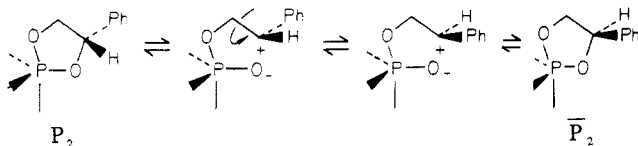


rotational freedom of the carbocation would obviously racemize P_2 (i.e., $P_2 \rightleftharpoons \bar{P}_2$), and through pseudorotation, P_1 is also racemized; therefore, oxyphosphonium betaines A and B would be without stereochemical integrity as would the final product, styrene oxide.



The thermodynamic facility for closure of chains to three- and five-membered rings is often quite similar, and there exists extensive documentation for a host of different reactions.²⁴ Because of the similarity in energetic considerations, we anticipated that the cyclodehydration of chiral 1,4-diols would show regioselectivity for tetrahydrofuran formation paralleling that observed for the conversion of chiral 1,2-diols to epoxides (assuming the R' group is the same). We examined the reaction of (*R*)-(-)-pentane-1,4-diol (**3**) with DTPP, TPP- CCl_4 - K_2CO_3 , and TPP-($\text{C}_2\text{H}_5\text{CO}_2\text{N}$)₂ and found that (*R*)-(-)-2-methyltetrahydrofuran was the predominant enantiomer reflecting largely retention of stereochemistry at C2. The percent regioselection ranged from 81-88% and is in accord with the results for formation of (*S*)-propylene oxide (vide supra).

Acknowledgment is made to the National Science Foundation (Grant CHE-78-05921) for support of this research. We also thank Dr. David L. Harris for recording some of the ¹³C NMR spectra related to this work. We are grateful to M & T Chemicals, Inc., for generous samples of triphenylphosphine.

Registry No. 1, 4254-15-3; 2, 25779-13-9; 3, 56718-04-8; DTPP, 18509-25-6; TPP, 603-35-0; CCl_4 , 56-23-5; K_2CO_3 , 584-08-7; ($\text{C}_2\text{H}_5\text{COON}$)₂, 1972-28-7; styrene oxide, 67253-49-0; diethyl peroxide, 628-37-5; (*S*)-(-)-ethyl lactate, 687-47-8; (*S*)-(+)-mandelic acid, 17199-29-0; glutamic acid, 56-86-0; (*S*)-(+)-propylene oxide, 16088-62-3; (*R*)-(-)-2-methyltetrahydrofuran, 63798-13-0; (*S*)-(+)-styrene oxide, 20780-54-5.

Supplementary Material Available: Full experimental details for compounds 1-3 (5 pages). Ordering information is given on any current masthead page.

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Allylboronate Synthesis. Synthesis of a β -Alkoxy Carbanion Equivalent

Summary: A stereospecific synthesis of allylboronates has been developed by the reaction of vinylolithium reagents with α -chloroboronic esters. This approach enables the inclusion of diverse substitution patterns as well as the

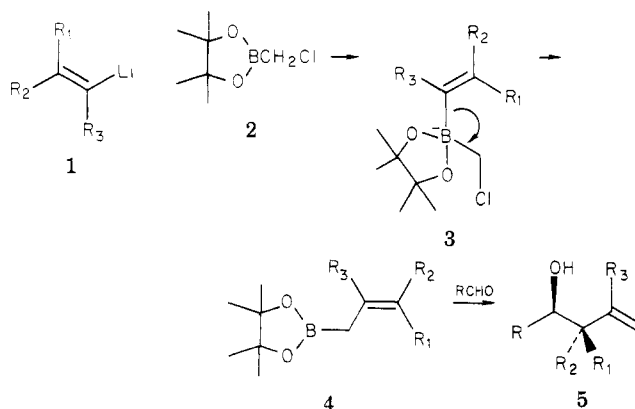
inclusion of a variety of functional groups.

