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New highly efficient aziridine-functionalized tridentate sulfinyl catalysts for enantioselective diethylzinc addition to carbonyl compounds

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ABSTRACT

New tridentate enantiomerically pure heteroorganic catalysts, containing hydroxyl, sulfinyl, and aziridine moieties, have proven to be highly efficient in the enantioselective diethylzinc addition to aryl and alkyl aldehydes to give the desired products in very high yields (up to 99%) and with ee's up to 97%. The influence of the stereogenic centers located on the sulfinyl sulfur atom and in the aziridine moiety on the stereochemical course of the reaction are discussed.

1. Introduction

The asymmetric diethylzinc addition to aldehydes is one of the most intensely investigated reactions leading to the formation of C–C bonds and serves as a test model for newly developed catalysts. Although over 20 years have already passed since the first report on the asymmetric addition of diethylzinc to benzaldehyde in the presence of leucinol, the search for new catalysts for this reaction still continues. The last comprehensive overview in this field was in 2001,[1] and has been followed by a number of new original reports. An inspection of the types of compounds that exhibit the best catalytic activity in this reaction clearly shows that the most efficient are chiral aminoalcohols. In most cases the stereogenic centers, which serve as a source of chirality, are located on carbon atoms, or the catalyst molecule is built on the basis of an axially chiral substrate. Although the stereogenic sulfinyl moiety is known to exert high asymmetric induction,[2] particularly when used as a chiral auxiliary, only a few catalysts have been used, which possess a stereogenic center located on the sulfur.[3–5] Moreover, in those cases only hydroxy sulfoximines[3] and hydroxy sulfoxides[4,5] were applied and proved to be rather poor catalysts.

Taking into account the findings that amino alcohols were the most efficient catalysts for this type of reaction, we decided to construct molecules which would have the structure of amino alcohols and bear an additional stereogenic sulfinyl group. To this end, we have recently synthesized a series of tridentate ligands, containing hydroxyl, sulfinyl, and amine moieties, via an enzyme-promoted desymmetrization of a prochiral bis-hydroxy sulfoxide, followed by attaching an enantiomeric amine.[6] However, the newly synthesized ligands, containing chiral open-chain amines turned out to be quite inefficient as catalysts and led to the product of the diethylzinc addition to benzaldehyde in yields of up to 55% and with ee's not exceeding 50%.[6]

In order to improve the efficiency of those catalysts via the introduction of other types of chiral amines, we turned our attention to enantiomeric aziridines, which are readily available from the corresponding amino alcohols obtained, in turn, by reduction of the appropriate natural aminoacids or their opposite enantiomers.[7] It is noteworthy that variously substituted aziridines have already been found to be very good catalysts for the title reaction.[8–13] Moreover, aziridines are known to efficiently coordinate organozinc compounds.[14–16] Therefore, we decided to apply the methodology developed by us earlier[6] to the synthesis of the new type of sulfinyl catalyst containing achiral and chiral aziridines.

2. Results and discussion

2.1. Synthesis of tridentate catalysts from monoacetate 1

The synthesis of tridentate catalysts **4a–d** comprised of three steps (Scheme 1). First, enantiomerically pure monoacetate **1** was mesylated with methanesulfonic anhydride in dichloromethane in the presence of triethylamine to form the mesyl derivative **2** in quantitative yield.[6] Mesylate **2** was then reacted with a series of enantiomerically pure aziridines[7] in chloroform in the presence of triethylamine leading to the products **3a–d** in chemical yields of 80–92%. Finally, the crude compounds **3a–d** were subjected to deacetylation with sodium methoxide in methanol to give products **4a–d** in yields 90–95%. The results are shown in Table 1.

