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Enantioselective synthesis induced by tetrathia-[7]-helicenes in conjunction with asymmetric autocatalysis

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Abstract—Highly enantioenriched 5-pyrimidyl alkanol was formed using tetrathia-[7]-helicenes as a chiral initiator in the enantioselective addition of diisopropylzinc to pyrimidine-5-carbaldehyde, in conjunction with asymmetric autocatalysis.

1. Introduction

Since the discovery of helicene,[1] a large number of carbo- and heterohelicenes have been developed in order to reveal the unique properties of these compounds. Recently, some potential applications of these helical compounds were discovered,[2] so the preparation of helicenes became an increasingly important subject. We and others have developed convenient syntheses of heterohelicenes.[3] Helicenes, especially heterohelicenes, are promising candidates for chiral auxiliaries and ligands for various asymmetric reactions because of their stable polyconjugated helical framework and the presence of heteroatoms, for example, sulfur, nitrogen, oxygen, etc. Although asymmetric syntheses and resolution methods to obtain carbo- and heterohelicenes in enantiomerically pure form have been reported,[3] examples of reactions using them as chiral sources in asymmetric reactions have rarely appeared. To the best of our knowledge, only chiral phosphine ligands with a helicene framework[4a,b] and a chiral diol with a bishelicene framework[4c] have been prepared and used as a chiral ligand or a chiral catalyst. A challenging problem is the study of a highly enantioselective synthesis using heterohelicenes as chiral inducers or catalysts.

Over the course of our study on asymmetric autocatalysis,[5,6] we found that the 5-pyrimidyl alkanols act as excellent asymmetric autocatalysts in the enantioselective addition of diisopropylzinc (*i*-Pr_2Zn) to the corresponding pyrimidine-5-carbaldehydes, and the reaction proceeds with significant amplification of chirality to generate highly enantioenriched 5-pyrimidyl alkanols.[7–9] In addition, we have reported that enantioselective diisopropylzinc addition to pyrimidine-5-carbaldehyde triggered with carbohelicenes, in conjunction with asymmetric autocatalysis, in which the coordination of carbohelicene with pyrimidine-5-carbaldehyde are proposed as one of the possible mechanisms.[10] On the other hand, the investigation of asymmetric autocatalysis in the presence of heterohelicenes enables to expand the utility of it as a chiral ligand of alkyl metal reagent by the interaction between heteroatoms in helicene and metals.[11]

Here, we report on a highly enantioselective addition of *i*-Pr_2Zn to pyrimidine-5-carbaldehyde **1** in the presence of tetrathia-[7]-helicene **3a**[3a,12] and its dipropyl derivative **3b**,[13] which has relatively high solubility in various organic solvents due to the effect of alkyl moiety (Scheme 1). Their helical framework controlled well the absolute configuration of the formed 5-pyrimidyl alkanol **2**.

The enantiomers of tetrathia-[7]-helicenes **3a** and **3b** used in the asymmetric autocatalysis as chiral inducers were obtained by the resolution of racemic **3a** and **3b** using HPLC with a chiral stationary phase (Chiralpak IA), the mobile phase being hexane/dichloromethane (19:1, v/v). In both

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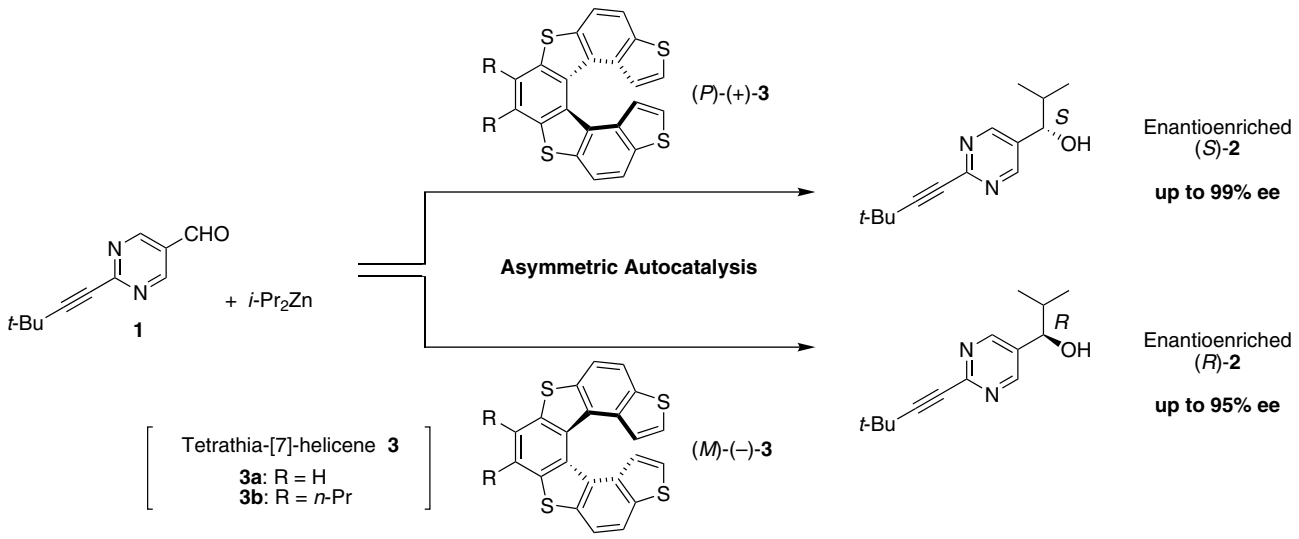

