be made. The literature contains some approaches to this linkage[7,8] and others can be imagined.[9] Therefore, we decided to test the cyclization of aryl ether **4**, which we expected to be available from the coupling of the appropriate phenol and cyclohexenediol derivative (see Scheme II).

Allylic alcohol **11b** was prepared in seven steps from commercially available *m*-methoxyphenethylamine (**6**). Birch reduction of phenethylamine **6**, tosylation of the amino group of the resulting nonconjugated dienol ether, and hydrolysis afforded enone **7**. N-Alkylation[10] followed by reduction of the keto group according to Luche's procedure[11] gave allylic alcohol **9**. Cyclohexenediol **11a** was prepared by epoxidation and regioselective isomerization[12] of the resulting epoxy alcohol **10** with $Ti(OiPr)_4$, according to the Sharpless protocol. Silylation of the less hindered hydroxyl group of *cis*-diol **11a** afforded the target monoprotected diol **11b**.

Alcohol **4b** was obtained by Mitsunobu coupling[13] of alcohol **11b** with phenol **12**[14] followed by removal of the silyl protecting group. This compound proved to be a suitable substrate for radical-initiated cyclization.

When heated with Bu_3SnH (0.035 M) and AIBN in benzene in a sealed tube (130 °C), bromoaryl ether **4b** underwent tandem cyclization followed by elimination of the *S*-phenyl radical to afford the tetracyclic styrene **5** (R = H) in 35% yield.[15,16]

With ready access to tosylamide **5**, we were now ready to consider the completion of the morphine skeleton by closure of ring IV. Of the methods available for the cleavage of sulfonamides, those which employ dissolving metal reducing conditions[17] seemed especially attractive for the task at hand. One could imagine that the nitrogen radical (or anion) generated by reductive detosylation of intermediate **5** might add to the β-carbon of the styrene moiety, affording dihydroisocodeine directly. *In fact, treatment of tosylamide 5 with Li/NH_3 in the presence of t-BuOH (-78 °C) did afford* (±)*-dihydroisocodeine* (**2**) *in 85% yield* (refer to Scheme I). This unprecedented closure[18] provides a remarkably simple solution to the final bond connection required for the morphine ring system.

Swern oxidation of dihydroisocodeine afforded (±)-dihydrocodeinone (**3**)[19] in 83% yield. When combined with the efficient procedures for the conversion of dihydrocodeinone to codeine (**1b**)[20] and the facile O-demethylation of codeine to morphine (**1a**),[21] Scheme I represents the formal total synthesis of (±)-codeine and (±)-morphine.

This synthesis illustrates the versatility of radical cyclization processes for the construction of multifunctional polycyclic compounds. In particular, it demonstrates the power of this methodology for "stitching" rings together to build convex ring systems. In addition, it introduces a new and convenient method for the joining of certain carbon-nitrogen bonds. It is potentially amenable to chiral synthesis, a modification which is currently being pursued in our laboratories.

Acknowledgment. This research was supported by the National Science Foundation and the National Institutes of Health. NMR spectra were acquired with a Bruker AM400WB spectrometer, purchased with funds from the National Science Foundation. The Kratos MS-80 mass spectrometer was purchased with funds from the Division of Research Resources of the National Institutes of Health.

Supplementary Material Available: Listings of experimental procedures and IR and ^1H NMR spectra for **2-5** and **7-12** (7 pages). Ordering information is given on any current masthead page.

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An Allyl Radical-Dioxygen Caged Pair Mechanism for *cis*-Allylperoxyl Rearrangements

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Rearrangements of allylperoxyl radicals have been known since the late 1950s,[1] yet the mechanism for this reaction is still open to debate.[2-6] Previous work has demonstrated that optically pure *trans*-allylperoxyl radicals derived from methyl oleate rearrange in a highly stereoselective process with minimal atmospheric oxygen incorporation, suggesting a concerted 2,3 free-radical pathway.[7,8] However, recent theoretical investigations on allylperoxyl radicals have failed to find a concerted transition state for the rearrangement, but rather support a dissociative process involving an allyl radical intermediate.[9] A mechanism consistent

