to comigrate with the less mobile isomer derived from thiazolidine **9**. The stereochemical retentions observed in these biosynthetic ring closures are in accord with previous observations on the incorporation of chiral valines into penicillin with overall retention[9,10,11,12] and with the incorporation of valine into tripeptide without loss of chirality at C-3.[13,14] Hence the relative stereochemistry at C-3 of peptide-bound valine is retained during conversion to penicillin, in agreement with our results on peptide analogues.

The selectivity of ring closure onto a methylene group was assessed by incubating the peptide **5**, from D-α-aminobutyric acid. This yielded a bioactive, penicillinase-sensitive product (Table I). That this was a mixture of C-2 epimers of demethylisopenicillin N was demonstrated by oxidation to a mixture of sulfonic acids, corresponding in electrophoretic mobility to those obtained from the diastereoisomers of 2-amino-3-mercaptobutyric acid. The major, less mobile isomer corresponded to that derived from **13**,[15] whose configuration has already been assigned.[16] This result, i.e., preferential formation of **14**, together with those from peptides **3** and **4** suggests that cyclization is favored to a penicillin with the larger group at C-2 in the β configuration.

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Deuterium Isotope Effects on Methyl Transfer to Alcohols. Possible Asynchronous Solvent Repolarization and Internal Structural Changes

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Recently we reported[1] that the H_2O/D_2O kinetic isotope effect (KIE) on methyl transfers to water (reaction 1: L = H, D; R = L) vanishes when the water is a dilute solute in aprotic solvents.

$$ROL + CH_3X \rightarrow \underset{L}{RO^+CH_3} + X^- \qquad (1)$$

We now have observed that alkyl substitution on the nucleophilic oxygen (reaction 1, R = CH_3, t-C_4H_9) causes large changes in this KIE; these changes would not be expected for a conventional S_N2 mechanism but are consistent with our suggestion[1,2] that the activation process for methyl transfer to L_2O is predominantly a fluctuation in the structure of the solvent surrounding the L_2O,CH_3X reactant pair.

The spectrophotometric method for measuring rate constants and the preparations of *S*-methythiophenium ($MeTh^+$) hexafluorophosphate and methyl perchlorate ($MeOClO_3$) were as previously[1] described. Commercial CH_3OL and t-C_4H_9OL were purified by vacuum line distillation from $Mg(OCH_3)_2$ and freshly calcined CaO, respectively. In each run, the absorbance change rate was first order, and the pseudo-first-order rate constants (k_ψ) were linear functions of ROL concentration (eq 2) when [ROL] < 1 M in CH_3CN.[3] Table I summarizes the KIE's observed at different concentrations of ROL in CH_3CN.

$$k_\psi^L = k_0 + k_1^L[ROL] \qquad (2)$$

For both $MeTh^+$ and $MeOClO_3$, Table I shows that KIE for CH_3OL is greater than KIE for L_2O. This is the *reverse* of the order expected for displacements proceeding by conventional S_N2 mechanisms, since L_2O has twice as many O–L bonds as CH_3OL.[4]

Table I also shows that, except for t-C_4H_9OL + $MeOClO_3$, KIE in CH_3CN is less than KIE in neat ROL. The KIE in CH_3CN should be a measure of the $R(L)O\cdots CH_3$ bond order in the transition state ($n_{OC}^\ddagger$),[1,4] and if $n_{OC}^\ddagger$ does not greatly increase as the ROL/CH_3CN solvent ratio changes, then the increase in KIE when the solvent changes from CH_3CN to ROL can be attributed to the τ_D/τ_H factor[1,5] (τ = effective dielectric relaxation time of solvent) which is present when reorganization of isotopically substituted solvent structure contributes to motion along the reaction coordinate. Estimates of $n_{OC}^\ddagger$ based on the KIE's in CH_3CN are listed in Table II. The range of these estimated $n_{OC}^\ddagger$ values is larger than would be expected if these reactions had the same mechanism as most previously studied methyl transfers; structure–reactivity relationships and KIE's suggest that structural changes in the nucleophile (or equivalently in the leaving group) do not commonly induce such large changes in the internal structure of S_N2 activated complexes, particularly for methyl transfers.[6-14] However, the directions and large magnitudes of

