placed in an oven-dried, 500-mL flask fitted with a side arm and a bent adaptor connected to a Hg bubbler. The flask was immersed in an ice-water bath, and to this was added 196 mL of precooled 2,3-dimethyl-2-butene (1.65 mol) dropwise over a period of 1 h via a double-ended needle. The reaction mixture was stirred for an additional 2 h at 0 °C, followed by stirring overnight at room temperature. The resulting CH_2Cl_2 solution was found to be 3.34 M in ThxBHBr–SMe_2, and ^{11}B NMR showed a clean doublet centered at δ 5.16 (J_{BH} = 123 Hz).

Reduction of Carboxylic Acids and Isolation of Products. The following procedure for the reduction of cyclohexanecarboxylic acid is illustrative. An oven-dried, 100-mL flask, fitted with a side arm and a bent adaptor connected to a Hg bubbler, was charged with 6.79 g (53 mmol) of cyclohexanecarboxylic acid and 35 mL of CS_2.[9] The flask was immersed in a cold bath and maintained at −20 °C. A precooled 3.2 M solution of ThxBHBr–SMe_2 in CH_2Cl_2 (16.6 mL, 53.1 mmol) was added dropwise with stirring. After complete evolution of the H_2, the cold bath was removed, and the reaction mixture was warmed to room temperature. An additional 1.1 eqiv of the reagent (18.3 mL, 58.3 mmol, 10% excess) was added, and the reaction mixture was stirred for 1 h at room temperature. Analysis of an aliquot with (2,4-dinitrophenyl)hydrazine indicated a yield of 99%.

The rest of the reaction mixture (50 mmol) was transferred via a double-ended needle to a flask containing 50 mL of cold water in an ice–water bath and was then hydrolyzed with vigorous stirring for 1 h at room temperature. The mixture was saturated with NaCl, and the separated organic layer was subjected to the $NaHSO_3$ isolation procedure.[2] The yield of pure distilled cyclohexanecarboxaldehyde was 5.0 g (89%): bp 160–161 °C (761 mm); n_D^{20} 1.4498.

Reduction of Carboxylic Acid Salts and Isolation of Products. The following procedure is for the larger scale reaction. In the assembly previously described were placed 10.9 g of lithium diphenylacetate (50 mmol) and 17 mL of CH_2Cl_2. Into the reaction mixture was injected 33.3 mL of the 3 M reagent solution (100 mmol), and the slurry was stirred for 3 h at room temperature. The reaction mixture was then hydrolyzed with 50 mL of cold water by stirring vigorously for 1 h at room temperature. The mixture was saturated with NaCl. After neutralization with a small amount of $NaHCO_3$, the separated organic layer was poured into 75 mL of a saturated aqueous $NaHSO_3$ solution, and 70 mL of THF was added. The mixture was stirred for 1 h, by which time the crystalline bisulfite adduct of diphenylacetaldehyde had precipitated. The solution was cooled in an ice–water bath to ensure complete crystallization of the adduct, which was then collected by filtration and washed with 3 × 25 mL of pentane. The adduct was placed in 40 mL of water, and then 50 mL of THF and 8 mL of a 37% CH_2O solution were added. The mixture was stirred for 1 h and saturated with $MgSO_4 \cdot 7H_2O$. The organic layer was separated and dried. Evaporation of volatiles gave 8.93 g of analytically pure diphenylacetaldehyde (91%), n^{20}_D 1.5892.

Acknowledgment. The research support provided by the Korea Science and Engineering Foundation is gratefully acknowledged. We appreciate the reviewers' kind comments.

