ner) for synthesizing interesting new compounds. This extends the chemistry of ferrocene by a basic new variant, and also opens a unique opportunity for the enantioselective-catalytic synthesis of planar-chiral ferrocene derivatives.

Experimental Section

General procedure for preparing the cyclization precursors **1**–**3** from the corresponding ferrocenyl alkanoic acids: To a stirred suspension of the ferrocenyl alkanoic acid (4.1 mmol) in anhydrous benzene (60 mL) was added sodium hydride (4.18 mmol) under an argon atmosphere, and after 10 min pyridine (2.61 mmol). The mixture was cooled to 0 °C, and freshly distilled oxalyl chloride (26.5 mmol) added dropwise. After complete addition stirring was continued for 30 min at 0 °C, for 30 min at room temperature, and finally for 1 h at 55 °C. After filtration of the reaction mixture through a short pad of silica gel, the solvent and excess oxalyl chloride were completely removed in vacuo. The dark brown residue was dissolved in Et_2O (15 mL), and a solution of diazomethane (35 ml, ≈0.6 M in Et_2O) added at 0 °C. After the reaction mixture was stirred for 20 min at 0 °C excess diazomethane and solvent were completely removed in vacuo, and the residue was purified by flash chromatography (hexane/EtOAc).

General procedure for the cyclization experiments summarized in Table 1: To a solution of the catalyst (34 μmol; 5 mol%) in the anhydrous solvent (10 mL) was added dropwise at RT under an atmosphere of argon a solution of the diazoketone (0.68 mmol) in the solvent (5 mL) within about 30 min. Gas evolution (N_2) indicated the decomposition of the diazo compound. After complete addition stirring was continued until complete conversion was reached (usually about one hour). After rapid filtration of the dark brown reaction mixture through a short pad of silica gel under argon, the solvent was completely removed in vacuo. The products were separated and purified by flash chromatography or radial chromatography (using a chromatotron) under an argon atmosphere.

Enantioselective cyclizations: Ligand **17** (17.3 μmol) was added to a solution of $Cu^{I}OTf$ (17 μmol; weighed in a glove box) in CH_2Cl_2 (3 mL) under an argon atmosphere. After the reaction mixture was stirred for 2 h at RT, the green solution of the catalyst was heated to reflux, and a solution of the substrate (**2** or **3**; 0.34 mmol) in CH_2Cl_2 (10 mL) slowly added within 2 h with a syringe pump (all under argon). After complete addition the brownish solution was heated at reflux for 20 min before being subjected to work-up as described above. The enantiomeric excess of the product was determined with HPLC (Daicel, Chiralcel OJ).

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[24] $[\alpha]_D^{20} = -68.8$, $c = 0.08$ in $CHCl_3$. The absolute configuration of the cyclization product (**14** oder *ent*-**14**) was not established; the enantiomeric excess was determined with HPLC using a Daicel Chiralcel OJ column.

Asymmetric Self-Replication of Chiral 1,2-Amino Alcohols by Highly Enantioselective Autoinductive Reduction**

Takanori Shibata, Tomohide Takahashi, Takashi Konishi, and Kenso Soai*

Dedicated to Professor Dieter Seebach on the occasion of his 60th birthday

Self-replication, like the chirality of the components, is one of the most characteristic features of living organisms. Therefore, self-replication of a chiral molecule is of much interest. The concept of self-replication, however, has not been applied in asymmetric synthesis; almost all conventional asymmetric syntheses require chiral auxiliaries with structures which differ from

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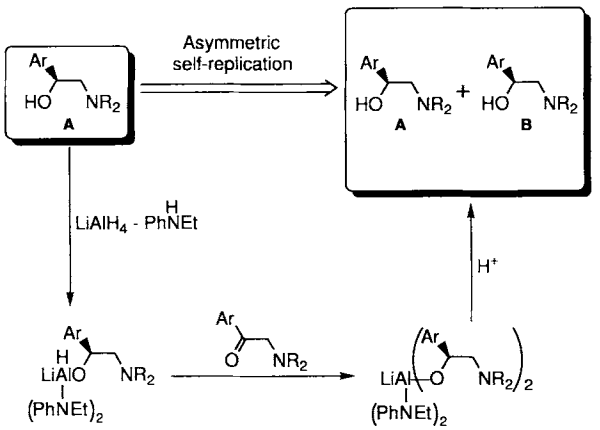
those of the chiral products. The only exceptions are the diastereoselective process with self-regeneration of a stereocenter,[1] the asymmetric autoinduction,[2] and the asymmetric autocatalytic reaction.[3] Asymmetric autoinduction has been limited to the enantioselective alkylation of aldehydes.[2] We report here the first highly enantioselective self-replication in an asymmetric autoinductive reduction in which the structure and configuration of the chiral ligand and product are identical.

