Table II. A Selection of pK_a Values Showing the Effect of Using Different log [H^+] Values for EAM, BOM and BSOM Calculations

		EAM		BOM		BSOM	
	HAFM[a]	pK_a[b]	pK_a[c]	pK_a[b,d]	pK_a[c,d]	pK_a[b,e]	pK_a[c,e]
phenyl methyl sulfoxideH^+[f]	−2.72	−2.32 ± 0.06	−2.47 ± 0.06	−2.29 ± 0.06	−2.37 ± 0.06	−2.40 ± 0.06	−2.55 ± 0.06
3-chlorophenyl methyl sulfoxideH^+[f]	−2.99	−2.79 ± 0.20	−2.94 ± 0.21	−2.55 ± 0.15	−2.63 ± 0.14	−2.86 ± 0.21	−3.02 ± 0.21
4-nitrophenyl methyl sulfoxideH^+[f]	−3.37	−2.76 ± 0.13	−2.91 ± 0.13	−2.78 ± 0.11	−2.87 ± 0.11	−2.82 ± 0.14	−2.98 ± 0.14
reserpiline[g]	−7.26	−6.09 ± 0.14	−6.23 ± 0.15	−6.07 ± 0.09	−6.05 ± 0.09	−6.28 ± 0.20	−6.45 ± 0.16
yohimbine[g]	−8.30	−6.22 ± 0.16	−6.38 ± 0.16	−6.68 ± 0.08	−6.67 ± 0.08	−6.33 ± 0.15	−6.49 ± 0.16
ajmalicine[g]	−8.31	−6.70 ± 0.12	−6.85 ± 0.12	−7.19 ± 0.11	−7.17 ± 0.11	−6.81 ± 0.12	−6.97 ± 0.13
3-methyl-2,4,6-trinitroanilineH^+[h]	−8.33	−6.95 ± 0.06	−7.99 ± 0.14	−8.30 ± 0.07	−8.30 ± 0.06	−7.88 ± 0.27	−9.07 ± 0.45
2,4,6-trinitroanilineH^+[h]	−10.03	−7.16 ± 0.18	−10.18 ± 0.14	−9.51 ± 0.09	−9.68 ± 0.06	−10.35 ± 0.19	−15.38 ± 0.35
3-bromo-2,4,6-trinitroanilineH^+[h]	−9.34	−8.14 ± 0.31	−10.47 ± 0.17	−10.48 ± 0.27	−10.15 ± 0.21	−11.02 ± 0.30	−14.42 ± 0.29

[a] References 13–15. [b] Calculated by using log [H^+] = log $M_{H_2SO_4}$; ref 16. [c] Calculated by using [H^+] from ref 12. [d] Calculated by using H_0 from ref 15. [e] Calculated by using X function from ref 17. [f] Calculated from UV data measured in aqueous H_2SO_4: values may differ from averaged values quoted elsewhere. Unpublished ionization ratio data supplied by Professor G. Scorrano. [g] Calculated from unpublished ionization ratio data supplied by Professor M. Balon. [h] Calculated from ionization ratio data of ref 15: values may differ from averaged values quoted elsewhere.

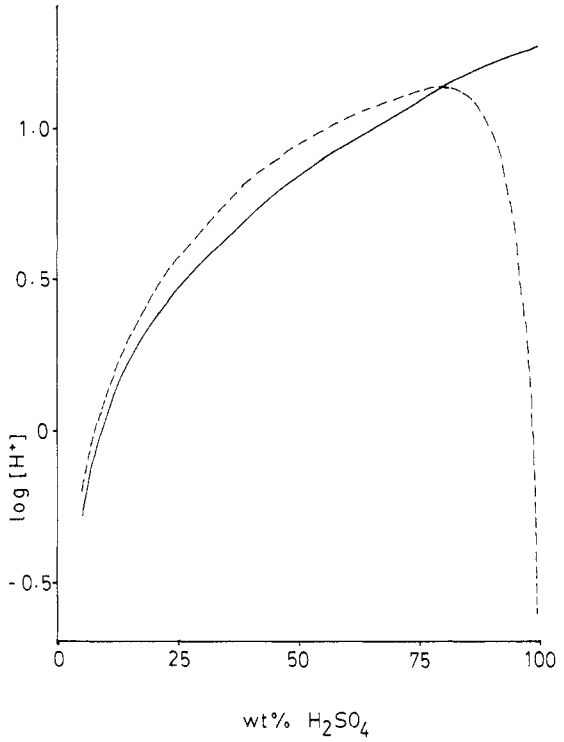

Figure 2. Comparison of operational log [H^+][12] (broken line) and log $M_{H_2SO_4}$[16] (solid line).

an ultimate quantitative solution to these problems.

