in high yield. It is notable that the conditions employed for silyl ether cleavage (5% aqueous HF in CH_3CN) gave **9a** directly with complete stereoselectivity at C8. This contrasts with other spiroketalization conditions explored by us in which the C8-epimeric spiroketal is formed in predominance.[17] Thus the tetracyclic ring system of (+)-phyllanthocin was established in ten synthetic steps, with six asymmetric centers present in their correct absolute configurations.

It remained to introduce the C3-methoxycarbonyl substituent, thereby establishing the last of the seven stereocenters of phyllanthocin. After failing to effect a satisfactory result via hydrozirconation-[18] or hydroboration-based[19] methodologies, we turned to hydroformylation[20] for this purpose. In terms of functionality and architecture, the silyl ether **9b** was clearly an unusually complicated hydroformylation substrate. In a representative experiment, 1.0 mmol of the cyclohexene **9b** in 10 mL of benzene was placed in a Parr bomb (45 mL capacity) which was then charged with 0.08 mmol of $[(COD)RhOAc]_2$,[21] followed by CO and H_2 (1:1) to 560 psi. After heating at 76 °C (bath temperature) for 3.25 h, chromatographic purification on silica gel gave the C3-α- and C3-β-formyl products **10a** and **10b** in 21% and 20% yields, respectively. A single C4-formyl product was also isolated in 12% yield. Stereoisomer **10a** could be equilibrated (NaOMe, MeOH, 25 °C, 80%) to a 2.3:1 mixture of C3 epimers in which **10b** predominated.

With the carbon skeleton established, complete with stereochemical details, the total synthesis was finished by straightforward cosmetic adjustments. Oxidation of the formyl residue in **10b** to the carboxylic acid with Jones reagent at 0 °C and treatment of the crude product with ethereal diazomethane afforded the methyl ester **10c** [mp 89.5–91 °C, $[\alpha]^{25}_D$ +101° (*c* 1.95, CH_2Cl_2)] in 93% yield. Cleavage of the C10-silyl ether gave in 96% yield the known[4] axial alcohol **10d** [mp 130–130.5 °C (sealed capillary), $[\alpha]^{25}_D$ +126° (*c* 1.23, $CHCl_3$)].[22] Cinnamoylation by the procedure of Williams[4b] gave in 82% yield (+)-phyllanthocin (1) [mp 129–129.5 °C, $[\alpha]^{26}_D$ +27.2° (*c* 2.04, $CHCl_3$)][23] which was identical with an authentic sample by standard spectroscopic and chromatographic criteria.[24]

Acknowledgment. We gratefully acknowledge the National Institutes of Health, the Alfred P. Sloan Foundation, the National Science Foundation (Presidential Young Investigator Award to S.D.B., Predoctoral Fellowship to J.E.C.), Stuart Pharmaceuticals, and the Rohm and Haas Company for generous financial support. High-field NMR spectra were obtained through the NSF Regional NMR Center at the University of South Carolina (CHE 82-07445). We thank Professor D. R. Williams (Indiana University) for providing spectral data for **1** and **10d** and an authentic sample of 1.

† Research Fellow of the Alfred P. Sloan Foundation; recipient of a National Science Foundation Presidential Young Investigator Award.

‡ National Science Foundation Predoctoral Fellow.

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Stereocontrol in the Intramolecular Diels–Alder Reaction. 7. Use of the Trimethylsilyl Group as a Removable Stereocontrol Element To Provide Greatly Enhanced Levels of Diastereoselection

Summary: Introduction of the trimethylsilyl group at defined locations in triene substrates which undergo closure by intramolecular Diels–Alder cycloaddition to both hydrindene and octalin systems results in greatly enhanced levels of diastereoselection.

