tained in 98% de by one crystallization from EtOAc/Skelly B (mp 138–139 °C). Pure samples of each diastereomer were obtained by semipreparative HPLC.

For the major diastereomer: mp 142.5–143 °C; $[\alpha]^{25}_D$ +104.2° (*c* 1.0, acetone); $^{13}$C NMR (75 MHz) δ 143.2 (s), 142.3 (s), 134.7 (s), 132.3 (s), 128.9 (d), 128.7 (d), 128.6 (d), 128.2 (d), 127.8 (d), 127.8 (d), 126.8 (d), 125.3 (d), 120.5 (d) 86.1 (d), 51.5 (d), 35.3 (t), 34.2 (t), 25.5 (t), 25.1 (t); $^1$H NMR (300 MHz) δ 7.79–7.75 (m, 2 H), 7.69 (d, *J* = 8.7 Hz, 1 H), 7.52–7.46 (m, 3 H), 7.40–7.38 (m, 3 H), 7.27–7.25 (m, 2 H), 6.84 (d, *J* = 8.4 Hz, 1 H), 4.62–4.55 (m, 1 H), 2.72–2.63 (m, 1 H), 2.42–2.38 (m, 1 H), 1.99–1.30 (br m, 7 H); IR 3020, 2920, 1850, 1470, 1440, 1340, 1130 cm$^{-1}$; MS-CI *m/z* 351 (M + H), 221, 193, 159; HRMS-CI *m/z* calcd for $C_{22}H_{23}O_2S$ ($M^+$ + H) 351.1419, found 351.1396.

For the minor diastereomer: mp 113–114 °C; $[\alpha]^{25}_D$ +124.5° (*c* 1.0, acetone); $^{13}$C NMR (75 MHz) δ 142.6 (s), 142.1 (s), 134.8 (s), 132.4 (s), 129.1 (d), 128.9 (d), 128.2 (d), 128.0 (d), 127.8 (d), 126.8 (d), 126.5 (d), 126.1 (d), 120.8 (d), 81.6 (d), 50.4 (d), 35.2 (t), 34.1 (t), 25.6 (t), 25.1 (t); $^1$H NMR (300 MHz) δ 7.86–7.81 (m, 3 H), 7.74–7.71 (dd, 1 H), 7.59–7.54 (m, 2 H), 7.27–7.11 (m, 4 H), 7.10–7.00 (m, 2 H), 4.35–4.20 (m, 1 H), 2.82–2.72 (m, 1 H), 2.54–2.49 (m, 1 H), 1.92–1.72 (m, 4 H), 1.50–1.26 (m, 3 H); IR 3020, 2920, 1590, 1470, 1440, 1340, 1130 cm$^{-1}$; MS-CI *m/z* 351 (M + H), 175, 159, 133; HRMS-CI *m/z* calcd for $C_{22}H_{23}O_2S$ ($M^+$ + H) 351.1419, found 351.1395.

**(+)-*trans*-2-Phenylcyclohexyl Methanesulfinates (3c).** The procedure above was followed except with 2.25 g (22.8 mmol) of methanesulfinyl chloride (prepared from dimethyl disulfide),[11] 1.85 mL (22.8 mmol) of pyridine, and 1.0 g (5.7 mmol) of (+)-*trans*-2-phenylcyclohexanol. Isolation as above afforded 1.08 g (80%) as a 9:2 mixture of diastereomers (α 1.20, 4:1 Skelly B/EtOAc). Pure samples of each diastereomer was obtained by semipreparative HPLC. The major diastereomer was assigned the *S* configuration at sulfur based on its conversion to (*S*)-methyl *p*-tolyl sulfoxide (see below). In this case, the minor diastereomer was less soluble and it was not possible to purify the major isomer by recrystallization. Conversly, the *R* isomer could be obtained in 98% de and 62% recovery by two crystallizations of a 1.49-g sample of a 3:7 mixture of major to minor diastereomer.