We examined the asymmetric autoinductive reduction of α-amino ketones with lithium aluminum hydride[4] modified with a chiral 1,2-amino alcohol and an achiral additive[5] (Scheme 1).



Scheme 1. Asymmetric self-replication of 1,2-amino alcohols; **A** = chiral ligand, **B** = reaction product. The ligand **A** recovered at the end of the reaction and the product **B** have the same structure and configuration.

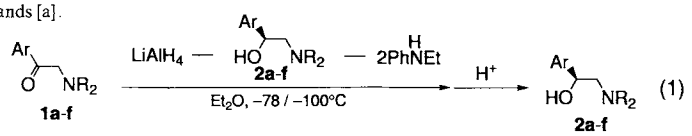
2-Morpholinoacetophenone (**1a**) was reduced with chirally modified $LiAlH_4$, which was prepared in situ from $LiAlH_4$, chiral (*S*)-2-morpholino-1-phenylethanol (**2a**),[6] and *N*-ethylaniline[5a] in Et_2O at −78 °C. The 1,2-amino alcohol (*S*)-**2a** was obtained in 95.8% *ee* (Table 1, entry 1), which means that (*S*)-**2a** was newly formed in 78.8% yield with 82.4% *ee*.[7] This result implies that **1a** was enantioselectively reduced by chiral **2a** to give new **2a** with the same (*S*) configuration. Lowering the reaction temperature (from −78 to −100 °C) improved the chemical yield, and the enantioselectivity remained high (entry 3). When the chiral ligand (*R*)-**2a** was used instead of (*S*)-**2a**, (*R*)-**2a** was formed in 88.6% yield with 84.7% *ee* (entry 4). Entries 5–9 in Table 1 demonstrate that a variety of N-heterocycles can be used as amino substituents. The reactions of (*S*)-**2c** and **2d** at −78 °C[8] were also examined (entries 6 and 7); the enantioselectivity was very high in both cases (87.9 and 90.1% *ee*, respectively).[9] The *ee* values of products **2a**–**2d** (entries 4–7) were easily improved to greater than 99.5% *ee* by a single recrystallization. This means that, by the self-replication of amino alcohols used as chiral ligands, increased amounts of chiral amino alcohols were obtained with no loss of enantiomeric purity. With (*S*)-2-(*N*,*N*-dibenzylamino)-1-phenylethan-1-ol (**2e**) as the chiral ligand, product **2e** formed in moderate optical yields (72.2% *ee*, entry 8). Entry 9 shows the results of the reaction with (*S*)-1-(4-methylphenyl)-2-pyrrolidinoethan-1-ol (**2f**).[10]

The present method demonstrates the first highly enantioselective autoinductive reduction in which the product has the same structure and configuration as the chiral ligand; a separation of the product from the chiral ligand is therefore unnecessary. This system could introduce a new concept into asymmetric reduction.