Sir: A large volume of research during the past 10 years has been devoted to exploration of the intramolecular Diels–Alder reaction as a protocol for the stereocontrolled synthesis of complex molecules.[1,2] However, very high levels of diastereoselection (≥20:1) have not been attainable in most instances, since the nonbonded interactions responsible for the diastereoselection are of insufficient magnitude.[3,4] Only when steric and stereoelectronic factors are reinforcing or when the system is amenable to the use of Lewis acid catalysis do the observed levels of stereoselection reach those found for other types of C–C bond forming reactions.[5,6] Since we, as well as others,[7,8] have concluded that a few specific nonbonded interactions are the primary determinants of the stereochemical outcome, we set out to establish whether introduction of sterically demanding groups as control elements,[9] whose presence magnify the controlling nonbonded interaction in the transition states leading to the undesired stereoisomers, would permit the very high levels of diastereoselection

(17) For example, if, after cleavage of the *p*-methoxybenzyl ether, the silyl ether was cleaved with *n*-Bu_4NF in THF, no spiroketalization occurred. Induction of spiroketalization with CF_3CO_2H in CH_2Cl_2 led to a 1:3.8 mixture of **9a** and its C8 epimer. These isomers are readily distinguished by IR (intramolecular H-bond in **9a** to C8-axial oxygen) and ^1H NMR.

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(22) Although our mp and optical rotation data for **10d** differ from those reported [lit.[4b] mp 104 °C (sealed capillary), $[\alpha]^{26}_D$ +143.9° (*c* 0.79, $CHCl_3$)], the high field ^1H NMR and ^{13}C NMR data indicate that the two samples are identical.

(23) The literature values for the mp and $[\alpha]_D$ of (+)-phyllanthocin are as follows: mp 126–127 °C, $[\alpha]^{24}_D$ +25.2° (*c* 2.00, $CHCl_3$);[1] mp 120–121 °C, $[\alpha]^{33}_D$ +23.81° (*c* 1.26, $CHCl_3$);[2] mp 118–120 °C, $[\alpha]^{24}_D$ +24.9° (*c* 1.86, $CHCl_3$).[4b]

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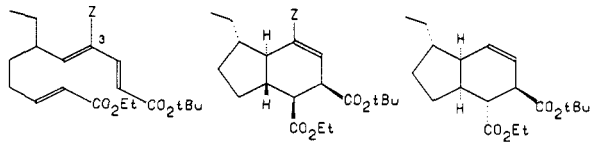
(9) The trimethylsilyl group (TMS) has been employed previously as a sterically demanding group to orient flexible chains for other types of synthetic transformations: Hasan, I.; Kishi, Y. *Tetrahedron Lett.* **1980**, *21*, 4229. Tomioka, H.; Suzuki, T.; Oshima, K.; Nozaki, H. *Tetrahedron Lett.* **1982**, *23*, 3387. Narula, A. S. *Tetrahedron Lett.* **1982**, *23*, 5579.

0022-3263/85/1950-3421$01.50/0

obtainable by other methodologies.[10]

To test this idea, we have examined two cases whose stereochemical outcome was already well-characterized, so that a direct assessment of the effects of the stereocontrol element would be possible. The first case is that of triene **1**, whose cycloaddition afforded the ester **2** as the major

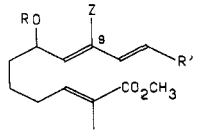


1, Z = H; **6**, Z = TMS

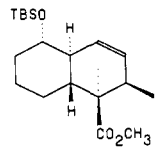
2, Z = H; **7**, Z = TMS

3

stereoisomer in a ratio of 4:1 with the minor isomer being the related cis ring isomer **3**.[11] A series of related trienes have been examined by other groups during synthetic studies on X-14547A and the stereoselectivities observed consistantly fall in the range of 4–8:1.[12] An even more demanding system is triene **4**, whose cyclization has been studied by Roush in work directed toward chlorothricolide.[13-15] Cyclization of **4** afforded a mixture of all four possible diastereomers in which the required isomer **5** comprised only 15% of the mixture.