Second, no completely systematic algorithm for calculation of pK_a values can replace the process involved in the HAFM where an arbitrary decision is made on what range of slope parameters constitutes obedience to a given acidity function, to what extent these deviations represent real differences, and to what extent are the experimental errors. Therefore, for the EAM to function optimally, it is necessary that the log I values employed in eq 2 are measured to the greatest possible accuracy.

Note Added in Proof. A referee has pointed out that, while the HAFM does not make an explicit reference to log [H^+], the EAM and BOM do. Moreover, Cox and Yates use [H^+] = [H_3O^+], which they describe as operational [H^+] values,[12] while Bunnett and Olsen refer to [H^+] as the sulfuric acid molarity.[3] The difference between these two definitions is greatest at high acidities, as shown in Figure 2. We have found that, in practice, the choice of log [H^+] values has generally only a small effect on the pK_a values calculated by any one method, usually well within experimental error, at the lower acidities. This is demonstrated in Table II, where the same data are treated, using the two definitions of log [H^+], by the EAM and BOM and the X function method recently published by Bagno, Scorrano, and More O'Ferrall (BSMOM).[17] For data measured in low to medium acid concentrations, the pK_a values calculated by one method using the different log [H^+] values are generally within 1 standard deviation of each other. This is also true for the BOM at high acidities, but not for the X function methods (EAM, BSMOM). This is another factor, therefore, which at the present time makes use of these extrapolative methods somewhat uncertain.

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(17) Bagno, A.; Scorrano, G.; More O'Ferrall, R. A. *Rev. Chem. Intermed.* **1987**, *7*, 313. This X function was derived from the data used by Cox and Yates[12] and a new set of log [H^+] values designed to incorporate all species capable of protonating the organic base. It is useful to note that these log [H^+] values are numerically almost identical with the log of the sulfuric acid molarity used for the BOM.[16]

Formation of Chiral Alkoxy Dienes in Wittig/Michael Reactions of 2,3,5-Tri-*O*-benzyl-D-arabinose

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In the course of our program[1] to develop new antidiabetic drugs based on carbohydrates, we prepared *C*-arabinofuranosides **1a** and **1b** by treatment of 2,3,5-tri-*O*-benzyl-D-arabinose (**2**) with (cyanomethylene)triphenylphosphorane[2] (86% yield as a 76:24 mixture of **1a** and **1b**, see Scheme I).[1c] Cyclization of the intermediate olefin **3** had occurred spontaneously under the reaction condi-

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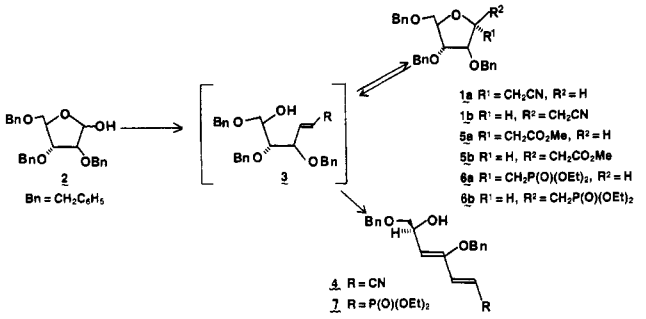
0022-3263/87/1952-4800$01.50/0

