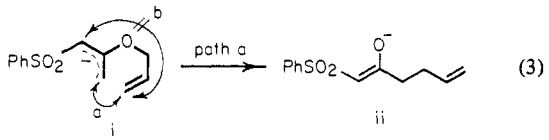
the observed rearrangements.[13,14] It is noteworthy that none of the regioisomeric rearrangement product (arrow b, eq 3) was



(3)

observed. This probably derives from the fact that the primary product of rearrangement, path a, is a β-keto sulfone anion, ii, while the product of path b would be a simple ketone enolate.[15,16] The direct formation of stabilized enolates of the type ii has obvious implications for organic synthesis.

We have examined the effects of methyl substitution and double-bond position in the allyl vinyl ether system on the regioselectivity and rate of the rearrangement. Those results are shown in Table I.[17] Experiments 2 and 4 demonstrate that the high regioselectivity of the anionic rearrangements was in *no way affected by the position of double bond in the vinyl ether portion.*[18] This lends further support to the intervention of allylic anions as the rearranging species. We have observed an interesting dependence of the reaction rate on the position of methyl substitution. Substrates **1b** and **1c** (experiments 3 and 5), derived from crotyl and methallyl alcohol, rearranged more slowly than **1a**, with the effect being greater for **1c**, which required heating to 62 °C for complete reaction. The yields were not seriously affected, and the regioselectivity was still very high. The yields of β-keto sulfones **2b** (experiments 3 and 4) have been corrected for minor amounts of phenylsulfonyl acetone (actual yields in parentheses), which were formed by a competing reaction[19] from **1b** (**1b′**). Contrariwise, **1d′** (derived from 3-buten-2-ol) rearranged much faster and in higher yield than any of the previous substrates. While the total yield was very good, we did isolate 3% of the regioisomeric β-keto sulfone **2d′**.[20] The potassium salt of **1d′** rearranges even at *room temperature* in comparable yield. HPLC analysis of the reaction product again showed ca. 8% of **2d′**. While still only a minor component, a regioisomer formed exclusively from **1d′** is intriguing. Experiments are in progress to determine the importance of steric effects in this side reaction. Substitution on the *vinyl unit* is apparently of no consequence in controlling the regioselectivity but has a remarkable accelerating effect on the rate of the rearrangement. The potassium salt of **1e** spontaneously rearranged at room temperature in excellent yield (experiment 8). Finally, the only structural limitation we have found thus far is the incompatibility of phenyl substituents as shown in experiment 9. Substrate **1f** produced dihydrofuran **3**, which is not derived from a rearrangement but rather is an addition to the styryl double bond followed by a series of proton shifts.

The effects of methyl substitution on the rates of these reactions are qualitatively similar to those effects observed by Ireland[3h,i] in the ester enolate Claisen rearrangement. Further, if one considers methyl groups to be π donors, then there is again qualitative agreement with Carpenter's predictions.[2f]

In summary, we have documented that first examples of a carbanion-accelerated Claisen rearrangement[21] that occurs under mild conditions (50 °C or less) in good yield (69–91%) and with excellent regioselectivity to produce β-keto sulfones. We are currently investigating (1) other carbanion stabilizing substituents (phenylsulfinyl, phenylsulfenyl), (2) relative and absolute stereochemical control, and (3) general methods for the preparation of precursors.

Acknowledgment. We thank the Research Corp. for financial support. This work was in part supported by the University of Illinois NSF Regional Instrumentation Facility (NSF CHE 79-16100) and the University of Illinois Mass Spectrometry Laboratory (NIH PHS HHS GM 27029). M.A.H. thanks the Campbell Soup Co., the University of Illinois, and the Eastman Kodak Company for fellowships.