For the major diastereomer, (+)-(1*S*,2*R*)-*trans*-2-phenylcyclohexyl (*S*)-methanesulfinate: mp 36–38 °C; $[\alpha]^{25}_D$ +25° (*c* 2.0, acetone); $^{13}$C NMR (75 MHz) δ 142.8 (s), 128.3 (d), 127.9 (d), 126.8 (d), 85.7 (d), 51.3 (d), 44.0 (q), 34.9 (t), 33.2 (t), 25.4 (t), 25.0 (t); $^1$H NMR (300 MHz) δ 7.34–7.17 (m, 5 H), 4.23–4.14 (m, 1 H), 2.66–2.56 (m, 1 H), 2.30–2.23 (m, 1 H), 2.08 (s, 3 H), 1.98–1.25 (br m, 7 H); IR 3010, 2850, 1065, 1050 cm$^{-1}$; MS-CI *m/z* 239.3 (M + H), 159.2; HRMS-CI *m/z* calcd for $C_{13}H_{19}O_2S$ (M + H) 239.1106, found 239.1074.

The minor diastereomer, (+)-(1*S*,2*R*)-*trans*-2-phenylcyclohexyl (*R*)-methanesulfinate, could be obtained in diastereomerically pure form (analytical HPLC) by fractional crystallization from Skelly B: mp 56–56.5 °C; $[\alpha]^{25}_D$ +157° (*c* 2.0, acetone); $^{13}$C NMR (75 MHz) δ 142.7 (s), 128.5 (d), 127.7 (d), 126.5 (d), 79.7 (d), 50.2 (d), 43.7 (q), 34.1 (t), 33.9 (t), 25.5 (t), 24.8 (t); $^1$H NMR (250 MHz) δ 7.34–7.17 (m, 5 H), 4.36–4.26 (m, 1 H), 2.77–2.67 (m, 1 H), 2.31–2.26 (m, 1 H), 2.20 (s, 3 H), 2.00–1.25 (m, 7 H); IR 3015, 2980, 2850, 1450, 1250, 1065, 1050 cm$^{-1}$; MS-CI *m/z* 239.3 (M + H), 159.2; HRMS-CI *m/z* calcd for $C_{13}H_{19}O_2S$ (M + H) 239.1106, found 239.1053.

**(+)-*trans*-2-Phenylcyclohexyl 2-Propanesulfinates (3d).** The procedure above was followed with 3.5 g (27.6 mmol) of 2-propanesulfinyl chloride (prepared from diisopropyl disulfide),[11] 2.2 mL (27.6 mmol) of pyridine, and 1.22 g (6.9 mmol) of (+)-*trans*-2-phenylcyclohexanol and a 4-h reaction time. Isolation as described above afforded a quantitative yield of a 9:2 mixture of diastereomers (α 1.13, 4:1 Skelly B/EtOAc). Pure samples of each diastereomer were obtained by preparative and semipreparative HPLC.

For the major diastereomer, (+)-(1*S*,2*R*)-*trans*-2-phenylcyclohexyl 2-propanesulfinate: mp 32.5–33 °C; $[\alpha]^{25}_D$ +51.7° (*c* 1.0, acetone); $^{13}$C NMR (75 MHz) δ 143.0 (s), 128.3 (d), 127.8 (d), 126.6 (d), 84.9 (d), 51.3 (d), 35.0 (t), 34.0 (t), 25.5 (t), 25.0 (t), 13.4 (q), 13.3 (q); $^1$H NMR (300 MHz) δ 7.30–7.15 (m, 5 H), 4.26–4.19 (m, 1 H), 2.64–2.60 (m, 1 H), 2.37–2.21 (m, 1 H), 1.94–1.30 (br m, 7 H), 0.80 (d, *J* = 7.2 Hz, 3 H), 0.76 (d, *J* = 7.2 Hz, 3 H); IR 3020, 2920, 2850, 1440, 1250, 1110 cm$^{-1}$; MS-CI *m/z* 267 (M + H), 159, 109; HRMS-CI *m/z* calcd for $C_{15}H_{23}O_2S$ (M + H) 267.1419, found 267.1396.