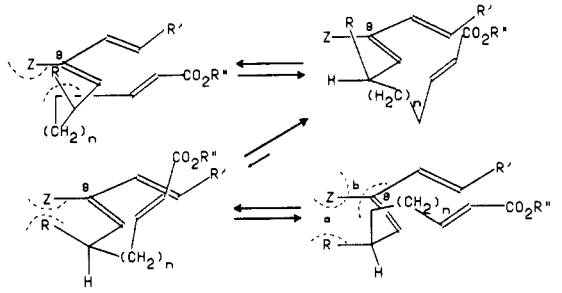


4, Z = H; R = TBS; R′ = CH_3
9, Z = TMS; R = MOM; R′ = $(CH_2)_3OBn$



5

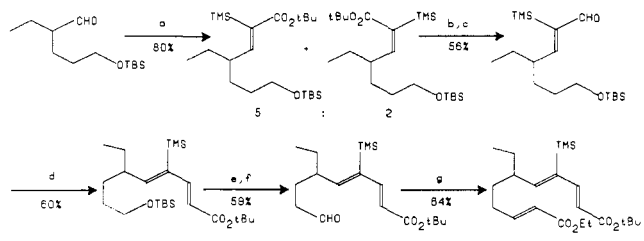
The controlling nonbonded interactions in the closure of trienes **1** and **4** are those which destabilize the syn transition states via $A_{1,3}$ strain (a)[16] and the exo transition states (b), as shown in eq 1, by interaction with the sub-



(1)

stituent Z at C-9. Thus, we chose to introduce the trimethylsilyl (TMS, Me_3Si) group at C-9 to attempt to enhance stereoselection since it is bulky (A = 2.4 kcal/mol)[17] and selectively removable under mild conditions by protodesilylation.[18] The preparation of **6** is outlined in Scheme I.[19-21]

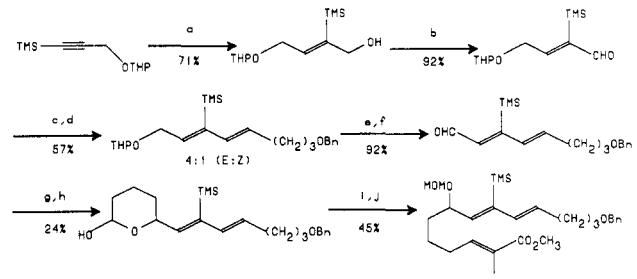
Scheme I[a]



6

[a] Reagents: (a) $LiC(Me_3Si)_2CO_2$-*t*-Bu/$MgBr_2$-Et_2O/THF, −78 °C → room temperature, H_3O^+; (b) DIBAL (2.2 equiv)/hexane, −78 °C, 2 h; (c) PDC (1.5 equiv)/CH_2Cl_2, room temperature, 2 h; (d) *t*-$BuO_2CCH_2PO(OEt)_2$/NaH/THF, −30 °C → room temperature, 2 h; (e) 5% (aq) HF/CH_3CN, room temperature, 10 min; (f) PDC (1.5 equiv)/CH_2Cl_2, room temperature, 22 h; (g) $EtO_2CCH_2PO(OEt)_2$/NaH/THF, −30 °C → room temperature, 2 h.

Scheme II[a]



