(ii) Reduction of 3a gave **3b**, bp 100 °C (20 mm) [lit.[2] bp 98 °C (17mm)], in 69% yield. ^{1}H NMR data for **3b** are identical with those reported in the literature.[10]

(iii) Reduction of 4a gave **4b**: bp 115 °C (18 mm); 70% yield; ^{1}H NMR ($CDCl_3$) δ 1.0–1.6 (m, 6 H, H10, H2, H3), 2.05 (m, 4 H, H1, H4, H4a, H8a), 2.82 (m, 2 H, H5, H8), 4.33 (s, 2 H, methylene CH_2), 5.66 (t, 2 H, H6, H7). Anal. Calcd for $C_{13}H_{16}$: C, 90.64; H, 9.36. Found: C, 90.58; H, 9.40.

(iv) Reduction of 5a gave **5b**: bp 79–80 °C (4.7 mm); 80% yield; ^{1}H NMR δ 1.2–1.75 (m, 3 H, H11, H12 syn to the aromatic ring), 2.25 (br s, 2 H, H4a, H9a), 2.83 (m, 3 H, H1, H4, H12 anti to the aromatic ring), 3.20 (s, 2 H, H9, H10), 6.10 (t, 2 H, H2, H3), 6.80–7.20 (m, 4 H, aromatic). Anal. Calcd for $C_{16}H_{16}$: C, 92.25; H, 7.75. Found: C, 92.06; H, 7.50.

This product was identical, in every respect, to that formed from the direct, thermal addition of cyclopentadiene to benzonorbornadiene.[11]

Reduction of 6a gave **6b**, bp 65–72 °C (20 mm) [lit.[4b] bp 58–68 °C (17 mm)], in 70% yield.

Reduction of 7a gave **7b** (from methanol), mp 99 °C, in 62% yield, identical in every respect to authentic material.[9]

Acknowledgment. We gratefully acknowledge the Australian Research Grants Committee for support of this work. We thank Dr. H. K. Patney for helpful discussions and Mr. M. J. Oliver for some assistance.

Registry No. **1a**, 77-47-4; **1b**, 695-77-2; **2a**, 28068-44-2; **2b**, 28068-45-3; **3a**, 309-00-2; **3b**, 15914-94-0; **4a**, 71871-91-5; **4b**, 71871-92-6; **5a**, 52420-67-4; **5b**, 71885-02-4; **6a**, 19448-78-3; **6b**, 875-04-7; **7a**, 71927-69-0; **7b**, 71871-93-7; 7-methylenenorbornene, 694-69-9; dimethanooctahydronaphthalene, 15914-93-9; benzonorbornadiene, 4453-90-1; sodium, 7440-23-5; ethanol, 64-17-5.

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Stereospecific Synthesis of (+)-Decahydro-$\alpha,\alpha,4a\beta$-trimethyl-β-cyclopropa-[*d*]naphthalene-7β-methanol[1]

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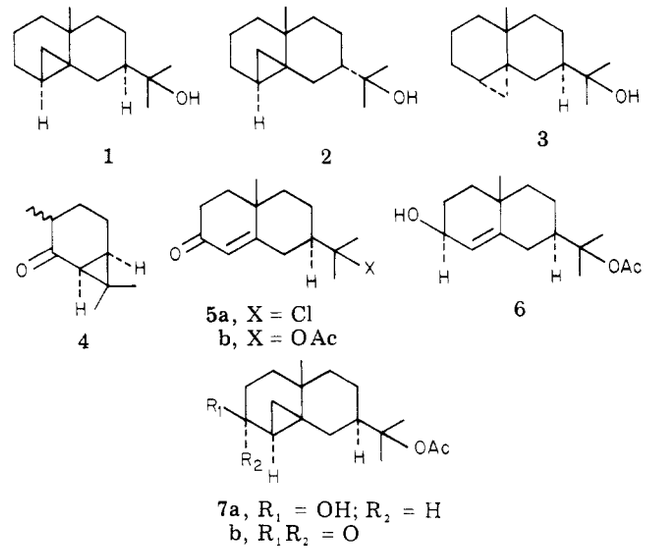
Recently, Moss and co-workers[2] reported the synthesis of the tricyclic alcohols **1** (β,β-cycloeudesmol) and **2** (β,α-cycloeudesmol). Independent syntheses of **1** and **2** as well as the tricyclic alcohol **3** (α,β-cycloeudesmol) have also been reported by Ando, Sayama, and Takase.[3] Compounds **1–3** are diastereomers of the structure assigned to cycloeudesmol, an antibiotic cyclopropane containing sesquiterpene, which was isolated from the marine alga *Chondria oppositiclada* Dawson by Fenical and Sims.[4] We wish to report an alternative stereospecific synthesis of the optically active alcohol **1** from the readily available terpene (−)-2-carone (**4**).[5]



