common with the other allene oxides we have characterized.[6] From the coupling constants, the 5,11,14 double bonds are known to be cis, a significant point in relation to subsequent experiments. The chirality at C-8 was determined following hydrogenation (H_2/PtO_2 in hexane, 10 min at −15 °C). The major product was identified as (*S*)-8-hydroxyeicosanoate,[11] which (given that the priority order is reversed on hydrogenation) indicates that the allene oxide is of the 8*R* configuration. Minor products included methyl eicosanoate, (*RS*)-9-hydroxyeicosanoate, the 8-oxo and 9-oxo analogues, and small amounts 8,9-diol.

An interesting facet of the biphasic enzymic reaction is that the recoveries are better if the mixture is emulsified, although this is not required with an analogous reaction in flaxseed. Notably, the allene oxide is likely to be a substrate for additional enzymic transformations in the intact tissue, and this may entail its biosynthesis in a "protected" hydrophobic millieu.

Allene oxides are known to undergo a facile rearrangement to cyclopentenones.[12] Recently it was shown that allene oxides with a cis olefin in conjugation with the epoxyene will form a cyclopentenone with cis geometry of the side chains, whereas with a trans bond, ring closure leads to side chains in the trans configuration.[12c] We detected an 11-cis to 11-trans isomerization of the α-ketol product **4** to **5** in incubations of *P. homomalla*.[13] The [1-^{14}C]α-ketol **4** was converted to **5** (yield: 26%) during a 20-min incubation at room temperature (pH 8, 3 mg/mL acetone powder). A pertinent question is whether **5** also arises from an allene oxide with an 11-trans double bond. From an incubation containing α-ketols **4** and **5** in the ratio of 85:15, 11-cis:11-trans, the cyclopentenone **3** was found to be 98.5% cis geometry. Thus, the allene oxide precursor is predicted to have almost exclusively an 11-cis double bond,[12] in accord with the direct structural analysis. There does remain, however, the possibility that the allene oxide **2** is a substrate for this type of transformation under the natural conditions of biosynthesis. Other mechanisms of biosynthesis involving lipoxygenase metabolism of the cyclopentenone **3**[2] or the allene oxide **2**[14] have been proposed.

PGA_2 and PGE_2 have the 8*R* configuration at the juncture of the top side chain, and therefore it is highly significant that the lipoxygenase pathway in *P. homomalla* forms an 8*R*-hydroperoxide, and, as shown here, the 8*R*-HPETE is converted to an 8*R*,9-epoxyallene (Scheme I). The biosynthetic pathway must include a cyclization of the allene oxide with retention of configuration at C-8, and, either during the cyclization or in a later isomerization, a cyclic product with a trans geometry of the side chains must be formed. Another mechanism involving the 15-lipoxygenase metabolism of 8*R*-HPETE to 8,15-DiHPETE prior to formation of an allene oxide[2] is as yet not substantiated by the experimental results.[3]

Acknowledgment. The cooperation of the Department of Development & Natural Resources of the Cayman Islands in collection of samples of *P. homomalla* is gratefully acknowledged and Dr. David G. Anderson for collections in Florida. Professor Thomas M. Harris and Steven Baertschi participated in many invaluable discussions and provided the excellent NMR data. I also thank Dr. Jin K. Cha for helpful discussions, Christiana Ingram for contributions to the α-ketol analyses, and Dr. Ian A. Blair for providing the excellent mass spectrometry facilities. This work is supported by NIH grants DK-35275 and ES-00267.

