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Limonene oxide derived aziridinyl alcohols as highly efficient catalysts for asymmetric additions of organozinc species to aldehydes

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ABSTRACT

The facile synthesis of a series of novel catalysts bearing a tertiary hydroxyl group and an aziridine moiety as chelating centers constructed on the scaffold derived from limonene oxide is described. The newly prepared compounds have been tested for the enantioselective addition of diethylzinc and phenylethynylzinc to aryl and alkyl aldehydes, affording the corresponding chiral alcohols in very high chemical yields (up to 96%) and with excellent ee's of ca. 95%.

1. Introduction

The enantioselective carbon–carbon bond formation using organometallic reagents is a commonly used methodology for the synthesis of nonracemic compounds.[1–5] Moreover, the asymmetric additions of zinc organic species (especially diethylzinc) are recognized as one of the most effective methods for generating enantiomerically pure secondary alcohols.[6–9] In the search for novel enantiomerically pure ligands, the chiral pool of terpenes is of enormous synthetic value.[10–12] Recently we have described the preparation of a series of enantiomerically pure aziridine alcohols derived from salicylic and 2-hydroxymethylbenzoic acid, (*S*)-mandelic and (*S*)-lactic acid, and their excellent catalytic effectiveness in the asymmetric addition of diethylzinc and phenylethynylzinc to aliphatic and aromatic aldehydes.[5,13,14]

According to literature protocols, some β-amino alcohols derived from limonene oxide have been used for the asymmetric addition of diethylzinc[9] and phenylethynylzinc[15] to aldehydes, affording the corresponding chiral alcohols in high chemical yields and with ee's ranging from moderate[15] (ca. 40%) to high[9] (up to 80%).

With all of the aforementioned results in mind and taking into account the fact that aziridines exhibit a special affinity to coordinate with organozinc species,[16–18] we decided to synthesize a series of novel compounds using limonene oxide as a key building block, and to test their catalytic activity toward the asymmetric addition of diethylzinc and phenylethynylzinc to various aliphatic and aromatic aldehydes.

2. Results and discussion

2.1. Synthesis of the ligands

The straightforward one-step synthesis of our new ligands is depicted in Scheme 1. As previously described,[15,19] the *cis*-diastereomer of (*R*)-(+)-limonene oxide can be isolated from the commercially available (1:1) diastereomeric mixture of limonene oxides. Selective epoxide ring opening with aziridines allows for the formation of β-aziridine alcohols from the *trans*-epoxide. The above 1:1 mixture of diastereomers of (+)-limonene oxide was reacted with a series of enantiomerically pure aziridines **3a–d** in refluxing water for 24 h in the presence of 1% triethylamine following the protocols described by Steiner *et al.*[9] and Spreitzer et al.[20]

The chemical yields and specific rotations of the newly synthesized ligands **4a–d** are collected in Table 1.

Using this reaction we were unable to synthesize a series of (1*S*,2*S*,4*R*)-stereoisomers of β-amino alcohols **4a–d** derived from (+)-limonene oxide. This observation was in full agreement with the literature report.[9]

2.2. Screening of the ligands

In order to check the catalytic activity of novel ligands **4a–d** in the asymmetric additions of organo zincs to aldehydes, we selected the reactions of diethylzinc and phenylethynylzinc with benzaldehyde as the model transformations (Scheme 2). The reactions were carried out under standard conditions (in toluene or THF, respectively),[21,22] and the results are shown in Table 2.