Registry No. 1 (R = CH_3), 64-19-7; 1 (R = CH_3, aldehyde), 75-07-0; 1 (R = $H_2C{=}C(CH_3)$, 79-41-4; 1 ($H_2C{=}C(CH_3)$), 78-85-3; 1 (R = $(CH_2)_2CH_3$), 107-92-6; 1·Na (R = $(CH_2)_2CH_3$), 156-54-7; 1-Li (R = $(CH_2)_2CH_3$), 21303-03-7; 1 (R = $(CH_2)_2CH_3$, aldehyde), 123-72-8; 1 (R = $(CH_2)_4CH_3$), 142-62-1; 1·Na (R = $(CH_2)_4CH_3$), 10051-44-2; 1·Li (R = $(CH_2)_4CH_3$), 16577-51-8; 1 (R = $(CH_2)_4CH_3$, aldehyde), 66-25-1; 1 (R = $(CH_2)_8CH_3$), 334-48-5; 1·Na (R = $(CH_2)_8CH_3$), 1002-62-6; 1·Li (R = $(CH_2)_8CH_3$), 20336-95-2; 1 (R = $(CH_2)_8CH_3$, aldehyde), 112-31-2; 1 (R = $(CH_2)_{16}CH_3$), 57-11-4; 1·Na (R = $(CH_2)_{16}CH_3$), 822-16-2; 1·Li (R = $(CH_2)_{16}CH_3$), 4485-12-5; 1 (R = $(CH_2)_{16}CH_3$, aldehyde), 638-66-4; 1 (R = $CH(CH_3)_2$), 79-31-2; 1·Na (R = $CH(CH_3)_2$), 996-30-5; 1·Li (R = $CH(CH_3)_2$), 25179-23-1; 1 (R = $CH(CH_3)_2$, aldehyde), 78-84-2; 1 (R = $CH_2CH(CH_3)_2$), 503-74-2; 1·Na (R = $CH_2CH(CH_3)_2$), 539-66-2; 1·Li (R = $CH_2CH(CH_3)_2$), 556-25-2; 1 (R = $CH_2CH(CH_3)_2$, aldehyde), 590-86-3; 1 (R = $C(CH_3)_3$), 75-98-9; 1·Na (R = $C(CH_3)_3$), 1184-88-9; 1 (R = $C(CH_3)_3$, aldehyde), 630-19-3; 1 (R = $C_6H_5CH_2$), 103-82-2; 1 (R = $C_6H_5CH_2$, aldehyde), 122-78-1; 1 (R = $(C_6H_5)_2CH$), 117-34-0; 1·Na (R = $C_6H_5)_2CH$), 1716-11-6; 1·Li (R = $(C_6H_5)_2CH$), 25017-18-9; 1 (R = $(C_6H_5)_2CH$, aldehyde), 947-91-1; 1 (R = $(C_6H_5)_3C$), 595-91-5; 1 (R = $(C_6H_5)_3C$, aldehyde), 42365-04-8; 1 (R = $HO_2C(CH_2)_2$), 110-15-6; 1 (R = $HO_2C(CH_2)_2$, aldehyde), 638-37-9; 1 (R = $(CH_2)_8CO_2H$), 693-23-2; 1·2Na (R = $(CH_2)_8CO_2H$), 17265-14-4; 1·2Li (R = $(CH_2)_8CO_2H$), 19370-86-6; 1 (R = $(CH_2)_8CHO$, dialdehyde), 38279-34-4; 1 (R = $ClCH_2$), 79-11-8; 1 (R = $ClCH_2$, aldehyde), 107-20-0; 1 (R = $BrCH_2$), 79-08-3; 1 (R = $BrCH_2$, aldehyde), 17157-48-1; 1 (R = C_6H_5), 65-85-0; 1·Na (R = C_6H_5), 532-32-1; 1·Li (R = C_6H_5), 553-54-8; 1 (R = C_6H_5, aldehyde), 100-52-7; 1 (4-$HO_2CC_6H_4$ = R), 100-21-0; 1·2Na (4-$HO_2CC_6H_4$ = R), 10028-70-3; 1·2Li (4-$HO_2CC_6H_4$ = R), 42596-02-1; 1 (R = 4-$OHCC_6H_4$, aldehyde), 623-27-8; 1 (R = 3-$O_2NC_6H_4$), 121-92-6; 1 (R = 3-$O_2NC_6H_4$, aldehyde), 99-61-6; 1 (R = 2-ClC_6H_4), 118-91-2; 1 (R = 2-ClC_6H_4, aldehyde), 89-98-5; 1 (R = 3-ClC_6H_4), 535-80-8; 1 (R = 3-ClC_6H_4, aldehyde), 587-04-2; 2, 124-04-9; 2 (aldehyde), 1072-21-5; 3, 4224-70-8; 3·Na, 50530-06-8; 3·Li, 51568-15-1; 3 (aldehyde), 57978-00-4; 4, 3724-65-0; 4·Na, 21988-86-3; 4·Li, 110419-20-0; 4 (aldehyde), 4170-30-3; 5, 621-82-9; 5·Na, 538-42-1; 5·Li, 110419-19-7; 5 (aldehyde), 104-55-2; 6, 150-13-0; 6 (aldehyde), 556-18-3; 7, 619-65-8; 7 (aldehyde), 105-07-7; 4-$H_3COC_6H_4CHO$, 123-11-5; 4-$H_3COC_6H_4CO_2Na$, 536-45-8; 4-$H_3COC_6H_4CO_2Li$, 16090-04-3; 4-ClC_6H_4CHO, 104-88-1; 4-$ClC_6H_4CO_2Na$, 3686-66-6; 4-$ClC_6H_4CO_2Li$, 16090-16-7; 4-$O_2NC_6H_4CHO$, 555-16-8; 4-$O_2NC_6H_4CO_2Na$, 3847-57-2; 4-$O_2NC_6H_4CO_2Li$, 18393-32-3; Me_2S, 75-18-3; $(H_3C)_2C{=}C(CH_3)_2$, 563-79-1; cyclopropanecarboxylic acid, 1759-53-1; cyclopropanecarboxaldehyde, 1489-69-6; cyclopropanecarboxylic acid sodium salt, 155-22-6; cyclopropanecarboxylic acid lithium salt, 110419-17-5; cyclohexanecarboxylic acid, 98-89-5; cyclohexanecarboxaldehyde, 2043-61-0; cyclohexanecarboxylic acid sodium salt, 136-01-6; cyclohexanecarboxylic acid lithium salt, 16090-10-1; α-naphthoic acid, 86-55-5; α-naphthaldehyde, 66-77-3; α-camphoric acid disodium salt, 74543-12-7; α-camphoric acid dilithium salt, 110419-18-6; α-camphoraldehyde, 69804-91-7; thexylbromoborane–dimethyl sulfide complex, 109620-28-2; monobromoborane–dimethyl sulfide complex, 55652-52-3.