(10) ^{1}H NMR of **2**, methyl ester (400 MHz, in hexane-d_{14} at −50 °C) was assigned from the 2-D COSY and decoupling experiments with peaks at δ 5.865 for H11 (t, $J_{10,11} = J_{11,12} = 11$ Hz, 1 H), 5.70 for H10 (d, 1 H), 5.55–5.37 for H5 and 6 (m, $J_{5,6} = 11$ Hz, 2 H), 5.365–5.270 for H14 and 15 (m, $J_{14,15} = 10.5$ Hz, 2 H), 5.085 for H12 (dt, $J_{11,12} = 11$ Hz, $J_{12,13} = 7$ Hz, 1 H), 3.525 for OCH_3 (s, 3 H), 3.465 for H8 (t, $J_{7,8} = 5.5$ Hz, 1 H), 2.85 for H13 (t, $J_{12,13} = J_{13,14} = 7$ Hz, 2 H), 2.575–2.485 for H7a and 2.43–2.36 for H7b (m, $J_{6,7} = 7$ Hz, $J_{7a,7b} = 15$ Hz, 2 H), 2.20 for H2 (t, 2 H), 2.10–2.03 for H4 and H16 (m, 4 H), 1.65 for H3 (p, 2 H), with H17–20 obscured by the residual CHD and CHD_2 in the hexane.

(11) Benzyl 8-hydroxyeicosanoate was resolved on a 25 × 0.46 cm Chiralcel OB column (Baker): solvent, 1% isopropyl alcohol in hexane; flow rate, 0.3 mL/min; 8*S* enantiomer at 17.2 mL (96% of area) and 8*R* at 20.0 mL (4%). Note that hydrogenation changes the R and S assignment of the 8-hydroxyl; thus, 8*R*-HETE is converted to (*S*)-8-hydroxyeicosanoic acid.

(12) (a) Grimaldi, J.; Bertrand, M. *Tetrahedron Lett.* **1969**, *38*, 3269–3272. Grimaldi, J.; Bertrand, M. *Bull. Soc. Chim. Fr.* **1971**, 957–962. (b) For recent work and further references, see: Doutheau, A.; Sartoretti, J.; Gore, J. *Tetrahedron* **1983**, *39*, 3059–3065. Doutheau, A.; Gore, J.; Diab, J. *Ibid.* **1985**, *41*, 329–338. (c) Kim, S. J.; Cha, J. K. *Tetrahedron Lett.* **1988**, *29*, 5613–5616.

(13) The ^{1}H NMR (400 MHz) of **5** in $CDCl_3$ was assigned by 2-D COSY and decoupling experiments with peaks at δ 5.61–5.54 for H12 and 5.56–5.48 for H11 (m, $J_{11,12} = 15.2$ Hz), at 5.65–5.45 for H5 and 5.45–5.35 for H6 (m, $J_{5,6} = 10.2$ Hz), at 5.50–5.40 for H15 and 5.40–5.30 for H14 (m, $J_{14,15} = 10.8$ Hz), with other resonances at 4.29 for H8 (dd, 1 H), 3.23 for H10 (m, 2 H), 2.79 for H13 (m, 2 H), 2.65–2.55 for H7a and 2.45–2.35 for H7b (m, 2 H), 2.37 for H2 (t, 2 H), 2.13 for H4 (q, 2 H), 2.02 for H16 (q, 2 H), 1.73 for H3 (p, 2 H), 1.40–1.30 for H17 (m, 2 H), 1.37–1.25 for H18 and H19 (m, 4 H), and 0.88 for H20 (t, 3 H). On normal phase and reversed phase HPLC, **5** chromatographed immediately after **4**, and the two isomers gave essentially identical mass spectra. The absolute configuration of **5** was measured using a method described before[3] as a 40% enantiomeric excess of the 8*S* configuration; the 11-*cis* α-ketol **4** recovered from the same incubation was 54% enantiomeric excess of 8*S*.

(14) Brash, A. R.; Baertschi, S. W.; Ingram, C. D.; Harris, T. M. *Adv. Prostaglandin, Thromboxane, Leukotriene Res., 19* Raven Press: New York, 1989; in press.