Scheme I



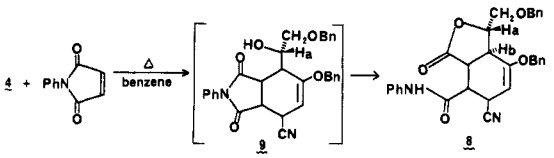
tions. The observed stereoselectivity in this reaction was attributed to a kinetic preference.[1c] Additionally, **1a** and **1b** have been prepared from **2** and the anion derived from diethyl (cyanomethyl)phosphonate (1.1 molar equiv), using either lithium hexamethyldisilazide (LiHMDS) or NaHMDS (1.1 molar equiv) in yields of 70% and 54%, respectively. However, treatment of **2** with diethyl (cyanomethyl)phosphonate (1.1 molar equiv) in the presence of 2.7 molar equiv of LiHMDS led to a 43–72% yield of diene **4**. The Wittig reaction product **3** had lost the elements of benzyl alcohol. An NOE NMR experiment on **4** revealed an *E,Z* geometry, as shown, indicating that anti (E2) elimination occurred. Diene **4** was also prepared from **1a** and **1b** upon treatment with LiHMDS (1.1 molar equiv, ca. 50%).

Although **4** was fairly stable to normal manipulations, it partially decomposed to unidentified products over a 4-month period at room temperature; storage in the cold (<0 °C) is recommended. The facile elimination of benzyl alcohol from **3** in the presence of a base is probably due to the acidity of the proton on C-4, which is enhanced by the cyano group (C-1). Esters **5a** and **5b** undergo base-catalyzed equilibration uneventfully (presumably through an olefin such as **3**).[1c] Reaction of **2** with the anion of tetraethyl methylenebisphosphonate leads to a 50–80% yield of the expected *C*-glycosides as a 2:1 ratio of **6a** and **6b**.[3] When we performed this reaction, some diene **7** (ca. 15%, unstable at room temperature) was also isolated and partially characterized (^{1}H NMR, UV). The identification of **4** and **7** by their UV absorption was particularly useful because they have large extinction coefficients (**4**, ϵ 10 228 at 261.5 nm; **7**, ϵ 10 225 at 250 nm).[4]

Several reactions have been described that produce dienes from perbenzylated, pyranose sugars.[5-7] Additionally, the elimination of benzyl alcohol during sodium borohydride reduction was observed with benzylated furanoses as well as pyranoses.[6]

Since diene **4** is only a single stereo and geometrical isomer, it is an excellent substrate for an asymmetric Diels–Alder reaction.[8-11] Also, **4** bears an alkoxy substituent cis to the asymmetric carbon, which can lead to pronounced stereochemical bias upon reaction with the alkene.[9] Diene **4** underwent a Diels–Alder reaction with *N*-phenylmaleimide to give a 51% yield of a major substance, assigned as lactone **8**, and a 16% yield of a minor isomeric substance (see Scheme II). The major product arose by transacylation in the originally formed endo cycloadduct **9**.[8,9] The stereoselectivity in the Diels–Alder reaction to give lactone **8** is that predicted by Franck,[8] and is the same sense as reported for an analogous reaction of a diene bearing a *Z*-alkoxy group (similar to **4**).[9] Our structural assignment was based on IR and NMR data. Both the ^{13}C and ^{1}H NMR data for **8** indicated a single stereoisomer, and ^{1}H NOE experiments confirmed the trans arrangement of H_a and H_b. The 90.6-MHz ^{13}C NMR spectrum displayed three singlets (the two carbonyl carbons and one of the olefinic carbons) at 176.6, 167.0, and 155.0 ppm, respectively, supporting a single substance.

Scheme II



Although the other possible diastereomer (the minor isomer in Franck's[8] work) was neither identified nor detected, the minor isomeric material was assigned as a maleimide adduct. This material diastereomerically related to **9** was not a diastereomer of **8** and did not suffer transacylation even under prolonged heating at 80 °C. We suggest that it is the isomer derived from addition of *N*-phenylmaleimide to the opposite face of **4**, which should be less prone to cyclization[8] since a trans 5,6 ring junction is unfavorable. If this assignment is correct, the facial selectivity in the Diels–Alder reaction of **4** with *N*-phenylmaleimide would be 3.2:1, which still reflects a bias for trans isomer **8**.