(9) We have observed that CS_2 seems to be essential in the solvent for the H_2 evolution step. CS_2 readily disssolves HBr, which is formed from the reaction of carboxylic acid and reagent. The dissolved HBr then reacts with thexyl(acyloxy)borane, the undesired intermediate, and thus converts it into the desired intermediate thexyl(acyloxy)bromoborane.

One-Step Preparation of the 3,3′-Dimer of Precocene II

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The antijuvenile hormones precocenes I (1) and II (2) have been shown to induce precocious metamorphosis when applied to larval stages of insects.[1,2] As a consequence of this effect several analogues of both compounds have been synthesized in a search for compounds with better activity.[3,4] We have prepared several dimers,[5,6] the

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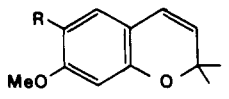
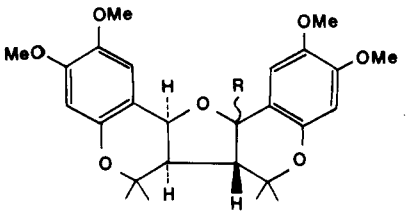
most representative being dimers B (3) and C (4), which have been obtained by treatment of precocene II (2) with Lewis acids.[5]



1 R=H

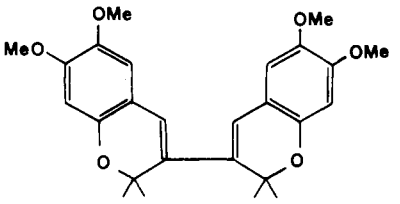
2 R=OMe



3 R=α-H

4 R=β-H

Continuing with this work, we describe here the preparation of the 3,3′-dimer (5) of precocene II (2), obtained in one step and with good yield. This compound has not been previously obtained and may prove to be of potential interest as an antijuvenile hormone.