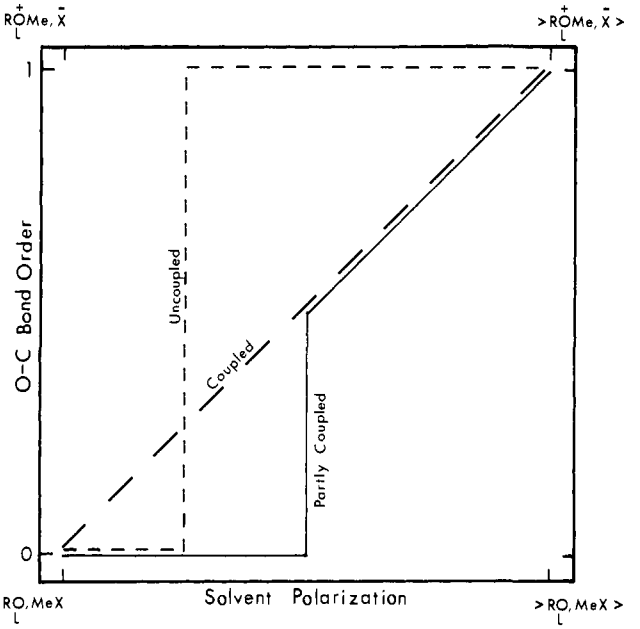

Figure 1. Example of possible reaction paths. The scale on the solvent polarization axis is defined by requiring that all structures along the diagonal path have solvent polarization in equilibrium with internal structure.

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Table I. KIE's on Displacements by ROL[a,b]

CH_3X	ROL	k_1^H/k_1^D [c,e] (dilute ROL)[f]	k_ψ^H/k_ψ^D [d,e] (20 mol % ROL)[f]	k_ψ^H/k_ψ^D [a,e] (neat ROL)
$MeTh^+$	L_2O	1.01_5	1.00_9	1.12_8
	CH_3OL	1.04_6	1.05_2	1.14_8
	t-C_4H_9OL	1.07_0	1.07_0	1.12_2
$MeOClO_3$	L_2O	0.99_5[g]		1.19_9
	CH_3OL	1.20_3	1.20_6	1.28_8
	t-C_4H_9OL	1.11_4	1.11_9	1.10_8

[a] See supplementary material for absolute rate constants, statistical parameters, and examples of data used to evaluate k_1^H/k_1^D. [b] At 25 °C, unless otherwise indicated. [c] k_1^L is the second-order constant defined by eq 2 for reaction in a dilute solution of ROL. [d] k_ψ^L is the pseudo-first-order constant. [e] Standard deviations of KIE's are all less than 1%. [f] In CH_3CN. [g] Measured at 35 °C.

Table II. Approximate Values of $n_{OC}^{\ddagger}$ [a]

CH_3X	$n_{OC}^{\ddagger}$ L_2O	CH_3OL	t-C_4H_9OL
$MeTh^+$	0.02	0.10	0.15
$MeOClO_3$	−0.01	0.40	0.23

[a] Estimated from KIE's in CH_3CN (Table I), using $n_{OC}^{\ddagger} \sim -\log \mathrm{KIE}/[2\log \phi(OL^+)]$ for L_2O and $n_{OC}^{\ddagger} \sim -\log \mathrm{KIE}/\log \phi(OL^+)$ for CH_3OL and t-C_4H_9OL.[4] Values of $\phi(OL^+)$ were taken as 0.69 for ROL = L_2O (the value for L_3O^+ in L_2O)[26] and 0.63 for ROL = CH_3OL or t-C_4H_9OL (the value for $CH_3OL_2^+$ in CH_3OL).[27] Uncertainties in $n_{OC}^{\ddagger}$ arising from uncertainties in KIE values are ca. ±0.02; those arising from uncertainties in $\phi(OL^+)$ may be larger.

the differences between $n_{OC}^{\ddagger}$ values in Table II both agree with expectations based on a model previously applied[5] to solvent–solute coupling in proton transfers.

Possible reaction paths implied by that model are shown in Figure 1: vertical displacements on that figure indicate changes in internal structure and charge distribution; horizontal displacements indicate changes in solvent polarization; the diagonal path includes all structures intermediate between reactant and product which have equilibrium solvation. If the forces which couple changes in solvent polarization to changes in internal structure are sufficiently strong, the reaction will proceed by a *coupled* (diagonal) mechanism, since deviations from the diagonal path will have prohibitively high free-energy costs. Weak coupling forces will allow an *uncoupled* mechanism in which solvent polarization can change at its natural (rotational) rate while internal bonding changes and the accompanying charge transfer occur at a faster (vibrational) natural rate.[15,17]

When the methyl transfer is to L_2O, the small, strongly hydrogen-bonding L_2O^+ moiety in the product will be very tightly solvated, and such tight solvation implies a strong coupling force between solvent polarization and internal charge distribution. Thus this coupling force will be much stronger after the transfer in the equilibrated successor complex ($L_2O^+CH_3,X^-$) than before transfer in the precursor complex (L_2O,CH_3X). If this difference in strength is sufficiently large, the reverse reaction ($X^-,CH_3OL_2^+ \rightarrow$) will start along the diagonal path, while the forward reaction ($L_2O,CH_3X \rightarrow$) is not so constrained. Furthermore, a sufficiently weak coupling force will allow a fluctuation in solvent polarization to occur around L_2O,CH_3X with a lower free-energy cost than that for the diagonal path along which bond making and breaking accompany the solvent structural fluctuation and contribute to the free-energy change. The complete path must be the same for both forward and reverse reactions; thus, for reactions with a sufficiently large change in coupling force, the mechanism will approximate the *partly coupled* path (Figure 1) which we have proposed[18] for methyl transfer to L_2O.[19]