Asymmetric Allylboration with *B*-Allyl-2-(trimethylsilyl)borolane

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Stereoselective addition of an acetate unit to the prostereogenic[1] carbonyl carbon of an aldehyde is achieved by a variety of aldol reactions.[2] A synthetic equivalent of this fundamental carbon–carbon bond-forming process is asymmetric allylboration, for which several chiral reagents have been devised in recent years.[3] The magnitude of asymmetric induction achieved by these reagents lies, with few exceptions, in the range 70–90% ee (see Table I) for representative (achiral) substrates.[4] In our synthetic studies on polyketide natural products we have been faced with a problem of raising the enantioselectivity of allylboration to 95% ee or higher so as to gain stereochemical control of the reaction sufficient for double asymmetric synthesis.[3e-f,5] We describe herein the preparation of homochiral (*R* and *S*)-*B*-allyl-2-(trimethylsilyl)-borolanes (*R*)-**1** and (*S*)-**1**, reagents which possess high reactivity and meet the above demand for enantioselectivity. In practice, **1** is generated in situ from its air-stable precursor, the synthesis of which is simple and practical. Of particular interest is the mechanistic course of this asymmetric reaction effected by the monosubstituted borolane derivative **1** of C_1 symmetry rather than the disubstituted analogues of C_2 symmetry (e.g., *trans*-2,5-dimethylborolane) reported earlier from these laboratories.[6]

(1) Mislow, K.; Siegel, J. *J. Am. Chem. Soc.* **1984**, *106*, 3319.

(2) (a) Braun, M. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 24 and references cited therein. (b) Masamune, S.; Sato, T.; Kim, B. M.; Wollmann, T. A. *J. Am. Chem. Soc.* **1986**, *108*, 8279.

(3) (a) Herold, T.; Hoffmann, R. W. *Angew. Chem., Int. Ed. Engl.* **1978**, *17*, 768. (b) Hoffmann, R. W. *Pure Appl. Chem.* **1988**, *60*, 123. (c) Brown, H. C.; Jadhav, P. K. *J. Am. Chem. Soc.* **1983**, *105*, 2092. (d) Jadhav, P. K.; Bhat, K. S.; Perumal, P. T.; Brown, H. C. *J. Org. Chem.* **1986**, *51*, 432. (e) Roush, W. R.; Walts, A. E.; Hoong, L. K. *J. Am. Chem. Soc.* **1985**, *107*, 8186. (f) Roush, W. R.; Banfi, L. *J. Am. Chem. Soc.* **1988**, *110*, 3979.

(4) The reagent described in ref 3f exhibits excellent enantioselectivity (>95% ee) but suffers from low reactivity and thus may not be a practical reagent for natural product synthesis.

(5) (a) Masamune, S.; Choy, W.; Petersen, J. S.; Sita, L. R. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 1. For studies on double asymmetric allylboration, see: (b) Roush, W. R.; Palkowitz, A. D.; Palmer, M. A. J. *J. Org. Chem.* **1987**, *52*, 316. (c) Roush, W. R.; Palkowitz, A. D. *J. Am. Chem. Soc.* **1987**, *109*, 953. (d) Brown, H. C.; Bhat, K. S.; Randad, R. S. *J. Org. Chem.* **1987**, *52*, 319.

(6) Masamune, S. In *Stereochemistry of Organic and Bioorganic Transformations*; Bartmann, W., Sharpless, K. B., Eds.; VCH: 1986; p 49.

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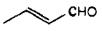
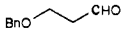
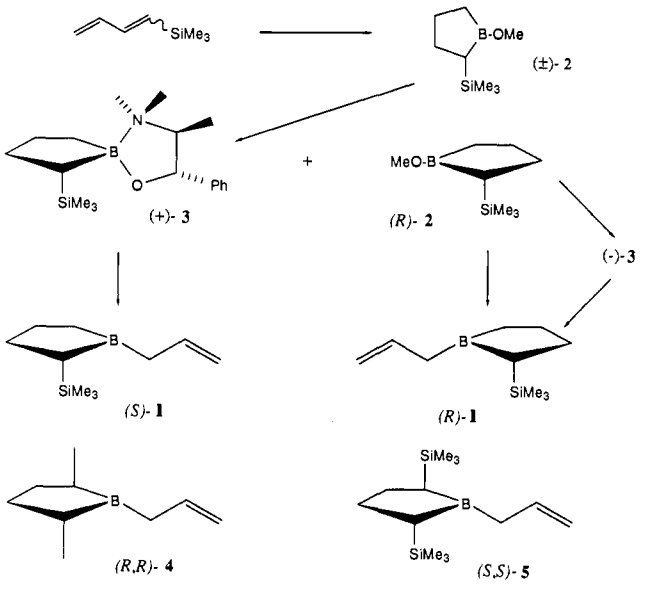
Table I. Asymmetric Allylboration with Reagent (*S*)-**1** of 99.4% ee