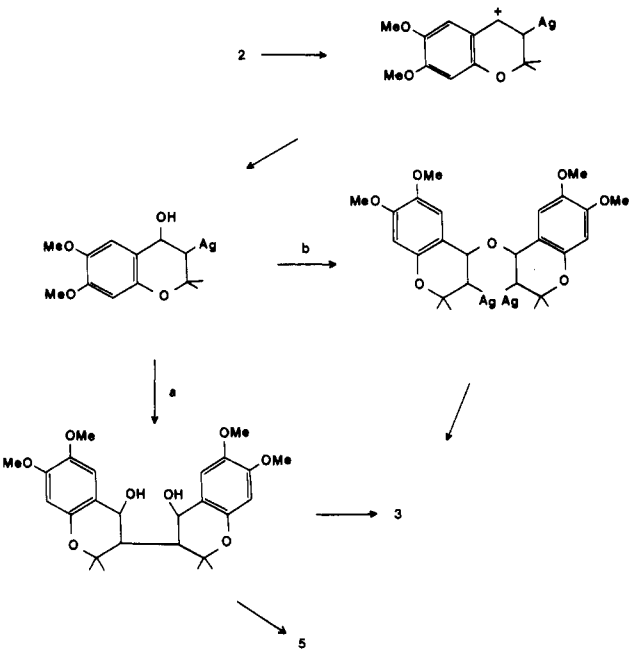
5

Treatment of precocene II (2) with dry ferric trichloride in acetic acid afforded 5. Its structure was given on the basis of the following considerations: Its high-resolution mass spectrum was in accordance with the formula $C_{26}H_{30}O_6$, and the UV spectrum had bands at 236, 286, and 337 nm. Its ^1H NMR spectrum showed that it was a substance of symmetrical structure. The aromatic protons (δ 6.45 and 6.55, each 2 H, s) were assigned to the C-8 and C-5 hydrogens, respectively, while the vinylic hydrogens were assigned to the δ 6.19 (2 H) singlet. Other signals observed in this spectrum were two pairs of methoxyls and four methyl groups.

When the dimer C (4) was treated with $FeCl_3$/AcOH under the same conditions as precocene II (2), only the starting material was recovered, while the action of this reagent on dimer B (3) afforded dimer C (4, 80%) and the 3,3′-dimer (5, 10%). The dimeric compound 5 was also obtained in good yield (80%) by treatment of dimer C (4) in benzene with a mixture of acetic acid, acetic anhydride, and perchloric acid. The formation of 5, from 3 or 4, also indicated that the dimeric structure 5 is the correct one and not an alternative one with a 4,4′-bond.

The proposed mechanism for the formation of dimers B (3) and C (4) by reaction of 2 with $SiO_2/AgNO_3$ or $SiO_2/FeCl_3$ is shown in Scheme I.[5] The formation of the 3,3′-dimer in the treatment of precocene II with ferric

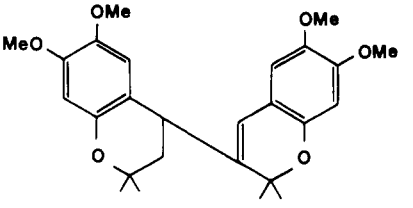
Scheme I



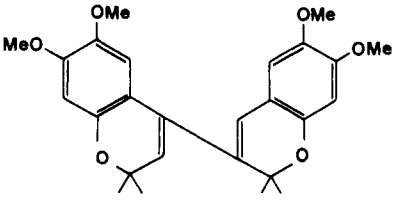
trichloride/acetic acid now vouches for route a.