The results in Table 2 clearly show that all of new aziridines **4a–d** derived from (+)-limonene oxide are prone to catalyze the selected model reactions and give the desired products **5a** and **6a**

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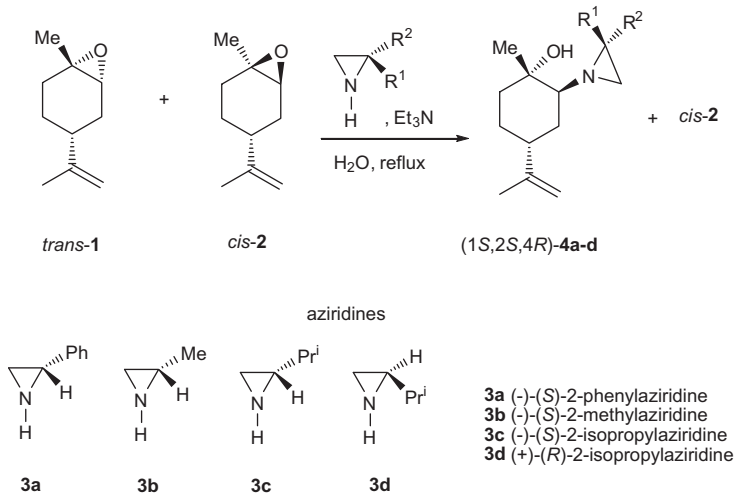
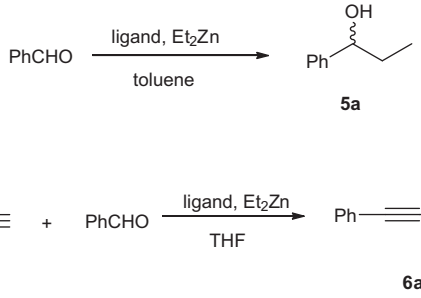


Scheme 1. Synthesis of ligands **4a–d**.

Table 1
One-step synthesis of ligands **4a–d**

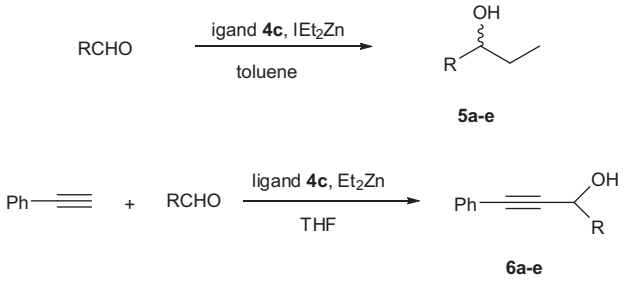
Entry	Ligand	Yield (%)	$[\alpha]_D$[a]
1	**4a**	82	−14.8
2	**4b**	85	+9.3
3	**4c**	83	+11.2
4	**4d**	82	−2.0

[a] In chloroform at room temperature (*c* 1).



Scheme 2. Asymmetric addition of diethyl- and phenylethynylzinc to benzaldehyde.

in high chemical yields (>90%) and with excellent enantiomeric excesses of >90%. The results also suggest that the presence of a stereogenic center on the aziridine moiety is not of crucial importance with respect to the stereochemical outcome of the catalyzed reactions (this is in contrast to our previous findings in which the aziridine moiety exerts a decisive influence on the absolute configuration of the chiral product[5,13,14,21,22]). A series of experiments promoted by diastereomeric ligands **4c** and **4d** gave products that exhibited comparable specific rotation values and the same absolute configuration (Table 2, entries 3 and 4) and this supported this assumption. The absolute configurations at the newly generated stereogenic centers of **5a** and **6a** were assigned according to the literature reports.[23]



Scheme 3. Additions of diethyl- and phenylethynylzinc to aldehydes in the presence of ligand **4c**.

2.3. Asymmetric additions of diethyl- and phenylethynylzinc to selected aldehydes in the presence of ligand 4c

With the novel effective ligands in hand, we next determined the scope of the catalytic activity of selected ligand **4c**. Thus, it was used for the title transformations performed with a series of aldehydes as shown in Scheme 3. The results are summarized in Table 3.