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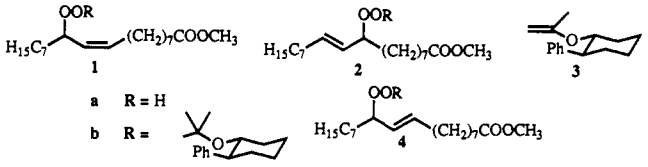
Table I. Composition of ^{18}O- and ^{16}O-Labeled Trans 9(*R*)- and 9(*S*)-Hydroperoxide **2a** Formed from Methyl (*R*)-11-Hydroperoxyoctadec-9(*Z*)-enoate (**1a**) as a Function of Solvent Viscosity

aliquot	solvent	1 − (mole fraction of cis 11-OOH **1a**)[a]	^{16}O (*R*)-**2a**[b]	^{16}O (*S*)-**2a**	^{18}O (*R*)-**2a**	^{18}O (*S*)-**2a**
1	hexane	0.444	85.6	1.1	6.1	7.2
	dodecane	0.461	90.3	1.2	4.3	4.2
	octadecane	0.462	95.8	0.8	1.8	1.6
2	hexane	0.583	78.7	0.8	10.2	10.3
	dodecane	0.592	84.9	1.8	7.1	6.2
	octadecane	0.562	91.2	1.3	3.9	2.7
3	hexane	0.655	69.4	1.6	14.2	14.8
	dodecane	0.661	82.1	2.3	7.7	7.9
	octadecane	0.630	90.4	1.9	3.9	3.8

[a] Mole fraction of cis 11-hydroperoxide **1a** as determined by HPLC–UV detection. [b] Percentage of ^{16}O- and ^{18}O-labeled trans 9(*R*)- and 9(*S*)-hydroperoxide **2a** calculated from the mole fraction of each enantiomer as determined by HPLC of perketal derivative and the mole fraction of ^{16}O- and ^{18}O-labeled **2a** as determined by HPLC–CIMS.

with experiment and theory involves an allyl radical–dioxygen solvent caged pair that collapses with stereocontrol. We report here that optically pure *cis*-allylperoxyl radicals rearrange with stereoselectivity and some incorporation of labeled oxygen, and that these processes depend on solvent viscosity, observations that are consistent with the caged pair mechanism.

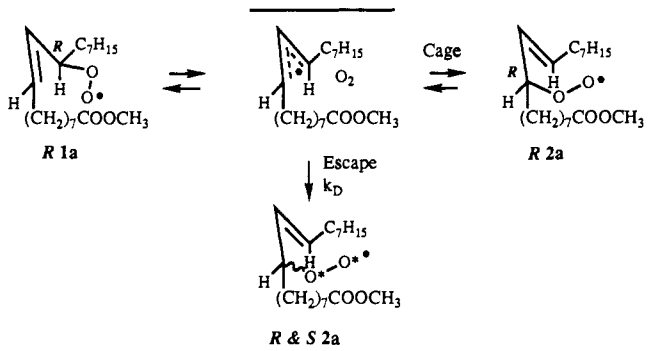
Chiral perketals of *cis*-oleate hydroperoxides can be resolved to optical purities of greater than 99% enantiomeric excess by the use of reverse-phase chromatography[10] (solvent: 2% tetrahydrofuran in acetonitrile). Thus, the perketal derivative **1b** of methyl (*R*)-11-hydroperoxyoctadec-9(*Z*)-enoate elutes before the corresponding cis 11(*S*) diastereomer on reverse-phase chromatography. Removal of the perketal with mild acid affords enantiomerically pure hydroperoxide, **1a**.



Rearrangement of the cis 11(*R*)-hydroperoxide in hexane (η = 0.27 cP, 40 °C) was initiated by 10 mol % di-*tert*-butyl hyponitrite and performed under an atmosphere of 99% pure $^{18}O_2$ at 40 °C. Loss of **1a** and formation of the rearrangement products trans 9-hydroperoxide **2a** and the corresponding trans 11-hydroperoxide **4a** were monitored by HPLC–UV detection, and hydroperoxide fractions were taken and converted to the perketals by reaction with optically-pure **3**.[8,11] Perketal analysis allows determination of the configuration of the stereocenters of **1b**, **2b**, and **4b**.[12,13] The rearrangement was also carried out under an atmosphere of $^{18}O_2$ in dodecane (η = 1.07 cP, 40 °C) and octadecane (η = 2.86 cP, 40 °C). Labeled oxygen incorporation analysis was carried out by HPLC–CIMS by comparison of m/z = 311 vs 313 (^{18}O) ions of $MH^+ - H_2O$ for the hydroperoxides.