Registry No. **1a**·K, 82352-25-8; **1a′**, 82352-26-9; **1b**·K, 82352-27-0; **1b′**, 82352-28-1; **1c**·K, 82352-29-2; **1d′**·K, 82352-30-5; **1e**·K, 82352-31-6; **1f**, 82352-32-7; **2a**, 80945-31-9; **2b**, 82352-33-8; **2c**, 82352-34-9; **2d**, 82352-35-0; **2e**, 82352-36-1; **3**, 82352-37-2; ii, 82352-38-3; $PhSO_2CH{=}C{=}CH_2$, 2525-42-0; $PhSO_2CH_2C{\equiv}CH$, 2525-40-8; *p*-Tol-$SO_2CH{=}C{=}C(CH_3)_2$, 82352-39-4; 2-propen-1-ol sodium salt, 20907-32-8; phenylsulfonylacetone, 5000-44-2; crotyl alcohol, 6117-91-5; methyallyl alcohol, 513-42-8; 3-buten-2-ol, 598-32-3.

(13) The potassium salt of **1a** is soluble in HMPA, and the reaction is homogeneous throughout, excluding surface-catalyzed processes.

(14) Accurate kinetic measurements are in progress to determine the factor of acceleration.

(15) Based on pK_a values *alone* there is a ca. 20 kcal/mol advantage (at 50 °C) for the formation of β-keto sulfone anions compared to ketone enolates. Compare, e.g., pk_a's: acetone[15a] (26.5) vs. phenylsulfonyl acetone[15b] (12.5). (a) Matthews, W. D.; Bartmess, J. E.; Bordwell, F. G.; Cornforth, F. J.; Drucker, G. E.; Margolin, Z.; McCallum, R. J.; McCollum, G. J.; Vanier, N. R. *J. Am. Chem. Soc.* **1975**, *97*, 7006. (b) Bordwell, F. G.; Van der Puy, M.; Vanier, N. R. *J. Org. Chem.* **1976**, *41*, 1883.

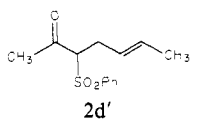
(16) This is, obviously, a product-side, ground-state argument. It remains to be seen if frontier molecular orbital coefficients in the HOMO of the (phenylsulfonyl)allyl anion would predict the same result.

(17) The starting sulfones are new compounds[10] and were prepared by one of the following routes: (a) **1** (a–c, f), alumina-catalyzed addition of the appropriate allylic alcohol to $PhSO_2CH{=}C{=}CH_2$; (b) **1** (a, b, d), addition of the sodium or potassium salt of the appropriate allylic alcohol to $PhSO_2CH_2C{\equiv}CH$; (c) **1e**, same as method b but with *p*-Tol-$SO_2CH{=}C{=}C(CH_3)_2$.

(18) This is in contrast to the behavior of the related α,β- and β,γ-unsaturated sulfoxides. Cookson, R. C.; Gopalan, R. *J. Chem. Soc., Chem. Commun.* **1978**, 608.

(19) The origin of the phenylsulfonyl acetone is still unclear. Control experiments showed that it was not arising in the workup.

(20) The structure of **2d′** is supported by its ^1H NMR (220 MHz) spectrum.



2d′

(21) For other examples of anionically accelerated Claisen rearrangements see (a) Frihard, C. R.; Leonard, N. J. *J. Am. Chem. Soc.* **1973**, *95*, 7174; **1974**, *96*, 5874. (b) Schultz, A. G.; Napier, J. *J. Chem. Soc., Chem. Commun.* **1981**, 224.

Origin of Huisgen's Factor "*x*": Staggering of Allylic Bonds Promotes Anomalously Rapid Exo Attack on Norbornenes

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Norbornene and many derivatives are attacked by a variety of reagents preferentially from the exo face of the double bond.[2-5] Preferred exo attack has been attributed variously to steric effects,[2] torsional effects,[6] or to "nonequivalent orbital extension".[7] The

(1) Present addresses: (a) New South Wales Institute of Technology, Sydney, Australia; (b) University of Catania, Catania, Italy.