Experimental Section

General Methods. ^{1}H NMR spectra were recorded on a Bruker AM-360WB (360 MHz) spectrometer. ^{13}C NMR spectra were obtained on a JEOL FX-60Q (15.0 MHz) or a Bruker AM-360WB (90.6 MHz) spectrometer as indicated. All NMR experiments were conducted in $CDCl_3$ with tetramethylsilane as an internal standard. Elemental analyses were performed by Atlantic Microlab, Inc. (Atlanta, GA). TLC analysis was conducted on Whatman MK6F 200-μm silica gel plates. Chemical ionization mass spectra (CI-MS) were recorded on a Finnigan 3300-6100 system with methane as the reagent gas. Compound **2** was purchased from Pfanstiehl Laboratories (Waukegan, IL).

***C*-Glycosides 1a and 1b.** The preparation of **1a** and **1b** from **2** and (cyanomethylene)triphenylphosphorane has already been described.[1c] The preparation of **1a** and **1b** from diethyl (cyanomethyl)phosphonate is presented below.

To a solution of **2** (0.42 g, 1.0 mmol) and diethyl (cyanomethyl)phosphonate (0.25 g, 1.4 mmol) in 5 mL of THF at −78 °C was added 1.1 mL of 1 M LiHMDS in THF. The reaction was allowed to warm to 0 °C by replacing the acetone–dry ice bath with an ice–water bath. After stirring for 2 h at 0–15 °C, the reaction mixture was treated with water, the product was extracted into ether, dried ($MgSO_4$), and concentrated. The crude product was chromatographed by preparative TLC (ethyl acetate/hexane, 3:7) to give 310 mg of **1a** and **1b** as an oil (70%), identical in all respects with **1a** and **1b** prepared previously. ^{13}C NMR indicated a 74:26 ratio of **1a** to **1b**.[1c] The reaction of **2** and diethyl (cyanomethyl)phosphonate with NaHMDS proceeded in the same

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(4) The wavelength at which maximal absorption occurs for **4** and **7** is very close to that predicted by Woodward's rules (calculated to be 250 nm). Pasto, D. J.; Johnson, C. R. *Organic Structure Determination*; Prentice-Hall, Inc., Englewood Cliffs, NJ, 1969; p 96.

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manner to give a 56% yield of **1a** and **1b**.

Diene 4. A. Preparation from 2. To a solution of **2** (18.94 g, 45 mmol) and diethyl (cyanomethyl)phosphonate (8.77 g, 45 mmol) in 175 mL of THF at 0 °C was added 100 mL of 1 M LiHMDS in THF. After stirring for 4.5 h at ambient temperature, considerable starting material remained by TLC, so additional phosphonate (0.88 g, 4.5 mmol) and 1 M LiHMDS in THF (20 mL, 20 mmol) were added at room temperature and the solution was stirred overnight. The reaction mixture was then treated with water, and the product was extracted into ether, washed with brine, dried ($MgSO_4$), filtered, and concentrated. The crude product was chromatographed on a Waters Prep-500 HPLC (ethyl acetate/hexane, 35:65) to give 6.50 g of **4** (43%): $[\alpha]^{20}_D$ +22.9° (c 0.20, CCl_4); MS, *m/e* 336 (M + 1); UV (MeOH) λ_{max} 261.5 nm (ϵ 10 228); ^1H NMR δ 2.47 (m, 1 H), 3.34 (dd, 1 H, *J* = 7.0, 9.6 Hz, C_7H_a), 3.45 (dd, 1 H, *J* = 3.7, 9.6 Hz, C_7H_b), 4.51 (m, 2 H), 4.69 (m, 1 H, *J* = 3.7, 7.0, 8.6 Hz, C_6H), 4.80 (m, 2 H, $PhCH_2O$ on C_4), 5.42 (d, 1 H, *J* = 8.6 Hz, C_5H), 5.58 (d, 1 H, *J* = 16.1 Hz, C_2H), 6.77 (d, 1 H, *J* = 16.1 Hz, C_3H), 7.3 (m, 10 H) [2D-NOESY data indicated a positive interaction of the resonance at 6.77 ppm with those at 5.58 and 5.42 ppm, as well as the resonance at 5.58 ppm with those at 4.80 and 6.77 ppm, indicating the *E,Z* geometry]; 15.0-MHz ^{13}C NMR δ 66.0 (d, C-6), 73.1 (t), 73.5 (t), 74.3 (t), 98.1 (d, C-3), 117.4 (q, C-1), 125.6 (d), 127.0 (d), 127.8-128.7 (10 C), 136.3 (q), 137.7 (q), 145.8 (d, C-5), 152.5 (q, C-4). Anal. Calcd for $C_{21}H_{21}NO_3$: C, 75.21; H, 6.31; N, 4.18. Found: C, 74.46; H, 6.30; N, 4.15.

