Asymmetric Synthesis of Cyclopentenes by [3 + 2] Annulations between Vinylcarbenoids and Vinyl Ethers

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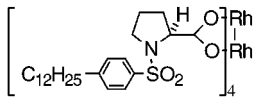
Received April 20, 1998

Rhodium *N*-(dodecylbenzenesulfonyl)prolinate [(Rh_2(*S*-DOSP)$_4$ (**1**)]-catalyzed decomposition of diazobutenoates in the presence of vinyl ethers results in highly diastereoselective and enantioselective synthesis of donor/acceptor-substituted vinylcyclopropanes. Diethylaluminum chloride-induced rearrangement of these vinylcyclopropanes results in the formation of cyclopentenes with excellent control of diastereoselectivity and for the fused cyclopentenes good control of absolute stereochemistry. This study illustrates the synthetic utility of Rh_2(*S*-DOSP)$_4$ as a chiral catalyst for vinylcarbenoid cyclopropanations and the remarkable diastereocontrol that is possible in the diethylaluminum chloride-induced ring expansion of donor/acceptor-substituted vinylcyclopropanes.

The development of synthetic methodologies for the stereoselective construction of five-membered rings has been a very active area of research.[1,2] A particularly fruitful area has been annulation strategies involving carbenoid intermediates. A [4 + 1] annulation strategy has been reported based on the intramolecular cyclopropanation of dienes followed by rearrangement of the resulting vinylcyclopropanes.[1,3] This methodology has been used in elegant syntheses of several fused cyclopentanoid terpenes such as retigeranic acid.[3b] Originally harsh thermal conditions were required to induce vinylcyclopropane rearrangement,[1b,3] but since then a number of milder methods have been developed.[3−10] Two notable methods result in vinylcyclopropane rearrangement with excellent control of relative stereochemistry. Danheiser and co-workers[9] reported an anion-accelerated vinylcyclopropane rearrangement which generates cyclopentenols with control of stereochemistry at up to three stereogenic centers. Corey and co-workers[10] have developed an intramolecular cyclopropanation of a diene followed by Et_2AlCl-catalyzed vinylcyclopropane rearrangement as a stereoselective method to form highly functionalized fused cyclopentenes and used this chemistry in a number of natural product syntheses.

We recently reported a two-step process, related to that used by Corey, for the construction of functionalized cyclopentenes with control of relative stereochemistry at three contiguous stereogenic centers (Scheme 1).[11] The first step is a highly stereoselective cyclopropanation of vinyl ethers by vinylcarbenoids, and this is followed by a vinylcyclopropane rearrangement that proceeds formally in a suprafacial retention mode. Since then, we have developed rhodium *N*-(dodecylbenzenesulfonyl)prolinate [Rh_2(*S*-DOSP)$_4$ (**1**)] as an efficient catalyst for asymmetric cyclopropanations.[12] This opens up the potential for the [3 + 2] annulation between vinylcarbenoids and vinyl ethers to become a highly enantioselective route to cyclopentenes. A study aimed at the development of such a process is the basis of this paper.



Rh_2(*S*-DOSP)$_4$, **1**

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S0022-3263(98)00738-5 CCC: $15.00
Published on Web 08/21/1998

Scheme 1

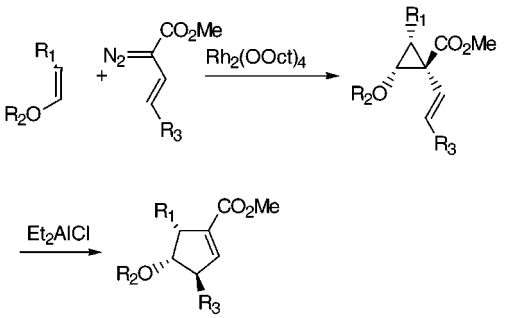
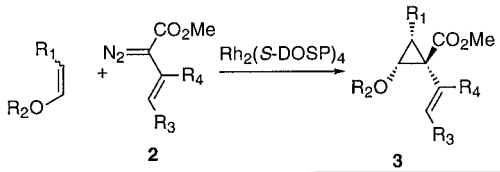


