% hydrogen peroxide in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 2.2 mg (6.5 μmol) of ligand **IV** (reaction time 16 h) gave 0.16 g (65%) of compound **B** with $[\alpha]_D$ = –1.4° (c = 1.0, EtOH), *ee* = 8.1%.

k. Oxidation of 228 mg (1 mmol) of sulfide **A** with 1.9 ml (1 mmol) of ClO_2 in chloroform (c = 0.52 M) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 2.2 mg (6.5 μmol) of ligand **IV** (reaction time 16 h) gave 0.185 g (76%) of compound **B** with $[\alpha]_D$ = –1.6° (c = 1.0, EtOH), *ee* = 8.9%.

l. Oxidation of 228 mg (1 mmol) of sulfide **A** with 7.7 ml (1 mmol) of an aqueous solution of ClO_2 (c = 0.13 M) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 2.2 mg (6.5 μmol) of ligand **IV** (reaction time 16 h) gave 0.17 g (69%) of compound **B** with $[\alpha]_D$ = –2.8° (c = 1.0, EtOH), *ee* = 14.7%.

m. Oxidation of 228 mg (1 mmol) of sulfide **A** with 93 μl (1 mmol) of 33% hydrogen peroxide in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 1.9 mg (5.8 μmol) of ligand **V** (reaction time 16 h) gave 0.19 g (77%) of sulfoxide **B** with $[\alpha]_D$ = –10.0 (c = 1.0, EtOH), *ee* = 48.3%.

n. Oxidation of 228 mg (1 mmol) of sulfide **A** with 8.1 ml (1 mmol) of ClO_2 in chloroform (c = 0.124 M) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 1.9 mg (5.8 μmol) of ligand **V** (reaction time 16 h) gave 0.21 g (87%) of compound **B** with $[\alpha]_D$ = –2.2° (c = 1.0, EtOH), *ee* = 11.0%.

o. Oxidation of 228 mg (1 mmol) of sulfide **A** with 9.2 ml (1 mmol) of an aqueous solution of ClO_2 (c = 0.108 M) in the presence of 1 mg (4 μmol) of $VO(acac)_2$ and 1.9 mg (5.8 μmol) of ligand **V** (reaction time 16 h) gave 0.195 g (80%) of compound **B** with $[\alpha]_D$ = –0.3° (c = 1.0, EtOH), *ee* = 1.5%.

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