trisilene has a twisted Si–Si double bond probably as a consequence of the eclipsed arrangement of the two *t*Bu$_2$MeSi groups attached to the unsaturated silicon atoms, whereas the *t*Bu$_3$Si-substituted cyclotrigermene has a planar Ge–Ge double bond. The torsional angle of Si4-Si1-Si2-Si5 is 31.9(2)°. The Si=Si bond length of **3** is 2.138(2) Å, which is one of the shortest distances among the Si–Si double-bonded species reported so far (2.138 – 2.261 Å).[11] The Si–Si single bond lengths in **3**, especially the bond distances between the saturated silicon atom in the three-membered ring and the substituents (2.401(3) Å for Si3–Si6 and 2.403(3) Å for Si3–Si7), are somewhat longer than the normal Si–Si bond length (2.34 Å). The three-membered ring is almost an isosceles triangle with bond angles of 62.8(1), 63.3(1), and 53.9(1)°.

Experimental Section

3: A mixture of **1** (1.01 g, 2.01 mmol) and **2** (1.78 g, 4.19 mmol) in toluene (10 mL) was added to a dispersion of sodium (0.95 g, 41.3 mmol) in toluene (10 mL) at room temperature. The reaction was followed by gas chromatography, with **2** disappearing after three hours. At this time 44 % of **1** remained. The resulting salts and excess sodium were removed by filtration, and then the solvent was removed in vacuo to give a dark red solid, which contained cyclotrisilene **3** in 34 % yield based on the amount of **1** that had reacted. Pure **3** (130 mg, 9.1 %) was obtained as red-orange crystals by recrystallization of the reaction mixture from hexane. Air-sensitive red-orange crystals; m.p. 207 – 209 °C; MS (EI, 70 eV): *m/z* (%): 655 ([$M^+ - t$Bu], 0.2), 555 ([$M^+ - t$Bu$_2$MeSi], 8.6), 73 (100), 59 (11); ^{1}H NMR ([D$_6$]benzene, tetramethylsilane (TMS)): $\delta = 0.42$ (s, 6 H), 0.47 (s, 6 H), 1.20 (s, 36 H), 1.29 (s, 36 H); ^{13}C NMR ([D$_6$]benzene, TMS): $\delta = -4.7$, -2.5, 22.2, 23.2, 29.9, 31.2; ^{29}Si NMR ([D$_6$]benzene, TMS): $\delta = -127.3$, 7.9, 27.1, 97.7; UV/Vis (hexane): λ_{max} (ε) = 466 (440), 297 (sh, 1490), 259 (sh, 3610), 223 (7490).

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[10] A single crystal (0.50 × 0.35 × 0.15 mm) of **3** was sealed in a capillary glass tube, and diffraction data collected at 120 K on a Mac Science DIP2030 Image Plate Diffractometer with a rotating anode (50 kV, 90 mA) employing graphite-monochromatized Mo$_{K\alpha}$ radiation (λ = 0.71073 Å). The final R factor was 0.071 ($Rw = 0.136$) for 4291 reflections with $I > 3\sigma(I)$. Crystal data: C$_{36}$H$_{84}$Si$_7$, $M_r = 713.67$, monoclinic, space group $P2_1/c$, $a = 24.063(4)$, $b = 11.629(2)$, $c = 17.715(3)$ Å, $\beta = 110.435(1)°$, $V = 4645(2)$ Å^3, $Z = 4$, $\rho_{calcd} = 1.018$ g cm^{-3}. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-118470. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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Kinetic Influences on Enantioselectivity for Non-Diastereopure Catalyst Mixtures

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It has been noted that a partially resolved chiral catalyst or auxiliary may be responsible for a reaction product of significantly higher enantiomeric excess than expected from combination of the separate reactions of the enantiopure catalysts. This nonlinear behavior has been termed an "asymmetric amplification" of product enantioselectivity, and Kagan and co-workers were the first to describe such behavior.[1] Numerous other catalytic and stoichiometric examples have since been reported.[2] This phenomenon has obvious implications for practical organic synthetic strategies, since the cost of producing highly enantiopure catalysts is often cited as a limitation to the broader commercial exploitation of asymmetric catalysis. Indeed, the observation of a nonlinear effect in a chiral borane reagent was recently used to advantage in the pharmaceutical production of an LTD$_4$ antagonist for treatment of asthma (LTD$_4$ = Leukotriene D$_4$).[3]