		% ee (abs config) (% yield)			% ee (abs config) (% yield)			
entry	aldehyde	(*S*)-**1**[a]	**4**[b]	**5**[c]	A[d]	B[e]	C[f]	D[g]
1		96 (R) (80)	93 (R) (86)		86 (R) (71)	91 (R) (76)		
2		96 (S) (85)	85 (S) (72)	71 (S) (82)	90 (S) (86)	97 (S) (73)		
3		97 (S) (90)	86 (S) (91)		83 (S) (88)	88 (S) (80)	82 (S)	96 (S)
4		96 (S) (92)	88 (S) (88)				87 (R) (72)	97 (S) (40)
5					96 (S) (81)		71 (S) (78)	85 (S)
6		97 (S) (85)						
7		92 (S) (84)						

[a] Reaction (Et_2O, −100 °C, 3 h) of 0.8 mmol of aldehyde with reagent prepared from (+)-**3** (1.0 mmol) and allylmagnesium bromide (0.9 mmol). Entries 1–4 utilized reagent (*S*)-**1** prepared in situ, entries 6 and 7 utilized distilled (*S*)-**1** (1.2 equiv in Et_2O, −100 °C, 3 h). Yields in entries 1 and 2 were determined by capillary GC; entries 3–7 are isolated yields of homoallylic alcohols following oxidative workup and silica gel chromatography. Product % ee was determined by conversion to the (*S*)-Mosher's acetate and analysis by HPLC (entries 2, 3, 4) or ^1H NMR (entries 1, 6, 7). Absolute configuration was assigned on the basis of comparison with the (*S*)-Mosher's acetates of authentic samples obtained using *B*-allyldiisopinocampheylborane as described in ref 3d. [b] Reagent **4** prepared in situ from (*R*,*R*)-2,5-dimethyl-*B*-methoxyborolane (Masamune, S.; Kim, B.; Petersen, J. S.; Sato, T.; Veenstra, S. J.; Imai, T. *J. Am. Chem. Soc.* **1985**, *107*, 4549) and allylmagnesium bromide in Et_2O. Reaction in Et_2O (−78 °C, 1 h) followed by warming to 25 °C over 1 h (Kim, B. M. Ph. D. Thesis, MIT, 1988; product % ee corrected for reagent of 92.8% ee). [c] Reagent **5** prepared in situ from (*S*,*S*)-2,5-bis(trimethylsilyl)-*B*-methoxyborolane and allylmagnesium bromide in Et_2O. (The absolute configuration of this reagent was determined by X-ray analysis of a crystalline derivative. Short, R. P. Ph. D. Thesis, MIT, 1989; product % ee corrected for reagent of 96% ee). Reaction in Et_2O (−78 °C, 1 h). [d] Reagent A: *B*-allyldiisopinocampheylborane prepared from (+)-α-pinene (ref 3d). [e] Reagent B: *B*-allyldiisocaranylborane prepared from (+)-3-carene, $[\alpha]^{23}_D$ +18° (neat), (ref 3d). [f] Reagent C: diisopropyl tartrate modified *B*-allylboronate (ref 3e, entries 3 and 5 utilized (+)-DIPT; entry 4 utilized (−)-DIPT). [g] Reagent D: (*R*,*R*)-*N*,*N*′-dibenzyl-*N*,*N*′-ethylenetartramide modified *B*-allylboronate (ref 3f). Entry 3: −78 °C, 67 h, 60% conversion; entry 4: −78 °C, 47 h, 48% conversion; entry 5: −78 °C, 47 h, 75% conversion.

