Table I. Conditions and Yields for the Preparation of Ketol Phosphates and Phosphoryloxy Lactones with **4a**[a]

reactant (mmol)	solvent (mL)	time (temp)	product (% yield)[c-e]
PhCOMe (10.4)	MeCN (45)	3 h, 20 min (reflux)	**5a** (59)
MeCOMe (ca 135; 10 mL)	MeCN (40)	30 min (reflux)	**5b** (81)
cyclopropyl methyl ketone (12.1)	MeCN (40)	2 h, 56 min (reflux)	**5c** (59)
cyclohexanone (10.5)	CH_2Cl_2 (35)	7 h, 35 min (room)	**5d** (62)
$CH_2(COPh)_2$ (5.0)	CH_2Cl_2 (40)	15 min (room)	**5e** (90)
CH_2 = $CH(CH_2)_2CO_2H$ (7.8)	CH_2Cl_2 (40)	2 h, 40 min (room)	**6a** (55)
CH_2 = $CHCH_2CH(Me)CO_2H$ (6.7)	CH_2Cl_2 (40)	8 h, 43 min (room)	**6b** (64)
CH_2 = $CHCH(OH)CH_2CO_2H$ (15.0)[b]	CH_2Cl_2 (45)	24 h (room)	**6c** (12.5)

[a] **4a** (5.04 mmol). [b] **4a** (15.0 mmol). [c] Yields rounded off to nearest percent. [d] The phosphates gave satisfactory (±0.4%) elemental (C, H) analyses, sometimes after a second attempt, except **5e** which was a bit off on carbon (calcd. 68.64, found 69.17, 69.00, same sample). [e] **5a**, **5b**, and **6b** were oils with some coloration; **5c**, **5d**, **5e**, **6a**, and **6c** were solids.

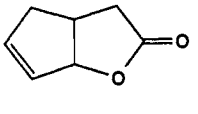
Table II. Selected Spectral Data for Ketol Phosphates and Phosphoryloxy Lactones

	IR[a] (cm^{-1}) P=O, C=O, OH	NMR[b]		
		300 MHz ^1H[c] (mult, J_{HP})	^{13}C[e,f] (mult, J_{CP})	^{31}P[g] (mult, J_{PH})
5a	1292, 1709	5.45 (d, 10.1)	69.8 (d, 5.7), 191.1 (d, 5.7)	-11.6 (t, 10.2)
5b	1292, 1740	4.70 (d, 9.5)	71.6 (d, 6.2), 201.5 (d, 6.6)	-12.2 (t, 9.4)
5c	1296, 1724	4.87 (d, 9.5)	71.7 (d, 6.5), 203.4 (d, 6.8)	-12.2 (t, ca. 9.3)
5d	1285, (1304, sh), 1732	4.91-5.05 (m)	81.3 (d, 6.2), 203.5 (d, 4.1)	-12.7 (d, 8.3)
5e	1296, 1682, 3453	6.79 (d, 8.8)	84.0 (d, 6.3), 190.3 (d, 5.2)	-13.0 (d, 8.5)
6a	1292, 1775	4.22-4.36 (m, 1 H) 4.36-4.51 (m, 1 H)	77.3 (d, 8.0), 176.2 (s)	-12.0 (s)[h]
6b	1292, 1775	4.26-4.34 (m, 1 H) 4.38-4.51 (m, 1 H)[d]	74.8 (d, 8.0), 75.3 (d, 7.8) 178.3 (s), 179.1 (s)	-11.98 (s), -12.04 (s)[h]
6c	1290, 1783, 3540	4.2-4.7 (m's, 5 H)	81.3 (d, 6.3), 174.8 (s)	-10.4 (s)[h]

