Steric and Electronic Effects in Conformational Preferences of **C1-Oxygenated Chiral Alkenes**

Benjamin W. Gung,* Jason P. Melnick, Mark A. Wolf, and Amanda King

Department of Chemistry, Miami University, Oxford, Ohio 45056

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A variable temperature NMR study shows that the benzyl protective group on the hydroxy function of a chiral allylic alcohol enhances the CH eclipsed form (I). On the other hand, various silyl ethers enhance the preference for the CO eclipsed conformer. However, when both the allylic R group and the hydroxy protective group are bulky (R = tert-butyl, P = TIPS), the staggered conformation of the chiral alkene becomes preferred. An acetate group does not have an apparent effect on the conformational preference of the protected allylic alcohol. These facts are explained in terms of steric and electronic interactions.

Introduction

It is known that the preferred conformations about a $C_{sp2}-C_{sp3}$ bond are the eclipsed forms; i.e., the allylic C-X linkage is coplanar with the double bond, I-III.¹⁻⁴ The staggered form (IV) has been identified as an energy maximum. For example, the staggered form of propene and 1-butene is the barrier to rotation around the C_{sv2} - C_{sp3} bond and has relative energies of 1.81 and 1.75 kcal/ mol,^{1b,d} respectively. The staggered form of 3-buten-2-ol has also been identified as an energy maximum.^{1f,4} Two types of interactions have been suggested in the literature, which account for the stabilities in rotational isomers about a $C_{\rm sp2}-C_{\rm sp3}$ bond. One theory emphasizes the repulsive interactions. As pointed out by Wiberg for acetaldehyde, $^{\rm 1d}$ the energy barriers in a $C_{\rm sp2}-C_{\rm sp3}$ rotamer have the same origin as in ethane, which are the results of torsional strain. The other theory stresses stabilizing hyperconjugative effects,^{1f} such as the overlap between $\pi_{C=C}$ and σ^*_{CO} and between σ_{CH} and $\pi^*_{C=C}$ orbitals. Both effects may contribute to the stabilities of individual conformers. The effect of torsional strain appears to be more obvious since conformation with eclipsed single bonds, such as IV, is energy maximum. In this paper, we report some exceptions. We will show that the staggered form (IV) could become the preferred conformation if steric effects are dominant.

The vinyl substituent (R', eq 1) has a profound influence on the relative stability of the ground state conformation of an (E)-chiral alkene.^{1f} We have previously



reported that the CH eclipsed form (I) is preferred for ordinary chiral allylic alcohols $(\mathbf{R}' = alkyl group)$ and the CO eclipsed form (II) is preferred for γ -hydroxy- α , β unsaturated esters ($\mathbf{R'} = \mathbf{CO}_2\mathbf{Et}$).⁴

More recently, we have found that the protective group

(P, eq 1) of the allylic hydroxy function in a chiral alkene influences the conformational preferences.⁵ The tertbutyldimethylsilyl (P = TBDMS) ether was found to promote the CO eclipsed form while a methyl ether (P =CH₃) enhances the CH eclipsed form.⁵ This observation was rationalized in terms of the difference in the oxygen lone pair size between the silvl and the methyl ethers. The lower energy $\pi^*(SiR_3)$ (relative to $\pi^*(CH_3)$) enables the oxygen lone pair electrons in a silvl ether to delocalize between the silicon and the oxygen atoms.⁶ Thus, for the CO eclipsed silyl ether, there should be a less repulsive interaction between the π_{C-C} orbital and the lone pair electrons on the allylic oxygen than for the corresponding conformation in methyl ether. In order to evaluate the validity of this rationalization, a conformational study of chiral allylic alcohols protected with benzyl, acetate, and various silyl groups has been carried out. It has been found that the benzyl ethers behave almost identically to methyl ethers while allylic acetates have no apparent effect on conformational preference. The various silvl ethers show the same preference as the TBDMS ether except when both the allylic R group and the protective group are very bulky. In the latter cases, a staggered form was observed, and these compounds exhibit limited conformational flexibility. It is potentially useful if the protective groups can be used to control diastereofacial selectivity since they may be easily manipulated.⁷ More than 20 years ago. Corey reported the use of a bulky allylic hydroxy protective group (tribenzylsilyl ether) to direct the π -facial selection in a prostaglandin A synthe-

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sis.⁸ This study has uncovered some previously unknown directing effects of the hydroxy protective groups on alkene conformation.