(2) Brown, H. C.; Hammar, W. J.; Kawakami, J. H.; Rothberg, I.; Van der Jugt, D. L. *J. Am. Chem. Soc.* **1967**, *89*, 6381. Brown, H. C.; Kawakami, J. H.; Liu, K.-T. *Ibid.* **1973**, *95*, 2209.

(3) Electrophiles and cycloaddends: Freeman, F. *Chem. Rev.* **1975**, *75*, 439. Allen, A. D.; Tidwell, T. T. *J. Am. Chem. Soc.* **1982**, *104*. 3145.

(4) Nucleophiles: Richey, H. G., Jr.; et al. *J. Org. Chem.* **1980**, *45*, 5027, 5042. See also: Corey, E. J.; Shibasaki, M.; Nicolau, K. C.; Malmstein, C. L.; Samuelson, B. *Tetrahedron Lett.* **1976**, 737. Baldwin, S. W.; Tomesch, J. C. *J. Org. Chem.* **1974**, *39*, 2382.

(5) Radicals: Davies, D. I.; Parrot, M. J. *Tetrahedron Lett.* **1972**, *2719* references therein.

(6) Schleyer, P. v. R. *J. Am. Chem. Soc.* **1967**, *89*, 701.

(7) Inagaki, S.; Fujimoto, H.; Fukui, K. *J. Am. Chem. Soc.* **1976**, *98*, 4054.

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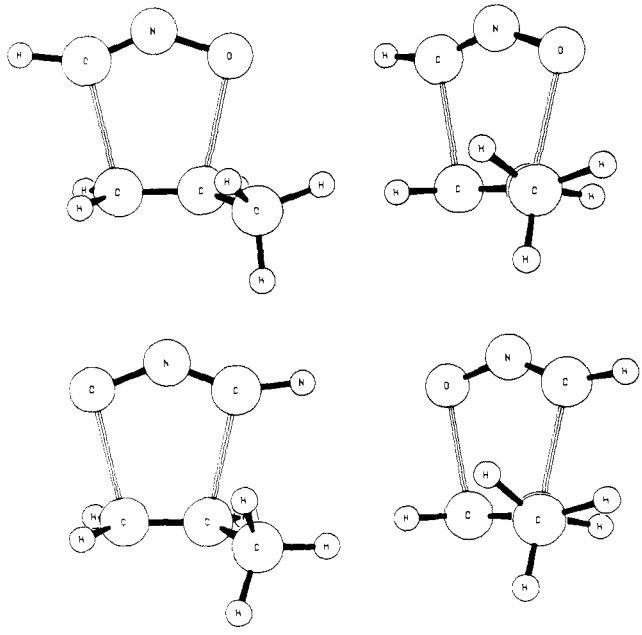

Figure 1. Preferred methyl rotations in model transition structures for cycloaddition of fulminic acid to propene to form 5-methyl- and 4-methylisoxazolines.[15] The drawings on the right are Newman projections looking down the CC bond from the methyl group.

phenomenon is also inextricably entwined with the classic nonclassical norbornyl cation problem:[8] the acceleration of exo attack on norbornene might arise from the hyperconjugative interaction of the strained 1–6 bond with the π bond. Huisgen recently found that cycloadditions to the exo face of norbornene are unusually rapid.[9] By correcting for the acceleration expected to result from the release of strain upon cycloaddition, Huisgen estimated that norbornene is 5–200 times more reactive in room-temperature cycloadditions than can be accounted for on the basis of strain release effects; thus, the activation energies for cycloadditions to norbornene are anomalously low by 1–3 kcal/mol.[9] Huisgen attributed this 1–3 kcal/mol to factor "*x*".[9] We report a consistent explanation of both reactivity and stereoselectivity phenomena observed in cycloadditions to norbornenes and an experimental verification of this theory.