The rearrangement is stereoselective with the product, **2a**, being of the same configuration as the starting material and having significant (but low, 6–7%) ^{18}O incorporation. Product enantiomer distribution and ^{18}O incorporation for the rearranged hydroperoxide **2a** formed in hexane, dodecane, and octadecane are presented in Table I as a function of the extent of rearrangement. There is a significant viscosity dependence on labeled oxygen incorporation and stereoselectivity for the rearrangement, higher

Scheme I



solvent viscosity giving rise to higher stereoselectivity and lower oxygen incorporation in the rearrangement products. Data presented in Table I are for product **2a** while similar trends are observed for the rearrangement product **4a** (e.g., for aliquot 1, hexane, the ratio **2a**/**4a** is 1.13 and the ratio of (*S*)-**4a**/(*R*)-**4a** is 9.75).

The simplest explanation for these oxygen-labeling and stereochemical studies as a function of solvent viscosity is a rearrangement that proceeds by an allyl radical–dioxygen caged pair. The proposed mechanism, presented in Scheme I, involves an intermediate allyl radical–triplet dioxygen caged pair which can either undergo coupling with stereochemical control or escape by diffusion and reaction with oxygen with loss of stereochemistry.[14] Allyl radicals that diffuse into solution incorporate $^{18}O_2$ with racemization of configuration at the stereocenter of **2a**, evidence for a planar allyl radical intermediate that has escaped the initial solvent cage. As solvent viscosity is increased, the diffusional rate constant, k_D, decreases, resulting in a decrease in escape product and an increase in cage product. As a consequence, in viscous solvents, less atmospheric oxygen incorporation is observed for the rearrangement. The peroxyl products that are formed in the solvent cage are apparently formed with a high degree of stereoselectivity. Thus, the decrease in escape product in viscous solvents correlates directly with an increase in cage product that is formed with high stereoselectivity. From the enantiomeric excess determined for ^{16}O-labeled **2a** in each solvent, a caged product averaged retention of configuration of 97% enantiomeric excess can be calculated when half the starting **1a** is consumed (Table I, aliquot 2: 98%, hexane; 95.8%, dodecane; 97.2%, octadecane).

In light of the results in the allylperoxyl rearrangement, we examined the dienyl hydroperoxides derived from methyl linoleate. A dissociative mechanism proceeding by an intermediate pentadienyl radical has been supported by experiment,[15,16] and it was

(10) The trans 9-hydroperoxide undergoes further rearrangement to the trans 11-hydroperoxide. These studies have also been carried out on the trans 11-hydroperoxide, and we find results similar to those for the trans 9-hydroperoxide **2a**.

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our intent to determine if the dienylperoxyl rearrangement mechanism also involved a radical caged pair. Cage effect studies investigating ^{18}O incorporation as a function of solvent viscosity demonstrate ^{18}O incorporation approaching 100% in hexane and slightly decreased incorporation of atmospheric oxygen in octadecane. This implies a much smaller cage effect with pair escape dominating pair collapse and gives supportive evidence that, in contrast to the allyl radical, the pentadienyl radical reacts with molecular oxygen more slowly than the diffusion-controlled rate.[17,18]

Both theoretical investigations[9] and cage effect studies point to a dissociative mechanism for the allylperoxyl rearrangement. Solvent viscosity studies have previously been used to provide evidence for caged radical pair intermediates,[19,20] and a radical–dioxygen pair should have reactivity similar to that of a caged radical pair since the collapse of both pairs occurs at or near the diffusion-controlled rate. In contrast to pairs of radicals that couple with loss of stereochemistry in isotropic media,[21] collapse of the radical–dioxygen pair apparently occurs in solution with high stereoselectivity. These results demonstrate the importance of solvent viscosity on peroxyl radical rearrangements and suggest that viscosity effects might affect peroxyl radical rearrangements in biological systems of high microviscosity such as lipid bilayers.

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Formation of a Novel P–B–N–C Ring via an Intramolecular C–H Activation Process