[a] Neat oils (**5a**, **5b**, **6b**); solid films (**5c**, **5d**, **6a**); CH_2Cl_2 (**5e**); Nujol (**6c**). [b] Solvent was $CDCl_3$ for all NMR spectra; chemical shifts given in ppm and coupling constants given in Hz. [c] α-Hydrogens of **5a-e**; C-5 hydrogens of **6a** and **6b**; C-3, C-4, C-5, and O-H hydrogens of **6c**. [d] D_2O added. [e] Chemical shifts relative to $CDCl_3$ at 77.0 ppm. [f] α-Carbon and carbonyl carbon. [g] Referenced to a sample of 85% H_3PO_4 (sealed capillary) in $CDCl_3$. [h] Proton-decoupled spectra (coupled spectra exhibit poorly resolved multiplets).

was obtained, apparently as a single diastereomer (^1H, ^{13}C, ^{31}P NMR), but even the best yield was low (12.5%). Unfortunately, the C-3, C-4, and C-5 hydrogens give rise to a set of complex multiplets, and the stereochemistry of **6c** has not yet been assigned. We note, in this context, that the bromolactonization of 3-hydroxy-4-pentenoic acid has been reported to give *threo*-5-bromo-3-hydroxy-4-pentanolactone (57% yield) free of the erythro diastereomer.[9] Clarification of the stereochemistry of **6c** and efforts to improve the yield with -OH protected 3-hydroxy-4-pentenoic acid will be reported later.

When 2-cyclopentene-1-acetic acid was treated with **4a**, the unsaturated lactone **7** was isolated in ca. 50% yield[10] consistent with the behavior of **1** with the same acid.[3] Efforts to prepare the six-membered lactonol phosphate **8** from 5-hexenoic acid and **4a** have been only partially successful; the spectra (IR, PMR) of the crude product are indicative of **8**, but purification has not been achieved, and the material appears to be somewhat unstable.



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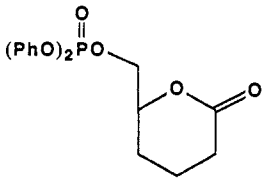

8

(7) Ramirez and his co-workers have prepared a variety of α-phosphoryloxycarbonyl compounds, ketol phosphates among them, from 1,3,2-dioxaphospholenes and 1,3,2-dioxaphosphole-2-oxides, see: (a) Ramirez, F.; Desai, N. B. *J. Am. Chem. Soc.* **1960**, *82*, 2652. (b) Swank, D.; Caughlan, C. N.; Ramirez, F.; Madan, O. P.; Smith, C. P. *J. Am. Chem. Soc.* **1967**, *89*, 6503. (c) Ramirez, F.; Kugler, H. J.; Patwardhan, A. V.; Smith, C. P. *J. Org. Chem.* **1968**, *33*, 1185. (d) Ramirez, F.; Bhatia, S. B.; Bigler, A. J.; Smith, C. P. *J. Org. Chem.* **1968**, *33*, 1192. (e) Ramirez, F.; Glaser, S. L.; Bigler, A. J.; Pilot, J. F. *J. Am. Chem. Soc.* **1969**, *91*, 496. (f) Ramirez, F.; Bauer, J.; Telefus, C. D. *J. Am. Chem. Soc.* **1970**, *92*, 6935. (g) Ramirez, F.; Maracek, J. F.; Ugi, I. *J. Am. Chem. Soc.* **1975**, *97*, 3809. (h) Ramirez, F.; Maracek, J. F. *Synthesis* **1985**, 449.

(8) This experiment was proposed by Professor Stephen D. Darling.

(9) Nakaminami, G.; Shioi, S.; Sugiyama, Y.; Isemura, S.; Shibuya, M.; Nakagawa, M. *Bull. Chem. Soc. Jpn.* **1972**, *45*, 2624.

(10) Minor impurities were present (NMR analysis).

Finally, we have also prepared [hydroxy((bis(benzoyloxy)-phosphoryl)oxy)iodo]benzene (**4b**) (88% yield) and note it reacts similarly to **4a** with ketones and pentenoic acids.[11]

Acknowledgment. We thank the Dow Chemical Company for partial financial support.

(11) Preliminary studies with acetone, cyclohexanone, 4-pentenoic acid, and 2-methyl-4-pentenoic acid have been conducted. Thus far, only the dibenzyl phosphate of acetone has been obtained "analytically pure" (i.e., ±0.4% C, H).