Scheme 1. Asymmetric autocatalysis initiated by tetrathia-[7]-helicenes **3**.

cases of thiahelicenes **3a** and **3b**, the enantiomers with right-handed helicity (*P*-configuration) have shorter retention times, and, in turn, the enantiomers with left-handed (*M*-configuration) show longer retention times, respectively.[14]

First, we examined the enantioselective addition of *i*-Pr_2Zn to pyrimidine-5-carbaldehyde **1** in the presence of chiral tetrathia-[7]-helicene **3a**. As shown in Table 1, in the presence of (*P*)-(+)-tetrathia-[7]-helicene **3a** with 98% ee, (*S*)-pyrimidyl alkanol **2** with 90% ee was obtained in 92% yield (entry 1). On the other hand, in the presence of (*M*)-(−)-tetrathia-[7]-helicene **3a** with 90% ee, (*R*)-**2** with 89% ee was formed in 91% yield (entry 2). Thus, the absolute configurations of the resulting pyrimidyl alkanol **2** were found to be dependent on those of the chiral thiahelicene **3a** used. When thiahelicene **3a** with low ee (10% and 12% ee) was used as a chiral inducer, pyrimidyl alkanol **2** with high ee (75% and 88% ee, respectively) was obtained with the same correlations of the absolute configurations between **3a** and **2** (entries 3 and 4). Even thiahelicene **3a** with only 2% and 3% ee were found to serve as chiral initiators in the asymmetric autocatalysis, producing (*S*)-**2** and (*R*)-**2** with 83% and 85% ee, respectively (entries 5 and 6). The results were reproducible (entries 7 and 8).

Next, the enantioselective addition of *i*-Pr_2Zn to pyrimidine-5-carbaldehyde **1** in the presence of enantioenriched dipropyltetrathia-[7]-helicenes **3b** was examined. The results are summarized in Table 2. (*P*)-(+)-Dipropyltetrathia-[7]-helicene **3b** (99% ee), used as a chiral initiator, gave (*S*)-**2** with 99% ee in 88% yield (entry 1). On the contrary, (*M*)-(−)-**3b** afforded (*R*)-**2** with 95% ee in 88% yield

Table 1. Enantioselective synthesis of pyrimidyl alkanol **2** in the presence of tetrathia-[7]-helicene **3a**

Entry[a]	Tetrathia-[7]-helicene **3a**		Pyrimidyl alkanol **2**		
	Config.	ee (%)[b]	Yield (%)[c]	ee (%)[d]	Config.
1[e]	(*P*)-(+)	98	92	90	*S*
2[e]	(*M*)-(−)	90	91	89	*R*
3	(*P*)-(+)	10	92	75	*S*
4	(*M*)-(−)	12	91	88	*R*
5	(*P*)-(+)	2	92	83	*S*
6	(*M*)-(−)	3	89	85	*R*
7	(*P*)-(+)	2	84	83	*S*
8	(*M*)-(−)	3	86	66	*R*

[a] Unless otherwise noted, the molar ratio used was tetrathia-[7]-helicene **3**/pyrimidine-5-carbaldehyde **1**/*i*-Pr_2Zn = 0.02:0.42:0.86. The aldehyde **1** and *i*-Pr_2Zn were added in three separate portions.

[b] The ee value was determined using HPLC employing a chiral stationary phase (Chiralpak IA column [4.6 × 250 mm], eluent: hexane/dichloromethane = 19:1 [v/v], flow rate: 0.7 mL min^{-1}, 254 nm UV detector, room temperature, retention time: 24.7 min for (+)-**3a**, 28.0 min for (−)-**3a**).

[c] Isolated yield.

[d] The ee value was determined using HPLC employing a chiral stationary phase (Chiralcel OD-H).

[e] The molar ratio used was **3**/**1**/*i*-Pr_2Zn = 0.02:0.84:1.57. Aldehyde **1** and *i*-Pr_2Zn were added in four separate portions.