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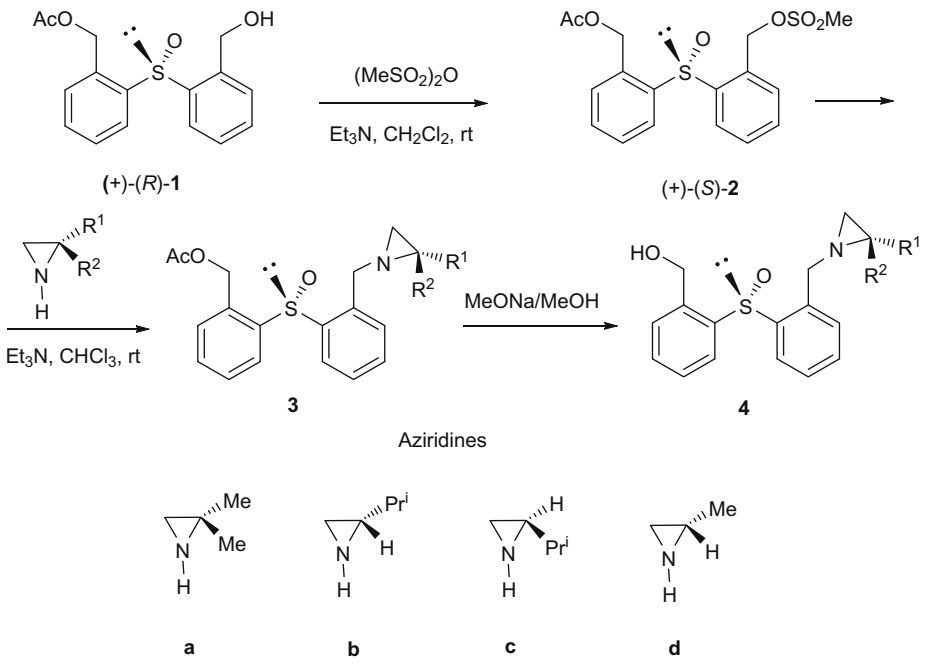


Scheme 1. Synthesis of chiral aziridine-substituted tridentate catalysts.

Table 1
Synthesis of products **3** and **4**

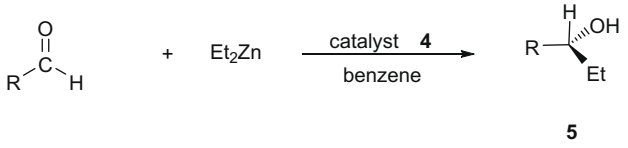
Entry	Aziridine			Products **3**	Products **4**		
	Symbol	R^1	R^2	Yield (%)	Yield (%)	$[\alpha]_D$[a]	Absolute configuration
1	**a**	Me	Me	80	95	−1.8	(R_S)
2	**b**	*i*-Pr	H	90	93	+2.3	(R_SS_C)
3	**c**	H	*i*-Pr	88	91	−2.0	(R_SR_C)
4	**d**	Me	H	92	90	+3.5	(R_SS_C)

[a] In chloroform (*c* 1).

2.2. The use of catalysts 4 in the asymmetric addition of diethylzinc to aldehydes

Optically active compounds **4a**–**d** constitute enantiomerically pure diastereomeric tridentate ligands/catalysts, and their three nucleophilic centers can bind organometallic reagents in a stereoselective manner. Therefore, it seemed reasonable to assume that they would effectively catalyze the asymmetric addition of diethylzinc to aldehydes (Scheme 2). The results are shown in Table 2.

Inspection of Table 2 reveals several interesting findings. First, the formation of enantiomerically enriched alcohols **5** in the presence of the catalyst **4a**, containing achiral 2,2-dimethylaziridine, means that the stereogenic sulfinyl moiety must exert asymmetric induction (though moderate), since it is the sole source of chirality in this catalyst (entries 1, 5 and 9). However, if compared with the catalysts containing open-chain amine moieties described previously,[6] catalyst **4a** leads to the formation of the opposite enantiomer of the product **5** (R = Ph). This allows us to conclude that the mode of chelation of diethylzinc must be different in each catalyst type. Thus, the aziridine moiety must coordinate diethylzinc much more strongly than the open-chain amine fragment, simultaneously lowering the involvement of the hydroxy group, yet cooperating with the sulfinyl part of the catalyst molecule. This is in agreement with the next results, in which two diastereomeric catalysts **4b** and **4c**, constructed from opposite enantiomers of 2-isopropylaziridine, lead to the formation of the opposite enantiomers of alcohols **5** with high enantiomeric excess. The small differences in their ee values may be explained in terms of 'match' and 'mismatch' interactions with the stereogenic sulfinyl center. Interestingly, the original aziridines, when deprived of the sufinyl and hydroxy moieties, did not catalyze the title reaction at all.



Scheme 2. Asymmetric addition of diethylzinc to aldehydes.