Table 2
Screening of ligands **4a–d**

Entry	Ligand	Product **5a**				Product **6a**			
		Yield (%)	$[\alpha]_D$[a]	ee (%)[b]	Abs. config.	Yield (%)	$[\alpha]_D$[a]	ee (%)[b]	Abs. config.[c]
1	**4a**	96	+42.3	94	(*R*)	92	−4.8	91	(*S*)
2	**4b**	92	+40.5	90	(*R*)	89	−4.5	88	(*S*)
3	**4c**	97	+43.1	96	(*R*)	96	−4.9	95	(*S*)
4	**4d**	91	+40.0	89	(*R*)	88	−4.4	86	(*S*)

[a] In chloroform at room temperature (*c* 1).
[b] Determined using chiral HPLC.
[c] According to the literature data.[23]

Table 3
Additions of diethyl- and phenylethynylzinc to aldehydes in the presence of ligand **4c**

Entry	R	Products **5a–e**				Products **6a–e**			
		Yield (%)	$[\alpha]_D$[a]	ee (%)[b]	Abs. config.[c]	Yield (%)	$[\alpha]_D$[a]	ee (%)[b]	Abs. config.[c]
1	Ph	97	+43.1	96	(*R*)	96	−4.9	95	(*S*)
2	2-$MeOC_6H_4$	95	+48.1	92	(*R*)	91	+7.5	90	(*S*)
3	*n*-Pr	92	−6.5	93	(*R*)	92	−3.2	92	(*S*)
4	4-BrC_6H_4	94	+8.3	95	(*R*)	93	−3.9	94	(*S*)
5	2-MeC_6H_4	92	+41.5	93	(*R*)	90	+11.3	91	(*S*)

[a] In chloroform at room temperature (*c* 1).
[b] Determined using chiral HPLC.
[c] According to the literature data.[4,21,22,24]

The results shown in Tables 2 and 3 clearly indicate that the selected ligand **4c** should be considered as a highly effective catalyst for the title reactions to give the desired chiral alcohols in excellent yields and ee's. Moreover, careful analysis of the absolute configurations of the products shown in Table 3 suggests that the attack of organozinc species promoted by **4c** always takes place from the same side, which is in full agreement with the literature transition state models proposed for diethylzinc[9] and alkynylzinc[15] additions catalyzed by alcohols derived from (+)-limonene oxide. The use of limonene amino alcohols with a (1*S*,2*S*,4*R*)-absolute configuration [from (+)-limonene oxide] led to the formation of the (*R*)-configured adducts of diethylzinc and the (*S*)-configured adducts of phenylethynylzinc, with the aldehydes being compatible with the facial selectivity in both cases.[9,15]

3. Conclusion

Chiral ligands of type **4** derived from (+)-limonene oxide containing five stereogenic centers were found to be highly efficient catalysts for the enantioselective addition of diethyl- and phenylethynylzinc to various aldehydes. The stereogenic centers located on the terpene moiety exerted a decisive influence on the stereochemistry of the reactions leading to the desired alcohols with the same absolute configuration [(*R*) in the case of diethylzinc and (*S*) in the case of phenylethynylzinc].

4. Experimental

4.1. General

Unless otherwise specified, all reagents were purchased from commercial suppliers and used without further purification. Tetrahydrofuran and toluene were distilled from sodium benzophenone ketyl radical. ^1H and ^{13}C NMR spectra were recorded on a Bruker instrument at 600 and 151 MHz, respectively, with $CDCl_3$ as the solvent and relative to TMS as the internal standard. Data are reported as s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, b = broad. Optical rotations were measured on a Perkin–Elmer 241 MC polarimeter with a sodium lamp at room temperature (*c* 1). Melting points were determined on a MELTEMP apparatus and are uncorrected. Column chromatography was carried out using Merck 60 silica gel. TLC was performed on Merck 60 F_{254} silica gel plates. Visualization was accomplished with UV light (254 nm) or using iodine vapor. The enantiomeric excess (ee) values were determined by chiral HPLC (Knauer, Chiralcel OD). Aziridines **3a–d** were prepared according to the literature procedures.[25,26]

4.2. Synthesis of the ligands 4a–d—general procedure[9,20]

The *cis*/*trans*-(+)-limonene oxide mixture (0.23 g, 1.5 mmol) was mixed with the corresponding enantiomerically pure aziridine (9.5 mmol) and triethylamine (0.15 mL) in deionized water (5 mL) and the reaction mixture was refluxed for 24 h. Next, water was evaporated in vacuo and the residue was subjected to column chromatography (silica gel, hexane with ethyl acetate in gradient) to afford the corresponding products **4a–d**. Chemical yields and optical rotation values are shown in Table 1.