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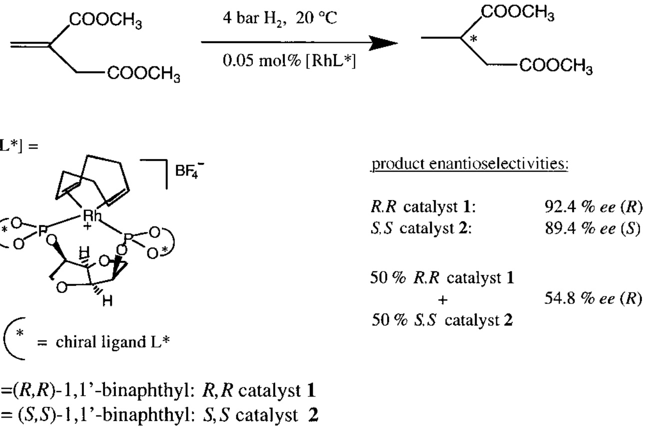
A related nonlinear effect was discussed by Kagan and co-workers[4] for reactions using "pseudo-enantiomeric" catalysts, which they defined as diastereomeric catalyst species that lead to opposite enantiomers as reactions products in asymmetric catalytic reactions. It was observed that the product enantioselectivity in the asymmetric dihydroxylation of olefins did not correlate linearly with the composition of the chiral ligands employed. The use of equal amounts of two ligands which separately gave products of opposite configuration with high enantioselectivity resulted in a product ratio of greater than 95 % in favor of the product preferred by one of the ligands. It was noted that such competitive experiments may be useful for comparing different catalytic species as well as for investigating potential interactions between chiral species.

Here we highlight a further kinetic aspect of nonlinear behavior[5] in asymmetric catalysis for reactions using mixtures of non-enantiopure catalysts. It is shown that when the concentration dependence of the reaction rate is not the same for each catalyst species in a mixture, profound changes in enantioselectivity may be possible over the course of a reaction. Thus it may be misleading to consider a single value of product enantioselectivity measured at the reaction endpoint. This value will represent a convolution of the complex kinetic behavior as a function of reaction progress, making the extraction of important mechanistic information difficult.

Asymmetric hydrogenation using rhodium catalysts with novel diastereomeric diphosphite ligands provides an experimental example to illustrate such a case of complex kinetic behavior. The hydrogenation of prochiral olefins using transition metal catalysts containing chiral phosphane ligands was the first commercially successful example[6] of asymmetric catalysis and has been extensively investigated.[7] A large number and variety of chiral complexes has since been synthesized, and many have been shown to exhibit high chiral efficiency in asymmetric hydrogenation reactions. In our investigations of novel chiral diphosphite ligands using carbohydrate building blocks, a series of ligands was synthesized based on 1,4:3,6-dianhydro-D-mannitol.[8] Catalysts **1** (*R,R* catalyst) and **2** (*S,S* catalyst) shown in Scheme 1 were prepared from [Rh(cod)$_2$]BF$_4$ (cod = cyclooctadiene), 1,4:3,6-dianhydro-D-mannitol, and (*R*)- or (S)-binaphthol. As a test reaction, the asymmetric hydrogenation of the dimethyl ester of itaconic acid (Scheme 1) was studied using these two catalysts separately and in mixtures. When used separately, the two catalysts gave high and opposite enantioselectivities for the product and quantitative yield under mild reaction conditions (*R,R* catalyst **1**: 92.4 % *ee*, *R* product; *S,S* catalyst **2**: 89.4 % *ee*, *S* product). It was confirmed that the enantioselectivity obtained by use of either pure catalyst alone did not vary with conversion of substrate. A 1:1 mixture of the two catalysts also gave quantitative yield, but, interestingly, the reaction provided the *R* product in 54.8 % *ee*.



Scheme 1. Reaction conditions and product enantioselectivities for the asymmetric hydrogenation of the dimethyl ester of itaconic acid using [RhL*] catalysts.