For the minor diastereomer, (+)-(1*S*,2*R*)-*trans*-2-phenylcyclohexyl 2-propanesulfinate: $[\alpha]^{25}_D$ +196.6° (*c* 0.77, acetone); $^{13}$C NMR (75 MHz) δ 142.8 (s), 128.2 (d), 127.8 (d), 126.5 (d), 79.7 (d), 54.8 (d), 50.6 (d), 34.6 (t), 33.3 (t), 25.7 (t), 24.9 (t), 13.6 (q), 13.4 (q); $^1$H NMR (300 MHz) δ 7.31–7.16 (m, 5 H), 4.33–4.26 (m, 1 H), 2.75–2.67 (m, 1 H), 2.49–2.31 (m, 2 H), 1.98–1.87 (m, 2 H), 1.79–1.74 (m, 1 H), 1.62–1.37 (m, 4 H), 0.96 (d, *J* = 2.4 Hz, 3 H), 0.94 (d, *J* = 2.4 Hz, 3 H); IR 3020, 2930, 2850, 1440, 1110 cm$^{-1}$; MS-CI *m/z* 267 (M + H), 159, 109; HRMS-CI *m/z* calcd for $C_{15}H_{23}O_2S$ (M + H) 267.1419, found 267.1423.

**(*S*)-Methyl *p*-Tolyl Sulfoxide.** To a solution of 400 mg (1.68 mmol) of (+)-(1*S*,2*R*)-*trans*-2-phenylcyclohexyl (*S*)-methanesulfinate in 5 mL ether at 0 °C was added a solution of *p*-tolylmagnesium bromide, prepared from 0.41 mL (3.36 mmol) of 4-bromotoluene and 200 mg (8.40 mmol) of magnesium turnings, over 10 min. The reaction mixture was stirred for 1 h at rt after completed addition. The mixture was quenched with saturated $NH_4Cl$ solution, extracted with ether twice, washed with saturated NaCl solution, and then dried over $MgSO_4$. The crude sulfoxide was purified by silica gel chromatography with 7:3 Skelly B/EtOAc, affording 200 mg (76%) of a white solid, mp 73.5–74 °C (lit.[8] for *R* enantiomer, mp 73–74.5 °C): $[\alpha]^{25}_D$ −143° (*c* 2, acetone) [lit.[8] for *R* enantiomer, $[\alpha]^{20}_D$ +145.5° (acetone)]; $^{13}$C NMR (75 MHz) δ 142.6 (s), 141.5 (s), 130.0 (d), 123.5 (d), 44.0 (q), 21.4 (q); $^1$H NMR (300 MHz) δ 7.54 (d, *J* = 8.1 Hz, 2 H), 7.32 (d, *J* = 8.1 Hz, 2 H), 2.70 (s, 3 H), 2.41 (s, 3 H).

**(*S*)-*p*-Phenoxyphenyl *p*-Tolyl Sulfoxide.** To a solution of 150 mg (48 mmol) of (+)-(1*S*,2*R*)-*trans*-2-phenylcyclohexyl (*S*)-*p*-toluenesulfinate in 5 mL ether at 0 °C was added over 10 min a solution of (*p*-phenoxyphenyl)magnesium bromide [prepared from 0.17 mL (96 mmol) of 4-bromophenyl phenyl ether and 62 mg (240 mmol) of magnesium turnings]. The reaction mixture was stirred for 1 h at rt and then quenched by the addition of a saturated $NH_4Cl$ solution. The mixture was extracted with ether twice, and the combined organic layers were washed with saturated NaCl solution and then dried over $MgSO_4$. The crude sulfoxide was purified by silica gel chromatography with 7:3 Skelly B/EtOAc, affording 103 mg (70%) of a white solid: mp 116–116.5 °C; $[\alpha]^{25}_D$ −2.0° (*c* 2, acetone); $^{13}$C NMR (75 MHz) δ 160.1 (s), 155.6 (s), 142.4 (s), 141.4 (s), 139.3 (s), 129.9 (d), 126.9 (d), 124.7 (d), 124.4 (d), 119.8 (d), 118.5 (d), 21.3 (q); $^1$H NMR (300 MHz) δ 7.56 (d, *J* = 8.7 Hz, 2 H), 7.52 (d, *J* = 8.1 Hz, 2 H), 7.35–7.30 (m, 2 H), 7.41 (d, *J* = 8.7 Hz, 2 H), 7.16–7.11 (m, 2 H), 7.00 (d, *J* = 8.7 Hz, 2 H), 6.98 (d, *J* = 8.1 Hz, 2 H); IR 3020, 1575, 1480, 1220, 1040 cm$^{-1}$; MS-EI *m/z* 308 (M), 260, 201, 185, 91, 77; HRMS-EI *m/z* calcd for $C_{19}H_{16}O_2S$ 308.0871, found 308.0865.