*Ligand **4a** (colorless oil):* ^1H NMR ($CDCl_3$): δ = 1.40 (s, 3H), 1.55–1.59 (m, 1H), 1.60 (s, 3H), 1.60–1.66 (m, 4H), 1.73–1.77 (m, 1H), 1.80–1.89 (m, 2H), 2.03 (d, *J* = 3.6 Hz, 1H), 2.18–2.25 (m, 1H), 2.43 (dd, *J* = 3.6, 5.5 Hz, 1H), 4.59–4.61 (m, 1H), 4.63–4.65 (m, 1H), 7.28–7.38 (m, 5H); ^{13}C NMR ($CDCl_3$): δ = 20.9 (CH_3), 26.7 (CH_{2az}), 28.3 (CH_2), 32.9 (CH_3), 35.0 (CH_2), 37.8 (*C*H), 38.1 (CH_2), 41.1 (CH_{az}), 72.3 (*C*H), 73.6 (C_q), 108.3 (CH_2), 126.3 ($2C_{ar}$), 126.7 (C_{ar}), 128.2 ($2C_{ar}$), 140.5 ($C_{q\ ar}$), 150.3 (C_q); MS (CI): *m*/*z* 272 (M+H); HRMS (CI): calcd for $C_{18}H_{25}NO$: 272.1246; found 272.1250.

*Ligand **4b** (colorless oil):* ^1H NMR ($CDCl_3$): δ = 1.21 (d, *J* = 6.0 Hz, 3H), 1.23 (d, *J* = 6.0 Hz, 1H), 1.33 (s, 3H), 1.37–1.39 (m, 1H), 1.44 (d, *J* = 3.6 Hz, 1H), 1.48–1.59 (m, 3H), 1.62–1.67 (m, 2H), 1.76 (s, 3H), 1.74–1.80 (m, 2H), 2.01–2.08 (m, 1H), 2.43–2.50 (m, 1H), 4.72–4.76 (m, 2H); ^{13}C NMR ($CDCl_3$): δ = 18.2 (CH_{3az}), 21.0 (CH_3), 26.9 (CH_{2az}), 28.2 (CH_2), 30.8 (CH_2), 32.1 (CH_3), 35.1 (CH_2), 37.9 (*C*H), 38.1 (CH_{az}), 72.0 (*C*H), 73.7 (C_q), 108.3 (CH_2), 150.4 (C_q); MS (CI): *m*/*z* 209 (M+H); HRMS (CI): calcd for $C_{13}H_{22}NO$: 209.1276; found 209.1273.

*Ligand **4c** (colorless oil):* ^1H NMR ($CDCl_3$): δ = 0.89 (d, *J* = 6.9 Hz, 3H), 1.02 (d, *J* = 6.7 Hz, 3H), 1.06 (d, *J* = 6.7 Hz, 1H), 1.33 (s, 3H), 1.38–1.47 (m, 4H), 1.49–1.53 (m, 1H), 1.59–1.64 (m, 3H), 1.65–1.72 (m, 1H), 1.75 (s, 3H), 1.78–1.83 (m, 1H), 1.93–2.00 (m, 1H), 2.44–2.50 (m, 1H), 4.73–4.74 (m, 2H); ^{13}C NMR ($CDCl_3$): δ = 18.1 (CH_{3az}), 20.9 (CH_{3az}), 21.3 (CH_3), 26.6 (CH_{2az}), 26.8 (CH_2), 27.1 (CH_{az}), 29.8 (CH_2), 32.1 (CH_3), 35.1 (CH_2), 38.1 (CH_{az}), 47.9 (*C*H), 72.6 (*C*H), 73.0 (C_q), 108.7 (CH_2), 149.7 (C_q); MS (CI): *m*/*z* 238 (M+H); HRMS (CI): calcd for $C_{15}H_{27}NO$: 238.1650; found 238.1660.