Model calculations indicate that there is a significant tendency for the allylic bonds of acyclic alkenes to adopt a staggered arrangement with respect to partially formed bonds in the transition states of addition to alkenes.[10] This "staggering effect" has been confirmed by calculations on the transition structures for hydroboration, hydride attack, and hydrogen atom attack on propene.[11] In each transition structure, the methyl group rotates so as to be staggered both with respect to the partially formed bond to C_2 of propene and to the bonds to the partially pyramidalized carbon undergoing attack. We have also optimized the methyl group rotations in model transition states for fulminic acid cycloadditions to propene in both orientations.[12] These are shown in Figure 1. Rotation of the methyl group by 180°, to form an "eclipsed" structure, causes 2.5 and 4.6 kcal/mol increases in energy for the top and bottom structures shown in the figure, respectively. Although the preferred direction of attack of electrophiles, radicals, nucleophiles, and concerted cycloaddends differs,[11] the preference for "staggered" over "eclipsed" transition structures is always 2–3 kcal/mol.

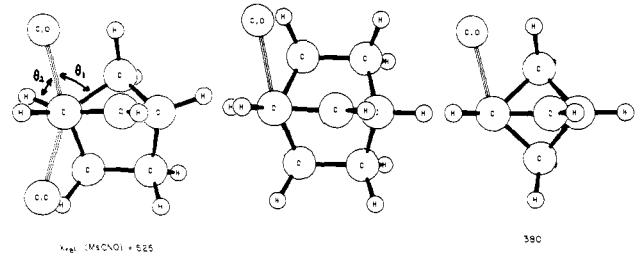

Figure 2. Directions of bond formation (striped bond) for attack of C or O of fulminic acid on norbornene, bicyclooctene, and bicyclohexene. The relative rates of attack of mesitonitrile oxide[9] are shown below the figures.[15,16]

Table I. Dihedral Angles between Partially Formed HCNO–Alkene Bonds and Allylic Bonds

bicyclic hydrocarbon	dihedral angles[a] X···CCC[b]	X···CCH[c]
norbornene		
exo attack	69	54
endo attack	34	98
bicyclo[2.2.2]oct-2-ene	36	78
bicyclo[2.1.1]hex-2-ene	60	76
bicyclo[3.2.1]oct-6-ene		
exo attack	77	41
endo attack	18	110

[a] X is the C or O terminus of fulminic acid (see Figure 2). [b] C is the bridging carbon attached to the bridgehead carbon. [c] H is the allylic hydrogen attached to the bridgehead C.

In rigid polycyclic systems, the allylic bonds are fixed with respect to the forming bonds. When attack of the reagent occurs so that the partially formed bonds are staggered with respect to the allylic bonds, the activation energy of the reaction should be 2–5 kcal/mol lower per forming bond than when the partially formed bonds are eclipsed with respect to the allylic bonds. The situation is demonstrated in Figure 2 for concerted cycloadditions of fulminic acid to norbornene (exo and endo), bicyclo[2.2.2]-octene, and bicyclo[2.1.1]hexene.[13,14] The partially formed bonds in the transition states for the concerted cycloadditions of nitrile oxides to these bicyclic alkenes are represented here by the striped bonds to C,O. These bond directions are taken from the fulminic acid–propene transition structures shown in Figure 1. The dihedral angles between the partially formed bonds and the allylic CC and CH bonds are listed in Table I. Whereas exo attack on norbornene can occur with nearly ideal staggering of the partially formed bonds with respect to the CC and CH bonds to the bridgehead carbons (dihedral angles 69 and 54°), attack on the endo face of norbornene, on bicyclo[2.2.2]octene, or, to a slight extent, on bicyclo[2.1.1]hexene involves greater eclipsing with the bond to the bridging carbon. The alkene pyramidalization we have noted[15] also contributes slightly to the staggered arrangement in norbornene reactions. Figure 2 also shows the relative rate differences reported by Huisgen et al. for the concerted cycloaddition of mesitonitrile oxide to these molecules.[9] The trends in rate are nicely rationalized by the extent of staggering in the transition states.