When considering the unusual ability of the aziridine ring to exert the observed high asymmetric induction, it seems reasonable to take into account its capability of forming relatively stable invertomers. Thus, although the inversion process is very fast for simple amines, it is much slower in aziridines,[17] whose invertomers can in certain cases be even physically separated.[18] The inversion barrier for aziridine is $\Delta G^{\#}$ = 17.2 kcal/mol.[19] Interestingly, in the case of the adducts containing the 2,2-dimethylaziridine ring (both ligand **4a** and its *O*-acetyl precursor **3a**), their ^{1}H and ^{13}C NMR spectra clearly indicate the presence of both invertomers: a high inversion barrier around the nitrogen atom causes non-equivalence of both the methyl groups and the ring hydrogen atoms (see Section 4). A similar phenomenon has already been reported.[20] However, in case of the adducts containing 2-monosubstituted aziridine rings

Table 2
Asymmetric addition of diethylzinc to aldehydes in the presence of **4**

Entry	R	Catalyst	Alcohol **5**			
			Yield (%)	$[\alpha]_D$[a]	ee[b] (%)	Absolute configuration
1	Ph	**4a**	50	−22	50	(*S*)
2	Ph	**4b**	99	−45.7	97	(*S*)
3	Ph	**4c**	99	+44.0	93	(*R*)
4	Ph	**4d**	95	−44.2	94	(*S*)
5	2-$MeOC_6H_4$	**4a**	46	−25.1	48	(*S*)
6	2-$MeOC_6H_4$	**4b**	94	−50.4	96	(*S*)
7	2-$MeOC_6H_4$	**4c**	95	+50.4	96	(*R*)
8	2-$MeOC_6H_4$	**4d**	92	−48.1	92	(*S*)
9	*n*-Pr	**4a**	40	+3.2	45	(*S*)
10	*n*-Pr	**4b**	89	+6.4	90	(*S*)
11	*n*-Pr	**4c**	87	−6.4	90	(*R*)
12	*n*-Pr	**4d**	84	+5.8	82	(*S*)

[a] In chloroform (*c* 1).
[b] Determined using chiral HPLC.

3b–d and **4b–d** the inversion barrier is not that high; hence, the presence of both invertomers is not so clearly observable. Moreover, the pyramidal inversion at the aziridine nitrogen can be halted by metal complexation, for example, by zinc.[21] This may result in the stereoinduced formation of an additional stereogenic center on nitrogen, enhancing the stereoselectivity of the diethylzinc addition to carbonyl compounds. The work aimed at the isolation and determination of the structure of zinc complexes with ligands **4**, which could allow us to gain more information in this matter, are currently in progress in our laboratories.

3. Conclusions

The chiral tridentate ligands, containing two stereogenic centers, one located on the sulfinyl sulfur atom, the other on the carbon atom in the aziridine moiety, were found to be very efficient catalysts for the enantioselective diethylzinc addition to aldehydes. Each enantiomer of the product may be obtained by using easily available diastereomeric ligands. The stereogenic centers located on the aziridine moieties exerted a decisive influence on the absolute configuration of the products. However, the influence of the sulfinyl stereogenic center cannot be neglected, since the reaction is stereoselective even if the ligand bears an achiral aziridine moiety. The mode of chelation of the diethylzinc molecule must be different for the aziridine-containing ligands when compared with those containing open-chain amine moieties.

4. Experimental

4.1. General

NMR spectra were recorded on a Bruker instrument at 200 MHz with $CDCl_3$ and CD_3OD as solvents. Optical rotations were measured on a Perkin–Elmer 241 MC polarimeter (*c* 1). Column chromatography was carried out using Merck 60 silica gel. TLC was performed on Merck 60 F_{254} silica gel plates. The enantiomeric excess (ee) values were determined by chiral HPLC (KNAUER, Chiralpak AS).

Aziridines **a–d** were synthesized according to the procedure reported in the literature.[7]

4.2. Synthesis of tridentate ligands 4a–d—general procedure

Enantiomerically pure monoacetate **1** ($[\alpha]_D$ = +60, ee >99, 1 g, 3.29 mmol) was dissolved in dichloromethane (20 mL) and triethylamine (0.4 g, 3.95 mmol) and mesyl anhydride (0.69 g, 3.95 mol) were added. The mixture was stirred at room temperature during 2 h. After this time, water was added and both phases were separated. The aqueous phase was extracted with dichloromethane (3×). After drying over anhydrous magnesium sulfate and evaporation of solvent, the mesyl derivative **2** was obtained (1.257 g, 100%, $[\alpha]_D$ = +12.4). ^{1}H NMR ($CDCl_3$): δ = 1.93 (s, 3H), 2.92 (s, 3H), 5.33 (AB, 2H), 5.47 (s, 2H), 7.59–7.86 (m, 8H). MS (CI): *m*/*z* 383 (M+H). HRMS (CI) calcd for $C_{17}H_{19}O_6S_2$: 383.4512; found 383.4587.

