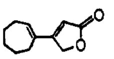
Table I. Silylative Cyclocarbonylation of **1**[a]

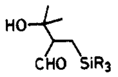
	acetylenic alcohol					product			
						β-lactone[c]		propenal[d]	
entry	no.	R^1	R^2	silane	base[b]	no.	yield, %	no.	yield, %
1	**1a**	H	H	Me_2PhSiH	Et_3N	**2a**	0	**6a**	83
2	**1b**	H	Me	Me_2PhSiH	Et_3N	**2b**	15	**6b**	76
3	**1b**	H	Me	Me_2PhSiH	DBU	**2b**	54	**6b**	26
4	**1b**	H	Me	$^tBuMe_2SiH$	DBU	**5b**	79	**9b**	6
5	**1c**	Me	Me	Me_2PhSiH	none	**2c**	0	**6c**	94
6	**1c**	Me	Me	Me_2PhSiH	pyridine	**2c**	0	**6c**	67
7	**1c**	Me	Me	Me_2PhSiH	Et_3N	**2c**	43	**6c**	52
8[e]	**1c**	Me	Me	Me_2PhSiH	DABCO	**2c**	52[f]	**6c**	0[g]
9	**1c**	Me	Me	Me_2PhSiH	DBU	**2c**	81	**6c**	0
10[h]	**1c**	Me	Me	Et_3SiH	Et_3N	**3c**	64	**7c**	6
11	**1c**	Me	Me	$^iPr_3SiH$	Et_3N	**4c**	33	**8c**	0
12	**1c**	Me	Me	$^tBuMe_2SiH$	Et_3N	**5c**	86	**9c**	0
13	**1d**	$-(CH_2)_4-$		Me_2PhSiH	Et_3N	**2d**	0	**6d**	71
14	**1d**	$-(CH_2)_4-$		$^tBuMe_2SiH$	DBU	**5d**	68	**9d**	0
15	**1e**	$-(CH_2)_5-$		Me_2PhSiH	DBU	**2e**	85	**6e**	0
16	**1f**	$-(CH_2)_6-$		Me_2PhSiH	DBU	**2f**	86	**6f**	2

[a] Reactions were conducted in a pressure bottle containing benzene, an acetylenic alcohol (1 equiv), a silane (1 equiv), a base (1 equiv), and $Rh_4(CO)_{12}$ (0.001 equiv) at 100 °C for 2 h under CO pressure (15–40 kg/cm^2) on a scale of 1–2 mmol. [b] DABCO: 1,4-diazabicyclo[2.2.2]octane. The quantity of DBU was reduced to 0.1 equiv. [c] References 5 and 7. [d] Reference 5. The ratio of isomers was evaluated for entries 1 (*Z*:*E* = 37:63), 2 (*Z*:*E* = 65:35), 5 (*Z*:*E* = 97:3), 6 (*Z*:*E* = 100:0), and 13 (*Z*:*E* = 97:3). [e] The reaction time is 17 h. [f] *Z*:*E* = 79:21. [g] Compund **19a** (R_3Si = Me_2PhSi, 32%) was isolated. [h] The reaction time is 25 h. Compound **19b** (R_3Si = Et_3Si, 20%) was also isolated.

under water gas shift conditions gave butenolide **16**[5] in 44% yield with the concomitant formation of some unidentified materials.



16



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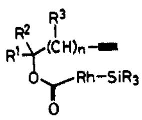
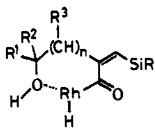