Ferric chloride on silica gel has been used in the dehydration and rearrangement of alcohols,[7-9] in the cleavage of acetals and ketals,[10] and in the coupling of phenol ethers.[11,12] On the other hand, the ether cleavage with ferric chloride/acetic anhydride has been noted,[13] while the vinylic coupling with ferric chloride via a Diels–Alder adduct has also been observed.[14]

In addition, we prepared the compound 7, an isomer of 5, by dehydrogenation with DDQ of the 3,4′-dimer 6 of precocene II (2), obtained from acid dimerization[5,15] of precocene II (2).



6



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Experimental Section

Melting points are uncorrected. NMR spectra were taken of solutions in $CDCl_3$ on a Bruker WP 200SY or AC 80 and IR spectra of solutions in $CHCl_3$ on a Perkin-Elmer 681. A UV spectrum in ethanol was taken on a Perkin-Elmer 550 SE. Mass spectra were determined on a VG ZAB-2F.

Reaction of Precocene II (2) with Iron(III) Chloride/Acetic Acid. Precocene II (**2**, 40 mg) was treated with ferric trichloride (80 mg) in acetic acid (0.5 mL) at room temperature for 4 h. The usual workup and chromatography of the residue eluting with light petroleum ether/ethyl acetate (4:1) afforded **5**: 26 mg; IR 3010, 3000, 2920, 2820, 1610, 1500, 1460, 1450, 1440, 1270,1130, 1010 cm^{-1}; UV, 236 nm, 286, 337; ^1H NMR (200 MHz) δ 1.52 (12 H, s), 3.82 and 3.84 (each 6 H, s), 6.19, 6.45, and 6.55 (each 2 H, s); MS, m/z (rel intens) 438 (M^+, 60), 423 (100), 219.5 (2), 219 (9), 211.5 (2), 211 (1), 204.5 (10), 204 (38), 196.5 (2), 196 (6); mass spectrum, exact mass calcd for $C_{26}H_{30}O_6$ 438.2034, found m/z 438.2042.

Reaction of Dimer B (3) with Iron(III) Chloride/Acetic Acid. Dimer B (**3**, 23 mg) was treated with ferric trichloride/acetic acid as above. Chromatography of the mixture of products obtained eluting with light petroleum ether/ethyl acetate (4:1) afforded dimer C (**4**, 20 mg) and the 3,3'-dimer (**5**, 3 mg).

Treatment of Dimer C (4) with Acetic Anhydride/Perchloric Acid. The dimer C (**4**, 300 mg) in benzene (1 mL) was treated with acetic acid (1 mL), acetic anhydride (1 mL), and perchloric acid (one drop) for 2 h. Usual workup and chromatography gave **5** (217 mg).

Treatment of the Dimer 6 with DDQ. The dimer **6** (150 mg)[15] in benzene (50 mL) was treated with DDQ (150 mg) at reflux for 5 h. The solution was filtered, washed with 2 M sodium hydroxide solution, and evaporated. The residue was crystallized from methanol affording **7**: 90 mg; mp 150–152 °C, IR 3000, 2980, 2960, 2810, 1610, 1500, 1460, 1450, 1440, 1400, 1380, 1350, 1290, 1270, 1240, 1190, 1130, 1080, 1010, 950, 900, 840 cm^{-1}; ^{1}NMR (200 MHz) δ 1.39 (3 H, s), 1.41 (9 H, s), 3.73, 3.81, 3.83, and 3.84 (each 3 H, s), 5.34 (1 H, s, H-4), 6.20 (1 H, s, H-3'), 6.44, 6.47, 6.57, and 6.72 (each 1 H, s, Ph-H); MS, m/z (rel intens) 438 (M^+, 70), 423 (100), 269 (13), 243 (33), 232 (14), 219 (9), 211.5 (5), 204 (52); mass spectrum, exact mass calcd for $C_{26}H_{30}O_6$ 438.2042, found m/z 438.2029.