Scheme I



A sequence of three reactions, hydroboration of 1-(trimethylsilyl)-1,3-butadiene[7] (BH_3·SMe_2, Et_2O, 25 °C), methanolysis, and thermal isomerization,[10] all executed in one pot, provides racemic *B*-methoxy-2-(trimethylsilyl)borolane (±)-**2** in 45% yield, bp 70–72 °C/20 mm (Scheme I). Treatment of (±)-**2** with (1*S*,2*S*)-(+)-*N*-methylpseudoephedrine (0.5 equiv) in pentane results in the diastereoselective formation of an air-stable, crystalline complex (+)-**3**, mp 115–116.5 °C, with (*S*)-**2**. The complex, which separates from solution at −20 °C in *quantitative* yield, is obtained as an ca. 40:1 mixture of diastereomers and apparently reflects the thermodynamic stability of (+)-**3** relative to the complex incorporating (*R*)-**2**. The filtrate contains mainly (*R*)-**2**, from which (−)-**3** is readily obtained. *This remarkably efficient resolution process thus permits the synthesis of either enantiomer of* ***1*** *in >90% yield from racemic* ***2***.[9] The structure of (+)-**3** has been determined by X-ray crystallographic analysis[11] of a single crystal grown from ether at −20 °C. The absolute configuration of the borolane moiety is thus established by relation to the known stereostructure of the amino–alcohol employed for the resolution.

Addition of 0.9 equiv of allylmagnesium bromide to (+)-**3** or (−)-**3** in ether generates (*S*)-**1** or (*R*)-**1**, respectively, for subsequent allylboration with an aldehyde.[13] Results of the reaction with

(7) Carter, M. J.; Fleming, I.; Percival, A. *J. Chem. Soc., Perkin Trans. 1* **1981**, 2415. This reference describes the synthesis of isomerically pure (*E*)-1-(trimethylsilyl)-1,3-butadiene. We employ a more efficient synthesis from 1,3-butadienylmagnesium chloride[8] and chlorotrimethylsilane which provides 1-(trimethylsilyl)-1,3-butadiene as a mixture of *Z*- and *E*-isomers. Dienes prepared by either route give identical results in the subsequent hydroboration–isomerization.[9]

(8) Ishii, T.; Kawamura, N.; Matsubara, S.; Utimoto, K.; Kozima, S.; Hitomi, T. *J. Org. Chem.* **1987**, *52*, 4416.

(9) Full experimental details and physical data for all new compounds (including (*S*)-**1** of 99.4% ee) are available as Supplementary Material.

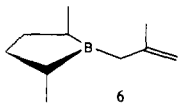
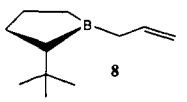
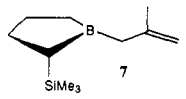
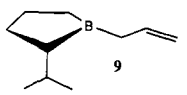
(10) Brown, H. C.; Negishi, E. *Tetrahedron* **1977**, *33*, 2331. For a discussion of the directing effect of silicon in the hydroboration of vinylsilanes, see: Soderquist, J. A.; Brown, H. C. *J. Org. Chem.* **1980**, *45*, 3571.