Consider the standard free-energy ($G°$) profile above this path. Motion starting from reactants along the initial (horizontal) leg is away from equilibrium solvation and must result in a monotonic increase in $G°$; the transition state at the maximum in $G°$ must therefore lie *at* or *past*[21] the point where the path turns vertical. Along that second (vertical) leg, two factors contribute to the $G°$ profile: as the internal structure approaches the diagonal, the system approaches the minimum in a $G°$ well for solvent–solute interactions, and this tends to decrease $G°$; bonding changes add in a barrier, first increasing and eventually decreasing $G°$. Since the force constants for this well and this barrier have opposite signs, the net curvature of the $G°$ profile along this vertical leg will be smaller than for a diagonal mechanism. Finally, along the third (diagonal) leg, the $G°$ profile is the same as for a coupled mechanism. If the transition state lies on this third leg, the rate behavior will be indistinguishable from that of a reaction which follows a conventional diagonal path. However, if the transition state lies on the second (vertical) leg, the rate can behave unconventionally.

Now apply these arguments to the KIE and estimated $n_{OC}^{\ddagger}$ patterns shown in Tables I and II. (1) In these reactions, coupling forces should *decrease* as the nucleophile and leaving group vary in the order $L_2O > CH_3OL > t$-C_4H_9OL (steric hindrance of solvation increases), and $SC_4H_4 > OClO_3^-$ (methyl transfer from S^+ to ROL moves a charge; transfer from $OClO_3$ only increases a dipole moment). Thus t-C_4H_9OL + $MeOClO_3$ should have the weakest coupling forces and should be the reaction most likely to follow the completely uncoupled path; that path is consistent with the KIE for this *single* reaction being the same in ROL as in CH_3CN.[22] (2) The estimated $n_{OC}^{\ddagger}$ values for the other five reactions behave as if those reactions follow partly coupled mechanisms and have their transition states along the vertical leg of the path. Increasing steric hindrance of solvation will destabilize more polar (product-like) internal structures; therefore[23] the transition state should shift upward along that vertical leg, increasing $n_{OC}^{\ddagger}$ in the order $L_2O < CH_3OL < t$-C_4H_9OL; this is the observed order of the estimated $n_{OC}^{\ddagger}$ values (Table II). (3) For a transition state on the vertical leg, both the small curvature of the $G°$ profile and the vertical orientation of the path will increase the sensitivity of $n_{OC}^{\ddagger}$ to structural perturbations;[9,23-25]

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this is in accord with the wide range of variation observed for the estimated $n_{OC}^{\ddagger}$ values.

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Supplementary Material Available: Numerical values of rate constants and examples of fits of observed k_ψ's to eq 2 and of evaluations of k_1^H/k_1^D (3 pages). Ordering information is given on any current masthead page.

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Homolytic Substitution at Carbon: 1,3- and 1,5-Ring Closures in Organotin-Substituted Radicals

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Pivotal to many studies of possible S_H mechanisms at saturated carbon[1] have been reactions involving 1,3-ring closure[2] or cleavage.[3] Remotely functionalized organometallics, particularly organotins,[4] have been shown to undergo facile, stereospecific (inversion),[5] heterolytic 1,3-ring closures; however the scope and mechanisms of the analogous homolytic 1,3-elimination reactions are much less clear. Kaplan and Drury[6] reported that cyclopropane is formed quantitatively by the Ph_3SiH-initiated reaction of γ-haloorganotins:

$$Ph_3SnCH_2CH_2CH_2Cl \xrightarrow[225\ ^\circ C]{3\%\ Ph_3SiH} C_3H_6 + Ph_3SnCl \quad (1)$$

In such cases a dichotomy exists in which bimolecular homolytic substitution (S_H2) at either the metal or the halogen (depending upon the chain carrier) leads to possible cyclopropane-precursor radicals:

$$M\text{-}CH_2CH_2CH_2\text{-}X \xrightarrow{X\cdot} M\text{-}X + \dot{C}H_2CH_2CH_2X \rightarrow C_3H_6 + X\cdot \quad (2)$$

$$M\text{-}CH_2CH_2CH_2\text{-}X \xrightarrow{M\cdot} M\text{-}X + M\text{-}CH_2CH_2\dot{C}H_2 \rightarrow C_3H_6 + M\cdot \quad (3)$$

In this work we demonstrate that not only are S_H2 reactions at tin competitive with remote atom-abstraction reactions but that γ- and ϵ-trimethyltin-substituted alkyl radicals undergo 1,3- and 1,5-ring closures (S_Hi) efficiently.