B. Preparation from 1a and 1b. To a 76:24 mixture of **1a** and **1b** (110 mg, 2.5 mmol) in 5 mL of THF was added 0.25 mL of 1 M LiHMDS in tetrahydrofuran. After 2 h of stirring at ambient temperature, the reaction mixture was treated with ether, washed with water, dried ($MgSO_4$), filtered, and concentrated. The crude material was chromatographed by preparative TLC (ethyl acetate/hexane, 3:7) to give 40 mg of **4** (48%).

Diels-Alder Reaction of 4 with *N*-Phenylmaleimide. A solution of **4** (1.0 g, 3.0 mmol) and *N*-phenylmaleimide (0.78 g, 4.5 mmol) in 10 mL of benzene was heated at reflux for 21 h. The solution was concentrated, and 10% of the product was purified on silica gel (ethyl acetate/hexane; 45:55). The major band was isolated, giving 77 mg of **8** (51% yield). With the exception of the isomer described below, each UV-active or H_2SO_4-charring impurity constituted <5% of the product. Upon standing the product solidified (mp 80-95 °C, dec). The lactone structure was inferred by ^1H NMR, IR, and literature precedence.[8,9] A 2D-COSY NMR experiment was run on this compound in order to make the ^1H assignments listed below; $[\alpha]^{20}_D$ -104° (c 0.70 CH_2Cl_2); MS, *m/e* 509 (M + 1), 537 (M + 29); IR (KBr) λ_{max} 3300-3550 cm^{-1} (N-H stretch); 2900-3000: 2250 (CN); 1777 (lactone stretch); 1678, 1600 (amide stretch), 1547 (N-H bend); 1497; 1444; 1200. ^1H NMR δ 3.16 (t, 1 H, *J* = 4.8 Hz, *CH*CONH), 3.30 (ddd, 1 H, *J* = ca. 0.5, ca. 1.0, 8.8 Hz, H_b coupled with C=C*H*, H_a, and $CHCO_2$),[12] 3.64 and 3.81 (both dd, *J* = 2.2, 11.0 Hz, 1 H each, CH_2OCH_2Ph coupled geminally and with H_a), 3.8 (m, 1 H, $CHCO_2$), 3.90 (ddd, 1 H, *J* = 5.2, 5.2, 1.5 Hz, *CH*CN coupled with *CH*CON and C=C*H*), 4.47 (d, 1 H, *J* = 12.0 Hz), 4.58 (d, 1 H, *J* = 12.0 Hz), 4.73 (d, 1 H, *J* = 11.2 Hz), 4.80 (d, 1 H, *J* = 11.3 Hz), 4.89 (q, 1 H, *J* = 2.3 Hz, H_a), 5.01 (dd, 1 H, *J* = ca. 0.3, 5.3 Hz, C=C*H*), 7.12 (t, 1 H, *J* = 7.3 Hz, 4-*H*PhNH), 7.2-7.4 (m, 12 H), 7.60 (d, 2 H, *J* = 8.6 Hz, 2-*H*PhNH), 10.0-10.4 (br s, 1 H, N*H*) [Irradiation of the resonance at 3.63 ppm resulted in enhancements of 5% and 8% at 3.33 and 4.88 ppm, respectively. Irradiation at 3.83 ppm gave enhancements of 9% and 10% at 3.33 and 4.88 ppm, respectively]; 90.6-MHz ^{13}C NMR δ 27.4 (d), 39.8 (d), 43.5 (d), 70.0 (t), 71.2 (t), 73.7 (t), 81.1 (d), 92.1 (d), 118.1 (q), 120.7 (d), 127-129 (d, 14C), 135.5 (q), 137.2 (q), 137.6 (q), 155.0 (q), 167.0 (q), 176.6 (q). Anal. Calcd for $C_{31}H_{28}N_2O_5$: C, 73.21; H, 5.55; N, 5.51. Found: C, 73.79; H, 6.03; N, 5.20. High-resolution mass spectrum, *m/e* calcd for $C_{31}H_{28}N_2O_5$ 508.19982, found 508.19240.

