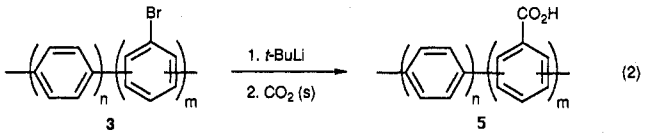
coated with films of both compounds **3** and **4**. Anodic peak potentials (E_{pa}) for the oxidation were at 1.44 and 1.45 V, respectively.[21]

Additionally, we used the lithiated polymer to prepare functionalized derivatives. For example, **3** was lithiated as described above and quenched with dry ice to afford the carboxylated polymer **5** with one carboxylic acid moiety per three aryl units (eq 2).[22] The FTIR (KBr) spectrum was free of the C–Br stretch



(2)

at 1074 cm^{-1} with the major stretch at 1686 cm^{-1} for the carbonyl moiety. The O–H stretch was weak presumably due to restricted hydrogen bonding in the solid. This procedure could have applications for the synthesis of functionalized polymers for self-doped conducting systems with fast electrochromic switching times and the fabrication of polymer-based batteries with high charge storage capacities.[23]

We do not have a clear understanding of the mechanism of the aryl couplings. The surprising aspect is that **3** unquestionably exhibits a predominance of para linkages while much of the bromide content is retained. Migrations of lithium and bromide in bromo lithio heteroaromatics are known under the base catalyzed halogen dance (BCHD) conditions.[24] The Taylor approach to PPP involving 1,4-dichloro-2-butene as a promoter for the polymerization of (4-bromophenyl)magnesium bromide may involve similar electron-transfer phenomena.[6] Additionally, the copolymerization of 2,5-dilithiothiophene with 2,5-dibromothiophene to afford poly(thiophene) has been reported.[25] However, as we described here, the addition of HMPA dramatically facilitates the aryl–aryl coupling process. A study of the scope and mechanism[26] of the polymerization as well as the detailed electrical and thermal analyses of the materials is in progress.

Acknowledgment. We thank the Department of the Navy, Office of the Chief of Naval Research, Young Investigator Program (N00014-89-J-3062), and the National Science Foundation (RII-8922165) for their generous support of this work. We also thank Professor R. Philp (University of South Carolina), Dr. R. Beckerbauer (Du Pont), and Dr. A. Diaz (IBM) for helpful suggestions. The scanning electron microscope was purchased with a grant from the National Science Foundation (BIR-8805143).

(21) Recorded relative to $Ag/AgNO_3$ (0.01 M) in CH_3CN at 50 mV/s scan rate with 0.1 M tetraethylammonium perchlorate (TEAP) as the electrolyte and a Pt working electrode. For related studies on oligo(phenylenes), see: (a) Diaz, A.; Crowley, J.; Bargon, J.; Gardini, G. P.; Torrance, J. B. *J. Electroanal. Chem.* **1981**, *121*, 355. (b) Meerholz, K.; Heinze, J. *Angew. Chem., Int. Ed. Engl.* **1990**, *29*, 692.

(22) Calculated elemental data for one CO_2H unit per three aryl units, $C_{19}H_{12}O_2$: C, 83.82; H, 4.41. Found: C, 76.10; H, 4.99.

(23) (a) Patil, A. O.; Ikenoue, Y.; Colaneri, N.; Chen, J.; Wudl, F.; Heeger, A. J. *Synth. Met.* **1987**, *20*, 151. (b) Patil, A. O.; Ikenoue, Y.; Wudl, F.; Heeger, A. J. *J. Am. Chem. Soc.* **1987**, *109*, 1858. (c) Ikenoue, Y.; Chiang, J.; Patil, A. O.; Wudl, F.; Heeger, A. J. *J. Am. Chem. Soc.* **1988**, *110*, 2983. (d) Ikenoue, Y.; Uotani, N.; Patil, A. O.; Wudl, F.; Heeger, A. J. *Synth. Met.* **1989**, *30*, 305. (e) Havinga, E.; van Horsen, L.; ten Hoeve, W.; Wynberg, H.; Meijer, E. W. *Polym. Bull. (Berlin)* **1987**, *18*, 277. (f) Tsai, W.; Jang, G. W.; Rajeshwar, K. *J. Chem. Soc., Chem. Commun.* **1987**, 1776. (g) Shi, S.; Wudl, F. *Marcromolecules* **1990**, *23*, 2119.