Mesylate **2** (0.5 mmol) was dissolved in chloroform (10 mL) and triethylamine (0.5 mmol) and corresponding aziridine (0.5 mmol) was added. The mixture was stirred at room temperature for 72 h. After this time chloroform was evaporated and the residue was purified via preparative TLC (chloroform/methanol 20:1) to give derivatives **3a–d**. Their chemical purity was determined on the basis of TLC, ^{1}H NMR, and ^{13}C NMR spectra. They were subjected to ensuing deacetylation without further analysis and purification.

4.2.1. Compound 3a (as a mixture of invertomers)

^{1}H NMR ($CDCl_3$): δ = 1.10–1.30 (m, 7H), 1.66, 1.78 (2s, 1H), 1.79, 2.00 (2s, 3H), 3.60–3.80 (m, 2H), 5.10–5.45 (m, 2H), 7.35–7.85 (m, 8H). ^{13}C NMR ($CDCl_3$): δ = 16.58 (*C*H_3), 20.46 (*C*H_3CO), 26.06 (*C*H_3), 36.44, 36.75 (2*C*), 40.55, 40.81 (2CH_2), 52.82, 53.34 (2CH_2N), 62.14, 62.34 (2CH_2O), 125.81, 125.96 (2C_{ar}), 126.54, 126.62 (2C_{ar}), 127.66, 127.69 (2C_{ar}), 128.06, 128.21 (2C_{ar}), 129.34, 129.37 (2C_{ar}), 129.44, 129.59 (2C_{ar}), 131.22, 131.22 (2C_{ar}), 131.34, 131.40 (2C_{ar}), 134.51, 134.74 (2*C*), 139.19, 139.43 (2*C*), 140.26, 140.82 (2*C*), 142.00, 142.37 (2*C*), 170.28 (*C*=O).

4.2.2. Compound 3b

^{1}H NMR ($CDCl_3$): δ = 0.79 (d, *J* = 6.4 Hz, 3H), 0.80 (d, *J* = 6.2 Hz, 3H), 1.08–1.16 (m, 1H), 1.19–1.28 (m, 2H), 1.50 (d, *J* = 3.4 Hz, 1H), 3.30–3.48 (m, 2H), 5.20 (d, *J* = 3.1 Hz, 1H), 5.35 (d, *J* = 3.1 Hz, 1H), 7.35–7.55 (m, 6H), 7.60–7.80 (m, 2H). ^{13}C NMR ($CDCl_3$): δ = 19.30 (*C*H_3), 20.29 (*C*H_3CO), 20.57 (*C*H_3), 30.90 (*C*H), 32.70 (*C*H), 46.91 (*C*H_2), 61.45 (*C*H_2N), 62.38 (*C*H_2O), 125.97 (C_{ar}), 126.65 (C_{ar}), 128.13 (C_{ar}), 129.21 (C_{ar}), 129.36 (C_{ar}); 129.42 (C_{ar}), 131.07 (C_{ar}), 131.19 (C_{ar}), 134.74 (*C*), 138.41 (*C*), 141.75 (*C*), 142.65 (*C*), 170.21 (*C*=O).

4.2.3. Compound 3c

^{1}H NMR ($CDCl_3$): δ = 0.68 (d, *J* = 6.4 Hz, 3H), 0.79 (d, *J* = 6.6 Hz, 3H), 1.03–1.15 (m, 1H), 1.12–1.25 (m, 1H), 1.29 (d, *J* = 6.1 Hz, 1H), 1.61 (d, *J* = 3.0 Hz, 1H), 1.94 (s, 3H), 3.43 (d, *J* = 14.1 Hz, 1H), 3.64 (d, *J* = 14.1 Hz, 1H), 5.14 (d, *J* = 13.0 Hz, 1H), 5.30 (d, *J* = 13.0 Hz, 1H), 7.35–7.53 (m, 5H), 7.55–7.85 (m, 3H). ^{13}C NMR ($CDCl_3$): δ = 19.28 (*C*H_3), 20.34 (*C*H_3CO), 20.58 (*C*H_3), 31.03 (*C*H), 32.76 (*C*H), 46.97 (*C*H_2), 60.61 (*C*H_2N), 62.38 (*C*H_2O), 126.03 (C_{ar}), 126.43 (C_{ar}), 126.72 (C_{ar}), 128.16 (C_{ar}), 129.40 (2C_{ar}), 129.53 (C_{ar}),

131.25 (C_{ar}), 134.48 (*C*), 138.59 (*C*), 141.32 (*C*), 142.47 (*C*), 170.26 (*C*=O).