The reaction of trimethyl(3-methoxypropyl)tin[7] (**1**), with a variety of photochemically generated H abstractors ($Ph_2CO/h\nu$; $(t\text{-BuO})_2/h\nu$; $FeCl_3/h\nu$; $PhCOMe/h\nu$) in benzene at 22 °C leads to the formation of cyclopropyl methyl ether, tetrahydrofuran, and methyl propyl ether (Table I). The reaction sequence shown in eq 4-12 accounts for the observed products in terms of competitive H-abstraction reaction (eq 4 and 5) and carbon-tin cleavage (eq 6) followed by product-forming steps. The detailed mechanism of eq 6, in particular, could involve either direct substitution or an initial electron transfer to the ketone triplet, followed by collapse of an organotin cation-ketyl anion radical pair to the intermediates shown. In either case, the net result is a bimolecular radical-induced cleavage of the carbon-tin which we refer to herein as an S_H2 reaction. The similar yields of acyclic and cyclic ethers suggest that the intermolecular substitution reaction at tin and H abstraction from the ether α carbons of **1** are competitive and that subsequent 1,3- or 1,5-ring closure reactions occur with similar facility. The relative yields of cyclopropyl methyl ether and tetrahydrofuran may reflect the greater reactivity of methylene vs. methyl hydrogens toward abstraction and/or the greater reactivity of tetrahydrofuran toward subsequent reactions. Under these conditions neither **1**, **2**, nor **5** give rise to detectable amounts of methyl radical products (methane or dimethylorganotins). This is the only reported homolytic 1,5-ring closure reaction of organotin compounds and shows some analogy to the heterolytic reactions of organotins.[4b]

Table I. Reaction of Trimethyl(3-methoxypropyl)tin with H Abstractors[a]

H abstractor	% conversion[b]	rel product distribution MPE[c]	CPME[d]	THF[e]
$Ph_2CO/h\nu$[f]	48	1.0	1.1	1.0
$Ph_2CO/h\nu$	33	1.0	0.33	>0.08
$PhCOMe/h\nu$	49	1.0	0.21	0.30
$(t\text{-BuO})_2/h\nu$	43[g]	1.0	0.88	0.65
$FeCl_3/h\nu$[h]	37	1.0	0.5	>0.05

[a] In degassed benzene at 0.67 M, 22 °C, 72 h, 200-W medium pressure Hg arc. [b] By difference from recovered 1. Mass balances for other products exceed 70%. [c] MPE = methyl propyl ether. [d] CPME = cyclopropyl methyl ether. [e] THF = tetrahydrofuran. [f] $[Ph_2CO] = 0.67$ M, $[1] = 0.22$ M. Me_6Sn_2 and benzopinacol isolated and identified by NMR spectroscopy and mixed melting point, respectively. Yields >85% in both cases, corrected for conversion. [g] 48 h. [h] Reference 21.

$$1 + Ph_2CO^*(T) \longrightarrow \underset{\mathbf{1a}}{Me_3SnCH_2CH_2CH_2\dot{O}CH_2} + Ph_2\dot{C}OH \quad (4)$$

$$1 + Ph_2CO^*(T) \longrightarrow \underset{\mathbf{1b}}{Me_3SnCH_2CH_2\dot{C}HOCH_3} + Ph_2\dot{C}OH \quad (5)$$

$$1 + Ph_2CO(T)^* \longrightarrow \underset{\mathbf{1b}}{\dot{C}H_2CH_2CH_2OCH_3} + Ph_2\dot{C}OSnMe_3 \quad (6)$$

$$\mathbf{1b} \longrightarrow \underset{\mathbf{1c}}{\text{cyclopropyl-}OCH_3} + Me_3Sn\cdot \quad (7)$$

$$\mathbf{1a} \longrightarrow \text{tetrahydrofuran} + Me_3Sn\cdot \quad (8)$$

$$\mathbf{1c} + \text{solvH (or 1)} \longrightarrow CH_3CH_2CH_2OCH_3 + \text{solv}\cdot\ (\text{or 1a,1b}) \quad (9)$$

$$Me_3SnO\dot{C}Ph_2 \rightleftharpoons Me_3Sn\cdot + Ph_2CO\ (\text{ref 8}) \quad (10)$$

$$2\,Me_3Sn\cdot \longrightarrow (Me_3Sn)_2 \quad (11)$$

$$2\,Ph\dot{C}OH \longrightarrow (Ph_2COH)_2 \quad (12)$$

Reports on the mechanism of trichloromethyl abstractions from 2-(trimethylstannyl)butane,[9] the rate constant for *tert*-butoxy abstraction from the α position of tetraethyltin,[10] the absolute rate constants for benzophenone triplet and *tert*-butoxy abstractions

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