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(25) Fujisaka, T.; Masaoka, T.; Inoue, T. Japan Patents 63,234,022 and 63,234,023; *Chem. Abstr.* **1989**, *110*, 1461711 and 1461712.

(26) For low-temperature NMR studies of Li–halogen exchange reactions in the presence of HMPA, see: (a) Reich, H. J.; Green, D. P. *J. Am. Chem. Soc.* **1989**, *111*, 8729. (b) Reich, H. J.; Green, D. P.; Phillips, N. H. *J. Am. Chem. Soc.* **1989**, *111*, 3444. (c) Reich, H. J.; Reich I. L.; Phillips, N. H. *J. Am. Chem. Soc.* **1985**, *107*, 4101. (d) Reich, H. J.; Phillips, N. H. *J. Am. Chem. Soc.* **1986**, *108*, 2102.

Studies on DNA-Cleaving Agents: Synthesis of a Functional Dynemicin Analogue[1]

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Received September 7, 1990

Neocarzinostatin, esperamicin, and calicheamicin are structurally unprecedented DNA-cleaving agents that operate putatively through the triggerable generation of diyl intermediates.[2] Recently, the groups of Konishi and Clardy[3a] reported the structure of dynemicin (**1**) (Scheme I), the newest member of this emerging class of chemotherapeutic leads. Dynemicin exhibits potent inhibitory activity against various tumor cell lines and in vivo activity in P388 leukemia and B16 melanoma inoculated mice.[3b] Dynemicin is proposed[1a,3,4] to be activated for DNA lesion through reductive cleavage of its epoxide ring. Addition to the resultant anthraquinone methide would then provide the activated enediyne **2**. In this overall process, carbons 2, 3, 8, and 7 initially fixed in an anti-like conformation by the epoxide ring in **1** are released to assume a gauche-like conformation in **2**, thereby allowing for facile cycloaromatization[5] to a diyl (**3**) capable of effecting lesions at proximate nucleotide sites.[6] We describe herein the first synthesis of an analogue of dynemicin that fully emulates the acid-inducible activation and cycloaromatization behavior exhibited by dynemicin itself.[3c]

Our approach to analogue design was based on the view that the diyl-generating capability of dynemicin could be mimicked by various dihydroquinoline epoxides spanned by an enediyne bridge (bold face in **1**). Activation of such systems was expected to arise through modification or cleavage of various aryl substituents or nitrogen protecting groups, which by increasing electron density in the surrogate C ring would result in epoxide cleavage. Importantly, this approach would allow for activation under a variety of chemical or physiological conditions.[7]

Synthesis of CDF-ring analogues of **1** started with reduction of commercially available aldehyde **4**[8] (Scheme II). Treatment of the resultant alcohol with the magnesium salt of (trimethylsilyl)acetylene and $ClCO_2Me$ gave the 1,2-addition product **5** along with minor amounts of the 1,4-addition product.[9] Attempts to

(1) (a) Presented in part at the 200th National Meeting of the American Chemical Society, Washington, DC, 1990; paper ORGN 161. (b) For the previous study in this series, see: Wender, P. A.; McKinney, J. A.; Mukai, C. *J. Am. Chem. Soc.* **1990**, *112*, 5369.