(11) Crystallographic data for $C_{18}H_{32}BONSi$: orthorhombic space group $P2_12_12_1$; a = 13.902 (2) Å, b = 14.093 (3) Å, c = 9.715 (2) Å, V = 1903.3 (7) Å^3, Z = 4, ρ_{calcd} = 1.11 g/cm^3. The intensities of 2522 reflections ($2\theta_{max}$ = 55°) were collected on a Rigaku AFC6R diffractometer at −65 °C using Mo Kα radiation (μ = 1.28 cm^{-1}). An empirical absorption correction (DIFABS)[12] was applied which resulted in transmission factors ranging from 0.91 to 1.39. The data were corrected for Lorentz and polarization effects. The structure was solved by direct methods. Full-matrix least-squares refinement on 2081 observed reflections [$I > 2.00\sigma(I)$] and 199 variable parameters with anisotropic thermal parameters for all non-hydrogen atoms and fixed positional and thermal parameters for hydrogen atoms converged with R = 0.054 (R_w = 0.072) and GOF = 2.76. The largest peak in the final difference map was 0.31 eÅ^{-3}. All calculations were performed using the TEXAN crystallographic software package of Molecular Structure Corporation. Tables of bond angles and distances are available as Supplementary Material.

(12) Walker, N.; Stuart, D. *Acta Crystallogr., Sect. A: Found. Crystallogr.* **1983**, *A39*, 158.

(13) Formation of an allylborane from an amino–alcohol complex by this method has not been previously reported.

Table II. Allylboration of Isobutyraldehyde with Reagents 6-9 at -78 °C

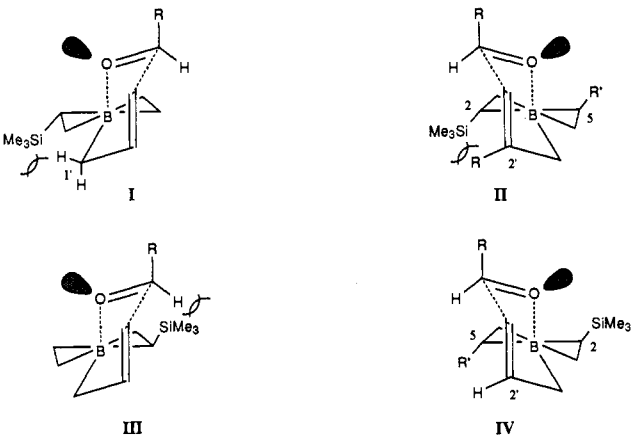
	% ee (% yield)		% ee (% yield)
 6	27 (80)	 8	72 (95)
 7	81 (78)	 9	24 (87)

(*S*)-**1**, (*S,S*)-2,5-dimethyl-*B*-allylborolane (**4**), and (*S,S*)-2,5-bis(trimethylsilyl)-*B*-allylborolane (**5**) are summarized together with those recorded for other known chiral reagents (Table I). It is evident that the enantioselectivity of **1** is uniformly high with all achiral aldehydes examined and superior to even **4** and **5**, which were previously thought to be ideally designed for this type of asymmetric induction.[14]

This unexpected result demands some explanations. Since the two faces of the borolane are diastereotopic, approach of the aldehyde from both faces is considered. Thus, provided that (1) the reaction proceeds through a six-membered chelate ring and (2) the aldehyde is coordinated with the larger group anti to the developing B-O bond,[15] four different transition states I-IV are subject to evaluation.

Examination of the absolute configuration of the products obtained employing reagent **1** of established absolute configuration leads immediately to the exclusion of transition states I and III,

Transition States



since these lead to products of configuration opposite to that obtained. A distinction between the two remaining transition states II and IV is drawn by the information provided in Table I, entry 2. The notable reduction in enantioselectivity from 96% ee (with **1**) to 71% ee with **5** may be taken as a manifestation of the steric interaction between C(2′)-H and C(2)-(Me_3Si), indicated in II (R = H), as this repulsion is unavoidable in either II or IV (R = H, R′ = Me_3Si) with **5** but does not exist in IV (R = R′ = H) with **1**.[16] This interpretation is in harmony with the observations revealed in Table II, which show that methyl substitution at C(2′) of **4** (methallylborane **6**)[9] results in significantly reduced enantioselectivity (27% ee) relative to the unsubstituted allyl derivative **4** (85% ee, Table I, entry 2). The 15% ee decrease observed with **7**[9] (2′-methyl substitution on **1**) therefore likely reflects the interaction of the C(2′)-methyl with C(5)-H in IV (R = Me, R′ = H) rather than that of the C(2′)-methyl with C(2)-(Me_3Si) in II (R = Me, R′ = H). Thus, IV appears to be energetically favored relative to II.