9

[a] Reagents: (a) DIBAL (1.1 equiv)/$PhCH_3$-Et_2O, 0 °C → 35 °C, 3 h; CH Li (1.1 equiv)/LiBr/Et_2O, 0 °C, $(CH_2O)_n$ (1.15 equiv), room temperature, 48 h; (b) $(COCl)_2$ (1.1 equiv)/Me_2SO (2.2 equiv)/Et_3N (5 equiv), CH_2Cl_2, −55 °C; (c) $PhCH_2O(CH_2)_4SO_2Ph$ (1 equiv)/*n*-BuLi (1 equiv)/THF, −50 °C, 0.33 h; aldehyde, −78 °C, 0.5 h; PhCOCl (1 equiv), −78 °C → 0 °C, 0.5 h; pH 7 buffer; (d) 5.65% Na(Hg) (6 equiv)/Et_2O-CH_3OH (3:1), −20 °C, 6 h; (e) Dowex CH_3OH, room temperature, 1 h; (f) $(COCl)_2$ (1.1 equiv), Me_2SO (2.2 equiv)/Et_3N (5 equiv)/CH_2Cl_2, 55 °C; (g) $(C_2H_5O)_2CHCH_2CH_2CH_2Br$ (1.1 equiv)/*t*-BuLi (2.2 equiv)/Et_2O, −78 °C → room temperature; TMEDA (1.1 equiv)/aldehyde (1 equiv), 0.5 h; (h) $(COOH)_2$ (0.66 equiv)/H_2O-THF (1:1), room temperature, 21 h; (i) $Ph_3P{=}C(CH_3)COOCH_3$/PhH, 80 °C, 3 h; (j) CH_3OCH_2Cl (6 equiv)/(*i*-Pr)$_2$NEt (6.7 equiv)/DMAP (catalytic)/CH_2Cl_2, room temperature, 15 h.

Thermolysis of **6** at 165 °C in toluene in the presence of BHT for 22 h afforded essentially a single cycloadduct,

(10) Professor W. R. Roush of the Massachusetts Institute of Technology has independently pursued investigations in this area. We thank Professor Roush for making the results of his studies available to us prior to publication and for valuable discussions of the problem.

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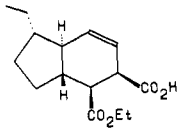
(19) All new substances exhibit spectral characteristics (IR, 300 MHz NMR, MS) consistant with their assigned structures and give satisfactory combustion or high resolution exact mass analytical data. Partial NMR data (in δ at 300 MHz). **6**: 7.31 (d, J = 16 Hz, 1 H), 6.92 (dt, J_1 = 16 Hz, J_2 = 6.6 Hz, 1 H), 6.11 (d, J = 9.9 Hz, 1 H), 5.74 (m, 2 H), 4.15 (q, J = 7 Hz, 2 H), 2.32 (m, 1 H), 2.14 (m, 2 H), 1.70–1.20 (m, 4 H), 1.50 (s, 9 H), 0.86 (t, J = 7 Hz, 3 H), 0.20 (s, 9 H). **7**: 5.98 (m, 1 H), 4.04 (q, J = 6 Hz, 2 H), 3.37 (m, 1 H), 2.55 (m, 1 H), 1.90 (m, 1 H), 1.80 (m, 2 H), 1.60 (m, 2 H), 1.36 (s, 9 H), 1.17 (m, 2 H), 0.86 (t, J = 7 Hz, 3 H), 0.05 (s, 9 H). **8**: 5.95 (d, J = 9 Hz, 1 H), 5.55 (m, 1 H), 4.00 (q, J = 6.6 Hz, 2 H), 3.54 (m, 1 H), 2.48 (m, 1 H), 2.10 (m, 1 H), 2.00–1.90 (m, 8 H), 1.08 (t, J = 6.6 Hz, 3 H), 0.78 (t, J = 6.6 Hz, 3 H). **9**: 7.37 (m, 5 H), 6.80 (t, J = 7.1 Hz, 2 H), 6.06 (d, J = 15 Hz, 1 H), 5.91 (d, J = 10 Hz, 1 H), 5.60 (dt, J_1 = 15 Hz, J_2 = 6.5 Hz, 1 H), 4.70 (m, 1 H), 4.50 (m, 2 H), 4.32 (m, 1 H), 3.76 (s, 3 H), 3.50 (t, J = 6 Hz, 2 H), 3.38 (s, 3 H), 2.20 (m, 4 H), 1.85 (s, 3 H), 1.80–1.40 (m, 6 H), 0.20 (s, 9 H). **10**: 7.35 (m, 5 H), 6.18 (d, J = 5.6 Hz, 1 H), 4.72 (d, J = 9 Hz, 1 H), 4.68 (d, J = 9 Hz, 1 H), 4.50 (s, 2 H), 3.61 (s, 3 H), 3.45 (t, J = 6 Hz, 2 H), 3.38 (s, 3 H), 3.22 (m, 1 H), 2.45 (m, 1 H), 2.35 (m, 1 H), 2.00–1.00 (m, 10 H), 1.09 (s 3 H), 0.09 (s, 9 H). **11**: 6.02 (d, J = 9.8 Hz, 1 H), 5.77 (dd, J_1 = 9.8 Hz, J_2 = 5.2 Hz, 1 H), 3.70 (s, 3 H), 3.63 (t, J = 6 Hz, 2 H), 3.38 (m, 1 H), 2.09 (m, 1 H), 2.00 (m, 1 H), 1.80–1.10 (m, 11 H), 1.20 (s, 3 H).