Experimental Section

The allylic alcohols 1a-d and 4a-d were prepared according to known procedures.⁵ The protection of the hydroxy group was carried out following procedures outlined in Scheme 1. For ordinary allylic alcohols, such as **1a-d**, the acetate and the benzyl groups are introduced by treating the alcohols directly with sodium hydride/benzyl bromide and acetyl chloride to generate the corresponding benzyl ethers (2a-d) and acetates (3a-d), respectively. However, the γ -hydroxy- α , β unsaturated esters 4a-d fail to produce the desired benzyl ethers when treated with sodium hydride and benzyl bromide. Accordingly, the alternative route starting with the benzyl ethers (2a-d) was employed. Ozonolysis of 2a-d followed by the addition of dimethyl sulfide and the Wittig reagent $Ph_3P=CHCO_2Et$ generates the desired products 6a-d. The reactions of the γ -hydroxy- α , β -unsaturated esters **4a**-**d**, with the various trialkylsilyl chloride in the presence of imidazole in DMF proceeded smoothly to produce the silvl ethers 7ad-10a,b.⁹ The introduction of the bulky triisopropylsilyl (TIPS) group to the isopropyl and tert-butyl derivatives (10c and 10d) was accomplished by using the more reactive TIPS trifluoromethanesulfonate (OTf) in the presence of 2,6-lutidine.¹⁰ The purity of the samples for the VT NMR experiments is judged to be > 95% based on the NMR spectra.

The ¹H NMR spectra of the chiral alkenes 2, 3, and 5-10were recorded on a Bruker 300 MHz NMR instrument at various temperatures. All data were obtained in CDCl₃. The VT NMR experiments all followed a general procedure. Typically, a 0.05 M solution of the sample in the solvent was placed in a high quality NMR tube. All samples were degassed using a glass pipette to bubble nitrogen or argon through the sample for ~ 30 s. The sample tube was then placed into the NMR probe using the heavy spinner. The air line responsible for spin was disconnected, and the delivery hose from the liquid nitrogen Dewar was connected to the NMR probe. The air line responsible for lifting the NMR tube and spinner out of the NMR probe was disconnected and the NMR probe was capped. The desired temperature was set on the variable temperature unit (console), and the self-tune procedure was initiated to calibrate the console. Following calibration, the temperature reading on the variable temperature console was allowed to stabilize. The sample was equilibrated for approximately 10-15 min at the set temperature. It is a common practice to raise the temperature slowly especially when approaching 325 K to avoid evaporation of the solvent. The accuracy of the temperature display on the console, which is measured from a thermocouple located inside the probe, was tested by measuring the chemical shifts of methanol where Δv was scaled to 220 MHz. Calibration of the temperature dependence of the separation (in hertz) between the OH resonance and the CH₃ resonance has been reported by Becker.¹³ The calibration results show a <1 K deviation in the temperature range employed.

The following steps were taken in order to improve the resolution of the spectra. A preliminary spectrum, which consisted of 16 scans and a memory size of 16K, was taken. After normal work up of the data, the spectral window was set to include only the signals for the compound under study. The window was typically 1800-2200 Hz. The size of memory was then set to 128K so that a total of 131 072 data points were collected, giving a digital resolution of 0.017 Hz. Typically 32 or more scans were collected at temperatures below 273K to improve the signal to noise ratios. The reported coupling constants were taken from the averaged values of the two coupled protons except when one proton signal was obscured by other peaks. For example, in the case of compound 7b, the H1 signal was obscured by the peaks from the ethoxy group. The ${}^{3}J_{12}$ coupling for 7b was assigned from the splitting patterns of H2 only. Selected chemical shifts (δ) and coupling constants (Hz) are compiled in the table in the supplementary material. The three-bond coupling constants ${}^{3}J_{12}$ are presented graphically in Figures 1–8.