Corner Attack on Cyclopropane by Deuteron and Mercuric Ions: An Example of Stereospecific Formation and Capture of Unsymmetrical Corner-Deuteriated/Mercurated Cyclopropane Intermediates

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The regiospecificity and stereochemistry of electrophilic carbon-carbon bond cleavage in cyclopropanes has been the subject of considerable investigation and speculation.[1] In general two possible reaction trajectories for electrophilic attack on cyclo-

(1) (a) Coxon, J. M.; Battiste, M. A. In *The Chemistry of the Cyclopropyl Group*; Rappoport, Z., Ed.; J. Wiley and Sons: London, 1987; Chapter 6. (b) Lambert, J. B.; Schulz, W. J.; Mueller, P. H.; Kobayashi, K. *J. Am. Chem. Soc.* **1984**, *106*, 792. (c) Battiste, M. A.; Coxon, J. M. *Tetrahedron Lett.* **1986**, *27*, 517. (d) Wiberg, K. B.; Kass, S. R. *J. Am. Chem. Soc.* **1985**, *107*, 988.

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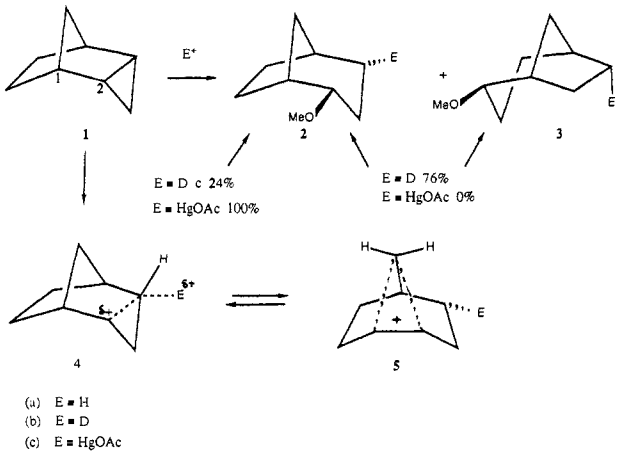
Scheme I



propanes have been differentiated for sufficiently dissymmetric substrates. Thus cleavage of the C–C bond syn (edge attack) or anti (corner attack) to the entering electrophile leads to retention or inversion, respectively, at the center of electrophilic attachment. For proton (or deuteron) promoted ring openings symmetrical corner- and edge-protonated cyclopropane intermediates have been frequently proposed to account for the predominance of inversion at the center of nucleophilic attack. Recently, however, stereochemical arguments[1c,2] and theoretical calculations[1d] have implicated a role for unsymmetrical corner-protonated cyclopropane type intermediates as major product controlling species in electrophilic opening of cyclopropanes. We now report definitive evidence for stereospecific formation and nucleophilic capture of unsymmetrical corner-deuteriated and -mercurated cyclopropanes. In addition we offer an explanation for corner attack by mercuric ions and protons and rationalize the contrasting behavior of oxidative addition by Ir, Pt, and Pd at the edge of cyclopropane.[3]

Incorporation of the cyclopropane ring into a fused polycyclic ring system often simplifies the stereochemical problems associated with determining reaction trajectories of the entering electrophile/nucleophile pair as illustrated, for example, in our previous studies of cyclopropane ring opening in *endo*-tricyclo[3.2.1.$0^{2,4}$]oct-6-ene.[4] In order to limit the number of potential mechanistic intermediates and/or reaction products the cyclopropyl substrate examined in this current study is the saturated analogue, *endo*-tricyclo[3.2.1.$0^{2,4}$]octane (**1**). Thus treatment of **1** with a catalytic quantity of *p*-toluenesulfonic acid in methanol at 80 °C for 7 days results in better than 80% conversion to a single major product with less than 2% of other products being detected. The structure of the major product was established as 2-*endo*-methoxybicyclo[3.2.1]octane (**2a**) by GPLC and spectral comparison with authentic **2a** (prepared by methylation of the known[5] alcohol with sodium amide, methyl iodide). The failure to detect any significant amounts of isomeric ethers testifies to the stereoelectronic precision of this remarkable ring-opening reaction which is mechanistically outlined in Scheme I (E = H).