Table 1. Asymmetric Synthesis of Vinylcyclopropanes 3



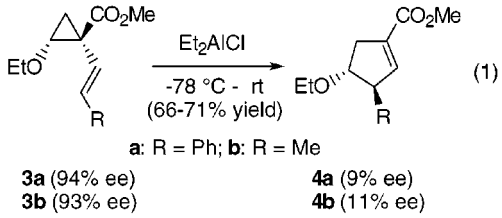
2 **3**

R_1	R_2	Diazo	R_3	R_4	Product	yield, %	ee, %
H	Et	**2a**	Ph	H	**3a**	71	94
H	Et	**2b**	Me	H	**3b**	75	93
Me	Et	**2a**	Ph	H	**3c**	51	65
cyclo-$(CH_2)_2$		**2a**	Ph	H	**3d**	84	86
cyclo-$(CH_2)_2$		**2b**	Me	H	**3e**	68	86
cyclo-$(CH_2)_3$		**2a**	Ph	H	**3f**	66	88
cyclo-$(CH_2)_3$		**2b**	Me	H	**3g**	57	87
cyclo-$(CH_2)_2$		**2c**	cyclo-$(CH_2)_3$		**3h**	49	76
cyclo-$(CH_2)_3$		**2c**	cyclo-$(CH_2)_3$		**3i**	53	88
		**2a**	Ph	H	**3j**	57	96

Results and Discussions

The first stage of the project was the asymmetric synthesis of a series of donor/acceptor[13]-substituted vinylcyclopropanes by reaction of vinyldiazoacetates with vinyl ethers. Previous studies have shown that the optimum conditions for asymmetric cyclopropanation using $Rh_2(S\text{-DOSP})_4$ as catalyst are low temperatures and nonpolar solvents.[12] Consequently, the asymmetric synthesis of the vinylcyclopropanes was carried out at −78 °C using pentane as solvent. Under these conditions, $Rh_2(S\text{-DOSP})_4$-catalyzed decomposition of vinyldiazoacetates **2** in the presence of various vinyl ethers resulted in the formation of a series of vinylcyclopropanes **3** with asymmetric induction in the range of 65−96% ee (Table 1). The absolute stereochemistry of these products is assigned on the basis of the predictive model we have developed to rationalize the asymmetric induction by $Rh_2(S\text{-DOSP})_4$.[12]

Having synthesized the vinylcyclopropanes with high enantioselectivity, the Et_2AlCl-catalyzed vinylcyclopropane rearrangements were then examined. Rearrangement of **3a** under the standard conditions that had been developed for the racemic series[11] (Et_2AlCl/CH_2Cl_2, −78 °C to RT, 3 h) afforded the trans cyclopentene **4a** in 71% yield (eq 1). As previously reported,[11] the diastereoselectivity of this rearrangement is excellent, and no evidence of the diastereomeric cyclopentene was seen in the crude reaction mixture. If the diastereoselectivity was due to zwitterionic intermediates that closed rapidly to the cyclopentene prior to any bond rotation, then retention of the high-asymmetric induction would have been expected during cyclopentene formation. This was not the case, however, because the cyclopentene **4a** was formed in only 9% ee. Similarly, rearrangement of the cyclopropane **3b** resulted in the formation of the cyclopentene **4b** in 11% ee. Modification of the reaction conditions for the conversion of **3a** to **4a** such as the use of a nonpolar solvent (Et_2AlCl/pentane, 0 °C 3 h) or higher temperature (Et_2AlCl/CH_2Cl_2, 0 °C for 10 min) gave only slightly better enantioselectivity (25% ee and 31% ee, respectively).

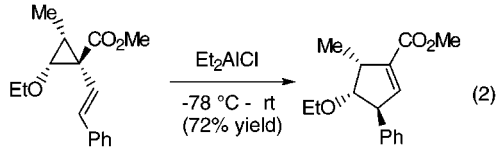


(1)

a: R = Ph; **b**: R = Me

3a (94% ee) **4a** (9% ee)
3b (93% ee) **4b** (11% ee)

Extension of the rearrangement studies to systems that contained three stereogenic centers was originally expected to form the cyclopentenes without racemization, because the third stereogenic center was expected to be unaltered during the rearrangement. This expectation proved not to be the case, however, because the rearrangement of **3c** (65% ee) resulted in the formation of the cycloptentene **5c** in 72% yield (eq 2) with excellent control of relative stereochemistry, but with considerable racemization (43% ee).



(2)

3c (65% ee) **5c** (43% ee)

In contrast to the above results, rearrangement of fused cyclopropanes resulted in the formation of fused cyclopentenes with very little, if any, racemization. In the case of the furanocyclopropanes **3d** and **3e**, rearrangement to the furanocyclopentenes **6d** and **6e** occurred without any detectable racemization (eq 3). In the case of the pyranocyclopropanes **3f** and **3g**, rearrangement to the pyranocyclopentenes **7d** and **7e** occurred with less than 5% racemization (eq 4).