**Acknowledgment.** Financial support of this research by the Robert A. Welch Foundation (F-626) and the National Institutes of Health (GM-31750) and a generous gift of (1*S*,2*R*)-2-phenylcyclohexanol from David L. Coffen of Hoffmann-La Roche, Nutley, NJ, are gratefully acknowledged.

(11) Douglass, I. B.; Norton, R. V. *Organic Syntheses*; Wiley: New York, 1973; Collect. Vol. V, p 708.

## [1,3]-Hydrogen Sigmatropic Rearrangements in Alkyl-Substituted Allenes

Daniel J. Pasto* and John E. Brophy

*Department of Chemistry and Biochemistry, University of Notre Dame, Notre Dame, Indiana 46556*

*Received December 27, 1990*

The suprafacial [1,3]-hydrogen sigmatropic rearrangement in alkenes is forbidden by orbital symmetry. The antarafacial [1,3]-hydrogen sigmatropic rearrangement in

(1) Woodward, R. B.; Hoffmann, R. *Angew. Chem., Int. Ed. Engl.* **1969**, *8*, 781.

0022-3263/91/1956-4554$02.50/0 

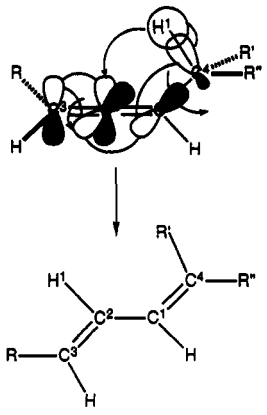
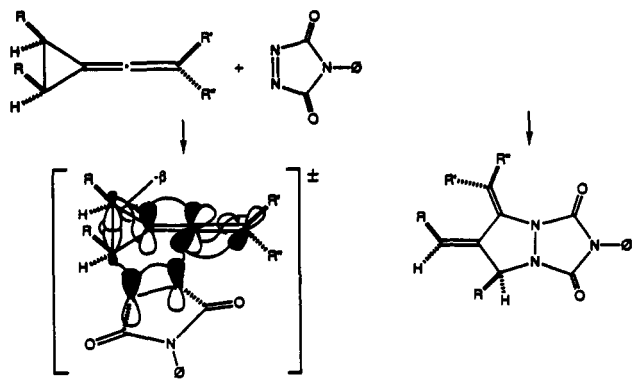


**Figure 1.** Diagram showing the interacting orbitals in the rearrangement of TBMA and the directions of rotation of the terminal carbon atoms leading to the formation of *trans*-TBB. The arrows indicate the direction of rotation about the $C^1$–$C^4$ and $C^2$–$C^3$ bonds, while the curved lines connecting the orbital lobes indicate the interacting orbital lobes in the transition state for the rearrangement in an allowed process.

alkenes is allowed by orbital symmetry; however, this type of rearrangement is geometrically impossible due to the inability to maintain overlap of the 1s AO of the migrating hydrogen atom with the necessary lobes of the terminal 2p AO's at the midpoint of the migration. The same holds true for such sigmatropic rearrangements in alkyl-substituted allenes. However, the unique geometry and symmetry of the orbitals in an alkyl-substituted allene provide for a uniquely different concerted pathway in which to accomplish a net concerted [1,3]-hydrogen sigmatropic rearrangement. This is illustrated in the orbital diagram shown in Figure 1. The orbitals of the $C^4$–$H^1$ bond lie in the plane of the $C^2$–$C^3$ $\pi$ bond, a favorable arrangement for the migration of $H^1$ to $C^2$. For such a migration to take place, however, 90° rotations about the $C^1$–$C^4$ and $C^2$–$C^3$ bonds must take place as indicated by the arrows about the bonds in the figure in order to form the two $\pi$ bonds of the product 1,3-butadiene. At first glance, processes of this type involving one, or more, 90° rotations about a bond for ultimate $\pi$-bond formation would appear to be very unlikely to occur.