When the acid-catalyzed ring-opening reaction of **1** was carried out in methanol-d_1, the ^{13}C NMR spectrum of the product showed deuterium incorporated at both C4 and C6 (Scheme I). From the intensities of the signals in the ^2H NMR spectrum at 1.43 and 1.35 ppm the ratio of deuterium at these sites (**2b**:**3b**) was established as 62:38, respectively.

Scheme II

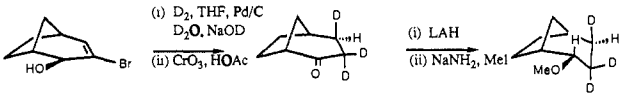


Chart I. σ Interaction of LUMO of Electrophile with Degenerate HOMO's of Cyclopropane

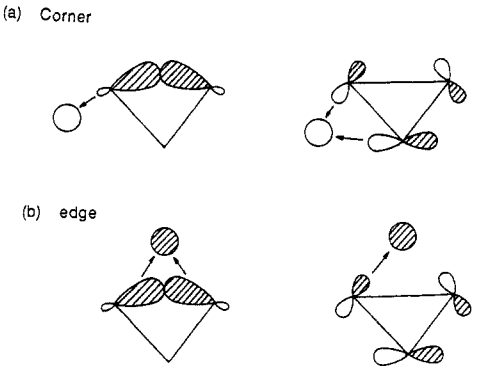
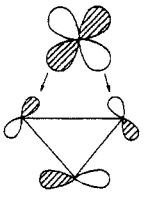


Chart II. Back Donation of d_π-Electrons to LUMO's of Cyclopropane



By contrast reaction of *endo*-tricyclo[3.2.$10^{2,4}$] octane with mercuric acetate in anhydrous methanol at room temperature gave exclusively 4-*endo*-acetoxymercurio-2-*endo*-methoxybicyclo-[3.2.1]octane (**2c**) in 95% yield. The stereochemistry is consistent with the observed ^1H NMR couplings (selective decoupling) and ^{13}C–^{199}Hg couplings.[7,8] The product arises by attack of the mercuric ion at the corner of the cyclopropane ring with concomitant attack by methanol at C4 with inversion. Reduction of the mercury adduct with sodium mercury amalgam in sodium deuteroxide (reaction conditions which in related systems have been shown[9] to give reduction with retention of configuration) affords 4-*endo*-deuterio-2-*endo*-methoxybicyclo[3.2.1]octane (**2b**). The ^2H NMR spectrum showed a signal at 1.43 ppm, and a two-dimensional ^1H–^{13}C heteronuclear correlation experiment identified the C4-exo-proton at 1.25 ppm. The stereochemistry of the deuterium was further confirmed by independent synthesis of an epimeric deuterio analogue as shown in Scheme II.

The stereochemistry of deuteron attack in the formation of ether **2b** from hydrocarbon **1** with deuteron in methanol-d_1 follows from the identity of this product with the reduction product of organomercurial **2c**. The stereochemistry of the deuterium at C6 in **3b** (1.35 ppm in the ^2H NMR spectrum) was established as endo from heteronuclear correlation experiments on the undeuteriated ether **3a** which exhibited connectivity of C6 with the exo and endo hydrogens at 1.65 and 1.35 ppm, respectively. The formation of the two deuteriated products **2b** and **3b** can be accounted for if reaction of deuteron occurs exclusively at the corner of the cyclopropane invoking rupture of the most substituted cyclopropane bond. The unsymmetrical corner-protonated intermediate **4b** can be attacked by methanol to give **2b** or collapse to the protonated species **5b**[10] which will be attacked with inversion equally at both