The potential range of this chemistry is seen in the reactions of the furanocyclopropanes **3h** and **3i** that are derived from a more elaborate vinyldiazomethanes (eq 5). Rearrangement of **3h** and **3i** resulted in the formation of the tricyclic products, **8h** and **8i**, with full control of relative stereochemistry. Moreover, **8h** is formed in 74% ee, while **8i** is formed in 86% ee.

The observation of significant racemization in the formation of the cyclopentene **5c** would require a reaction pathway for its formation in which equilibration of all three stereogenic centers of the starting cyclopropane can occur. The originally proposed mechanism,[11a] in which zwitterionic intermediates cyclized to cyclopentenes before bond rotation could occur, although consistent with the excellent control of relative stereochemistry fails to account for the racemization that occurs during these

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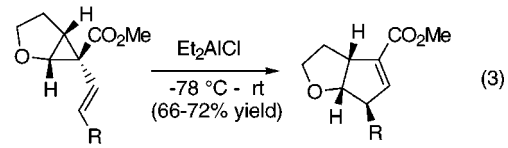
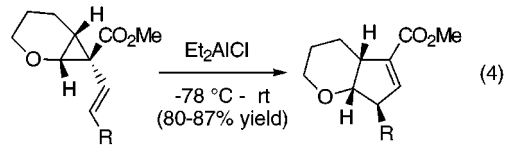


(3)

d: R = Ph; **e**: R = Me

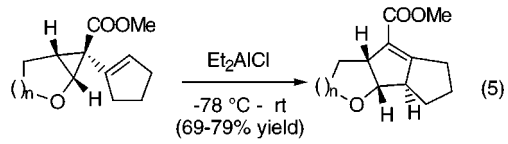
3d (86% ee) **6d** (86% ee)
3e (86% ee) **6e** (85% ee)



(4)

f: R = Ph; **g**: R = Me

3f (88% ee) **7f** (82% ee)
3g (87% ee) **7g** (84% ee)

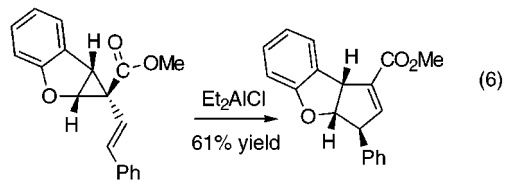


(5)

h: n = 1; **i**: n = 2

3h (76% ee) **8h** (74% ee)
3i (88% ee) **8i** (86% ee)

rearrangements. To further explore the factors that are behind the racemization, the rearrangement of **3j** (96% ee) that contains a benzylic stereocenter was examined (eq 6). This resulted in the formation of **9j** in 61% yield and 11% ee, which indicates that equilibration of the benzylic stereocenter is very facile under the vinylcyclopropane rearrangement reaction conditions.



(6)

3j (96% ee) **9j** (11% ee)

The absolute stereochemistry of **6d** was determined to be (1*R*, 5*R*, 8*S*) by the method illustrated in Scheme 2. Previously we had shown that cyclopropanation of dihydrofuran with a vinyldiazoacetate containing (*R*)-pantolactone as a chiral auxiliary resulted in the formation of the cyclopropane **10** in 66% de.[14] On prolonged standing **10** rearranged to the fused cyclopentene **11**, which recrystallized to a single diastereomer of **11**. The absolute configuration of **11** was determined to be (1*S*, 5*S*, 8*R*) by X-ray crystallography. Reduction of partially enriched **11** (85% de) resulted in the formation of the alcohol **12.** The HPLC retention time of the major enantiomer of **12** was the same as that of the minor enantiomer in **ent-12**, which was obtained by reduction of **6d**. Consequently, it can be concluded that the absolute stereochemistry for **6d** is (1*R*, 5*R*, 8*S*). This is the predicted major enantiomer that would be formed in

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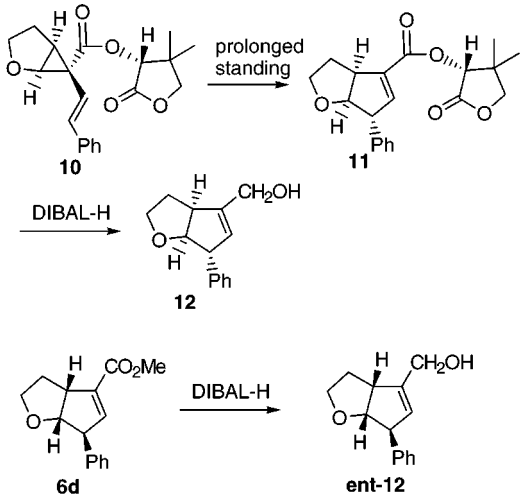
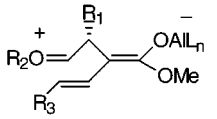
Scheme 2



Rh_2(*S*-DOSP)$_4$-catalyzed cyclopropanations,[12] followed by vinylcyclopropane rearrangement with only partial epimerization at the C-3 position. These results are also consistent with the general trend[12,13] that the stoichiometric chiral auxiliary (*R*)-pantolactone results in asymmetric induction in vinylcarbenoid reactions opposite that obtained using the chiral catalyst Rh_2(*S*-DOSP)$_4$ (**1**). The absolute stereochemistry of the other cyclopentenes is assigned by analogy with the result for **6d**.