17



18

In the absence of precise mechanistic details, the formation of lactones through the intermediacy of an alkoxycarbonyl complex **17** reminiscent of the palladium-catalyzed cyclocarbonylation[1a,b] of **10** is presumed. In the rhodium case, however, **18** may be proposed as the common intermediate to give both lactone and propenal derivatives on the bases of the following observations: (i) the isolated propenal **6c** was not transformed to the β-lactone **2c** under the carbonylation conditions, (ii) the presence of R_3SiH is crucial for smooth carbonylation, and (iii) the presence of methanol or 2-methyl-2-propanol does not cause an intermolecular alkoxycarbonylation in the silylformylation of 1-pentyne. A conformation such as that of **18** seems to be advantageous for lactone formation and for the release of severe steric repulsion caused by a bulkier silyl group and rhodium metal. Hydrogen gas accompanying the lactones was detected by mass spectroscopic analysis[10] of the gas recovered after the formation of **2c**. Hydrogen formation was also supported by the fact that an appreciable amount of a saturated aldehyde **19** was isolated after a lengthy reaction time (entries 8 and 10 in Table I).

The silyl-protected *exo*-methylene group linked to the α-position of lactones could be transformed to the methylene group by a known procedure.[11] The triorganosilyl group located at the *exo*-methylene carbon serves to stabilize these structures and to direct the regioselectivity in successive reactions. Further studies are in progress.

Supplementary Material Available: Experimental details for typical cyclocarbonylations and characteristic boiling or melting point, IR, and ^1H and ^{13}C NMR data for the products (4 pages). Ordering information is given on any current masthead page.

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New, Rational Molecular Design for Chiral Recognition Involving Application of Dual Hydrogen Bond Association

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The recognition of molecular chirality is a very important aspect of the field of molecular recognition. For small molecules, the association mode provides a basis for molecular design of the chiral selector. Thus, to date, various modes of association have been developed for attaining enantioselectivity.[1,2] We recently found a unique mode of dual hydrogen bonds in the crystal structure[3] of a 1:1 complex of (*R*,*R*)-*N*,*N*′-diisopropyltartramide (DIPTA)[4] and (*S*,*S*)-9,10-dimethyl-9,10-dihydrophenanthrene-9,10-diol (**1**). The observed mode has led to a new concept of "complementary twist" for molecular design in quest of chiral recognition. In the

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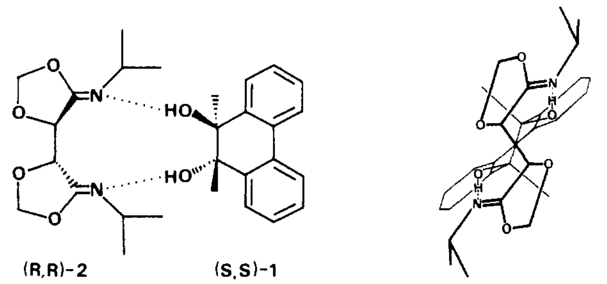
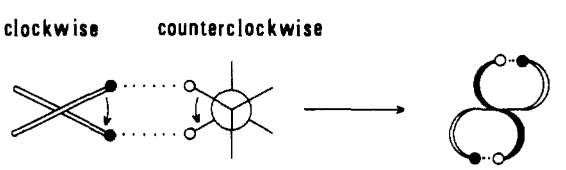


(R,R)-2 (S,S)-1

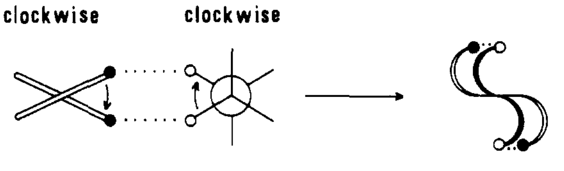
Figure 1. The proposed structure of the association of (*R*,*R*)-**2** and (*S*,*S*)-**1**.

present study, this concept was used to design (*R*,*R*)-4,4′-bi[5-(*N*-isopropylimino)-1,3-dioxolane] (**2**)[5] as a novel chiral selector that would show a high level of enantioselectivity toward *trans*-**1** in solution through dual hydrogen bonds, as illustrated in Figure 1.