*Ligand **4d** (colorless oil):* ^1H NMR ($CDCl_3$): δ = 0.83 (d, *J* = 6.8 Hz, 3H), 1.04 (d, *J* = 6.7 Hz, 3H), 1.08 (d, *J* = 6.7 Hz, 1H), 1.35 (s, 3H), 1.41–1.48 (m, 4H), 1.49–1.52 (m, 1H), 1.61–1.66 (m, 3H), 1.67–1.73 (m, 1H), 1.76 (s, 3H), 1.80–1.85 (m, 1H), 1.95–2.01 (m, 1H), 2.46–2.51 (m, 1H), 4.75–4.76 (m, 2H); ^{13}C NMR ($CDCl_3$): δ = 18.1 (CH_{3az}), 20.9 (CH_{3az}), 21.3 (CH_3), 26.6 (CH_{2az}), 26.9 (CH_2), 27.1 (CH_{az}), 29.8 (CH_2), 32.1 (CH_3), 35.1 (CH_2), 38.1 (CH_{az}), 47.9 (*C*H), 72.6 (*C*H), 73.0 (C_q), 108.7 (CH_2), 149.7 (C_q); MS (CI): *m*/*z* 238 (M+H); HRMS (CI): calcd for $C_{15}H_{27}NO$: 238.1650; found 238.1653.

4.3. Asymmetric addition of diethylzinc to aldehydes; general procedure[21]

Chiral catalysts of type **4** (0.1 mmol) in dry toluene (5 mL) were placed in a round-bottomed flask. The mixture was cooled to 0 °C and a solution of diethylzinc (1.0 M sln. in hexane, 3.0 mmol) was added under argon. After stirring for 30 min, an aldehyde (1.0 mmol) was added at 0 °C, and the mixture was stirred at room

temperature overnight. Next, a 5% HCl aqueous solution was added, the layers were separated, and the aqueous phase was extracted with diethyl ether (4 × 10 mL). The combined organic layers were washed with brine (10 mL) and dried over anhydrous $MgSO_4$. The solvents were evaporated off to afford the crude alcohols **5a**–**e**, which were purified via column chromatography on silica gel (hexane with ethyl acetate in gradient). The yields, specific rotations, enantiomeric excess values, and the absolute configurations of products **5a**–**e** are collected in Table 3. The spectroscopic data were in full agreement with those reported in the literature.[4,21–23]

4.3.1. (*R*)-(+)-1-Phenylpropanol 5a

Enantiomeric excess was determined by HPLC with a Chiralcel OD–H column (99:1 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 15.5 min, minor enantiomer t_R = 18.1 min; ee = 96%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[27]

4.3.2. (*R*)-(+)-1-(2-Methoxyphenyl)-1-propanol 5b

Enantiomeric excess was determined by HPLC with a Chiralcel OD–H column (98:2 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 30.3 min, minor enantiomer t_R = 27.4 min; ee = 92%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[28]

4.3.3. (*R*)-(-)-3-Hexanol 5c

Enantiomeric excess was determined by HPLC with a Chiralcel OD–H column (99:1 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 24.7 min, minor enantiomer t_R = 30.6 min; ee = 93%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[21]

4.3.4. (*R*)-(+)-1-(4-Bromophenyl)-1-propanol 5d

Enantiomeric excess was determined by HPLC with a Chiralcel OD–H column (98:2 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 25.5 min, minor enantiomer t_R = 22.1 min; ee = 95%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[28]

4.3.5. (*R*)-(+)-1-(2-Methylphenyl)-1-propanol 5e

Enantiomeric excess was determined by HPLC with a Chiralcel OD–H column (98:2 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 20.2 min, minor enantiomer t_R = 23.1 min; ee = 93%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[21]