The mechanistic interpretation of the stereochemical results is not fully defined. The original model[11a] involving the zwitterionic intermediates **13** which close to cyclopentenes prior to bond rotation is inconsistent with the racemization that was observed in **4a** and **4b**. Therefore, it appears that the zwitterionic intermediates **13** have sufficient lifetime, at least for several of the examples, that some bond rotation can occur prior to formation of the cyclopentenes. The control of relative stereochemistry that is observed in these rearrangements would then presumably be due to kinetically favored ring closures, which turn out to be highly stereoselective in this system. Even more puzzling is the observation of partial racemization of **5c** and **9j** that contained three stereogenic centers. The exact mechanism of this racemization in unknown, although it could conceivably occur via reversible proton transfers on the zwitterionic intermediate or alternative reversible cyclopropane ring opening reactions.



13

In summary the [3 + 2] annulation between vinylcarbenoids and vinyl ethers is an attractive stereoselective method for the synthesis of highly functionalized cyclopentenes. Excellent control of relative stereochemistry is possible, and in most of the fused cyclopentenes good control of absolute stereochemistry can be achieved. This study illustrates further the synthetic utility of Rh_2(*S*-DOSP)$_4$ as a chiral catalyst for vinylcarbenoid cyclopropanations and the remarkable diastereocontrol that is possible in the diethylaluminum chloride-induced ring expansion of donor/acceptor-substituted vinylcyclopropanes.

Experimental Section

General Procedures. ^{1}H NMR spectra were run at 300, 400, or 500 MHz and ^{13}C NMR spectra at 75.5 and 125.7 MHz with the sample solvent being $CDCl_3$. Pentane was dried over sodium metal. Column chromatography was carried out on silical gel 60 (230−400) mesh. The diazo compounds were prepared according to literature procedures.[15]

General Procedure for Rhodium (II)-Catalyzed Decomposition of Vinyldiazomethanes in the Presence of Alkenes. A mixture of the alkene (5 equiv) and Rh_2(*S*-DOSP)$_4$ (0.01 equiv) in pentane was cooled to −78 °C under argon. To this solution was added the vinyldiazomethane (1 equiv, 0.10−0.18 M) in pentane over 30 min. The mixture was stirred at −78 °C for 24 h. The reaction mixture was slowly warmed to room temperature. The solvent was evaporated under reduced pressure, and the residue was purified on silica using ether/petroleum ether as the eluent in the ratio specified. The amounts of diazo compound, rhodium(II), alkene, reaction solvent, and eluent ratio are presented in that order in abbreviated form.

(1*S*,2*R*)-Methyl (*E*)-2-ethoxy-1-((*E*)-2-phenylethenyl)-cyclopropane-1-carboxylate (3a):[11a] **2a** (300 mg, 1.48 mmol), Rh_2(*S*-DOSP)$_4$ (28 mg, 0.015 mmol), ethyl vinyl ether (0.71 mL, 7.4 mmol), pentane (60 mL) (5:95 to 10:90); yield 260 mg (71%); 94% ee, determined by HPLC (OJ column); flow rate 1.0 mL/min, 1% 2-propanol in hexane; UV 254 nm; T_R = 15 min (minor), 17 min (major); $[\alpha]^{25}_D = -26.0$ (*c* 2.0, MeOH).

General Procedure for the Et_2AlCl-Catalyzed Rearrangement of Cyclopropanecarboxylates. A solution of cyclopropanecarboxylate (1 equiv, 0.1−0.4 M) in CH_2Cl_2 was added dropwise to a stirred solution of Et_2AlCl (1 M in heptane, 2 equiv) in CH_2Cl_2 at −78 °C under argon. The cooling bath was left in place, and the reaction mixture was slowly warmed to room temperature over 3−4 h. After quenching with ethanol, water was added and the mixture was extracted twice with ether. The combined organic layers were washed with brine and dried ($MgSO_4$). The solvents were evaporated, and the residue was purified by column chromotography on silica gel using ether/petroleum ether as eluent in the ratio specified in the parentheses.