(2) DePuy, C. H.; Funfschilling, P. C.; Andrist, A. H.; Olson, J. M. *J. Am. Chem. Soc.* **1977**, *99*, 6297.
(3) Crabtree, R. H. *Chem. Rev.* **1985**, *85*, 245.
(4) (a) Battiste, M. A.; Coxon, J. M.; Simpson, G. W.; Steel, P. J.; Jones, A. J. *Tetrahedron* **1984**, *40*, 3137, and references cited therein. (b) While the structural features in such polycyclic systems will have a strong bearing on the observed regiochemistry of cyclopropane ring cleavage, any trajectory bias of the entering electrophile/nucleophile by the proximate structure might be expected to be limited to steric accessability.
(5) Belikova, N. A.; Bobyleva, A. A.; Kalinichenko, A. N.; Lipmaa, E.; Ordubadi, M. D. Pehk, T.; Plate, A. F. *Org. Mag. Reson.* **1976**, *8*, 74.
(6) Mass spectral analysis showed deuterium incorporation was >85%.
(7) J ^{13}C–^{199}Hg for **2c**: C1, 25; C2, 310; C3, 97; C4, 1634; C5, 66; C6, 65; C7, n.o.; C8, 310 Hz.
(8) Kitching, W.; Atkins, A. R.; Wickham, G.; Alberts, V. *J. Org. Chem.* **1981**, *46*, 563.
(9) Jensen, F. R.; Miller, J. J.; Cristol, S. J.; Beckley, R. S. *J. Org. Chem.* **1972**, *37*, 4341.

C1 and C2. The preferential attack on hydrocarbon **1** by the nucleophile at C2 as compared to C1 (62:38) clearly demonstrates that protonated intermediate **4b** must be trapped by the nucleophile at least to the extent of 24% before conversion to the symmetrical species **5b**.[11]

The observation of mercuric ion cleavage of the most substituted cyclopropane bond in this study is in direct conflict with previously stated rules for mercuric ion induced cyclopropane ring opening[12] and is in contrast to an earlier prediction[13] of the regioselectivity of this reaction. The unsymmetrical mercurated cation **4c** unlike the deuteriated analogue **4b** does not rearrange to the more symmetrical corner-protonated cation **5c**. Apparently a high degree of orbital interaction between C4 and C2 in the cation **4c** results in little charge development at C2. This reaction is therefore formally similar to that of alkenes with mercuric acetate[12] where skeletal rearrangement is not normally observed.

The favorable attack by the electrophiles deuteron and mercuric ion at the corner of the cyclopropane ring reflects the favorable interaction of both the degenerate HOMO's of the cyclopropane with the H 1s and d_σLUMO of the electrophile, respectively (Chart I (part a)). It should be noted for edge attack that while the HOMO/LUMO interaction is favorable for proton interaction with the symmetric Walsh orbital this is not the case with the unsymmetric orbital (Chart I (part b)). The preference for corner attack reflects the favorable HOMO/LUMO interaction for both degenerate molecular orbitals. A favorable interaction of the LUMO Walsh orbitals of cyclopropane with the d-orbitals of electron donor metals allows oxidative addition[14] at the edge of the cyclopropane (Chart II). This interaction compensates for the more favored σ-interaction at the corner of cyclopropane between the HOMO Walsh orbitals and the LUMO orbitals of the electrophile. For mercury the donor ability[15] of the d_π-orbitals is small and thus the d_πHOMO, cyclopropane LUMO interaction is unimportant, and the reaction stereochemistry parallels the reaction with deuteron.[16]

Acknowledgment. We acknowledge grants from the Research Committee of the New Zealand Universities Grants Committee.

(10) A small isotope effect will perturb the symmetry of this cation as represented. A functionally equivalent representation of symmetrical corner-protonated cation **5b** as two rapidly equilibrating unsymmetrical corner-protonated cations may also be considered for this intermediate.

(11) The stability of methoxy ether **2** to the reaction conditions was established by heating a sample of 3,3,4-*exo*-trideuterio-2-*endo*-methoxybicyclo[3.2.1]octane (cf. Scheme II) with *p*-toluenesulfonic acid for 7 days. The absence of rearrangement in the recovered starting material (^{13}C NMR) confirms the kinetic origin of the **2b**:**3b** ratio observed in the reaction of hydrocarbon **1** with acid in methanol-d_1.