(2) For lead references, see the following. Neocarzinostatin: (a) Edo, K.; Mizugaki, M.; Koide, Y.; Seto, H.; Furihata, K.; Otake, N.; Ishida, N. *Tetrahedron Lett.* **1985**, *26*, 331. (b) Napier, M. A.; Goldberg, I. H.; Hensens, O. D.; Dewey, R. S.; Liesch, J. M.; Albers-Schoenberg, G. *Biochem. Biophys. Res. Commun.* **1981**, *100*, 1073. (c) Myers, A. G.; Proteau, P. J.; Handel, T. M. *J. Am. Chem. Soc.* **1988**, *110*, 7212. Calicheamicin: (d) Lee, M. D.; Dunne, T. S.; Siegel, M. M.; Chang, C. C.; Morton, G. O.; Borders, D. B. *J. Am. Chem. Soc.* **1987**, *109*, 3464. Lee, M. D.; Dunne, T. S.; Chang, C. C.; Ellestad, G. A.; Siegel, M. M.; Morton, G. O.; McGahren, W. J.; Borders, D. B. *J. Am. Chem. Soc.* **1987**, *109*, 3466. Esperamicin: (e) Golik, J.; Clardy, J.; Dubay, G.; Groenewold, G.; Kawaguchi, H.; Konishi, M.; Krishnan, B.; Ohkuma, H.; Saitoh, K.; Doyle, T. W. *J. Am. Chem. Soc.* **1987**, *109*, 3461. Golik, J.; Dubay, G.; Groenewold, G.; Kawaguchi, H.; Konishi, M.; Krishnan, B.; Ohkuma, H.; Saitoh, K.; Doyle, T. W. *J. Am. Chem. Soc.* **1987**, *109*, 3462.

(3) (a) Konishi, M.; Ohkuma, H.; Tsuno, T.; Oki, T.; VanDuyne, G. D.; Clardy, J. *J. Am. Chem. Soc.* **1990**, *112*, 3715. (b) Konishi, M.; Ohkuma, H.; Matsumoto, K.; Tsuno, T.; Kamei, H.; Miyaki, T.; Oki, T.; Kawaguchi, H.; VanDuyne, G. D.; Clardy, J. *J. Antibiot.* **1989**, *42*, 1449. (c) After submission of this manuscript, the following synthetic studies on dynemicin analogues have appeared: Porco, J. A.; Schoenen, F.; Stout, T. J.; Clardy, J.; Schreiber, S. L. *J. Am. Chem. Soc.* **1990**, *112*, 7410. Nicoloau, K. C.; Hwang, C.-K.; Smith, A. L.; Wendeborn, S. V. *J. Am. Chem. Soc.* **1990**, *112*, 7416.

(4) (a) Semmelhack, M. F.; Gallagher, J.; Cohen, D. *Tetrahedron Lett.* **1990**, *31*, 1521. (b) Snyder, J. P.; Tipsword, G. E. *J. Am. Chem. Soc.* **1990**, *112*, 4040.

(5) Bergman, R. G. *Acc. Chem. Res.* **1973**, *6*, 25.

(6) Sugiura, Y.; Shiraki, T.; Konishi, M.; Oki, T. *Proc. Natl. Acad. Sci. U.S.A.* **1990**, *87*, 3831.

(7) For a recent example of nitrogen deprotection that would lend itself to an activation process, see: Senter, P. D.; Pearce, W. E.; Greenfield, R. S. *J. Org. Chem.* **1990**, *55*, 2975.

(8) The aldehyde was purchased from Aldrich Chemical Company.

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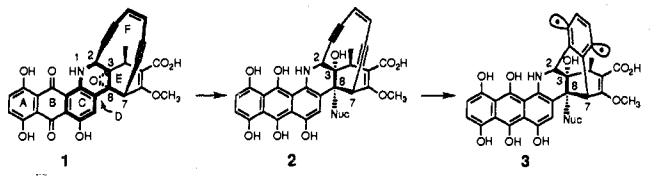
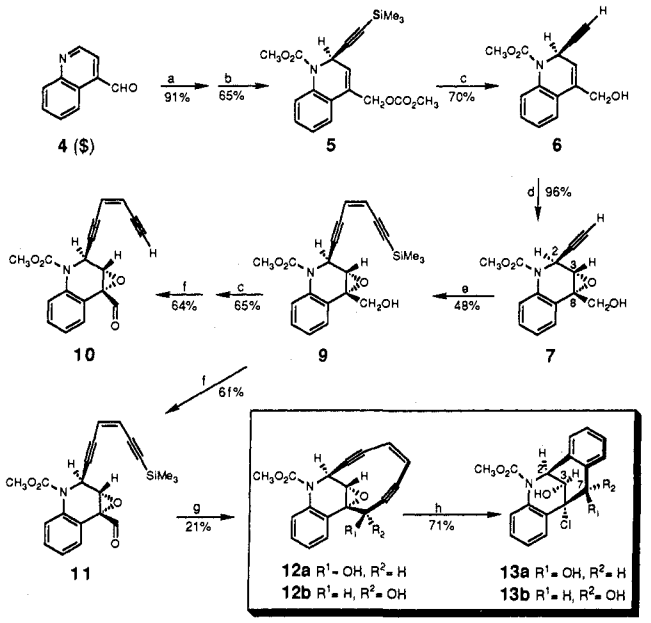
Scheme I