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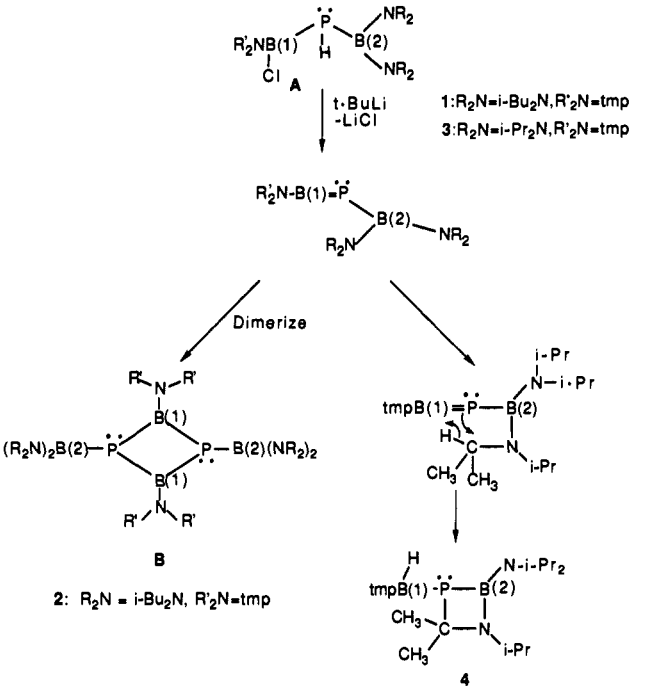
Recently, there has been new interest in the syntheses of phosphinoboranes, $R_2PBR'_2$,[1] but only limited chemistry of these species has been examined. We have previously found that tmpB(Cl)PH_2 (tmp = 2,2,6,6-tetramethylpiperidino), in combination with H_2PLi or *t*-BuLi, forms a diphosphadiboretane, $(tmpBPH)_2$,[2] and we assume dehydrohalogenation proceeds through a transient boraphosphene, tmpB=PH. Our interest here was to determine whether diborylphosphanes (A)[3] undergo dehydrohalogenation with formation of boraphosphenes $(R_2N)_2BP{=}B(NR'_2)$ and, via dimerization, synthetically useful diphosphadiboretanes (B).

Scheme I



The equimolar reaction of tmpB(Cl)P(H)B(N-*i*-Bu_2)$_2$ (**1**) and *t*-BuLi in hexane produces the anticipated diphosphadiboretane, $(i\text{-}Bu_2N)_2BPB(tmp)P[B(N\text{-}i\text{-}Bu_2)_2]B(tmp)$ (**2**).[4] On the other hand, combination of tmpB(Cl)P(H)B(N-*i*-Pr_2)$_2$ (**3**) and *t*-BuLi (1:1) leads to $[tmpB(H)]PB(N\text{-}i\text{-}Pr_2)N(i\text{-}Pr)C(CH_3)_2$ (**4**)[5] (Scheme I), which with $Fe_2(CO)_9$ (1:1) gives a yellow, crystalline complex, $Fe(CO)_4$·**4** (**5**).[6] Spectroscopic data[7] indicate that **4** is not a diphosphadiboretane and that its general structure is not affected by metal complexation. Therefore, the molecular structure of **5** was determined in order to elucidate the nature of **4**.[8] The structure (Figure 1) reveals a novel, planar, asymmetric, four-membered azacarbaphosphaboretane ring with $Fe(CO)_4$ and tmpBH fragments as exo substituents on the phosphorus atom.

The structure of **4** does not preclude formation of a boraphosphene during this reaction. However, if produced, it does not dimerize as does the transient boraphosphene formed from **1**. Instead, the P=B(1) bond apparently undergoes intramolecular

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(4) A hexane solution of **1** (18.1 mmol, 8.8 g) was cooled to −78 °C, and *t*-BuLi (10.7 mL, 1.7 M) was added dropwise. The mixture was stirred at −78 °C (2 h) and at 23 °C (16 h) and then filtered, and the solvent was vacuum evaporated. The residue deposited yellow crystalline solid (5.0 g, 61%) **2**, mp 167–169 °C.

(5) Addition of *t*-BuLi (1.2 mL, 1.7 M) to a cooled (−78 °C) hexane solution of **3** (0.9 g, 2.1 mmol), followed by stirring at −78 °C (2 h) and 23 °C (24 h), resulted in a cloudy, yellow solution that was filtered, and the solvent was removed by vacuum evaporation. The residue crystallized upon standing (23 °C). Two recrystallizations from cold hexane gave white solid **4** (0.50 g, 61%), mp 87–89 °C.

(6) A sample of **4** (0.60 g, 1.5 mmol) in 50 mL of hexane was combined with $Fe_2(CO)_9$ (0.56 g, 1.5 mmol) and stirred (3 days). Solvent and volatiles were removed by vacuum evaporation, and the residue was extracted with hexane (25 mL). The extract was filtered, concentrated, and cooled to −10 °C, and brown crystals of **5** (0.4 g, 47%) were collected; mp 154–156 °C.

(7) Characterization data for **2**, **4**, and **5** (microanalysis, MS, IR, and ^{31}P, ^{11}B, ^{13}C, and ^{1}H NMR) are provided in the supplementary material.

(8) Selected crystal data for **5**, $C_{25}H_{46}B_2N_3O_4PFe$: orthorhombic, *Pbca* with a = 18.373 (3) Å, b = 17.845 (4) Å, c = 19.172 (3) Å, Z = 8, R_F = 0.088 and R_{wf} = 0.063.

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