Sir: The isolation and structural elucidation of a diversity of biologically important propionate and acetate derived natural products from fungal and bacterial phyla has led to an intense effort in the development of methodology for the assembly of acyclic molecular substructures by a number of groups.¹

Our interest in the macrolides and ionophores has led us to explore the application of allylboronates in their synthesis, primarily because of their known ability to condense with aldehydes in a stereospecific manner,² their neutrality, their low reduction potential,³ their chemoselectivity,⁴ and the potential for securing them in a geometrically homogeneous form.⁵

In general, allylboronates are most conveniently prepared by the addition of a suitable Grignard or lithium reagent to borate esters, boron trihalides, and haloborate esters⁶ or by transmetalation of allyltin reagents with chloroborate esters.⁷ Although these approaches are experimentally simple, they suffer from a general lack of regio- and stereospecificity as well as the inability to include leaving groups in the δ -position of the allyl Grignard or lithium reagents due to elimination. In light of the

Scheme I



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(3) We have found that allylboronates react cleanly with α -alkoxy aldehydes whereas Cr(II)-derived allyl species result in reductive removal of the alkoide. Note entry 2 in Table I. Wuts, P. G. M.; Bigelow, S. S. *J. Org. Chem.*, in press. Rausch, W. R.; Harris, D. J.; Lesur, B. M. *Tetrahedron Lett.* 1983, 24, 2227.



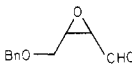










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Table I. Synthesis of Allylboronates and Their Reactions with Aldehydes

entry	vinyllithium	E/Z allylboronate ratio	% yield boronate ^b	RCHO	homoallylic alcohol	
					threo/erythro ratio ^c	% yield ^e
1		10:1	56	PhCHO	10:1	97
2		10:1			9:1 ^d	95
3		1:20	47	PhCHO	1:20	97
4			<i>a</i>	PhCHO		80
5		1:10	52	PhCHO	1:9	80
6			57	PhCHO	>100:1	90
7			<i>a</i>	PhCHO		56
8						
8		13:1	41	AcOCH ₂ CH ₂ CHO	13:1	71
9		13:1	<i>a</i>	PhSCH ₂ CH ₂ CHO	13:1	50
10		13:1	<i>a</i>	C ₅ H ₁₁ CHO	13:1	86
11		13:1	<i>a</i>	(<i>E</i>)-PhCH=CHCHO	13:1	62

^a Not isolated. ^b Yield of isolated distilled material based on chloromethaneboronate. ^c Ratios determined by 360-MHz NMR. ^d Represents ratio of Cram/anti-Cram addition. ^e Chromatographically isolated material.

above-mentioned limitations, we have explored an alternate route to allylboronates which overcomes these limitations. The approach was based on the pioneering work of Brown,⁸ Matteson,⁹ and others,¹⁰ who demonstrated the ability of α -halo boronic esters to undergo clean substitution reactions with a variety of nucleophiles.

Scheme I illustrates the synthesis of a variety of allylboronates from readily available vinyllithium reagents 1¹¹ and pinacol chloromethaneboronate 2.¹² The reaction

proceeds by initial addition of the vinyllithium to boron to form the ate complex 3 which then undergoes migration with elimination of chloride. Each of the resulting allylboronates 4 undergoes highly stereoselective condensations with aldehydes to give homoallylic alcohols 5. In all cases the threo/erythro selectivity corresponds to the isomeric purity of the precursor boronate as previously established.² The entire process proceeding from the vinyllithium 1 to homoallylic alcohols 5 may either be carried out as a two-step procedure in which the allylboronate is first isolated and then condensed with the aldehyde either neat or in methylene chloride solution or as a one-pot procedure where 4 is generated in situ in tetrahydrofuran or ether and then treated directly with the aldehyde. In employing the in situ procedure, the reactions with aldehydes are somewhat slower, presumably because of the complexing ability of the ether solvent.