(8) Brown, H. C. *Acc. Chem. Res.* **1973**, *6*, 377. See ref 6. Schleyer, P. v. R. *J. Am. Chem. Soc.* **1964**, *86*, 1854, 1856; **1967**, *89*, 699 for discussion of torsional effects on solvolysis.

(9) Huisgen, R.; Ooms, P. H. J.; Mingin, M.; Allinger, N. L. *J. Am. Chem. Soc.* **1980**, *102*, 3951. Huisgen, R. *Pure Appl. Chem.* **1981**, *53*, 171.

(10) Caramella, P.; Rondan, N. G.; Paddon-Row, M. N.; Houk, K. N. *J. Am. Chem. Soc.* **1981**, *103*, 2438. A similar effect was deduced empirically by Felkin (Cherest, M.; Felkin, H.; Prudent, N. *Tetrahedron Lett.* **1968**, 2199, 2205) and supported computationally by Anh (Anh, N. T.; Eisenstein, O. *Nouv. J. Chem.* **1977**, *1*, 161) for nucleophilic additions to carbonyl compounds.

(11) Paddon-Row, M. N.; Rondan, N. G.; Houk, K. N. *J. Am. Chem. Soc.*, in press.

(12) The transition structures were based on the published fulminic acid–acetylene transition state: Goddard, J.; Komornicki, A.; Schaefer, H. F., III *J. Am. Chem. Soc.* **1980**, *102*, 1763. The methyl groups were substituted for hydrogen and were optimized by using the STO-3G basis set.

(13) Geometries of the bicyclic hydrocarbons were obtained from the optimizations of these structures by using Allinger's MM2 program: Allinger, N. L. *J. Am. Chem. Soc.* **1977**, *99*, 8127.

(14) The figure was produced on the PROPHET Computer System, a national resource sponsored by the Division of Research Resources of the National Institutes of Health.

(15) Rondan, N. G.; Paddon-Row, M. N.; Caramella, P.; Houk, K. N., *J. Am. Chem. Soc.* 1981, *103*, 2436.

In 1967, Schleyer called attention to the consequences of the torsional arrangement about C_1C_2 bonds in norbornyl systems and concluded that "torsional effects thus favor exo over endo attack, and, by microscopic reversibility, exo over endo departure".[6] Although Schleyer focused attention on the relief of torsional strain involving the bridgehead CH bond, the general role of the transition-state torsional interactions was clearly noted.[16]

Does *anti*-periplanar interaction (hyperconjugation) of the strained C_1C_6 and C_4C_5 bonds with the π bond of norbornene also accelerate exo cycloaddition? An experimental test was designed as follows. Table I shows the dihedral angles for attack on bicyclo[3.2.1]oct-6-ene. The staggered arrangement of the allylic bonds in this molecule should make exo attack as rapid as that on norbornene. Tempering the staggering effect is the strain relief, which is 3.5 kcal/mol greater for norbornene than that for the [3.2.1] system. If *anti*-periplanar hyperconjugation also contributes to factor "*x*", then bicyclo[3.2.1]oct-6-ene should be much less reactive than norbornene. For example, the rate of acetolysis of *exo*-bicyclo[3.2.1]oct-6-yl tosylate is approximately the same as that of cyclohexyl tosylate,[17] which is, in turn, 477 times less reactive than *exo*-2-norbornyl tosylate.[18,19] These effects are geometrically interrelated, since the staggered arrangement of adjacent bonds automatically places one bond gauche to two vicinal bonds (minimizing closed-shell destabilization) and anti to a third (maximizing donor–acceptor stabilization), and both of these effects can contribute to torsional effects in substituted ethanes.[20] Nevertheless, a clear experimental distinction between staggering and hyperconjugation is possible in this case.