Scheme II[a]



[a](a) $NaBH_4$, CH_3OH, room temperature, 2 h; (b) 2.5 equiv of $Me_3SiC{\equiv}CMgBr$, then 3.0 equiv of CH_3O_2CCl, THF, 0 °C, 2 h; (c) K_2CO_3, CH_3OH, room temperature, 1 h; (d) *m*-CPBA, CH_2Cl_2, 0 °C, 20 min; (e) 0.1 equiv of $Pd(PPh_3)_2Cl_2$, 0.3 equiv of CuI, 3.0 equiv of *n*-$BuNH_2$, *cis*-ClCH=CHC≡$CSiMe_3$ (**8**), THF, room temperature, 2 h; (f) Dess–Martin periodinane, CH_2Cl_2, room temperature, 2 h; (g) CsF, CH_3CN, 0 °C, 3 h; (h) 0.6 N HCl/THF (1:1), 1,4-cyclohexadiene, room temperature, 2 h.

introduce the entire enediyne subunit[10] in this way were successful, but lower yields and less selective subsequent transformations eliminated the potential advantages of this more direct approach. The carbonate and silyl groups of **5** were removed in one operation with K_2CO_3 in MeOH. Treatment of the resultant product (**6**) with *m*-chloroperoxybenzoic acid at 0 °C provided exclusively epoxide **7** as expected on the basis of reagent approach control and confirmed by the coupling constant of 2.9 Hz between H-2 and H-3.[11,12] Coupling of the alkyne **7** with *cis*-chloro enyne **8**[13] was accomplished with catalytic Pd(0) and Cu(I) to provide enediyne **9**. Removal of the silyl group and oxidation of the carbinol with Dess–Martin periodinane[14] gave aldehyde **10**.

Scheme III

On the basis of independent studies reported by the Danishefsky, Kende, and Tius groups,[13,15] it was expected that ring closure in **10** could be initiated by deprotonation of the terminal alkyne. However, attempts to effect ring closure in this fashion resulted in extensive decomposition or recovery of starting material.[16] A solution to this problem was devised from the studies of Nakamura and Kuwajima[17] in which silylated alkynes were found to undergo desilylative carbonyl addition when treated with *n*-Bu_4NF in THF. While the intramolecular variant of this process has not been reported, we were gratified to find that aldehyde **11** upon treatment with CsF in CH_3CN at 0 °C provided alcohols **12a** and **12b** (2:1, respectively).[18]

In direct analogy with dynemicin,[3] when alcohol **12a** was dissolved in THF and treated with 0.6 N HCl in the presence of 1,4-cyclohexadiene, facile conversion (2 h, 25 °C) to the cycloaromatized product **13a** occurred in 71% yield. Similarly, alcohol **12b** gave product **13b** (ca. 70%). The stereochemical assignments for **13a/b** are based on NOE difference spectroscopy in which irradiation of the C-3 hydrogen in **13a** produces an enhancement (2.9%) of the C-7 hydrogen while no enhancement was observed in the corresponding experiment with **13b**. Both isomers exhibited an NOE enhancement at C-2 when C-3 was irradiated. Importantly, when **13a** was treated with *n*-Bu_3SnH, the reduction product **14** (53%) (Scheme III) was obtained, thereby confirming the position of chloride attachment and through decoupling experiments allowing assignment of the connectivity of carbons 2, 3, 8, and 7 in **13**. The cycloaromatization of alcohols **12a** and **12b** can also be achieved with other reagents. For example, independent treatment of **12a** and **12b** with HBr provided the bromides corresponding to **13a** and **13b**, respectively, while exposure of **12a** and **12b** to HOAc led to the slow formation of acetates **15a** (27%) and **15b** (41%) derived presumably from the initially formed tertiary acetates through transesterification. In accord with the pH-dependent activation behavior exhibited by this analogue series, **12a** reacts more rapidly in 0.6 N HCl/THF than in HOAc/THF and only very slowly in THF/H_2O at pH 7.5 (phosphate buffered). The intermediacy and trapping of a common *o*-quinone imine methide in these activation procedures is indicated by the slow formation of both **13a** and **15a** when **12a** is treated with benzyltriethylammonium chloride in HOAc/THF.