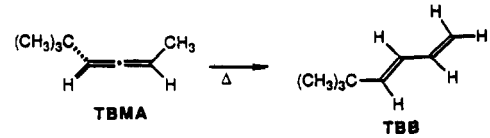
Our first discovery of a concerted process involving two simultaneous 90° rotations of groups was in the cycloaddition reactions of 4-phenyl-1,2,4-triazoline-3,5-dione with alkenylidenecyclopropanes, the detailed stereochemical and kinetic studies of which indicated the orbital interactions shown in the transition state for the reaction and the overall stereochemical consequences as illustrated in the following equation.[2]



The results of a subsequent PMO calculational study on the possible modes of (2 + 2) cycloaddition reactions of an allene with an alkene indicated that the six-electron [2 + (2 + 2)] cycloaddition process,[3] an allowed process according to the Hückel–Möbius concept,[4] which involves both $\pi$ bonds of the allene and a 90° rotation of the terminus of the allene, should be more favorable than the symmetry-allowed ($_\pi2_s$ + $_\pi2_a$) process.

In our studies on the (2 + 2) cycloaddition reactions of 1,1- and 1,3-dimethylallene we have discovered competitive [1,3]-hydrogen sigmatropic rearrangement reactions that we believe are concerted processes involving the orbital interactions shown in Figure 1, which are also allowed according to the Hückel–Möbius concept.[4] (The linked rotations indicated in Figure 1 result in some very interesting stereochemical consequences; however, the study of the stereochemistry of this process would require a substituted allene containing an asymmetric allene center and an asymmetric center at $C^4$!)

In an earlier study of the (2 + 2) cycloaddition reactions of 1,1-dimethylallene (11DMA) with various radiocophiles, it was observed that in the reactions with the much less reactive diethyl fumarate and maleate, cycloadducts of isoprene were formed.[5] It was proposed that the 11DMA undergoes a [1,3]-hydrogen sigmatropic rearrangement to form isoprene, which was then trapped by the diethyl fumarate or maleate.[5] Subsequent attempts to study the rearrangement of 11DMA were foiled by the greater tendency of the 11DMA to undergo cyclodimerization and oligomerization. Similarly, the attempted cycloaddition of 1,3-dimethylallene (13DMA) with diethyl or dimethyl fumarate in toluene solution results in extensive rearrangement of the 13DMA to 1,3-pentadiene, which is trapped by the fumarate.[6] In our current studies of the (2 + 2) cycloaddition reactions of 1-*tert*-butyl-3-methylallene (TBMA), a similar rearrangement has been observed in which the TBMA undergoes rearrangement to 1-*tert*-butyl-1,3-butadiene (TBB), which is then trapped by the dienophile (radiocophile) that is present.[7] The much lesser tendency of TBMA to undergo (2 + 2) cycloaddition[8] and cyclodimerization compared to other alkyl-substituted allenes has allowed us to study this interesting rearrangement process.



TBMA TBB

Dilute solutions of TBMA in bromobenzene-$d_5$ in sealed NMR tubes were suspended in the vapors of refluxing solvents at appropriate temperatures. The NMR spectra of the solutions were recorded periodically, and the ratios of TBMA to TBB were determined by integration of the NMR spectra. The NMR spectra taken during the course of the rearrangement reactions showed only the presence of TBMA and TBB. The trans isomer of TBB is the only isomer formed, the stereochemistry being assigned by the relatively large value of the coupling constant between the protons attached to $C_1$ and $C_2$ (15.41 Hz). There was no evidence for the formation of any other products.

Very good first-order kinetic plots of the data were obtained (see Figure 2). (The data obtained at 150° and

(2) Pasto, D. J.; Borchardt, J. K. *J. Am. Chem. Soc.* **1974**, *96*, 6944.

(3) Pasto, D. J. *J. Am. Chem. Soc.* **1979**, *101*, 37.
(4) Zimmerman, H. *Acc. Chem. Res.* **1971**, *4*, 272.
(5) Pasto, D. J.; Yang, S.-H. *J. Am. Chem. Soc.* **1984**, *106*, 152.
(6) Pasto, D. J.; Sugi, K. D. *J. Org. Chem.*, in press.
(7) Pasto, D. J.; Brophy, J. Unpublished observations.
(8) Pasto, D. J.; Warren, S. E. *J. Am. Chem. Soc.* **1982**, *104*, 3670.