Nonlinear effects of this type are most often interpreted as evidence for the interaction between different catalytic species in solution to form new species which exhibit chiral efficiencies different from the original catalysts. A second, simpler possibility is that in this case the reaction proceeded independently to a greater extent through a channel involving the *R,R* catalyst **1** than one involving the *S,S* catalyst **2**. This is analogous to the example discussed by Kagan and co-workers, although in that case they were not able to rule out the possibility of interaction between the catalytic species.[4] It will be shown how careful kinetic investigations allow us in the present example to choose definitively between these quite different mechanistic hypotheses.

The reaction rate curves shown in Figure 1 for reactions using the catalysts separately reveal that the catalysts exhibit strikingly different kinetic profiles. While the *S,S* catalyst **2** showed a monotonically decreasing reaction rate over the entire course of the reaction (positive-order kinetics in substrate concentration), the rate of the reaction using the *R,R* catalyst) **1** increased continually until very high substrate conversion (negative order kinetics in substrate concentration).

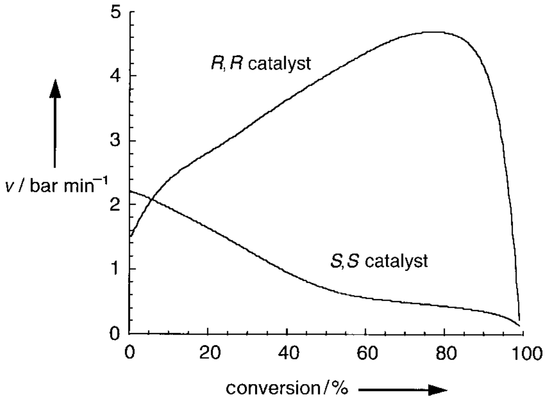


Figure 1. Reaction rate *v* as a function of the conversion of substrate for the *R,R* catalyst **1** and the *S,S* catalyst **2**. The reaction conditions are given in the text.

Heller et al.[9] have noted similar induction periods with negative-order kinetics in their studies of asymmetric hydrogenation reactions with catalysts containing COD as ligand. Since the hydrogenation of COD provides binding sites for the substrate, the induction period may be indicative of an increase in the concentration of catalytically active species over the course of the reaction. Why COD prehydrogenation should differ for the two diastereomeric catalysts remains unclear, however, and is the subject of further investigation.

The data from Figure 1 may be used to predict enantioselectivity as a function of reaction progress when a 1:1 mixture of the *R*,*R* catalyst **1** and the *S*,*S* catalyst **2** is used, under the assumption that the catalysts act independently in the mixture. Figure 2 shows this calculated enantioselectivity over the course of the reaction, predicting that an initial enantioselectivity of 16% *ee* for the *S* product would ultimately shift to a final enantioselectivity of 54% *ee* for the *R* product. The experimentally determined endpoint value of 54.8% *ee* for the *R* product is in excellent agreement with this prediction.

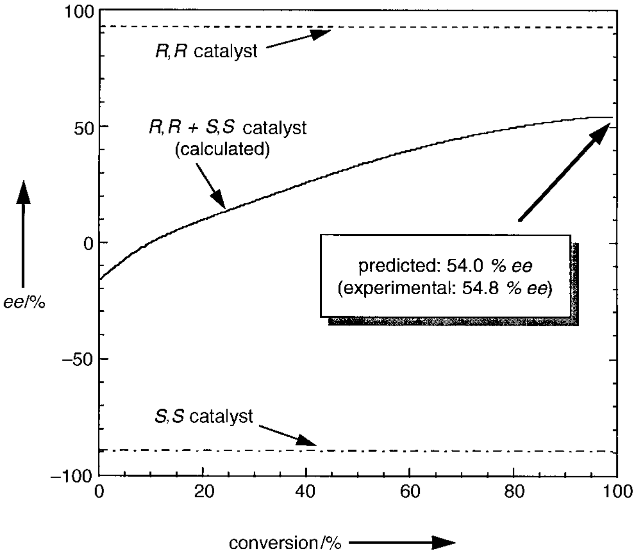


Figure 2. Experimental and calculated enantioselectivity as a function of conversion of substrate: – – –: experimentally determined for the *R*,*R* catalyst **1**; –·–: experimentally determined for the *S*,*S* catalyst **2**; ——: enantioselectivity calculated according to Equation (1) (see Experimental Section) for a mixture of 50% catalyst **1** and 50% catalyst **2**.