We have measured relative reactivities of bicyclo[3.2.1]oct-6-ene and norbornene toward mesitonitrile oxide[21] under competitive conditions[22] at 25 °C in CCl_4 solution. Norbornene is only 1.3 ± 0.2 times more reactive than bicyclo[3.2.1]oct-6-ene. Even if only 27% of the added strain relief in norbornene is felt in the cycloaddition transition state (Huisgen's most conservative estimate),[9] the activation energy of the norbornene reaction should be 1 kcal/mol lower than that for bicyclo[3.2.1]oct-6-ene. The difference is actually only 0.2 kcal/mol, so that factor "*x*" lowers the activation energy of cycloaddition to bicyclo[3.2.1]oct-6-ene by 0.8 kcal/mol more than it lowers the norbornene activation energy!

It might appear that staggering effects should allow monocyclic and acyclic systems to react as readily as norbornene in cycloadditions. However, the staggered conformations which are preferred in transition structures are different from the preferred conformations of acyclic and monocyclic alkenes. In order to achieve the preferred transition structure conformation, acyclic and monocyclic alkenes must distort in ways that introduce unfavorable interactions within the alkene moiety itself (e.g., boat for cyclohexene and a conformation with internal H–H repulsions for *cis*-3-hexene, both of which are not the preferred eclipsed lowest energy conformations). Norbornene must go through none of these gyrations. Also, such molecules are less strained and less electron rich than norbornene, and these factors contribute to reactivity with electrophilic species as well. Factor "*x*" arises from enforced staggering of allylic bonds in norbornene, not from "nonequivalent orbital extension" or hyperconjugative interactions. Indeed, there is no significant sp mixing even in highly pyramidalized alkenes, so that the staggering effects described here are the only remaining candidate for factor "x".

Acknowledgment. We are grateful to Professors Rolf Huisgen and Paul von Rague Schleyer for enlightening discussions, to the National Science Foundation for financial support of this research, and to the Swiss National Science Foundation for a fellowship to J.M.

Registry No. Norbornene, 498-66-8; bicyclo[2.2.2]oct-2-ene, 931-64-6; bicyclo[2.1.1]hex-2-ene, 822-41-3; bicyclo[3.2.1]oct-6-ene, 6491-96-9; mesitonitrile oxide, 2904-57-6; fulminic acid, 506-85-4.

(16) Our hypothesis differs in detail from that of Schleyer, who noted that the relief of torsional strain could accelerate solvolysis reactions (Schleyer, P. v. R. *J. Am. Chem. Soc.* **1964**, *86*, 1854). By contrast, we suggest that torsional interactions are as significant in transition states as they are in molecular ground states so that in attack on an sp^2 hybridized carbon in an alkene, carbonyl, or carbocation, staggered attack is favored over eclipsed. By microscopic reversibility, the formation of an sp^2 center should occur more rapidly from a staggered precursor than from an eclipsed precursor.

(17) Appleton, R. A.; Fairlie, J. C.; McCrindle, R.; Parker, W. *J. Chem. Soc. C* **1968**, 1716.

(18) Schleyer, P. v. R.; Donaldson, M. M.; Watts, W. E. *J. Am. Chem. Soc.* **1965**, *87*, 375.

(19) Hyperconjugation and bridging are undoubtedly significant in such solvolyses and in electrophilic additions to norbornenes.

(20) Brunck, T. K.;; Weinhold, F. *J. Am. Chem. Soc.* **1976**, *98*, 4392.

(21) Grundmann, C.; Richter, R. *J. Org. Chem.* **1968**, *33*, 476.

(22) Bast, K.; Christl. M.; Huisgen, R.; Mack, W. *Chem. Ber.* **1973**, *106*, 3312.

Highly Stereoselective Approaches to α- and β-*C*-Glycopyranosides

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Received May 14, 1982

Tetrahydropyrans derived from pyranosides via substitution at C1, i.e., *C*-glycopyranosides, occur as subunits of a variety of natural products[1] and are of potential interest as chiral intermediates and enzyme inhibitors.[2] Although stereoselective routes exist for both α- and β-*C*-glycopyranosides,[3] they suffer from low yields, poor selectivity, or lack of generality. Recent requirements related to our interest in the marine natural product palytoxin[4] led us to seek a general expeditious route from simple starting materials.