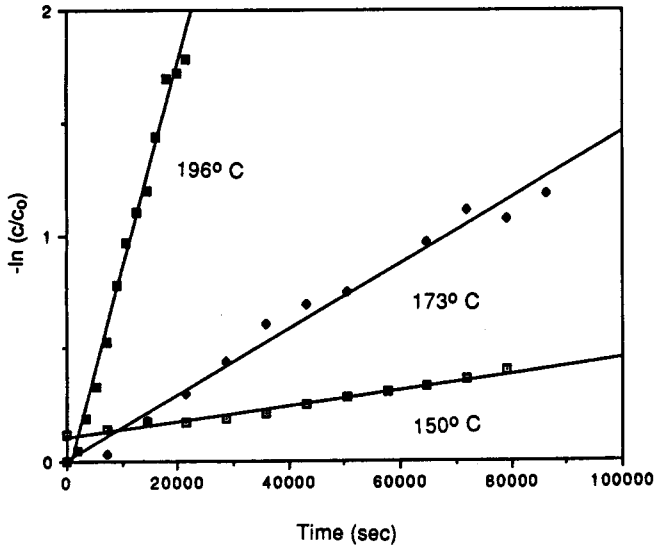



**Figure 2.** First-order kinetic plots for the rearrangement of TBMA to TBB at 150, 173, and 196 °C.

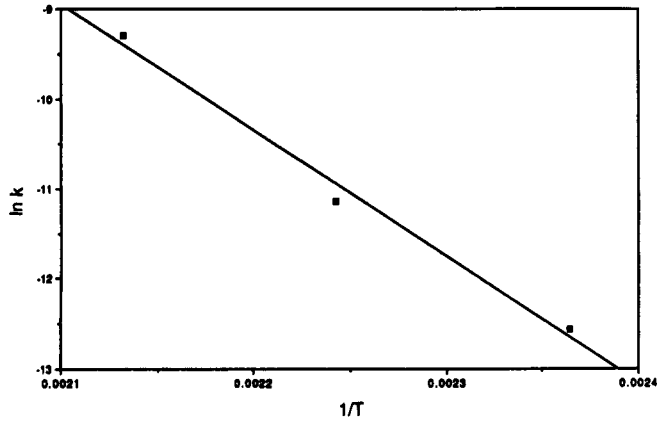



**Figure 3.** Plot of ln $k$ versus $1/T$ for the rearrangement of TBMA to TBB.

shown in Figure 2 are for data obtained at longer reaction times, which enabled more reliable integration of the NMR spectra and calculation of the TBMA to TBB ratios.) The plot of ln $k$ versus $1/T$ is shown in Figure 3, which gives values for the activation parameters for this reaction of $\Delta H^{\ddagger} = 27.9 \pm 1.0$ kcal mol$^{-1}$ and $\Delta S^{\ddagger} = +1.4 \pm 1.5$ eu. The linearity of this plot suggests that this reaction is a true unimolecular process and is not "catalyzed" by some adventitious acid or base that might be present in the solutions. The apparent driving force for this reaction is the loss of the very high positive heat of formation of the allenic carbon atom of the TBMA (+34.2 kcal mol$^{-1}$), the calculated change in the heat of formation, $\Delta H_f$, and entropy, $\Delta S_f$, for the reaction at 298 °C being −18.3 kcal mol$^{-1}$ and +16.4 eu ($\Delta G_{298}$ −23.2 kcal mol$^{-1}$),[9] both being highly favorable for the reaction. The rather high $\Delta H^{\ddagger}$ for the rearrangement compared to the rather exothermic nature of the process reflects the poor overlap between the 2p AO's on the terminal carbon atoms of the ultimate 1,3-diene, which are undergoing rotation in the transition state for the rearrangement.

## Experimental Section

The preparation of 1-*tert*-butyl-3-methylallene (TBMA) was carried out by the procedure of Elsevier and Vermeer.[10] 1-*tert*-Butyl-1,3-butadiene (TBB) has been previously prepared and characterized by Corey and Cane.[11]