Results and Discussion

The usual assumptions associated with the J-averaging method are also considered to be true in this study.¹¹ Thus, all coupling constants observed are averages from the three eclipsed rotamers. The coupling between H₁ and H₂ of the alkenes is assumed to be related to the dihedral angle $\tau_{\rm H1CCH2}$ following the Karplus relationship.¹² Thus, the observed coupling constants are related to the populations of different rotamers by the following equation.

$${}^{3}J_{\rm obs} = p_{\rm i}J_{\rm t} + p_{\rm iii}J_{\rm g} + p_{\rm iii}J_{\rm g'}$$
(2)

The p_i , p_{ii} , and p_{iii} are the fractional populations, and J_t , J_g , and $J_{g'}$ are the coupling constants characteristic

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of conformers I–III, respectively. The trans three-bond coupling $(J_{\rm t} \sim 10 \text{ Hz})$ is about five times as large as the gauche coupling $(J_{\rm g} \sim 2 \text{ Hz})$. Therefore, a change in the population of the CH eclipsed rotamer (p_i) will in turn cause a change in the observed coupling constant ${}^{3}J_{\rm obs}$. A greater observed ${}^{3}J_{\rm obs}$ indicates an increased population of conformer I. On the basis of the Boltzmann distribution law, eq 3, the more stable isomer becomes more

$$\frac{N_2}{N_1} = e \frac{(E_2 - E_1)}{kT}$$
(3)

populated at lower temperature. Thus, a greater observed coupling constant at lower temperatures indicates that the CH-eclipsed conformer is more stable, while a smaller ${}^{3}J_{obs}$ at lower temperatures indicates that the COeclipsed form is more stable. Due to steric interactions, the C–R eclipsed form **III** is assumed to be the highest energy conformer and only exists in a negligible amount.

The spin-spin coupling constants $({}^{3}J_{12}, \text{Hz})$ as a function of temperature for chiral allyl benzyl ethers $2\mathbf{a}-\mathbf{d}$ are plotted graphically in Figure 1. The generally large (7.5-9.3 Hz) and considerably greater coupling constants at lower temperatures indicate a preference for the CH-eclipsed conformer I. The extent of the preference for one rotamer can be estimated from the magnitude of the observed coupling constants. The ${}^{3}J_{obs}$ exhibited by the benzyl ethers $2\mathbf{a}-\mathbf{d}$ are slightly larger than those by the corresponding methyl ethers.⁵ However, the same trend is observed. Benzyl ether, like methyl ether, promotes the CH-eclipsed form.

The allyl acetates $3\mathbf{a}-\mathbf{d}$ also show preferences for the CH-eclipsed conformation, Figure 2. However, the preference is smaller than that exhibited by the benzyl ethers $2\mathbf{a}-\mathbf{d}$ as indicated by the average observed coupling constants shown in Figures 1 and 2. This fact can be explained by the electron-withdrawing character of the acetyl group. From our recent studies, it has been suggested that the major factor which destabilizes the CO-eclipsed form II is the repulsive interaction between the lone pair electrons on the allylic oxygen atom and the π electrons of the double bond since they are in close proximity in conformation II.⁵ We found that allyl silyl ethers tend to promote the CO-eclipsed form due to their shrunk size of the oxygen lone pair.⁵



Conformation II of the acetates $3\mathbf{a}-\mathbf{d}$ is expected to suffer less from the repulsion between the oxygen lone pair and the $\pi_{C=C}$ orbital since the acetyl group is an electron-withdrawing group (EWG). As the magnitude of the coupling shows (the acetates exhibit ~1 Hz smaller ${}^{3}J_{12}$ than the benzyl ethers at all temperatures), the acetates $3\mathbf{a}-\mathbf{d}$ have indeed a smaller preference for the CH-eclipsed form assuming the difference in ${}^{3}J_{12}$ is caused solely by the difference in rotamer population. The second most important influence on the magnitude of spin-spin coupling is the substituent electronegativity (the first being the dihedral angle). If the smaller ${}^{3}J_{12}$ of $3\mathbf{a}-\mathbf{d}$ were caused by the electron-withdrawing ability of the acetate function, the same effect should have been seen on compounds $5\mathbf{a}-\mathbf{d}$. The fact that the acetates



Figure 1. Spin-spin coupling constants $({}^{3}J_{12}, \text{Hz})$ as a function of temperature for chiral allyl benzyl ethers $2\mathbf{a}-\mathbf{d}$. The coupling constants increased considerably at lower temperatures indicate a preference for the CH-eclipsed conformer I.