It was hoped that stereochemical control could be realized by nucleophilic addition to the pyran oxonium ion derived from readily available tetrabenzylpyranose derivatives. This oxonium ion should preferentially accept nucleophiles from the α (axial) side due to the anomeric effect[5] from the ring oxygen (Figure 1).[6] By reductive process, i.e., Nu: = H^-, one could then obtain the opposite configuration at the anomeric center.[7] Herein is reported the successful realization of such an approach.

Thus, 2,3,4,6-tetrabenzylglucopyranose (**1**, Scheme I)[8] was reacted with allyltrimethylsilane[9] and boron trifluoride etherate in acetonitrile at ambient temperature for 3 h to yield a 10:1 mixture[10] of allylglucopyrans in 55% combined yield. Preparative thin-layer chromatographic separation allowed isolation of the α (axial) allylglucopyran **3**[11] and the β (equatorial) allylglucopyran

(1) For examples, see: McDonald, F. J.; Campbell, D. C.; Vanderah, D. J.; Schmitz, F. J.; Washecheck, D. M.; Burks, J. E.; van der Helm, D. *J. Org. Chem.* **1975**, *40*, 665. Connor, D. T.; Greenough, R. C.; von Strandtmann, M. *J. Org. Chem.* **1977**, *42*, 3664; **1978**, *43*, 5027.

(2) Shulman, M. L.; Shiyan, S. D.; Khorlin, A. Y. *Carbohydr. Res.* **1974**, *33*, 229. Cerretti, D. *Ibid.* **1981**, *94*, C10. Chmielewski, M.; BeMiller, J. N.; Cerretti, D. P. *Ibid.* **1981**, *97*, C1.

(3) Fraser-Reid, B.; Dawe, R. D.; Tulshian, D. B. *Can. J. Chem.* **1979**, *57*, 1746. Ireland, R. E.; Thaisrivongs, S.; Vanier, N.; Wilcox, C. S. *J. Org. Chem.* **1980**, *45*, 48. Hanessian, S.; Liak, T. J.; Dixit, D. M. *Carbohydr. Res.* **1981**, *88*, C14. Pougny, J.-R.; Nassr, M. A. M.; Sinaÿ, P. *J. Chem. Soc., Chem. Commun.* **1981**, 375. Dawe, R. D.; Fraser-Reid, B. *Ibid.* **1981**, 1180 and references cited therein. See also ref 2 above.

(4) Uemura, D.; Ueda, K.; Hirata, Y.; Naoki, H.; Iwashita, T. *Tetrahedron Lett.* **1981**, 2781. Moore, R. E.; Bartolini, G. *J. Am. Chem. Soc.* **1981**, *103*, 2491.

(5) For an example: "Anomeric Effect: Origin and Consequences"; Szarek, W. A., Horton, D., Eds.; American Chemical Society: Washington, D.C., 1979; ACS Symp. Ser. No. 87.

(6) *C*-Nucleoside precursors have been made by a similar approach; however, this is not strictly orbital control solely by the ring oxygen. Ogawa, T.; Pernet, A. G.; Hanessian, S. *Tetrahedron Lett.* **1973**, 3543. Deoxygenated pyrans have also been made by a similar approach from oxonium ions derived from acetylated glycals. The additions are primarily from the axial direction, although the ratios are at best 4:1: Dawe, R. D.; Fraser-Reid, B. *J. Chem. Soc., Chem. Commun.* **1981**, 1180.

(7) Recently such an oxonium ion was observed to add hydride axially through intramolecular reaction. Deslongchamps, P.; Rowan, D. D.; Rothier, N. *Can. J. Chem.* **1981**, *59*, 2787.

(8) Purchased from Sigma Chemical Co.

(9) Purchased from Petrarch Systems Inc.

(10) The ratio of stereoisomers was determined by chromatographic separation of the products.

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