4.4. Asymmetric addition of phenylethynylzinc to aldehydes; general procedure[22]

To a solution of ligand of type **4** (0.2 mmol) in THF (5 mL), was added a solution of diethylzinc (1.4 mL, 1.4 mmol, 1.0 M in hexane) at room temperature under argon. After the mixture was stirred at ambient temperature for 30 min, phenylacetylene (154 μL, 1.4 mmol) was added, and stirring was continued for another 30 min. The solution was then cooled to 0 °C (ice bath) and treated with the corresponding aldehyde (1.0 mmol), and the resulting mixture was stirred for 2 h at 0 °C and then overnight at room temperature. After completion of the reaction (TLC monitoring), it was quenched with 5% aqueous HCl. The resulting mixture was extracted with diethyl ether (4 × 10 mL) and the combined organic layers were washed with brine. After the organics were dried over anhydrous $MgSO_4$ the solvents were removed in vacuo. The residue was purified by column chromatography (silica gel, hexane with ethyl acetate in gradient) to afford the corresponding products **6a**–**e**. The yields, specific rotations, enantiomeric excess values, and the absolute configurations of the products **6a**–**e** are collected in Table 3. The spectroscopic data were in full agreement with those reported in the literature.[4,21–24]

4.4.1. (*S*)-(−)-1,3-Diphenyl-prop-2-yn-1-ol 6a

Enantiomeric excess was determined by HPLC with a Chiralcel OD–H column (98:2 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 11.8 min, minor enantiomer t_R = 21.2 min; ee = 95%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[29]

4.4.2. (*S*)-(+)-1-(2-Methoxyphenyl)-3-phenyl-prop-2-yn-1-ol 6b

Enantiomeric excess was determined by HPLC with a Chiralcel OD column (98:2 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 22.6 min, minor enantiomer t_R = 16.8 min; ee = 90%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[29,30]

4.4.3. (*S*)-(−)-1-Phenyl-hex-1-yn-3-ol 6c

Enantiomeric excess was determined by HPLC with a Chiralcel OD column (98:2 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 16.2 min, minor enantiomer t_R = 6.5 min; ee = 92%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[30]

4.4.4. (*S*)-(−)-1-(4-Bromophenyl)-3-phenyl-prop-2-yn-1-ol 6d

Enantiomeric excess was determined by HPLC with a Chiralcel OD column (98:2 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 40.1 min, minor enantiomer t_R = 10.8 min; ee = 94%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[30]

4.4.5. (*S*)-(+)-1-(2-Methylphenyl)-3-phenyl-prop-2-yn-1-ol 6e

Enantiomeric excess was determined by HPLC with a Chiralcel OD column (98:2 hexane/isopropanol, 1 mL/min); major enantiomer t_R = 22.1 min, minor enantiomer t_R = 9.6 min; ee = 91%. The absolute configuration was ascribed by comparison of the specific rotation value with the reported data.[29]