Also, the in situ procedure requires the use of 2 equiv of the vinyllithium 1 and chloride 2 since the yields of allylboronates are generally in the range of 50%. At this time we do not understand the limited yield of 50% which is in contradistinction to Brown's earlier results, but the reaction is entirely reproducible on both large and small scales. Matteson¹² recently noted in a related reaction that ZnCl₂ catalysis improved the yields of similar substitutions,

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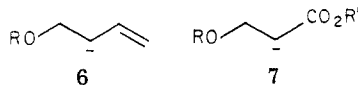
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but we have found that our reactions are not affected.

Examination of Table I reveals that the reaction proceeds with complete regio- and stereochemical control with a variety of vinylolithiums. The *Z/E* ratios were determined by examination of the 360-MHz NMR spectra and reflect those of the precursor vinyl halides. The ortho ester (entry 7) is of interest in that it provides ready access to α -methylene lactones. Of greater interest is the δ -alkoxyallylboronates (entries 8-11) in that these provide the first operational equivalent to α -alkoxy carbanions 6 and 7.¹⁴



In conclusion we have developed a regio- and stereo-specific synthesis of allylboronates which is operationally simple and may be used to prepare reagents with diverse substitution patterns. The attractiveness of this approach is further augmented by the large number of methods available for the preparation of stereochemically pure vinylolithium reagents¹¹ and precursor vinyl halides¹³ with a variety of functionality. We are currently exploring the application of this methodology in natural product synthesis.¹⁵

Registry No. (*E*)-1 ($R_1 = R_3 = H, R_2 = Me$), 6386-72-7; (*Z*)-1 ($R_1 = R_3 = H, R_2 = Me$), 6524-17-0; 1 ($R_1 = R_2 = H, R_3 = Me$), 6386-71-6; 1 ($R_1 = R_3 = Me, R_2 = H$), 57012-95-0; 1 ($R_1 = R_2 = H, R_3 = C(OEt)_3$), 87938-75-8; 1 ($R_1 = R_3 = H, R_2 = THPOCH_2$), 87938-76-9; 2, 83622-42-8; (*E*)-4 ($R_1 = R_3 = H, R_2 = Me$), 69611-02-5; (*Z*)-4 ($R_1 = R_3 = H, R_2 = Me$), 69611-01-4; (*E*)-4 ($R_1 = R_3 = Me, R_2 = H$), 87938-71-4; (*Z*)-4 ($R_1 = R_3 = Me, R_2 = H$), 87938-72-5; (*E*)-4 ($R_1 = R_3 = H, R_2 = THPOCH_2$), 87938-73-6; (*Z*)-4 ($R_1 = R_3 = H, R_2 = THPOCH_2$), 87938-74-7; 5 ($R = Ph, R_1 = R_2 = R_3 = H$) (isomer 1), 52922-10-8; 5 ($R = Ph, R_1 = R_2 = R_3 = H$) (isomer 2), 52922-19-7; 5 ($R = 3$ -(benzyloxymethyl)oxiran-2-yl) ($R_1 = R_3 = H, R_2 = Me$), 87938-62-3; 5 ($R = Ph, R_1 = R_3 = Me, R_2 = H$) (isomer 1), 87938-63-4; 5 ($R = Ph, R_1 = R_3 = Me, R_2 = H$) (isomer 2), 87938-64-5; 5 ($R = AcOCH_2CH_2, R_1 = R_3 = H, R_2 = THPOCH_2$) (isomer 1), 87938-67-8; 5 ($R = AcOCH_2CH_2, R_1 = R_3 = H, R_2 = THPOCH_2$) (isomer 2), 87984-11-0; 5 ($R = PhSCH_2CH_2, R_1 = R_3 = H, R_2 = THPOCH_2$) (isomer 1), 87938-68-9; 5 ($R = PhSCH_2CH_2, R_1 = R_3 = H, R_2 = THPOCH_2$) (isomer 2), 87984-12-1; 5 ($R = C_5H_{11}, R_1 = R_3 = H, R_2 = THPOCH_2$) (isomer 1), 87938-69-0; 5 ($R = C_5H_{11}, R_1 = R_3 = H, R_2 = THPOCH_2$) (isomer 2), 87984-13-2; 5 ($R = (E)$ -PhCH=CH, $R_1 = R_3 = H, R_2 = THPOCH_2$) (isomer 1), 87938-70-3; 5 ($R = (E)$ -PhCH=CH, $R_1 = R_3 = H, R_2 = THPOCH_2$) (isomer 2), 87984-14-3; PhCHO, 100-52-7; AcOCH₂CH₂CHO, 18545-28-3; PhSCH₂CH₂CHO, 27098-65-3; C₅H₁₁CHO, 66-25-1; (*E*)-PhCH=CHCHO, 14371-10-9; α -(2-cyclohexen-1-yl)benzenemethanol (isomer 1), 87938-65-6; α -(2-cyclohexen-1-yl)benzenemethanol (isomer 2), 87938-66-7; 1-cyclohexenyllithium, 37609-34-0; 3-[(benzyloxy)methyl]oxirane-2-carboxaldehyde, 87938-77-0; 2-(2-cyclohexen-1-yl)tetramethyl-2-bora-1,3-dioxacycloheptane, 87938-78-1.