Experimental Section

In the following, a typical experiment and the method used to calculate the yield and *ee* values of the newly formed 1,2-amino alcohol (Table 1, entry 3) are described. To a suspension (1 mL) of $LiAlH_4$ (47.4 mg, 1.25 mmol) in Et_2O was added a solution (14 mL) of (*S*)-**2a** (263.0 mg, 1.27 mmol, >99.5% *ee*) in Et_2O over a period of 10 min with vigorous stirring. The mixture was heated at reflux for 1 h, treated within 5 min with a solution (2 mL) of *N*-ethylaniline (309.0 mg, 2.55 mmol) in Et_2O, and heated again at reflux for an additional hour. After the reaction mixture was cooled to −100 °C, a solution (3 mL) of **1a** (102.7 mg, 0.50 mmol) in Et_2O was added dropwise. The reaction mixture was stirred at this temperature for 3–4 h, poured into 1 M hydrochloric acid (5 mL) to quench the reaction, and neutralized with a saturated aqueous solution of $NaHCO_3$ (15 mL) at 0 °C. The mixture was filtered over celite, the precipitate thoroughly washed with CH_2Cl_2 (30 mL), and the combined filtrate extracted with CH_2Cl_2. The extract was dried over anhydrous $MgSO_4$, and the solvent removed under reduced pressure. Purification of the crude product by flash column chromatography gave **2a** (355.5 mg) as a mixture of the newly formed product **2a** and the chiral ligand **2a** (263.0 mg). HPLC analysis of the mixture using a chiral column (DAICEL Chiralcel OD-H) provided an enantiomeric purity of 95.7% *ee*. Therefore, the mixture consisted of (*S*)-**2a** (347.9 mg) and (*R*)-**2a** (7.6 mg). The amount of newly formed **2a** was 355.5 − 263.0 = 92.5 mg (0.446 mmol, 89.2% yield), consisting of (*S*)-**2a** (347.9 − 263.0 = 84.9 mg) and (*R*)-**2a** (7.6 mg). The newly formed (*S*)-enriched amino alcohol **2a** had an enantiomeric purity of 83.6% *ee*.

Table 1. Asymmetric autoinductive reduction according to Equation (1) using 1,2-amino alcohols **2a**–**f** as chiral ligands [a].



Entry	Ar	−NR2	Chiral ligand (*ee*/%) [b]	*T* [°C]	*ee*/% [b,c]	Product [d] Yield	Product [d] *ee*/%
1	Ph	−N⟨⟩O	(*S*)-**2a** (>99.5)	−78	95.8 (*S*)	78.8	82.4 (*S*)
2	Ph	−N⟨⟩O	(*S*)-**2a** (>99.5)	−78	92.1 (*S*)	77.7	73.2 (*S*)
3	Ph	−N⟨⟩O	(*S*)-**2a** (>99.5)	−100	95.7 (*S*)	89.2	83.6 (*S*)
4	Ph	−N⟨⟩O	(*R*)-**2a** (>99.5)	−100	96.1 (*R*)	88.6	84.7 (*R*)
5	Ph	−N⟨⟩NBn	(*S*)-**2b** (>99.5)	−100	95.3 (*S*)	88.9	81.6 (*S*)
6	Ph	−N⟨⟩	(*S*)-**2c** (>99.5)	−78	97.7 (*S*)	73.0	87.9 (*S*)
7	Ph	−N⟨⟩	(*S*)-**2d** (>99.5)	−78	97.6 (*S*)	82.4	90.1 (*S*)
8	Ph	$-NBn_2$	(*S*)-**2e** (95.3)	−78	90.6 (*S*)	65.3	72.2 (*S*)
9	Tol	−N⟨⟩	(*S*)-**2f** (95.7)	−78	89.4 (*S*)	92.9	69.7 (*S*)

[a] Molar ratio **1**:$LiAlH_4$:**2**:*N*-ethylaniline for entries 1 and 3–9 1.0:2.5:2.5:5.0., for entry 2 1.0:1.8:1.8:3.6. [b] Enantiomeric purities were determined by HPLC analysis using a chiral column; >99.5% *ee* means that the minor peak was undetectable. [c] Enantiomeric purity of the total amount of isolated **2**. [d] The portion of **2** used at the beginning of the reaction as a chiral ligand was excluded in the calculation (see Experimental Section).

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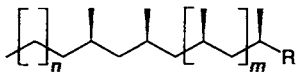
Chiral Methyl-Branched Surfactants and Phospholipids: Synthesis and Properties

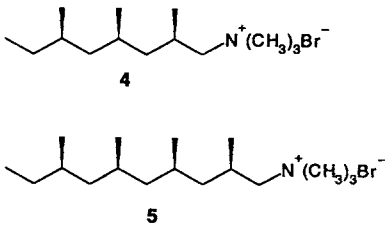
Michael Morr,* Jens Fortkamp, and Stefan Rühe