Ketone **4** has been previously converted into the bicyclic chloro enone **5a**[6a] by Michael addition to methyl vinyl ketone followed by treatment of the 1,5-diketone product with hydrogen chloride in ethanol to effect opening of the cyclopropane ring and aldol cyclization.[6b] Solvolysis of **5a** in acetic acid containing silver acetate gave **5b** in 42% yield. Reduction of **5b** with lithium tri-*tert*-butoxyaluminum hydride in ether gave exclusively the β allylic alcohol **6** in 86% yield.[7] The NMR spectrum of **6** showed a small coupling constant (ca. 1.0 Hz) between the vinyl proton and the adjacent proton on the carbon atom bearing the hydroxyl group. This was consistent with the assignment of the β configuration to the allylic hydroxyl group.[8] Allylic alcohol **6** was then converted into β-cyclopropanated derivative **7a** in 49% yield by using the Conia modification[9] of the Simmons–Smith reaction.[10] There is a considerable amount of literature precedent which indicates that the β-hydroxyl group in **6** should direct the cyclopropanation in the indicated manner.[2,3,8,10] The structural assignment of the tricyclic hydroxy acetate **7a** was verified by the similarity of its NMR spectral properties (see Experimental Section) to those of closely related tricyclic alcohols.[3,11] Jones oxidation of alcohol **7a** gave the tricyclic acetoxy ketone **7b**[12] in 52% yield. The synthesis of **1** was accomplished in 64% yield by Wolff–Kishner reduction of the carbonyl function of **7b** which was accompanied by

(1) Partial support of the research by a grant from the National Cancer Institute is gratefully acknowledged.
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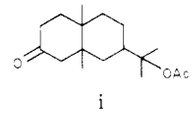
(12) In order to obtain chemical confirmation for the structural assignment of **7b** it was converted in good yield to a decalone derivative which had spectral properties completely consistent with the structure **i** by reductive cleavage of the cyclopropane ring with lithium in liquid ammonia (see W. G. Dauben and J. E. Deviny, *J. Org. Chem.*, **31**, 3794 (1966)).



i

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hydrolysis of the acetate group under the reaction conditions employed.

Alcohol 1 exhibited NMR and IR spectra properties identical with those reported previously.[2,3,13] Its spectral properties were clearly different from those obtained by Fenical and Sims for cycloeudesmol.[14]

Experimental Section[15]

Synthesis of Enone Acetate 5b. To a solution of 17.5 g (0.073 mol) of chloro enone **5a**[6a] in 300 mL of glacial acetic acid was added 14.0 g (0.084 mol) of silver acetate. The mixture was stirred in the dark at room temperature for 96 h. After removal of the silver chloride by filtration, the filtrate was diluted with 500 mL of water and extracted with four 100-mL portions of ether. The combined ethereal extracts were washed with six 100-mL portions of a saturated aqueous solution of sodium bicarbonate followed by two 100-mL portions of a saturated aqueous solution of sodium chloride. The organic layer was dried over anhydrous magnesium sulfate. After removal of the solvent in vacuo, the residual oil was chromatographed on Florisil by using 20% ether–hexane as eluent to afford 7.27 g (42%) of **5b**. Recrystallization from pentane gave an analytical sample: mp 66–67 °C; $[\alpha]^{25}_D$ +125.3° (c, 0.30, chloroform); UV λ_{max} (95% EtOH) 236 nm (ϵ 14 500); IR (CCl_4) 1730 (acetate C=O), 1678 (α,β-unsaturated C=O), 1618 cm^{-1} (conjugated C=C); NMR (CCl_4) δ 1.22 (s, 3 H, angular CH_3), 1.42 (s, 6 H, $C(CH_3)_2OAc$), 1.92 (s, 3 H, CH_3CO_2), 5.62 (s, 1 H, vinyl proton); mass spectrum, *m/e* (rel intensity) 204 (100, $(M - HOAc)^+$), 189 (31), 176 (61), 133 (51), 91 (42), 43 (94).

Anal. Calcd for $C_{16}H_{24}O_3$: C, 72.69; H, 9.15. Found: C, 72.72; H, 9.16.