The concept of "complementary twist"[6] is presented in eqs 1 and 2. On the basis of this mode of dual hydrogen bonds, the



(1)



(2)

following guidelines for molecular design were established. (1) The bicyclopentyl system is the fundamental structure of the chiral selector having C_2 symmetry. (2) The relative orientation of two five-membered rings is flexibly controlled by dipole–dipole repulsion between two C–O bonds adjacent to each other, thus making it possible to enhance the adaptability of the selector to dual hydrogen bonding with a selectand. (3) An *N*-isopropylimino group that functions as a hydrogen-bond acceptor, as well as a steric barrier, is incorporated into the five-membered ring in an exo manner. (*R*,*R*)-**2** embodies all these features. One of the favored conformations of (*R*,*R*)-**2** is considered to be that which provides an anti relationship between two bulky isopropyl substituents at imino nitrogens. In such a conformation, two bonds between dioxolane oxygens and asymmetric carbons have a gauche relationship, and the relative orientation of two iminodioxolane rings displays a propeller-like twist with counterclockwise rotation. In addition, the molecular shape of (*R*,*R*)-**2** in this conformation is closely related to that of (*R*,*R*)-DIPTA in the associate with (*S*,*S*)-**1**, whose structure was demonstrated by an X-ray crystal analysis.[3] Thus, on the basis of this crystal structure, a perspective view of the associate of (*R*,*R*)-**2** and (*S*,*S*)-**1** along the C_2 axis would be envisioned, as illustrated in Figure 1 (right), when dual hydrogen bonds between imino and hydroxyl groups occur. Due to the minimum steric interaction between the two components, a combination of association sites with counterclockwise and clockwise rotation should be complementary in this system. Accordingly, it is expected that (*R*,*R*)-**2** exerts *S*,*S* selectivity toward *trans*-**1**.

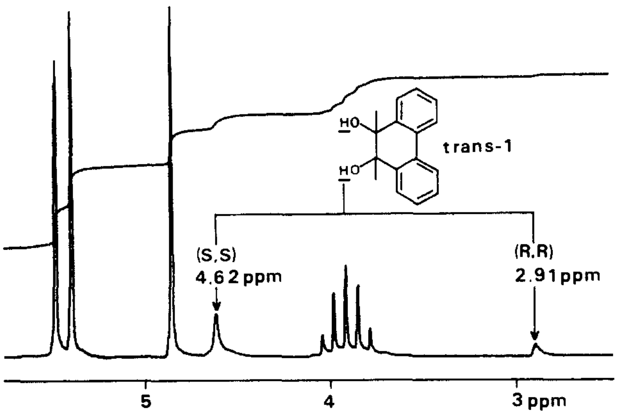


Figure 2. A partial ^{1}H NMR spectrum recorded at 100 MHz of *S*,*S*-enriched *trans*-**1** in $CDCl_3$ solution containing (*R*,*R*)-**2** at 23 °C((*R*,*R*)-**1**:(*S*,*S*)-**1** = 1:6, (*R*,*R*)-**2**:(*S*,*S*)-**1** = 5:2, 0.06 M in (*R*,*R*)-**2**). The arrows indicate the resonance of the hydroxyl protons of *trans*-**1**. The downfield singlet is assigned to the resonance of (*S*,*S*)-**1** on the basis of the correlation of the relative intensity of the signals with the enantiomeric composition of *trans*-**1**.