assigned the stereostructure 7 in 85% yield.[19,22] Examination of the crude reaction mixture by high field (300 MHz) NMR indicated that the mixture contained less than 1% of stereoisomeric adducts. We were unable to isolate any minor cycloadducts if they were present. *Thus, the stereoselectivity for the cyclization of 6 is ≥100:1, a 20-fold enhancement over 1.* We confirmed the stereostructure of 7 by conversion to acid 8. Removal of the Me_3Si group occurred smoothly, concomitant with cleavage of the *tert*-butyl ester, upon treatment of 7 with anhydrous HF(g) in CH_2Cl_2/(room temperature/15 min), affording 8 in 92% yield, identical with that prepared by cyclization of 1.[19,23]



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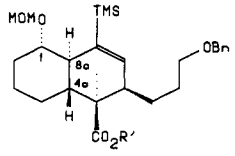
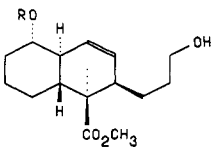
Encouraged by this result, we prepared triene 9, a much more demanding test, by the route outlined in Scheme II.[19] Introduction of the Me_3Si group and development of the required olefin geometry at an early stage were important elements of this successful approach.[24]

As expected, 9 proved to be significantly less reactive than the system 4 studied by Roush. Thermolysis of 9 at 180 °C for 24 h (concentration $\sim 10^{-4}$ M) afforded cleanly *a single cycloadduct* (>100:1) in 89% yield. If the reaction is conducted at higher concentrations (e.g. $\sim 10^{-2}$ M), significant amounts of bimolecular dimer form. The stereostructure of the adduct was tentatively assigned as 10 on the basis of difference decoupling experiments which established that the coupling constants $J_{1,8_a} = 10.4$ Hz and $J_{4_a,8_a} = 10.5$ Hz were consistent with a trans diaxial relationship between H_{8_a} and both H_1 and H_{4_a}.[19] Therefore in the cyclization of 9, a very demanding case, *stereocontrol resulting from introduction of the Me_3Si group provided an ~600-fold increase in stereoselectivity over 4.*

To confirm the structural assignments unequivocally, we converted 10 to diol ester 11, previously prepared and



10



11, R = H
12, R = MOM

correlated with a degradation product of chlorothricolide by Roush.[13] Treatment of 10 with BF_3–Et_2O (6 equiv/EtSH (12 equiv) for 24 h at room temperature resulted in concomitant cleavage of the benzyl and methoxymethyl ethers, as well as cleavage of the trimethylsilyl group to afford 11 in 84% yield.[19,25] This material was identical in all respects with a sample of 11 prepared from acid 12 by esterification (CH_2N_2/Et_2O) and cleavage of the methoxymethyl ether (BF_3–Et_2O/PhSH).[13,26]

Thus, use of strategically positioned stereochemical control elements constitutes a powerful protocol applicable to the preparation of hydrindene and octalin systems with essentially complete stereocontrol (≥100:1). The only apparent restriction is the currently limited number of general synthetic methods which permit ready introduction of the required trimethylsilyl group with high geometrical control.