Thus in this case, the observed nonlinear effect may be traced to differences in the kinetic profiles of the catalysts acting independently, and was not diagnostic of the formation of dimeric or higher order species from the original catalyst. This example points out that nonlinear behavior may result from complex kinetic behavior as well as from complex catalyst structure. Whenever two or more different asymmetric catalyst species compete in the same reaction, the possibility that they will follow different kinetic rate laws, and therefore exhibit a conversion dependence on enantioselectivity, cannot be excluded a priori.

In cases where an interaction between catalysts does result in the formation of new catalytic species, the possiblity of a kinetically based conversion dependence of enantioselectivity must still be considered. It is arguably quite likely that if a new catalyst species gives a product enantioselectivity different from that given by the original catalyst, the new species will also exhibit different reaction-rate characteristics. For example, signficant changes in substrate binding strength, and therefore in the kinetic rate law, might be expected for a *meso*-ML_RL_S species compared to the enantiopure ML_RL_R and ML_SL_S species in a typical ML_2 system exhibiting a strong deviation from linear behavior.[1, 2]

The example presented here demonstrates that profound consequences for enantioselectivity may ensue when asymmetric reactions are carried out using a mixture of catalysts following different reaction rate laws. In such a case, the enantioselectivity measured at the reaction endpoint will represent a convolution of the progressive kinetic behavior over the course of the reaction. Observation of a conversion-dependent enantioselectivity may thus be used as a diagnostic of complex kinetic behavior,[10] and information about reaction progress may provide important clues for a better mechanistic understanding of nonlinear effects in asymmetric catalysis.

Experimental Section

Details of the ligand and catalyst preparations are contained in reference [8], along with details of the characterization by NMR (^1H, ^{13}C, ^{31}P) and IR spectroscopy and elemental analysis. Preparation of the catalysts from the ligands was found to be very sensitive to conditions, and batchwise variations in catalyst activity were attributed to the difficulty in determination of the precise amount of catalytic complex formed. However, reactions reported here were reproduced at least twice in a 100-mL autoclave under the conditions shown in Scheme 1. Reaction progress was followed by monitoring the pressure drop in a hydrogen reservoir connected to the reactor through a dosing valve, which maintained the reactor at constant pressure. The reaction rate as a function of conversion of substrate was determined by calculating the derivative of the pressure uptake curve. Product enantioselectivity (ee_{x_A}) was calculated as a function of substrate conversion (x_A) for a reaction employing an equal mixture of the *R*,*R* catalyst **1** and the *S*,*S* catalyst **2** from their respective reaction rates ($r^{R,R}$ and $r^{S,S}$) and from the enantioselectivities found for the pure catalysts ($ee^{R,R}$ and $ee^{S,S}$) according to Equation (1).

$$ee_{x_A} = \frac{\left(\frac{1+ee^{RR}}{2}\int_0^{x_A} r^{RR}\mathrm{d}x_A + \frac{1+ee^{SS}}{2}\int_0^{x_A} r^{SS}\mathrm{d}x_A\right) - \left(\frac{1-ee^{RR}}{2}\int_0^{x_A} r^{RR}\mathrm{d}x_A + \frac{1-ee^{SS}}{2}\int_0^{x_A} r^{SS}\mathrm{d}x_A\right)}{\left(\frac{1+ee^{RR}}{2}\int_0^{x_A} r^{RR}\mathrm{d}x_A + \frac{1+ee^{SS}}{2}\int_0^{x_A} r^{SS}\mathrm{d}x_A\right) + \left(\frac{1-ee^{RR}}{2}\int_0^{x_A} r^{RR}\mathrm{d}x_A + \frac{1-ee^{SS}}{2}\int_0^{x_A} r^{SS}\mathrm{d}x_A\right)} \quad (1)$$

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Isolation and Spontaneous Resolution of Eight-Coordinate Stereoisomers**

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Björn Gustafsson, and Göran Hilmersson