(12) Wardell, J. L. In *Comprehensive Organometallic Chemistry*; Wilkinson, G., Stone, F. G. A., Abel, E. W., Eds.; Pergamon Press: Oxford, 1982; Vol. 2, p 863.

(13) Sokolov, V. I.; Reutov, O. A.; Rodina, N. B. *J. Organomet. Chem.* **1969**, *17*, 477.

(14) (a) Campbell, W. H.; Jennings, P. W. *Organometallics* **1983**, *2*, 1460. (b) Waddington, M. D.; Campbell J. A.; Jennings, P. W.; Campana, C. F. *Organometallics* **1983**, *2*, 1269. (c) Wiberg, K. B.; McClusky, J. V.; Schulte, G. K. *Tetrahedron Lett.* **1986**, *27*, 3083.

(15) Nyholm, R. S. *Proc. Chem. Soc.* **1961**, 273.

(16) (a) The generality of this conceptualized molecular orbital approach to the stereodifferentiation of electrophilic attack on cyclopropane is substantially upheld by a detailed examination of orbital interactions in hydrocarbon **1** and related systems (manuscript in preparation). (b) A prevalent opinion persisting in the area of acid-catalyzed ring opening of cyclopropanes is nicely summarized by the following referees observation: "...experiment and theory agree that the difference in energy between corner and edge protonated cyclopropanes is quite small. Therefore, the cited HOMO/LUMO interaction cannot contribute much. And, it is known from earlier studies that many of the cyclopropane orbitals are strongly perturbed on protonation—not just the HOMO." It is precisely these conclusions that the present experimental results, along with the theoretical calculations of Wiberg and Kass, call into question. Prior to the latter work the mechanistic role of the unsymmetrical corner-protonated cyclopropane had not been properly recognized. In particular, the optimized structures calculated for such unsymmetrical cations derived from methyl-substituted cyclopropanes are convincingly lower in energy than either the edge protonated or any of the symmetrical corner-protonated structures.[1a,d] No calculations have as yet been carried out on the unsymmetrical cation **4a**, but these results and our arguments in ref 1c suggest it is a reasonable intermediate not only for this system but also for cyclopropane itself.

Evidence for a Selenium Anomeric Effect? An Unusual Conformation of a Selenium Coronand

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The anomeric effect[1] has been the subject of intense investigation by both experimental and theoretical chemists alike.[2] While the existence of this conformational effect in X-C-Y systems containing first-row atoms has been widely accepted, the existence of significant anomeric interactions involving second- and lower-row atoms has been questioned recently.[3,4] We present herein unprecedented evidence for the existence of a third-row anomeric effect, based on an unusual solid-state conformation adopted by a selenium coronand.

In its generalized form, the anomeric effect refers to the torsional preferences about the C-X and C-Y bonds in $RXCH_2YR'$ molecules. The conformations increase in energy in the sequence gauche, gauche **1** < anti, gauche **2** < anti, anti **3** (Figure 1). The torsional behavior, bond length variations, and bond angle variations in $RXCH_2YR'$ have been rationalized both qualitatively[2,5] and quantitatively[6] by a perturbational molecular orbital (PMO) treatment that focuses on the stabilizing orbital interactions between the p-type nonbonding orbitals on X and Y, n_X and n_Y, with the acceptor orbitals, σ^*_{C-Y} and $\sigma^*_{C-X'}$ respectively. Whereas both these interactions may be expressed in **1**, symmetry considerations dictate that only the n_X-σ^*_{C-Y} is possible in **2** and neither interaction is possible in **3**. These hyperconjugative interactions account for the existence of the endo and exo anomeric effect[7,8] when the $RXCH_2YR'$ moiety is incorporated into a heterocyclohexane (Figure 1).