4.2.4. Compound 3d

^{1}H NMR ($CDCl_3$): δ = 1.15 (d, *J* = 6.0 Hz, 3H), 1.25–1.60 (m, 3H), 1.98 (s, 3H), 3.40–3.60 (m, 2H), 5.18–5.45 (m, 2H), 7.35–7.85 (m, 8H). ^{13}C NMR ($CDCl_3$): δ = 17.80 (CH_3), 20.65 (CH_3CO), 34.61 (*CH*), 35.25 (CH_2), 60.68 (CH_2N), 62.48 (CH_2O), 126.18 (C_{ar}), 126.63 (C_{ar}), 128.04 (C_{ar}), 128.62 (C_{ar}), 129.56 (2C_{ar}), 131.32 (2C_{ar}), 134.70 (*C*), 138.57 (*C*), 141.22 (*C*), 142.58 (*C*), 170.40 (*C*=O).

Compound **3** (0.15 mmol) was dissolved in methanol (2 mL) after which sodium (0.15 mmol) was slowly added. The mixture was stirred at room temperature for 0.5 h. After this time methanol was evaporated and the residue was separated by preparative TLC (chloroform/methanol 10:1) to give chiral tridentate ligands **4a**–**d**.

4.2.5. Compound 4a (as a mixture of invertomers)

^{1}H NMR ($CDCl_3$): δ = 1.05–1.45 (m, 7H), 1.70, 1.88 (2s, 1H), 3.05–3.70 (m, 1.5H), 4.00–4.85 (m, 2.5H), 6.00 (s, br, 1H), 7.15–7.65 (m, 7H), 7.90–8.20 (m, 1H). ^{13}C NMR ($CDCl_3$): δ = 17.49, 17.99 (2CH_3), 25.36, 26.31 (2CH_3), 38.22, 38.60 (2C_q), 40.38, 40.94 (2CH_2), 52.79, 54.21 (2CH_2N), 61.65, 61.87 (2CH_2O), 125.84, 126.13 (2C_{ar}), 126.53, 128.10 (2C_{ar}), 127.79, 128.33 (2C_{ar}), 129.33, 129.57 (2C_{ar}), 129.62, 129.73 (2C_{ar}), 129.84, 130.20 (2C_{ar}), 130.68, 130.93 (2C_{ar}), 131.68, 131.74 (2C_{ar}), 138.35, 138.99 (2*C*), 139.93, 140.24 (2*C*), 142.48, 142.60 (2*C*), 143.29, 144.05 (2*C*). MS (CI): *m*/*z* = 316 (M+H). HRMS (FAB) calcd for $C_{18}H_{22}NO_2S$: 316.1371; found 316.1372.

4.2.6. Compound 4b

^{1}H NMR ($CDCl_3$): δ = 0.63 (d, *J* = 6.5 Hz, 3H), 0.88 (d, *J* = 6.6 Hz, 3H), 1.05–1.15 (m, 1H), 1.50–1.65 (m, 1H), 1.69 (d, *J* = 6.6 Hz, 1H), 1.83 (d, J = 3.8 Hz, 1H), 2.69 (d, *J* = 12.5 Hz, 1H), 4.10 (d, *J* = 13.0 Hz, 1H), 4.44 (d, *J* = 13.0 Hz, 1H), 4.97 (d, *J* = 12.5 Hz, 1H), 6.90 (s, br, 1H), 7.20–7.60 (m, 7H), 8.20–8.35 (m, 1H). ^{13}C NMR ($CDCl_3$): δ = 19.46 (CH_3), 20.50 (CH_3), 30.97 (*CH*), 32.77 (*CH*), 48.19 (CH_2), 60.73 (CH_2N), 61.77 (CH_2O), 125.66 (C_{ar}), 127.96 (C_{ar}), 128.15 (C_{ar}), 129.78 (C_{ar}), 129.92 (C_{ar}), 130.45 (C_{ar}), 130.69 (C_{ar}), 131.58 (C_{ar}), 137.62 (*C*), 139.52 (*C*), 142.48 (*C*), 143.88 (*C*). MS (CI): *m*/*z* = 330 (M+H). HRMS (FAB) calcd for $C_{19}H_{24}NO_2S$: 330.1528; found 330.1527.