In summary, a synthesis of functional analogues of dynemicin is described that requires only seven steps and allows access to preparative quantities of acid-activatable, diyl-generating devices. This approach to the CDF-ring analogues of dynemicin should be generally applicable to the synthesis of other polycyclic analogues and amenable to variations in the protecting group on nitrogen and the substituents on the aromatic ring as desired for control over transport, intercalation, activation, and diyl formation. These acid-activatable analogues embody just one of several activation strategies[19] that could exploit chemistry peculiar to a cellular target site.

(9) (a) Yamaguchi, R.; Nakazono, Y.; Kawanisi, M. *Tetrahedron Lett.* **1983**, *17*, 1801. (b) Stadniichuk, R. F.; Pilyugin, G. T.; Petrenko, O. E. *Zh. Obshch. Khim.* **1970**, *40*, 1834.

(10) Danishefsky, S. J.; Yamashita, D. S.; Mantlo, N. B. *Tetrahedron Lett.* **1988**, *29*, 4681.

(11) The coupling constant for the other diastereomer was expected to be in the range of 5–8 Hz. All compounds that possess the benzylic epoxide were prone to decomposition when chromatographed on silica. Consequently, the yields given for purified products from **7** forward are minimum values.

(12) The numbering scheme used for the analogues is the same as that used for dynemicin in Scheme I and ref 3.

(13) Kende, A. S.; Smith, C. A. *Tetrahedron Lett.* **1988**, 29, 4217.

(14) (a) Dess, D. B.; Martin, J. C. *J. Org. Chem.* **1983**, *48*, 4155. (b) **CAUTION**: Hazards associated with the use of this reagent are described by J. B. Plumb and D. J. Harper in a letter to the editor (*Chem. Eng. News* **1990**, *68*(29), 3). While we have experienced no problems, caution should be observed in the use of this reagent. An alternative oxidant, TPAP (Griffith, W. P.; Ley, S. V.; Whitcombe, G. P.; White, A. D. *J. Chem. Soc., Chem. Commun.* **1987**, 1625) provided aldehyde **11**; however, the yield was low (20%) and required the chromatographic removal of significant amounts of impurities.

(15) (a) Danishefsky, S. J.; Mantlo, N. B.; Yamashita, D. S.; Schulte, G. *J. Am. Chem. Soc.* **1988**, *110*, 6890. (b) Tius, M. A.; Cullingham, J. M. *Tetrahedron Lett.* **1989**, *30*, 3749.

(16) Urethane-directed deprotonation of the propargylic hydrogen could compete with deprotonation of the alkyne. For a pertinent lead reference, see: Beak, P.; Snieckus, V. *Acc. Chem. Res.* **1982**, *15*, 306.

(17) Nakamura, E.; Kuwajima, I. *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 498.

(18) The *isolated* yield of the alcohols was approximately 20%. The 2:1 ratio of alcohols is consistent with the stereoselectivity observed in analogous reactions: Howe, G. P.; Wang, S.; Procter, G. *Tetrahedron Lett.* **1987**, *28*, 2629.

(19) Tietze, L. F.; Beller, M. *Liebigs Ann. Chem.* **1990**, 587.

Acknowledgment. This research was supported by a grant (CA31845) from the National Institutes of Health.

Supplementary Material Available: IR, NMR, and mass spectrometry data for compounds **7**, **11**, **12a**, **12b**, **13a**, **13b**, **14**, and **15a** (7 pages). Ordering information is given on any current masthead page.