Also isolated was 24 mg (16%) of an isomeric material that did not convert to **8** upon continued refluxing in benzene, although there was decomposition to several near-origin spots on TLC (ethyl acetate/hexane; 45:55). It did not have the N*H* singlet at 10-10.4 ppm in the ^1H NMR spectrum, as does **8**: mp 50-65 °C dec; MS, *m/e* 509 (M + 1); IR (KBr) λ_{max} 3280-3550 cm^{-1}, 2880-2970, 1777, 1693, 1601, 1545, 1499, 1445, 1162; ^1H NMR δ 2.7 (m, 2 H), 3.27 (m, ca. 1 H), 3.39-4.09 (m, ca. 4 H), 4.37-4.62 (m, 2 H), 4.66-4.87 (m, 2 H), 5.17 (m, ca. 1 H), 7.1 (d, ca. 1 H), 7.16-7.40 (m, ca. 12 H), 7.5-7.64 (m, ca. 1 H). High-resolution mass spectrum, *m/e* calcd for $C_{31}H_{28}N_2O_5$ 508.19982, found 508.20001.

Acknowledgment. We thank Dr. G. Caldwell and J. Masucci for mass spectral results and M. S. Mutter for ^1H NMR data. We also thank Professors R. W. McClard (Reed College) and R. W. Franck (Hunter College) for helpful suggestions.

Registry No. **1a**, 110116-77-3; **1b**, 110116-78-4; **2**, 37776-25-3; **4**, 110116-79-5; **8**, 110116-80-8; diethyl (cyanomethyl)phosphonate, 2537-48-6; *N*-phenylmaleimide, 941-69-5.

(12) Professor Franck (private communication, 1986) has indicated that the ^1H NMR data of his lactones[8] were actually for compounds epimeric at the carbon bearing the CONHPh functionality. The correct coupling constant for H_a and H_b of the trans-configured major isomer is 2.4 Hz (instead of 7.3 Hz).

Chemistry of 3,4-Epoxy Alcohols. Fragmentation Reactions

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Recent interest in the chemistry of 3,4-epoxy alcohols has centered on applications toward the synthesis of the taxane ring system,[1] on models for the biosynthesis of secolongifolene[2] and the antitumor xanthanolides,[3] and on studies related to the structural effects governing the formation of oxetanes[4] and fragmentation products.[5-7] Despite this attention, factors favoring fragmentation over other possible reaction pathways (e.g., addition to epoxide) need to be more clearly recognized.[5,6] To this end, we have treated selected 3,4-epoxy alcohols with excess aluminum isopropoxide and report herein some significant results.

1,2-Epoxy-4-hydroxy-4-methylnonane (**1**) was prepared by addition of allylmagnesium bromide to 2-heptanone followed by epoxidation of the tertiary alcohol with MCPBA.[5] Compound **1**, presumably a mixture of stereoisomers, was treated with 5 equiv of aluminum isopropoxide in refluxing toluene for 70 h. A complex mixture of products resulted, containing a 33% yield (by GC) of 2-heptanol, identified by GC/MS analysis and by GC comparison to authentic material. 2-Heptanol is formed in this reaction by a Grob-type fragmentation followed by a Meerwein-Ponndorf reduction of 2-heptanone.[8] We have reported that the action of potassium *tert*-butoxide converted **1** into the oxetane (by intramolecular nucleophilic addition) and the *tert*-butoxide adduct, with no detectable fragmentation.[5] Thus, for fragmentation to

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