The asymmetric induction brought about by **1** is largely of steric origin; any stereoelectronic component, if active, is not evident. The enhanced enantioselectivity exhibited by **1** relative to **8** and **9** (Table II) is due to the greater effective "reach" of trimethylsilyl versus *tert*-butyl and isopropyl, respectively, a consequence of the increased C-Si bond length present in **1**. Comparison of transition state IV with transition state I (which collapses to the minor enantiomer) suggests that the Me_3Si group of **1** senses the difference between C(1′)-H_2 and the (uncoordinated) syn lone pair of the aldehydic oxygen. Indeed, aldol reactions with *B*-(vinyloxy)-2-(trimethylsilyl)borolanes (replacing the C(1′)-H_2 by oxygen) proceed with virtually no enantioselection.[17]

Acknowledgment. We thank Dr. John C. Dewan for the crystallographic analysis of (+)-**3** and the National Institutes of Health (GM 35879) for financial support.

Supplementary Material Available: Detailed experimental procedures and tables of crystallographic data (positional parameters, *U* values, and bond distances and bond angles) for (+)-**3** (33 pages). Ordering information is given on any current masthead page.

(14) (a) Short, R. P.; Masamune, S. *Tetrahedron Lett.* **1987**, *28*, 2841. Garcia, J.; Kim, B. M.; Masamune, S. *J. Org. Chem.* **1987**, *52*, 4831.
(15) Reetz, M. T.; Hullmann, M.; Massa, W.; Berger, S.; Rademacher, P.; Heymanns, P. *J. Am. Chem. Soc.* **1986**, *108*, 2405.
(16) With reagent **4**, this interaction is between C(2′)-H and C(2)-Me. The smaller steric demand of Me relative to $SiMe_3$ is reflected in the higher enantioselectivity of **4** vs **5**.
(17) The boron enolate derived from 3-(3-ethyl)pentyl ethanethioate (ref 2b) and (*S*)-2-(trimethylsilyl)-*B*-chloroborolane was evaluated. Cho, J.-H.; Masamune, S., unpublished results.

Characterization of the Equilibrating Forms of the Aldehydic Abasic Site in Duplex DNA by ^{17}O NMR

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The enzymatic repair of chemical and physical damage to the bases in DNA is frequently initiated by hydrolysis of the N-glycosidic bond to the damaged base to yield an abasic site.[1,2] For example, the ubiquitous uracil-DNA glycosylase removes the uracil formed in DNA by spontaneous hydrolysis of the 4-amino group of cytosine, and UV endonuclease V from bacteriophage T_4 hydrolyzes one of the glycosidic bonds in a pyrimidine photodimer. The resulting abasic site is a mixture of aldehyde, hydrate, and cyclic hemiacetals. The abasic sites so generated are removed enzymatically from the DNA duplex by cleavage of the phosphodiester backbone at both sides of the abasic site followed by insertion of the proper nucleotide unit in the gap so produced.

While aldehydic abasic sites are important intermediates in DNA repair, little detailed information is available about their structure and chemical reactivity.[3,4] We recently used ^{13}C NMR to study defined DNA duplexes containing an abasic site specifically labeled with ^{13}C in the aldehydic carbon and concluded that the mixture of cyclic hemiacetals is the predominant form of the abasic site in the duplex, that the conformation of a duplex

(1) Lindahl, T. *Annu. Rev. Biochem.* **1982**, *51*, 61-87.
(2) Friedberg, E. C. *DNA Repair*; W. H. Freeman: New York, 1985.
(3) Jones, A. R.; Mian, A. M.; Walker, R. T. *J. Chem. Soc.* **1968**, 2042-2044.
(4) Lindahl, T.; Anderssen, A. *Biochemistry* **1972**, 3618-3623.

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