X-ray crystallographic analysis[9] of the selenium coronand, 1,3,7,9,13,15-hexaselenacyclooctadecane (**4**),[10] reveals that the ring has a very unusual irregular geometry, in sharp contrast to the regular quadrangular shapes normally exhibited by even-membered cycloalkane derivatives.[11] The two long sides of this elongated ring, shown in Figure 2, are distinctly different in

(1) (a) Edward, J. T. *Chem. Ind. (London)* **1955**, 1102. (b) Lemieux, R. U.; Chu, N. J. *Abstracts of Papers*, 133rd National Meeting of the American Chemical Society, San Francisco, CA; American Chemical Society: Washington, DC, 1958; Abstract 31N. (c) Chu, N. J. Ph.D. Thesis, University of Ottawa, 1959.

(2) For leading references see: (a) *Anomeric Effect. Origin and Consequences*; Szarek, W. A., Horton, D., Eds.; ACS Symposium Series 87; American Chemical Society: Washington, DC, 1979. (b) Kirby, A. J. *The Anomeric Effect and Related Stereoelectronic Effects at Oxygen*; Springer Verlag: Berlin, 1983. (c) Deslongchamps, P. *Stereoelectronic Effects in Organic Chemistry*; Wiley: New York, 1983. (d) Pinto, B. M.; Sandoval-Ramirez, J.; Sharma, R. D. *Tetrahedron Lett.* **1985**, 5235. (e) Pinto, B. M.; Sandoval-Ramirez, J.; Sharma, R. D.; Willis, A. C.; Einstein, F. W. B. *Can. J. Chem.* **1986**, *64*, 732. (f) Pinto, B. M.; Wolfe, S. *Tetrahedron Lett.* **1982**, 3687. (g) Pinto, B. M.; Schlegel, H. B.; Wolfe, S. *Can. J. Chem.* **1987**, *65*, 1658.

(3) Schleyer, P. v. R.; Jemmis, E. D.; Spitznagel, G. W. *J. Am. Chem. Soc.* **1985**, *107*, 6393.

(4) Anet, F. A. L.; Kopelevich, M. *J. Chem. Soc., Chem. Commun.* **1987**, 595.

(5) David, S.; Eisenstein, O.; Hehre, W. J.; Salem, L.; Hoffmann, R. *J. Am. Chem. Soc.* **1973**, *95*, 3806.

(6) Wolfe, S.; Whangbo, M.-H.; Mitchell, D. J. *Carbohydr. Res.* **1979**, *69*, 1.

(7) Praly, J.-P.; Lemieux, R. U. *Can. J. Chem.* **1987**, *65*, 213.

(8) Booth, H.; Khedhair, K. A.; Readshaw, S. A. *Tetrahedron* **1987**, *43*, 4699.

(9) $Se_6C_{12}H_{24}$: monoclinic, $P2_1/c$; T = 190 K; a = 20.154 (3) Å, b = 5.4292 (9) Å, c = 17.678 (3) Å, β = 110.66 (1)°; Z = 4; λ = 0.71069 Å; μ (Mo Kα) = 120.0 cm^{-1}; crystal dimensions 0.10 × 0.55 × 0.21 mm; transmission 0.083–0.330, corrected analytically; 2θ: 2–52°; data $I \geq 2.5\sigma(I)$, 2426; refined parameters, 163; $R_1 = \sum(|F_o| - |F_c|)/\sum|F_o| = 0.024$; maximum |shift/error| < 0.01; bond distances: Se–C 1.932 (6)–1.967 (6) Å, C–C 1.501 (9)–1.530 (9) Å; bond angles: C–Se–C 94.3 (3)–100.4 (3)°, Se–C–Se 116.0 (3)–118.6 (3)°, C–C–Se 108.9 (4)–116.6 (5)°, C–C–C 111.8 (4)–115.8 (5)°.

(10) Pinto, B. M.; Johnston, B. D.; Batchelor, R. J.; Einstein, F. W. B.; Gay, I. D. *Can. J. Chem.*, submitted for publication.

(11) Dale, J. *J. Chem. Soc.* **1963**, 93. Dale, J. *Top. Stereochem.* **1976**, *9*, 199.

0002-7863/88/1510-2990$01.50/0