Supplementary Material Available: Experimental details for entries 2 and 11 of Table I (2 pages). Ordering information is given on any current masthead page.

(14) Schlessinger has prepared the anion of a β -alkoxy carboxylate, but this is not an anion equivalent. See: Herrmann, J. L.; Schlessinger, R. H. *Tetrahedron Lett.* 1977, 4575. Seebach later exploited this chemistry. See: Seebach, D. In "Modern Synthetic Methods 1980"; Scheffold, R., Ed.; Verlag Chemie: Weinheim/Bergstr., Germany, 1980; pp 91-171.
(15) We acknowledge support of this work by NIH and NSF.

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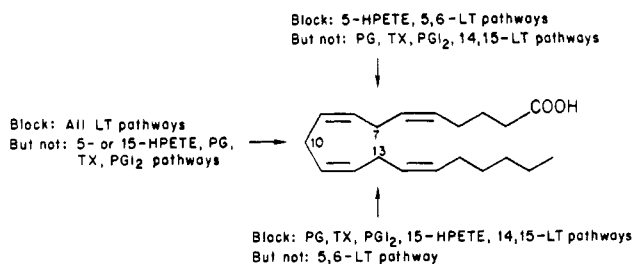
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Ethanoarachidonic Acids. A New Class of Arachidonic Acid Cascade Modulators. 1. Monoethano Compounds¹

Summary: The rational design, total synthesis, and preliminary biological data of ethanoarachidonic acids 1-3 are described.

Sir: Biosynthetic considerations of the various biologically active metabolites of arachidonic acid (AA) suggest that the major peroxidation pathways, including the cyclooxygenase pathway leading to prostaglandins and thromboxanes³ and the lipoxygenase pathways leading to mono- and polyhydroxyarachidonic acids and leukotrienes,⁴ begin with an enzymatic abstraction of a hydrogen radical from the bis-allylic position 7, 10, or 13. Therefore, by blocking one or more of these positions of arachidonic acid, it might be possible to "shut off" one or more peroxidation pathways at will.⁵ Such analogues of arachidonic acid should only undergo the "allowed" transformations and, furthermore, may prove to be selective inhibitors of certain enzymes of the AA cascade by successfully competing for receptors with the parent arachidonic acid or some of its early metabolites. This strategy for modulation of the AA cascade is summarized below.



As one of the most promising and convenient ways to block these active positions we considered the introduction of an ethano group in the form of a cyclopropane ring.⁶

(1) This work was partially disclosed at the 16th ACS-MARM meeting, Newark, DE, April 21-23, 1982.

(2) (a) Fellow of the A. P. Sloan Foundation, 1979-1983. (b) Recipient of a Camille and Henry Dreyfus Teacher-Scholar Award, 1980-1984. (c) J. S. Guggenheim Fellow, 1984. (d) NSF Minority Graduate Fellow, 1982-1985.

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