Biogenetically Inspired Stereospecific Synthesis of the Dienylvinylcyclopropane Gamete Attractant Dictyopterene B

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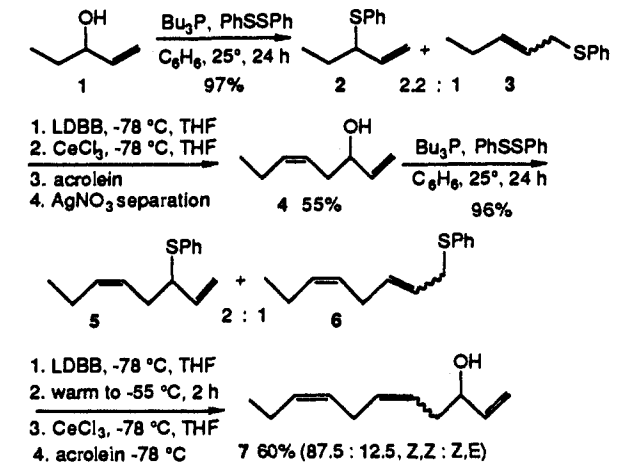
Although *cis,cis*-undeca-1,5,8-trien-3-ol (the *Z,Z* isomer of **7**) has been proposed as a biosynthetic precursor of the marine gamete attractants dictyopterene B (**10**), dictyopterene D (**13**), and the two $C_{11}H_{16}$ tetraenes **11** and **12**,[1,2] **7** has never been converted to these gamete attractants. We now disclose that such conversions can be executed in a highly efficient and stereospecific manner, especially with regard to the formation of **10**.

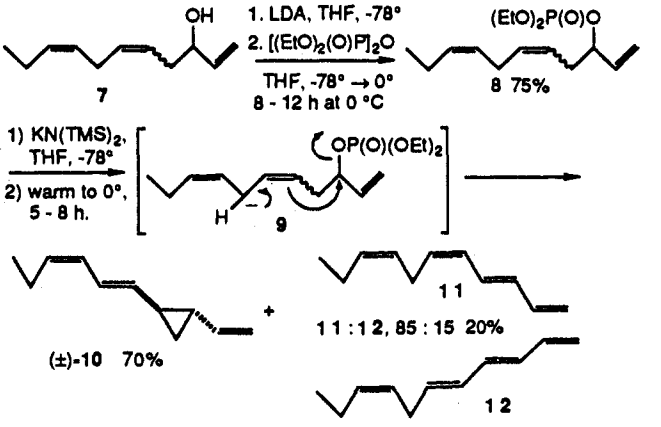
7 (mainly the *Z,Z* isomer) is now readily available by a modification and refinement (Scheme I) of the synthesis reported recently from this laboratory.[3] Commercially available 1-penten-3-ol (**1**) was efficiently converted[4] to a mixture of sulfides **2** and **3**, which was reductively lithiated[5] with lithium *p,p'*-di-*tert*-butylbiphenylide[6] (LDBB) followed by transmetalation with $CeCl_3$ and quenching of the resulting allylcerium(III) η^3 complex in situ with acrolein to afford 68% of a mixture of **4** and its trans isomer in a ratio of 89:11. The desired cis isomer **4** was separated from the trans isomer by flash chromatography using silica gel impregnated with a low concentration of $AgNO_3$ to provide a 55% yield from **2** and **3**. The alcohol **4** was subjected to the same reactions as **1** except that the intermediate allyllithium was warmed to −55 °C for 2 h in order to accomplish stereochemical equilibration, which was more sluggish than that of the allyl anion derived from **2** and **3**. The product **7** was a mixture of *Z,Z* and *Z,E* isomers in a ratio of 87.5:12.5. It was assumed that separation of the isomers of **7**, which was found to be very difficult at best, would be unnecessary since the internal double bond is destroyed during the ring closure to (±)-dictyopterene B (**10**) and that double bond that occurs in **7** in both cis and trans forms appears in the tetraenes **11** and **12** also as a mixture of cis and trans isomers. It should be noted that this route (four synthetic steps, 31% overall yield) to the putative biogenetic precursor **7** is the most efficient to date.[2,7]