Further studies extending this concept to other substitution patterns and to applications to natural products synthesis are currently in progress.

Acknowledgment. We gratefully acknowledge financial support for this investigation through grants from the National Science Foundation (CHE-81-19823) and the National Institute for General Medical Sciences of the NIH (GM-29290).

(20) For examples of the use of bis TMS esters in the Peterson reaction and the effects of counterion on geometry, see: Hartzell, S. L.; Rathke, M. W. *Tetrahedron Lett.* **1976**, 2737. Larcheveque, M.; Debal, A. *J. Chem. Soc., Chem. Commun.* **1981**, 877.

(21) The results of our studies on the origin of the observed geometric selectivity in the process will be reported elsewhere: Boeckman, R. K., Jr.; Chinn, R. L. *Tetrahedron Lett.*, in press.

(22) The rate of cyclization of 6 is ~10× slower than 1 ($t_{1/2} \simeq 3$ h (165 °C) vs. ≃0.33 h (150 °C)).

(23) Comparison was made with acid 8 obtained by cleavage of the *tert*-butyl ester of the major cycloadduct derived from 1 (150 °C, 3 h) with trifluoroacetic acid in CH_2Cl_2 at room temperature (0.5 h).

(24) The nature of the substrates in this case prevents introduction of the TMS group at a late stage by silylation of a carbanion. A kinetic method must be utilized to access the required trisubstituted TMS olefin since the *Z* isomer is much less stable than the related *E* isomer.

(25) Kieczykowski, G. R.; Schlessinger, R. H. *J. Am. Chem. Soc.* **1980**, *102*, 782.

(26) We thank Professor Roush for providing a generous sample of authentic acid 12 to permit the structural correlation.

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Alkylation of Pyridine in Free Radical Chain Reactions Utilizing Alkylmercurials[1]

Summary: Pyridines or *N,N,N′,N′*-tetramethyl-*p*-phenylenediamine will undergo a photostimulated free radical chain reaction with alkylmercury halides or carboxylates, yielding ring alkylated substitution products. Alkene mercuration products ($R^1CH(Y)CH(R^2)HgX$ with Y = HO, RO, CH_3CONH; X = Cl, CH_3CO_2, CF_3CO_2) can be used without isolation for the alkylation reaction.

Sir: Alkylmercury halides are convenient sources of free alkyl radicals in chain reactions.[2-4] Among the reactions which will regenerate R· from RHgX (Scheme I) are S_H2 attack at X (X = $PhCH_2$, H),[3,5] electron transfer (X = halogen, carboxylate),[2] and S_H2 substitution at Hg (X = alkyl, halogen).[3,4] Chain reactions involving electron transfer ($S_{RN}1$) have been observed in which primary, secondary, or tertiary alkyl radicals have been added to anions such as NO_2^-, N_3^-, $R^1R^2C{=}NO_2^-$, $R^1R^2C{=}C(O^-)Ph$, $Ph_2C{=}C{=}N^-$, $PhC(CO_2Et)_2^-$, or phthalimide$^-$ to generate $RA^{-}\cdot$ of Scheme I.[2] Neutral radicalphiles (πH) which can generate an easily oxidized adduct ($R\dot{\pi}H$), or an adduct readily converted to $R\pi^{-}\cdot$, can also participate in chain reactions of the $S_{RN}1$ type (Scheme II). Pyridines, quin-

(1) Electron Transfer Processes. 38. This work was supported by grants from the National Science Foundation (CHE-8119343) and Petroleum Research Fund (14784-AC4).

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