Figure 2. Spin-spin coupling constants $({}^{3}J_{12}, \text{ Hz})$ as a function of temperature for chiral allyl acetates 3a-d. The greater coupling constants at lower temperatures indicate a preference for the CH-eclipsed conformer I.

5a-**d** show slightly larger ${}^{3}J_{12^{r}}$ couplings than their parent alcohols supports the conclusion based on dihedral angle and rotamer population.

Nevertheless, the acetates 3a-d still prefer the CHeclipsed form. This is in contrast to the previously reported allyl *tert*-butyldimethylsilyl ethers (same structure as 3a-d except replacing acetates with the silyl group), which have no preference for either conformation at all.⁵ This difference shows that the acetyl group, although a σ -acceptor, does not reduce the lone pair size on oxygen as much as a silyl group. It also shows that a silyl group, although a σ -donor, is an excellent π -acceptor, which reduces the lone pair size on oxygen by delocalization. That is why the silyl ethers enhance the COeclipsed form (II).

The three-bond coupling constants ${}^{3}J_{12}$ for the acetate protected γ -hydroxy- α , β -unsaturated esters **5a**-**d** are presented in Figure 3. The magnitude of ${}^{3}J_{12}$ in this case indicates that the dihedral angle $\tau_{\rm H1CCH2}$ is in the neighborhood of 60°, consistent with a conformation close to either **II** or **III**. The virtual lack of an effect by the allylic substituent R suggests that conformation **II** is preferred. The acetate function does not override or enhance the vinyl substituent effect previously reported for the parent γ -hydroxy- α , β -unsaturated esters.⁵ By comparison, the acetate group appears to slightly reduce the preference for the CO-eclipsed form. The observed coupling constants for the parent γ -hydroxy- α , β -unsaturated esters are ~0.2 Hz smaller than those observed for **5a**-**d**.



Figure 3. Spin-spin coupling constants $({}^{3}J_{12}, Hz)$ as a function of temperature for acetates **5a**-**d**. The decreasing coupling constants at lower temperatures indicate a preference for the CO-eclipsed conformer **II**.



Figure 4. Spin-spin coupling constants $({}^{3}J_{12}, Hz)$ as a function of temperature for benzyl ethers **6a**-**d**. The greater coupling constants at lower temperatures indicate a preference for the CH-eclipsed conformer **I**.

The benzyl ethers **6a**-d exhibit opposite trends from their unprotected parent compounds, Figure 4. The ${}^{3}J_{12}$ increases as the temperature is lowered. Since the magnitude of coupling is in the range of 6.0-7.2 Hz, the increase in ${}^{3}J_{12}$ may be explained in terms of an increase in the population of conformer I, the CH-eclipsed form, or an increase in the population of the staggered form, IV, where the dihedral angle $\tau_{\rm H1CCH2}$ is 0. However, as described in the Introduction, it is known that the staggered form IV is an energy maximum, rather than a minimum, in propene^{1b} and other related allylic compounds.¹⁻⁴ Thus, the benzyl group, like methyl, overrides the vinyl substituent effect and promotes the CH-eclipsed form.

A striking similarity is apparent when the data in Figure 4 are compared to that previously reported for the corresponding methyl ethers.⁵ Since the benzyl group is larger than a methyl group, steric effect appears to be an unlikely factor in the promotion of the CH-eclipsed form. Instead, the evidence is consistent with the proposal that the lone pair electrons on oxygen are responsible for destabilizing the CO-eclipsed form. Thus, both methyl and benzyl groups promote the CH-eclipsed form due to the large lone pairs in these ether functions. On the other hand, despite of the bulky steric size, silyl ethers reduce the preference for the CH-eclipsed form because the smaller lone pair size in these ethers.⁵

The VT NMR data for the γ -hydroxy- α , β -unsaturated esters protected with various silyl groups (**7a-10d**) are presented in Figures 5–8. Except for compounds **10c** and **10d**, all silyl ethers favor the CO-eclipsed conformation. An enhancement for the preference of the CO-



Figure 5. Spin-spin coupling constants $({}^{3}J_{12}, Hz)$ as a function of temperature for the trimethylsilyl ethers **7a-d**. The smaller coupling constants at lower temperatures indicate a preference for the CO-eclipsed conformer II.