4.2.7. Compound 4c

^{1}H NMR ($CDCl_3$): δ = 0.58 (d, *J* = 6.4 Hz, 3H), 0.81 (d, *J* = 6.6 Hz, 3H), 0.98–1.15 (m, 1H), 1.35–1.50 (m, 1H), 1.59 (d, *J* = 6.6 Hz, 1H), 1.73 (d, *J* = 3.8 Hz, 1H), 2.68 (d, *J* = 12.6 Hz, 1H), 4.09 (d, *J* = 13.0 Hz, 1H), 4.37 (d, *J* = 13.0 Hz, 1H), 4.77 (d, *J* = 12.6 Hz, 1H), 6.75 (s, br, 1H), 7.15–7.70 (m, 7H), 8.10–8.20 (m, 1H). ^{13}C NMR ($CDCl_3$): δ = 19.85 (CH_3), 20.42 (CH_3), 30.81 (*CH*), 32.70 (*CH*), 47.97 (CH_2), 60.51 (CH_2N), 61.59 (CH_2O), 125.51 (C_{ar}), 128.06 (C_{ar}), 129.00 (C_{ar}), 129.35 (C_{ar}), 129.73 (C_{ar}), 130.11 (C_{ar}), 130.43 (C_{ar}), 131.55 (C_{ar}), 138.13 (*C*), 139.49 (*C*), 142.20 (*C*), 143.51 (*C*). MS (CI): *m*/*z* = 330 (M+H). HRMS (FAB) calcd for $C_{19}H_{24}NO_2S$: 330.1528; found 330.1526.

4.2.8. Compound 4d

^{1}H NMR ($CDCl_3$): δ = 1.25 (d, *J* = 5.5 Hz, 3H), 1.52 (d, *J* = 4.0 Hz, 1H), 1.59 (d, *J* = 6.5 Hz, 1H), 1.75–1.95 (m, 1H), 2.74 (d, *J* = 13.0 Hz, 1H), 4.13 (d, *J* = 13.0 Hz, 1H), 4.52 (d, *J* = 13.0 Hz, 1H), 4.80 (d, *J* = 13.0 Hz, 1H), 6.70 (s, br, 1H), 7.20–7.60 (m, 7H), 8.10–8.25 (m, 1H). ^{13}C NMR ($CDCl_3$): δ = 17.12 (CH_3), 34.60 (*CH*), 35.32 (CH_2), 60.87 (CH_2N), 61.58 (CH_2O), 125.77 (C_{ar}), 127.84 (C_{ar}), 128.32 (C_{ar}), 129.56 (C_{ar}), 129.74 (C_{ar}), 130.18 (C_{ar}), 130.76 (C_{ar}), 131.71 (C_{ar}), 137.70 (*C*), 139.84 (*C*), 142.20 (*C*), 143.43 (*C*). MS (CI): *m*/*z* = 302 (M+H). HRMS (FAB) calcd for $C_{17}H_{20}NO_2S$: 302.1214; found 302.1216.

4.3. Asymmetric addition of diethylzinc to aldehydes—general procedure

Chiral catalysts **4a**–**d** (0.1 mmol) and benzene (10 mL) were placed in a flask. To ensure dryness, 5 mL of benzene were distilled off. The mixture was cooled to 0 °C and a solution of diethylzinc (1.0 M solution in hexane, 3 mmol) was added under an argon atmosphere. After stirring for 0.5 h, an aldehyde (1 mmol) was added at 0 °C and the mixture was stirred at room temperature for 12 h. After this time, a 5% aqueous solution of hydrochloric acid was added to the mixture. Both layers were separated and the aqueous layer was extracted with diethyl ether (4×). The combined organic layers were washed with brine (10 mL) and dried over $MgSO_4$. The solvents were evaporated to give the crude alcohols **5**, which were purified by preparative TLC (ethyl acetate/hexane 1:7). They were identified by comparison of their ^{1}H NMR spectra with those reported in the literature. Their absolute configurations were determined in the same way: for the alcohol **5** (R = Ph),[6] for **5** (R = 2-$MeOC_6H_4$)[22] and for **5** (R = *n*-Pr).[23]

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