**Thermal Rearrangement of TBMA.** Solutions of 10 mg (0.09 mmol) of TBMA in 0.5 mL of bromobenzene-$d_5$ in NMR tubes were triply freeze–degassed, and the NMR tubes were sealed under reduced pressure. The NMR tubes were suspended in the refluxing vapors of nonane (150 °C), decane (173 °C), and undecane (196 °C). The NMR tubes were periodically removed from the vapors of the refluxing solvents, and the 200-MHz $^1$H NMR spectra of the solutions were recorded and integrated. Resonances corresponding to only TBMA and the product TBB were observed in the NMR spectra. The relative ratios of the TBMA and TBB were converted to relative concentrations, resulting in the first-order kinetic plots shown in Figure 2 ($k_{150}$ $3.49 \times 10^{-6}$ s$^{-1}$, $k_{173}$ $1.45 \times 10^{-5}$ s$^{-1}$, $k_{196}$ $9.15 \times 10^{-5}$ s$^{-1}$). 300-MHz $^1$H NMR spectrum of TBMA: ($CDCl_3$) $\delta$ 1.01 (s, 9 H), 1.65 (dd, $J$ = 6.82, 3.37 Hz, 3 H), 5.04 (dq, $J$ = 6.73, 3.37 Hz, 1 H), 5.09 (dq, $J$ = 6.73, 6.82 Hz, 1 H). 300-MHz $^1$H NMR spectrum of TBB: $\delta$ 0.96 (s, 9 H), 4.95 (dddd, $J$ = 10.11, 1.79, 0.76, 0.55 Hz, 1 H), 5.10 (dddd, $J$ = 16.92, 1.79, 1.03, 0.67 Hz, 1 H), 5.65 (dddd, $J$ = 15.41, 0.76, 0.68, 0.67 Hz, 1 H), 5.99 (dddd, $J$ = 15.41, 10.13, 1.03, 0.55 Hz, 1 H), 6.29 (dddd, $J$ = 16.92, 10.13, 10.11, 0.68 Hz, 1 H).

**Acknowledgment.** This research was supported by a grant from the National Science Foundation (CHE-8709725).

(9) Calculated by the method of Benson: Benson, S. W. *Thermochemical Kinetics*, 2nd ed.; John Wiley & Sons: New York, 1976).

(10) Elsevier, C. J.; Vermeer, P. *J. Org. Chem.* **1989**, *54*, 3726.
(11) Corey, E. J.; Cane, D. E. *J. Org. Chem.* **1969**, *34*, 3053.

# *gem*-Difluorination versus 1,2-Migration and Fragmentation in the Reaction of 2- and 3-Uloses with DAST. Influence of Stereochemistry at the Anomeric Carbon Atom

Anas El-Laghdach, Raouf Echarri, M. Isabel Matheu, M. Isabel Barrena, and Sergio Castillón*

*Departament de Quimica, Facultat de Ciencies Quimiques de Tarragona, Universitat de Barcelona, Psa. Imperial Tarraco 1, 43005 Tarragona, Spain*

Jordi Garcia

*Departament de Quimica Orgânica, Facultat de Quimica, Universitat de Barcelona, Martí i Franquès 1, 08028 Barcelona, Spain*

*Received October 1, 1990 (Revised Manuscript Received March 18, 1991)*

Introduction of fluorine into biologically active organic compounds is one of the most simple structural modifications in order to increase their activity.[1] Recently, many reagents[2] have been developed for the synthesis of mono- and *gem*-difluorinated compounds, looking for higher yields and selectivities. The difluoromethylene group has a steric profile similar to that of methylene and is frequently prepared from a carbonyl group; among the reagents commonly utilized for this transformation—$SF_4$,[3] $SeF_4$,[4] $MoF_6$,[5] $PhSF_3$,[6] and (diethylamino)sulfur trifluoride

(1) (a) Schlosser, M. *Tetrahedron* **1978**, *34*, 3. (b) *Fluorinated Carbohydrates, Chemical and Biochemical Aspects*; Taylor, N. F., Ed.; American Chemical Society: Washington DC, 1988.
(2) Gerstenberger, M. R. C.; Haas, A. *Angew. Chem., Int. Ed. Engl.* **1981**, *20*, 647.
(3) (a) Boswell, G. A.; Ripko, W. C.; Scribner, R. M.; Tullock, C. W. *Org. React. (N.Y.)* **1974**, *21*, 20–30. (b) Wang, C.-L. *J. Org. React. (N.Y.)* **1985**, *34*, 319.
(4) Kent, P. W.; Wood, K. R. British Patent 1968, 1 136075.
(5) Mathey, F.; Bensoam, J. *Tetrahedron* **1971**, *27*, 3965.
(6) Sheppard, W. A. *J. Am. Chem. Soc.* **1962**, *84*, 3058.

0022-3263/91/1956-4556$02.50/0 