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References

1. Liu, R.; Bai, X.; Zhang, Z.; Zi, G. *Appl. Organomet. Chem.* **2008**, *22*, 671–675.
2. Dabiri, M.; Salehi, P.; Kozehgary, G.; Heydari, A.; Esfandyari, M. *Tetrahedron: Asymmetry* **2008**, *19*, 1970–1972.
3. Gou, S.; Judeh, Z. M. A. *Tetrahedron Lett.* **2009**, *50*, 281–283.
4. Zhang, C.-H.; Yan, S.-J.; Pan, S.-Q.; Huang, R.; Lin, J. *Bull. Korean Chem. Soc.* **2010**, *31*, 869–873.
5. Rachwalski, M.; Jarzyński, S.; Jasiński, M.; Leśniak, S. *Tetrahedron: Asymmetry* **2013**, *24*, 689–693.
6. Noyori, R. *Asymmetric Catalysis in Organic Synthesis*; Wiley: New York, 1994. pp 255–297.
7. Noyori, R.; Kitamura, M. *Angew. Chem., Int. Ed. Engl.* **1991**, *30*, 49–69.
8. Pu, L.; Hong-Bin, Y. *Chem. Rev.* **2001**, *101*, 757–824.
9. Steiner, D.; Sethofer, S. G.; Goralski, C. T.; Singaram, B. *Tetrahedron: Asymmetry* **2002**, *13*, 1477–1483.
10. Bolm, C.; Zani, L.; Rudolph, J.; Schiffers, I. *Synthesis* **2004**, 2173–2180.
11. Krzemiński, M. P.; Wojtczak, A. *Tetrahedron Lett.* **2005**, *46*, 8299–8302.
12. Binder, C. M.; Bautista, A.; Zaidlewicz, M.; Krzemiński, M. P.; Oliver, A.; Singaram, B. *J. Org. Chem.* **2009**, *74*, 2337–2343.
13. Rachwalski, M.; Jarzyński, S.; Leśniak, S. *Tetrahedron: Asymmetry* **2013**, *24*, 421–425.
14. Leśniak, S.; Rachwalski, M.; Jarzyński, S.; Obijalska, E. *Tetrahedron: Asymmetry* **2013**. http://dx.doi.org/10.1016/j.tetasy.2013.09.008.
15. Watts, C. C.; Thoniyot, P.; Hirayama, L. C.; Romano, T.; Singaram, B. *Tetrahedron: Asymmetry* **2005**, *16*, 1829–1835.

16. Faure, R.; Loiseleur, H.; Bartnik, R.; Leśniak, S.; Laurent, A. *Cryst. Struct. Commun.* **1981**, *10*, 515–519.
17. Bartnik, R.; Leśniak, S.; Laurent, A. *Tetrahedron Lett.* **1981**, 4811–4812.
18. Bartnik, R.; Leśniak, S.; Laurent, A. *J. Chem. Res.* **1982**, *287*, 2701–2709.
19. Steiner, D.; Ivison, L.; Goralski, C. T.; Appell, R. B.; Gojkovic, J. R.; Singaram, B. *Tetrahedron: Asymmetry* **2002**, *13*, 2359–2363.
20. Pongprom, N.; Bachitsch, H.; Bauchinger, A.; Ettefagh, H.; Haider, T.; Hofer, M.; Knafl, H.; Slanz, R.; Waismeyer, M.; Wieser, F.; Spreitzer, H. *Monatsh. Chem.* **2010**, *141*, 53–62.
21. Leśniak, S.; Rachwalski, M.; Sznajder, E.; Kiełbasiński, P. *Tetrahedron: Asymmetry* **2009**, *20*, 2311–2314.
22. Rachwalski, M.; Leśniak, S.; Kiełbasiński, P. *Tetrahedron: Asymmetry* **2010**, *21*, 2687–2689.
23. Zhong, J.-C.; Hou, S.-C.; Bian, Q.-H.; Yin, M.-M.; Na, R.-S.; Zheng, B.; Li, Z.-Y.; Liu, S.-Z.; Wang, M. *Chem. Eur. J.* **2009**, *15*, 3069–3071.
24. Chen, Y.-J.; Lin, R.-X.; Chen, C. *Tetrahedron: Asymmetry* **2004**, *15*, 3561–3571.
25. Xu, J. *Tetrahedron: Asymmetry* **2002**, *13*, 1129–1134.
26. Buckley, B. R.; Patel, A. P.; Wijayantha, K. G. U. *J. Org. Chem.* **2013**, *78*, 1289–1292.
27. Hayashi, M.; Michigami, K. *Tetrahedron* **2013**, *69*, 4221–4225.
28. Kang, S.-Y.; Baek, J.; Ko, Y.-K.; Im, C.; Park, Y.-S. *Synlett* **2013**, 630–634.
29. Boobalan, R.; Chen, C.; Lee, G.-H. *Org. Biomol. Chem.* **2012**, *10*, 1625–1638.
30. Xu, Z.; Chen, C.; Xu, J.; Miao, M.; Yan, W.; Wang, R. *Org. Lett.* **2004**, *6*, 1193–1195.

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