As indicated in Figure 2, there was a large shift difference ($\Delta\delta$ = 1.71 ppm) in the resonance of the two hydroxyl protons of *trans*-**1** in the ^{1}H NMR spectrum of a $CDCl_3$ solution containing (*R*,*R*)-**2**. The downfield singlet was assigned to the resonance of (*S*,*S*)-**1**, suggesting that hydrogen bonds of the two hydroxyl groups of (*S*,*S*)-**1** with (*R*,*R*)-**2** occur to a greater extent. Taking advantage of the shift of hydroxyl protons of *trans*-**1** as the probe, a 6×10^{-3} M $CDCl_3$ solution[7] of each enantiomer of *trans*-**1** was titrated by (*R*,*R*)-**2** in the concentration range from 6×10^{-3} to 0.1 M at 23 °C. Analysis of the data assuming the formation of a 1:1 associate and using the least-squares method indicated the association constant $K = 25.0 \pm 0.6$ M^{-1} and the limiting chemical shift in the associate $\delta_{\text{assoc}} = 6.26 \pm 0.04$ ppm for the (*R*,*R*)-**2**–(*S*,*S*)-**1** system and $K = 5.8 \pm 0.6$ M^{-1} and $\delta_{\text{assoc}} = 5.08 \pm 0.21$ ppm for the (*R*,*R*)-**2**–(*R*,*R*)-**1** system. In both cases, a correlation coefficient of approximately 0.999 was obtained, thus supporting 1:1 stoichiometry for the association. These results clearly demonstrate the expected *S*,*S* selectivity of (*R*,*R*)-**2** toward *trans*-**1**, and the magnitude of enantioselectivity obtained here is large ($\Delta(\Delta G) = -874 \pm 76$ cal/mol).

In the IR spectrum of a 6×10^{-3} M $CDCl_3$ solution of (*S*,*S*)-**1**, an isolated and/or intramolecularly hydrogen bonded OH stretching band was observed at 3600 cm^{-1}. When the solution contained 1 equiv of (*R*,*R*)-**2**, a broad absorption from 3150 to 3450 cm^{-1} appeared ($A_1/A_2 = 3.8$). This absorption was considered due to an intermolecular N---HO bond[8] and rapidly grew as the ratio of (*R*,*R*)-**2** to (*S*,*S*)-**1** proceeded to 4:1 ($A_1/A_2 = 0.9$). In addition, the resonance of the imino carbon of (*R*,*R*)-**2** in a 0.05 M $CDCl_3$ solution of (*S*,*S*)- and (*R*,*R*)-**1** (0.025 M in (*R*,*R*)-**2**) was observed at 154.5 and 153.5 ppm, respectively. In a 1:1 mixture of (*R*,*R*)-**1** and (*R*,*R*)-**2** in $CDCl_3$ (6×10^{-3} M each), the resonance of the isopropyl methyl protons of (*R*,*R*)-**2** appeared at 1.12 ppm as a doublet (J = 6.4 Hz). The corresponding resonance in the (*S*,*S*)-**1**–(*R*,*R*)-**2** system appeared as a pair of doublets at 1.13 and 1.14 ppm (J = 6.4 Hz). This peak splitting in the latter system may possibly have resulted from

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(5) (*R*,*R*)-**2** was synthesized from (*R*,*R*)-DIPTA. The details will be published elsewhere. Data for (*R*,*R*)-**2**: mp 70–71 °C; $[\alpha]_D$ +126.9 (*c* 1.2, $CHCl_3$); ^{1}H NMR ($CDCl_3$) δ 1.12 (d, J = 6.4 Hz, 12 H), 3.89 (sep, J = 6.4 Hz, 2 H), 4.68 (s, 2 H), 5.37 (d, J = 0.7 Hz, 2 H), 5.47 (d, J = 0.7 Hz, 2 H); IR (KBr) 1730 (C=N) (s) cm^{-1}. Anal. Calcd for $C_{12}H_{20}N_2O_4$ (256.301): C, 56.24; H, 7.86; N, 10.93. Found: C, 55.97; H, 7.80; N, 10.84.

(6) When the relative orientation of two sites for dual hydrogen bonds displays a twist reflecting the absolute configuration, two combinations of association sites are possible according to the sense of rotation represented as clockwise or counterclockwise. The transient diastereomers formed in this manner are expected to be different in their stability, since each combination of association sites possessing a given orientation results in associates differing in steric interaction between the two components. It thus follows that the complementarity that leads to the selective formation of a more stable diastereomeric associate should exist in combinations of twists with given senses. That is, this type of association mode should elicit enantioselectivity. The general relationship between a combination of association sites with a given sense of rotation and the formation of a more stable diastereomer cannot be defined since the stability of the diastereomer depends on the arbitrary steric environment of both of its components.