Acknowledgment. We thank the CAICYT (Madrid) for financial support.

Registry No. **2**, 644-06-4; **3**, 89004-21-7; **4**, 89064-12-0; **5**, 110271-54-0; **6**, 17678-76-1; **7**, 110271-55-1.

Preparation of (3*S*,4*S*)-2,5-Dimethyl-3,4-hexanediol [(*S*)-DIPED] from (*R*,*R*)-Tartaric Acid via Trimethylsilyl Chloride Catalyzed Acetylation of a Hindered 1,4-Diol

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Directed asymmetric synthesis via α-chloro boronic esters is a powerful new synthetic tool.[1-3] The original chiral director used was pinanediol derived from α-pinene, and it was noted that oxygenated chiral directors such as diacetone mannitol or tartrate esters failed.[1] Pinanediol boronic esters are sterically hindered and difficult to hydrolyze.[1]

(*R*,*R*)-2,3-Butanediol boronic esters can be hydrolyzed and have desirable C_2 symmetry, though limited diastereoselectivity (20:1).[4] More recently we have reported 30:1 diastereomeric ratios from the use of (3*S*,4*S*)-2,5-dimethyl-3,4-hexanediol (diisopropylethanediol, DIPED, **7**) as chiral director.[5] The reagent was made efficiently by our previous diol synthesis,[2] but prior preparation of pinanediol was required. In view of the potential utility of DIPED in the α-chloro boronic ester synthesis, as well as in other syntheses which utilize simple chiral diols as directors,[6,7] we undertook the synthesis of DIPED from tartaric acid.

The quantitative conversion of L-tartaric acid to the methyl ester acetonide[8] (**1**) was followed by straightforward methylation with methylmagnesium bromide to yield the bis(tertiary diol) **2**.[9] After brief attempts to convert **2** to diolefin **5** via a methanesulfonate,[10] we undertook the acetate pyrolysis route.

Attempted acetylation of the very hindered diol **2** by standard means yielded mixtures containing substantial amounts of monoacetylated diol. Thus, acetyl chloride and dimethylaniline with **2** in ether refluxed overnight, conditions which convert *tert*-butyl alcohol to the acetate,[11] yielded the monoacetate of **2**. Acetic anhydride, 4-(dimethylamino)pyridine (1 equiv), and triethylamine (1.7 equiv) with **2** at 25 °C for 48 h[12] yielded a 2:1 mixture of the diacetate **3** and the monoacetate, but an attempt to improve the conversion to **3** by refluxing failed.

Trimethylsilyl chloride in acetic anhydride has been reported to be an efficient acetylating agent.[13] This combination with **2** at 25 °C overnight yielded a 3:2 mixture of monoacetate to diacetate **3** but gave exclusively **3** within a few hours at 85 °C. The original rationale for this process was a postulated formation of acetylium ion, CH_3CO^+,[13] and our results are perhaps consistent with the formation of a low equilibrium concentration of this intermediate. However, the large excess of reactants originally specified[13] is not necessary, and trimethylsilyl chloride is not consumed in the reaction. Nearly quantitative diacetylation was achieved with only a catalytic amount (10 mol %) of trimethylsilyl chloride. Without trimethylsilyl chloride, acetic anhydride reacted very slowly with **2** to form a small amount of monoacetate barely detectable by TLC.

Ester pyrolysis of **3** proved efficient at the optimum temperature and flow rate, but as the temperature rose

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(9) **2** has also been made by: Mash, E. A.; Nelson, K. A.; Heidt, P. C. *Tetrahedron Lett.* **1987**, *28*, 1865-1868. The yield was nearly quantitative on a small scale at much higher dilution than our procedure (Mash, E. A., personal communication, 1987).
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