While geometrical and optical isomers are numerous among four- and six-coordinate complexes, the isolation of such stereoisomers with coordination number eight remains a challenge to synthetic chemists. Eight-coordinate complexes are usually labile since the energy differences between the most frequent geometries are small, as is the energy barrier for interconversion between them.[1a] Thus only two pairs of eight-coordinate stereoisomers have been structurally characterized, namely, diastereomeric *cis*- and *trans*-$[SmI_2\{O(CH_2CH_2OCH_3)_2\}_2]$[2] and *cis*- and *trans*-$[ReH_2(mhp)_2(PPh_3)_2]PF_6$ (mhp = monoanion of 2-methyl-6-hydroxypyridine).[3] We now report the spontaneous resolution of configurationally chiral[1b] eight-coordinate complexes *Δ*-**1** and *Λ*-**1**[4] (dme = 1,2-dimethoxyethane). A racemic phase (**2**) exhibiting conformational isomers can also be isolated.[5]

Δ-$[SmI_2(dme)_3]$	*Δ*-**1**
Λ-$[SmI_2(dme)_3]$	*Λ*-**1**
rac-$[SmI_2(dme)_3]$	**2**

The chemistry of divalent lanthanides—established with Sm, Eu, and Yb and recently expanded into Tm[6]—is currently developing rapidly. In particular, following the pioneering work of Kagan et al.,[7] solutions of SmI_2 in THF have become commercially available and are finding an ever-increasing role as selective one-electron reducing reagents in a variety of reactions.[8] The dominant species in such solutions in THF is probably the seven-coordinate $[SmI_2(thf)_5]$, and similar pentagonal-bipyramidal coordination geometry has also been found in $[SmI_2(dme)(thf)_3]$ and $[SmI_2(dme)_2(thf)]$, which were isolated from mixtures of THF and DME.[9]

Samarium reacts smoothly[10] with 1,2-diiodoethane in DME at 50 °C to yield a dark blue solution of SmI_2. Crystals can be isolated from this solution both at ambient temperature and at -20 °C. X-ray crystallographic data[11] were collected using seven different crystals obtained from solutions at ambient temperature, which resulted in three crystal structures of *Δ*-**1** and four of *Λ*-**1** (see Figure 2). Assignment of the chiral space group $P2_1$ was unequivocal, and the absolute configuration determination was conclusive with no indication of racemic twinning. X-ray structure analyses[11] proved that the crystals obtained at −20 °C constitute a different phase—racemic **2** (see Figure 3).[5] It thus seems as if crystallization from the blue solution at −20 °C yields racemic **2**, while crystallization at ambient temperature results in a mixture of chiral crystals comprising either the *Δ*-**1** or the *Λ*-**1** enantiomer. The chiral crystals can be separated manually using a microscope with polarized light, but due to their intense color this procedure is not practical for larger quantities. Such hand-picked crystals can, however, be used to seed a supersaturated solution in order to exclusively crystallize one enantiomer from the racemate, a technique which is known as preferential crystallization.[12] We are presently searching for the optimal conditions for this procedure.

The enantiomers of **1** have the shape of a propeller, the shaft being represented by the I-Sm-I axis and the blades by the three Sm–dme rings (Figures 1 and 2). The enantiomers D-**1** and *Λ*-**1** then correspond to a right- and left-threaded propeller, respectively. There is a distortion in **1** such that the "propeller" axis is bent towards one "blade", which consequently is close to an equatorial conformation. The Sm–I and Sm–O distances are normal, that is, somewhat longer than in the seven-coordinate thf/dme complexes.[9] An analysis of the 28 angles formed by the donor atoms around Sm in **1** suggests that the coordination geometry is best described as a distorted hexagonal bipyramid, or alternatively as a distorted triangular dodecahedron.[13]

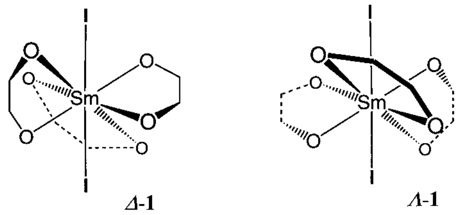

Figure 1. Schematic representation of the propeller-shaped enantiomers of **1** (methyl groups are excluded for clarity).

Samarium and the dme ligands in **1** form five-membered rings which exhibit chiral conformations. It is noteworthy that in *Δ*-**1** all three rings have the λ conformation (i.e., $\Delta\lambda\lambda\lambda$), while in *Λ*-**1** all three are δ conformers (i.e., $\Lambda\delta\delta\delta$). This can be illustrated by the fact that the C2–C3, C5–C6, and

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