The recent publication of the synthesis and unusual properties of spiro-surfactants by Menger et al.[1] prompted us to report the results of our work on chiral methyl-branched surfactants and phospholipids which are obtained by formal opening of spiro-compounds. For some time we have been engaged in the isolation of chiral methyl-branched fatty acids, for example, (2*R*,4*R*,6*R*)-2,4,6-trimethyloctanoic acid (**1**)[2] from the preen gland of the musk duck *Cairina moschata* as well as (2*R*,4*R*,6*R*,8*R*)-2,4,6,8-tetramethyldecanoic acid (**2**) and (2*R*,4*R*,6*R*,8*R*)-2,4,6,8-tetramethylundecanoic acid (**3**)[3] from



m	*n*	R	
1	0	0	COOH
2	1	0	COOH
3	1	1	COOH
lardolure	1	1	OCHO
norlardolure	1	0	OCHO

the preen gland of the domestic goose *Anser a.f. domesticus*. In addition to a number of new derivatives of these compounds—such as the mite pheromones lardolure and norlardolure[4, 5] as well as new, chiral ferro- and antiferroelectric liquid crystals with methyl side chains[6]—we were interested in the physical and chemical properties (such as critical micellization concentration (CMC)) of surfactants **4** and **5**, which are derived from



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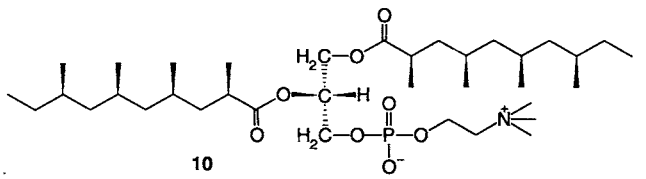
1 and **2**, respectively. We also focused our attention on the diacyl-*sn*-glcero-3-phosphatidylcholines **10**–**15**, which show high enantiomeric purity and a uniform chain length of ten carbon atoms, but varying numbers of methyl-substituted chiral



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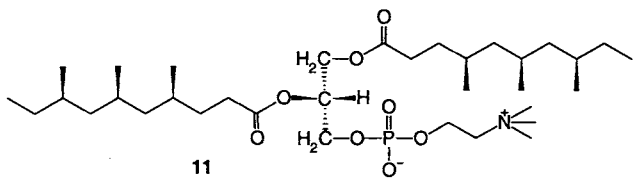

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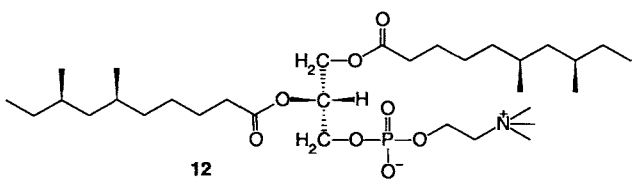

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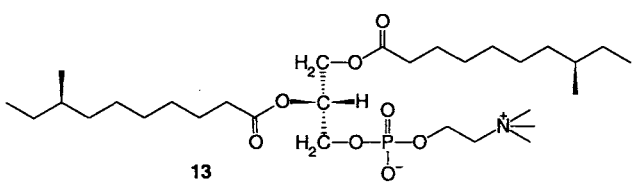

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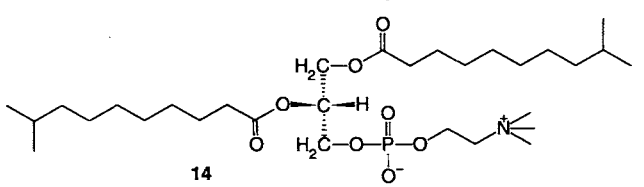

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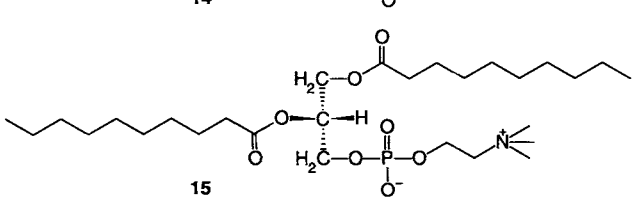

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centers. Properties required for their useful applications (e. g. as membrane constituents) were analysed by differential scanning calorimetry (DSC) to determine the main phase-transition temperatures, and with a Langmuir film balance to record π-*A*-isotherms.

The methyl esters **6** of **1** and **2** were allowed to react with lithium aluminum hydride to give alcohols **7** ($n = 0, 1$; yield 95%), which were converted by tosylation (via **8**) into the bromides **9** with LiBr in acetone (85%). The reaction of **9** with trimethylamine in ethanol at 80 °C (in a pressure vessel) afforded **4** and **5** in quantitative yield.

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