Figure 6. Spin-spin coupling constants $({}^{3}J_{12}, Hz)$ as a function of temperature for the triethylsilyl ethers **8a-d**. The smaller coupling constants at lower temperatures indicate a preference for the CO-eclipsed conformer II.



Figure 7. Spin-spin coupling constants $({}^{3}J_{12}, Hz)$ as a function of temperature for the triphenylsilyl ethers 9a-d. The decreasing coupling constants at lower temperatures for 9a-c indicate a preference for the CO-eclipsed conformer II. The consistently large coupling constants for 9d indicate a rigid CH-eclipsed conformer.

eclipsed form is apparent when comparing the data to their corresponding parent γ -hydroxy- α , β -unsaturated esters.⁵ This enhancement of preference for the COeclipsed form provides additional proof that the lone pairs on the oxygen atom of the silyl ethers have a reduced size, a theoretical prediction made by Jorgenson and Schreiber.¹⁴

A universal trend is observed for all of the alkenes studied. A bulkier allyl R group gives a larger average coupling constant, ${}^{3}J_{12}$. The gradual increase of coupling from the methyl derivatives (**7a**, **8a**, **9a**, and **10a**) to the *tert*-butyl derivatives (**7d**, **8d**, **9d**) (see Figures 1-8) can be explained in two ways. First, the observed effect may

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Figure 8. Spin-spin coupling constants ${}^{3}J_{12}$, Hz) as a function of temperature for the triisopropylsilyl ethers 10a-d. The smaller coupling constants at lower temperatures for 10a-c indicate a preference for the CO-eclipsed conformer I. The large and consistent coupling constants for 10d indicate a rigid CH-eclipsed conformer.

be a manifestation of the decrease in the population of conformation **III** at all temperatures. Secondly, it is also possible that it is the result of changes in the dihedral angles of the allylic carbon atom, $\phi_{\rm CO}$ and $\phi_{\rm CH}$, due to the steric requirement of the R group.



To expand on the second possibility, the dihedral angle ϕ_{CO} should increase when the allyl R is increased in steric bulk. This in turn should cause an increase in J_g based on the Karplus relationship. Since the population of the CO-eclipsed form (II) is greater than the CH-eclipsed form (I) for compounds 7–10, the net result should be an increase in the observed coupling constants, and this is indeed observed. However, currently we cannot rule out one of the two possibilities.

The most unusual results come from the compounds with extremely bulky substituents. The triphenylsilyl (TPS) and the TIPS ether of the *tert*-butyl derivatives **9d** and **10d** exhibit a constant, large coupling (\sim 8.0 Hz) at all temperatures (see Figures 7 and 8). This is a departure from the norm of all the other silyl ethers. It is obvious that the compounds **9d** and **10d** assume a conformation that would relieve the steric strain caused by both the allylic *tert*-butyl group and the bulky protective group. On the basis of the coupling constant, we suggest that instead of the normally favored CO-eclipsed form, compounds 9d and 10d maintain an equilibrium between the CH-eclipsed form and the staggered form. It is unusual to suggest that the staggered form (IV) is a minimum since it is normally a barrier. However, its presence for compounds 9d and 10d was confirmed by the observation that irradiation of H1 gave a 7.2% and 4.8% of enhancement of H2 and H3, respectively. Similarly, irradiation of H2 led to an enhancement of H1. Thus, steric effects are dominant in the conformations of 9d and 10d, which has raised the energies of the normally preferred eclipsed-form (II) to such a level that the staggered form (IV) becomes an energy minimum. Thus, it is possible to manipulate the conformations of the C1-oxygenated chiral (E)-alkenes by the hydroxy protective groups. Further study on the diastereofacial selectivity of these chiral alkenes is in progress.



Conclusions

Like methyl ethers, benzyl ether protective group promotes the CH-eclipsed form of the (E)-allylic chiral alcohols. The acetate group has no apparent effect on the conformational preference of a chiral alkene. Silyl ethers enhance the CO-eclipsed form by delocalizing the lone pair electrons on the allylic oxygen atom. Steric effects come into play when both the allyl R and the protective group are very bulky. In such cases, the staggered conformation becomes the predominant form.

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Supplementary Material Available: ¹H NMR data for compounds 2, 3, and 5-10 (9 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.

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