Dictyopterene B, the most abundant and interesting of these gamete attractants, was prepared from **7** in two steps. Treatment of the alkoxide derivative of **7** with tetraethyl pyrophosphate produced the phosphate ester **8**.[8] Upon addition of potassium bis(trimethylsilyl)amide to **8** and subsequent warming of the

Scheme I



Scheme II



reaction mixture, a remarkably stereospecific and efficient [1,2,(3),5]-elimination[9] occurred to provide (±)-dictyopterene B (**10**) in 70% yield (Scheme II).[10,11] The only separable byproduct isolated from the reaction by chromatography was an oil consisting of a mixture of the two natural tetraenes **11** and **12**. Interestingly, no production of the cis-disubstituted cyclopropane corresponding to **10** was formed since it is known[12] to rearrange at room temperature to dictyopterene D (**13**), which was not an observed product. Molecular models indicate that, in the transition state for the elimination leading to the cis-disubstituted cyclopropane, serious nonbonded interactions occur between the protons on the sp^2 carbon atoms closest to the developing ring; the transition state leading to **10** appears to be strain free.

Although S_N2 displacements of phosphate groups appear to be very rare, there is a well-documented procedure for cyclopropane formation that involves displacement of this group by an enolate anion in a special system in which the phosphate ester is generated by a rearrangement.[13] Allyl diethyl phosphates undergo nucleophilic displacement of the phosphate group by ligands of aluminum, but this process appears to follow an S_N1 course.[14]

(1) Moore, R. E. *Acc. Chem. Res.* **1977**, *10*, 40 and citations therein.
(2) After Moore made this suggestion, the acetate of *cis,cis*-**7** in the correct enantiomeric configuration was isolated from *Dictyopteris prolifera*. Yamada, K.; Tan, H.; Tatematsu, H.; Ojika, M. *Tetrahedron* **1986**, *42*, 3775 and citations therein.
(3) Guo, B.-S.; Doubleday, W.; Cohen, T. *J. Am. Chem. Soc.* **1987**, *109*, 4710.
(4) Nakagawa, I.; Hata, T. *Tetrahedron Lett.* **1975**, 1409.
(5) Cohen, T.; Bhupathy, M. *Acc. Chem. Res.* **1989**, *22*, 152.
(6) Freeman, P.; Hutchinson, L. *J. Org. Chem.* **1980**, *45*, 1924.
(7) A five-step route to racemic *cis,cis*-**7** in poor yield has been reported: Marner, F. J. Ph.D. Thesis, University of Cologne, Cologne, 1975. Cited in the following: Moore, R. E. In *Marine Natural Products*; Scheuer, P. J., Ed.; Academic Press: New York, 1978; Vol. 1, pp 43-124.
(8) Preparation of dialkyl phosphates from pyrophosphates: Chouinard, P. M.; Bartlett, P. A. *J. Org. Chem.* **1986**, *51*, 75 and citations therein.
(9) Review of such eliminative cyclizations: Kaupp, G. *Top. Curr. Chem.* **1988**, *146*, 58.
(10) The ^1H NMR, ^{13}C NMR, and mass fragmentation spectra of (±)-**10** were identical with the spectra of the natural gamete attractant, dictyopterene B.
(11) According to capillary GC and ^1H NMR (500 MHz), the isolated cyclopropane (±)-**10** was contaminated with ~5% of an unknown and inseparable impurity.
(12) Schneider, M. P.; Goldbach, M. *J. Am. Chem. Soc.* **1980**, *102*, 6114.
(13) Izydore, R. A.; Ghirardelli, R. G. *J. Org. Chem.* **1973**, *38*, 1790. Petter, R. C. *Tetrahedron Lett.* **1989**, *30*, 399.
(14) Kitagawa, Y.; Hashimoto, S.; Iemura, S.; Yamamoto, H.; Nozaki, H. *J. Am. Chem. Soc.* **1976**, *98*, 5030. Itoh, A.; Ozawa, S.; Oshima, K.; Sasaki, S.; Yamamoto, H.; Hiyama, T.; Nozaki, H. *Bull. Chem. Soc. Jpn.* **1980**, *53*, 2357.

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