(7) At this concentration, the self-association of *trans*-**1** was negligible.

(8) Bellamy, L. J. *The Infrared Spectra of Complex Molecules. Advances in Infrared Group Frequencies*; Chapman and Hall: London, 1980; Vol. II, Chapter 8.

restriction of free rotation about the single bond between the imino nitrogen and the isopropyl methine carbon due to the access of the (*S*,*S*)-**1** molecule. From the foregoing discussion, the involvement of the imino nitrogen of (*R*,*R*)-**2** in the association appears quite likely, and this would confirm the associated structure depicted in Figure 1 as responsible for the enantioselection noted in the present study.

In summary, we have developed a representative of a new chiral selector that employs the complementarity of twists in dual hydrogen bond association. We are currently extending the scope of enantioselection by the present selector to reveal the generality of this approach to molecular recognition.

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Oxidative Organic Electrochemistry: A Novel Intramolecular Coupling of Electron-Rich Olefins

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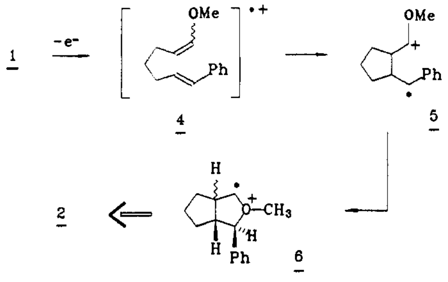
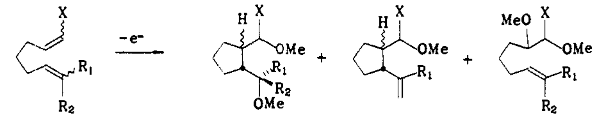
Oxidative cyclization reactions are of interest because they allow for the generation of carbon–carbon bonds and the formation of rings without a loss in the overall functionality of a molecule.[1,2] Organic electrochemistry would appear ideally suited for initiating such reactions because it can selectively oxidize electron-rich functional groups at preset potentials, under neutral conditions, and without the need for chemical reagents.[3] Unfortunately, there exist only a few examples of anodic reactions that lead to direct carbon–carbon bond formation.[4] In addition, only a handful of these reactions have been shown to be generally useful for initiating intramolecular cyclization reactions.[5,6] We report herein our initial efforts to develop the anodic coupling of electron-rich olefins for such a purpose.[7,8]

Scheme I



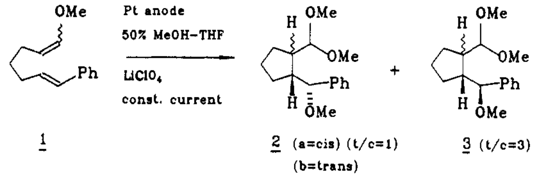
Scheme II[a]



7a. X = OMe, $R_1 = R_2 = CH_3$[a]	**8a.** 60% (t/c = 2)	**9a.** 17% (t/c = 4)	**10a.** --
7b. X = Ph, $R_1 = R_2 = CH_3$[a] (6% recovered)	**8b.** 15%	**9b.** 5%	**10b.** 25%
7c. X = OSi(i-Pr)₃, $R_1 = R_2 = CH_2$[b]	**8a.** 48% (X = OMe)	**9a.** 19% (X = OMe)	**10c.** --
7d. X = OSi(i-Pr)₃, R_1 = Ph, R_2 = H[c] (9% recovered)	**2/3.** 57% (X = OMe: **2/3** = 1.7) **8d.** 10% (X = OSi(i-Pr)₃, R_1 = Ph, R_2 = H)	**9d.** N.A.	**10d.** --

[a] Conditions: (a) Pt anode, MeOH, $LiClO_4$, 2,6l-lutidine, divided cell; (b) Pt anode, 50% MeOH/THF, $LiClO_4$, undivided cell; (c) Pt anode, 10% $MeOH/CH_3CN$, $LiClO_4$, undivided cell.