Methyl (4α,5α)-4-ethoxy-5-phenylcyclopent-1-ene-1-carboxylate (4a):[11a] **3a** (100 mg, 0.41 mmol) (10:90); yield 66 mg (66%); 9% ee, determined by HPLC (OD); flow rate 1.0 mL/min, 10% 2-propanol in hexane; UV 254 nm; T_R = 5.6 min (major), 6.6 min (minor); $[\alpha]^{25}_D = -11.9$ (*c* 2.0, $CHCl_3$).

(3*R*)-3-(4,4-Dimethyl-2-oxotetrahydrofuranyl)(1*S*,5*S*,8*R*)-8-Phenyl-4-oxabicylo[3.3.0]oct-6-ene-6-carboxylate (11). On standing, (3*R*)-3-(4,4-dimethyl-2-oxotetrahydrofuranyl) (1*S*,5*S*,6*S*)-6-(2-(*E*)-phenylethenyl)bicyclo[3.1.0]hexane-6-carboxylate (**10**) (66% ee)[14] rearranged into **11**. Recrystallization from ethyl acetate/hexanes gave diasteriomerically enriched **11** as a colorless solid (greater than 85% de by NMR): mp 115−116 °C; $[\alpha]^{25}_D = +108.7$ (*c* 0.26, $CHCl_3$); IR (neat) 3060, 3015, 2960, 2860, 1782, 1720, 1620 cm^{-1}; ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.50−7.10 (m, 5 H), 6.94 (br s, 1 H), 5.48 (s, 1 H), 4.55 (br d, 1 H, J = 6.3 Hz), 4.08 (s, 2 H), 4.02 (br d, 1 H, J = 1.9 Hz), 3.89−3.75 (m, 3 H), 2.24−1.99 (m, 2 H), 1.26 (s, 3 H), 1.16 (s, 3 H); ^{13}C NMR (50.3 M Hz, $CDCl_3$) δ 172.2, 163.1, 147.0, 140.7, 135.7, 128.9, 127.4, 127.1, 89.9, 76.2, 75.1, 67.2, 59.1, 49.0, 40.4, 31.0, 23.1, 20.1. Anal. Calcd for $C_{20}H_{22}O_5$: C, 70.16; H, 6.48. Found: C, 70.23; H, 6.52.

(1*S*,5*S*,8*R*)-6-(Hydroxymethyl)-8-phenyl-4-oxabicylo-[3.3.0]oct-6-ene (12). A solution of DIBAL-H (0.29 mL, 1 M in hexane) was added dropwise over 5 min to a stirred solution of **11** (33 mg, 0.096 mmol) in ether (1 mL) at −78 °C. After slowly warming to RT fo 2 h, the mixture was quenched with saturated aqueous ammonium chloride solution and extracted with ether. The ether layer was washed with saturated aqueous NaCl solution, dried ($MgSO_4$), and concentrated. The residue was purified by chromatography on silica using ether/petroleum ether (1:1) as eluent to give **12**: 5.2 mg (24% yield); 88% ee, determined by HPLC (OD); flow rate 1 mL/min, 10% 2-propanol in hexane; UV 254 nm; T_R = 12.7 min (major), 14.0 min (minor);^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32−7.10 (m, 5 H), 5.70 (br s, 1 H), 4.48 (d, 1 H, J = 6.3 Hz), 4.30 (m, 2 H), 3.85 (m, 2 H), 3.80 (m, 1 H), 3.48 (br t, 1 H), 2.03−1.87 (m, 2 H), 1.50 (m, 1 H); ^{13}C NMR (50.3 MHz, $CDCl_3$) δ 144.7, 143.0, 128.5, 128.0, 127.4, 126.6, 90.9, 67.2, 60.9, 58.2, 49.4, 29.7. Anal. Calcd for $C_{14}H_{16}O_2$: C, 77.75; H, 7.46. Found: C, 77.68; H, 7.51.

A similar DIBAL-H reduction of **6d** on a 0.9 mmol scale gave **ent-12** in 66% yield: $[\alpha]^{25}_D = -174.7$ (0.8%, $CHCl_3$); 86% ee, determined by HPLC (OD); flow rate 1 mL/min, 10% 2-propanol in hexane; UV 254 nm; T_R = 12.7 min (minor), 14.0 min (major).

Acknowledgment. Financial support of this work by the National Science Foundation (CHE 9726124) is gratefully acknowledged.

Supporting Information Available: Experimental data for **3b**−**j**, **4b**, **4c**, **6d**, **6e**, **7f**, **7g**, **8h**, **8i**, and **9j**, copies of ^{1}H NMR spectra of **6d**, and X-ray crystallographic data for **11** (16 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

JO980738N

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