Table 2. Enantioselective synthesis of pyrimidyl alkanol **2** in the presence of dipropyltetrathia-[7]-helicene **3b**

Entry[a]	Dipropyltetrathia-[7]-helicene **3b**		Pyrimidyl alkanol **2**		
	Config.	ee (%)[b]	Yield (%)[c]	ee (%)[d]	Config.
1	(*P*)-(+)	99	88	99	*S*
2	(*M*)-(−)	95	88	95	*R*
3	(*P*)-(+)	11	89	90	*S*
4	(*M*)-(−)	13	86	23[e]	*R*
5	(*P*)-(+)	2	88	83	*S*
6	(*M*)-(−)	3	88	48[f]	*R*

[a] The molar ratio used was **3b**/pyrimidine-5-carbaldehyde **1**/*i*-Pr_2Zn = 0.02:0.42:0.86. Aldehyde **1** and *i*-Pr_2Zn were added in three separate portions.

[b] See Table 1, footnote b (flow rate: 0.5 mL min^{-1}, retention time: 16.2 min for (+)-**3b**, 19.3 min for (−)-**3b**).

[c] Isolated yield.

[d] See Table 1, footnote d.

[e] The ee value can be easily amplified by consecutive asymmetric autocatalysis. See Ref. 7d.

[f] The ee value is the one after one more consecutive asymmetric autocatalysis was performed.

(entry 2). When thiahelicenes **3b** with low ee (11% and 13% ee) were used as chiral inducers, (*S*)-**2** and (*R*)-**2** with 90% and 23% ee were obtained in 89% and 86% yields, respectively (entries 3 and 4). Thiahelicene **3b** with only 2% ee induced the generation of (*S*)-**2** with 83% ee in 88% yield (entry 5).

The typical experimental procedure is as follows (Table 1, entry 5): All reactions were performed under an argon atmosphere at 0 °C. To a solution of (*P*)-(+)-tetrathia-[7]-helicene **3a** (8.0 mg, 0.02 mmol, 2% ee) and pyrimidine-5-carbaldehyde **1** (3.8 mg, 0.02 mmol) in toluene (2.5 mL), *i*-Pr_2Zn (0.06 mmol, 0.06 mL of 1.0 M toluene solution) was added dropwise over a period of 2 h, and the mixture was stirred overnight. After the addition of toluene (1.8 mL) and *i*-Pr_2Zn (0.16 mmol, 0.16 mL of 1.0 M toluene solution), a solution of aldehyde **1** (15.1 mg, 0.08 mmol) in toluene (1.0 mL) was added dropwise over a period of 2 h. After stirring for 3 h, toluene (7.2 mL) and *i*-Pr_2Zn (0.64 mmol, 0.64 mL of 1.0 M toluene solution) were added. Then, aldehyde **1** (60.2 mg, 0.32 mmol) in toluene (2.0 mL) was added dropwise over a period of 2 h and the mixture was stirred for an additional 2 h. The reaction was quenched with 1 M hydrochloric acid (2 mL), and neutralized with a saturated sodium hydrogen carbonate solution (6 mL). The mixture was filtered using Celite, and the filtrate extracted using ethyl acetate (three times). The combined organic layers were dried over anhydrous sodium sulfate and evaporated in vacuo. Purification of the residue using silica gel column chromatography (hexane/ethyl acetate = 1:0 to 2:1, v/v) gave (*S*)-5-pyrimidyl alkanol **2** (89.3 mg, 0.384 mmol) in the yield of 92%. The enantiomeric excess was determined to be 83% using HPLC with a chiral stationary phase (Chiralcel OD-H column, 4.6∅ × 250 mm, eluent: hexane/2-propanol = 19:1 [v/v], flow rate 1.0 mL min^{-1}, 254 nm UV detector, retention time 11.6 min for (*S*)-**2**, 17.9 min for (*R*)-**2**).

The enantioselectivity observed in this asymmetric reaction may be explained as follows: *i*-Pr_2Zn was coordinated with sulfur atoms of chiral thiahelicene to form chiral active zinc species.[11] Since these chiral species reacted with pyrimidine-5-carbaldehyde **1** in the initial stage of the reaction, a small enantiomeric excess was induced. Then, a subsequent asymmetric autocatalysis with an amplification of the ee afforded alkanol **2** (as zinc alkoxide) with a high ee, which showed the corresponding absolute configuration.

In summary, we have described a highly enantioselective asymmetric autocatalysis in the presence of tetrathia-[7]-helicenes **3**. Pyrimidyl alkanol with up to 99% ee was formed, with the absolute configuration corresponding to the helical chirality of the thiahelicenes used as the chiral inducers. We believe that these results expand significantly the opportunity of heterohelicenes to be used as chiral inducers.

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