These studies were initiated by examining the electrochemical behavior of compound **1**. To this end, *the anodic oxidation of 1 under constant-current conditions in an undivided cell using a platinum anode and a 1 N lithium perchlorate in 50% methanol–tetrahydrofuran electrolyte solution led to 68–73% isolated yields of cyclized products.* To our surprise, the reaction afforded a moderate degree of diastereoselectivity and gave rise to a 5.3:1 mixture of isomers at the benzylic carbon (compounds **2** and **3**). The cyclization also resulted in cis and trans isomers about the five-membered ring. The cis and trans isomers having the same stereochemistry at the benzylic carbon were identified by hydrolysis of the acetals and then epimerization of the products to a single aldehyde isomer. The stereochemistry at the benzylic carbon was determined by a single-crystal X-ray analysis of the 2,4-dinitrophenylhydrazone (2,4-DNP) derivative derived from the trans major aldehyde.[9]



(Yield = 68-73%, Ratio of 2 to 3 = 5.3/1)

[†] Author to whom correspondence concerning the X-ray analysis should be addressed.

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(4) (a) For reviews, see refs 3b–d (especially ref 3b, Appendix 1.1, and ref 1c, Chapter 4) as well as the following: Schafer, H. J. *Angew. Chem., Int. Ed. Engl.* **1981**, *20*, 911. For a couple of recent examples, see: (b) Yamamura, S.; Shizuri, Y.; Nakamura, K. *J. Chem. Soc., Chem. Commun.* **1985**, 530. (c) Yoshida, K.; Sakaguchi, K.; Isoe, S. *J. Org. Chem.* **1988**, *53*, 2525. Yoshida, J.; Sakaguchi, K.; Isoe, S. *Tetrahedron Lett.* **1986**, *27*, 6075.

(5) For a review, see ref 3c, section 4-3, and references therein.

(6) For some recent examples of anodic cyclizations, see: (a) Swenton, J. S.; Morrow, G. W. *Tetrahedron Lett.* **1987**, *28*, 5445. (b) Schafer, H. J.; Becking, L. *Tetrahedron Lett.* **1988**, *29*, 2797. (c) Yamamura, S.; Shizuri, Y.; Okuno, Y.; Shigemori, H. *Tetrahedron Lett.* **1987**, *28*, 6661.

(7) For reviews of intermolecular examples, see ref 4a. See also: (a) Belleau, B.; Au-Young, Y. K. *Can. J. Chem.* **1969**, *47*, 2117. (b) Fritsch, J. M.; Weingarten, H. *J. Am. Chem. Soc.* **1968**, *90*, 793. (c) Fritsch, J. M.; Weingarten, H.; Wilson, J. D. *J. Am. Chem. Soc.* **1970**, *92*, 4038. (d) Le-Moing, M. A.; Guillanton, G.; Simonet, J. *Electrochim. Acta* **1981**, *26*, 139. (e) Schafer, H. J.; Engels, R.; Steckhan, E. *Justus Liebigs Ann. Chem.* **1977**, 204. (f) Schafer, H. J.; Steckhan, E. *Tetrahedron Lett.* **1970**, 3835. (g) Schafer, H. J.; Steckhan, E. *Angew. Chem., Int. Ed. Engl.* **1974**, *13*, 472. (h) Schafer, H. J.; Steckhan, E.; Koch, D. *Chem. Ber.* **1974**, *107*, 3640. (i) Fox, M. A.; Akaba, R. *J. Am. Chem. Soc.* **1983**, *105*, 3460.

(8) In a somewhat related study, Shono has reported the anodic intramolecular coupling of enol acetates and olefins in moderate yields (18–40%). Shono, T.; Nishiguchi, I.; Kashimura, S.; Okawa, M. *Bull. Chem. Soc. Jpn.* **1978**, *51*, 2181.

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