Synthesis of Allylic Alcohol 6. To a suspension of 6.07 g (0.16 mol) of lithium aluminum hydride in 250 mL of anhydrous ether under nitrogen was added dropwise with stirring over 1.5 h 36.8 g (0.50 mol) of dry *tert*-butyl alcohol. After the addition was complete, the solution was stirred at room temperature for 45 min, and the ether was removed in vacuo. Anhydrous tetrahydrofuran (THF) (400 mL) was then added, and the mixture was stirred and cooled to 0 °C under nitrogen. A solution of 5.28 g (0.020 mol) of enone acetate **5b** in 100 mL of anhydrous THF was added dropwise with stirring over 1.2 h. After the addition was complete, the mixture was stirred at 0 °C for 5 h and at 25 °C for an additional 2 h. A saturated aqueous solution of ammonium chloride was then added dropwise, and the layers were separated. The aqueous layer was then extracted with two 100-mL portions of ether. The combined organic layers were washed with two 200-mL portions of a saturated aqueous solution of sodium chloride and dried over anhydrous magnesium sulfate, and the solvent was removed in vacuo to afford 6.52 g of crude material. The crude product was chromatographed on Florisil by using 20% ether–hexane as eluent to give 4.55 g (86%) of allylic alcohol **6**: bp 130 °C (0.025 mm) (bath temperature); $[\alpha]^{25}_D$ +41.6° (c 0.134, chloroform); IR (CCl_4) 3610, 3435 (OH), 1732 (acetate C=O), 1658 cm^{-1} (C=C); NMR (CCl_4) δ 1.07 (s, 3 H, angular CH_3), 1.38 (s, 6 H, $C(CH_3)_2OAc$), 1.92 (s, 3 H, CH_3CO_2), 2.98 (s, 1 H, OH), 4.03 (m, 1 H, CHOH), 5.30 (d, $J \simeq 1.0$ Hz, vinyl proton); mass spectrum, *m/e* (rel intensity) 206 (5, $(M - HOAc)^+$), 188 (33), 105 (39), 91 (47), 43 (100).

Anal. Calcd for $C_{16}H_{26}O_3$: C, 72.14; H, 9.84. Found: C, 72.05; H, 9.85.

Synthesis of the Tricyclic Hydroxy Acetate 7a. Zinc metal (3.4 g) was added with stirring at reflux temperature under nitrogen to a solution of 20 mg of silver acetate in 20 mL of glacial acetic acid. The hot mixture was stirred for 5 min, and the solid residue was washed with five 200-mL portions of anhydrous ether and dried by warming it under a positive nitrogen flow. Anhydrous ether (30 mL) was then added to the resulting zinc–silver couple, and 8.04 g (0.030 mol) of methylene iodide was added dropwise with stirring under nitrogen. An exothermic reaction began within a few minutes. The mixture was stirred for 30 min and then heated at reflux for 1 h. After the mixture had cooled to room temperature, a solution of 3.72 g (0.014 mol) of the allylic alcohol **6** in 20 mL of anhydrous ether was added dropwise with stirring over 30 min, and the mixture was stirred for 18 h at room temperature. A saturated aqueous solution of ammonium chloride (20 mL) was then added, and after being stirred for a few minutes, the aqueous and ethereal layers were separated. The aqueous layer was extracted with two 20-mL portions of ether. The ethereal extracts were combined and washed with 20 mL of a saturated aqueous solution of sodium chloride. The ether solution was dried over anhydrous magnesium sulfate, and the solvent was removed in vacuo to afford 3.05 g of a crude oil. The material was chromatographed on silica gel by using 50% ether–hexane as eluent to give 1.49 g (49%) of the tricyclic hydroxy acetate **7a**: bp 160 °C (0.07 mm) (bath temperature); $[\alpha]^{25}_D$ −46.3 (c 0.155, chloroform); IR (CCl_4) 3610 (OH), 1730 cm^{-1} (acetate C=O); NMR ($CDCl_3$, $CHCl_3$ reference) δ 0.09 (dd, J = 4.64 and 9.28 Hz, 1 H, cyclopropyl CH_2), 0.60 (dd, J = 4.64 and ca. 4.70 Hz, 1 H, cyclopropyl CH_2), 0.97 (s, 3 H, angular CH_3), 1.34 and 1.36 (2 s, 6 H, $C(CH_3)_2OAc$), 1.92 (s, 3 H, CH_3CO_2), 4.33 (t of m, CHOH); mass spectrum, *m/e* (rel intensity) 220 (1, $(M - HOAc)^+$), 202 (57), 187 (34), 159 (62), 148 (41), 105 (55), 81 (58), 43 (100).

Anal. Calcd for $C_{17}H_{28}O_3$: C, 72.92; H, 10.06. Found: C, 72.99; H, 10.12.

Synthesis of the Acetoxycyclopropyl Ketone 7b. The allylic alcohol **7a** (2.83 g, 0.010 mol) was dissolved in 150 mL of acetone, and the solution was cooled to 0 °C. Jones reagent (2.5 mL of a 2.67 M solution of chromium trioxide in sulfuric acid–water) was then added dropwise with stirring, and after the addition was complete, the mixture was stirred for 10 min at 0 °C. Isopropyl alcohol was then added dropwise until the dark orange color of the excess reagent disappeared. The mixture was then poured into 150 mL of a saturated aqueous solution of sodium chloride. The aqueous layer was extracted with two 100-mL portions of ether. The combined ethereal extracts were washed with four 50-mL portions of a saturated aqueous solution of sodium bicarbonate and one 50-mL portion of a saturated aqueous solution of sodium chloride. The organic layer was dried over anhydrous magnesium sulfate, and the solvent was removed in vacuo to give 2.90 g of the crude oxidation product. This material was chromatographed on silica gel by using 30% ether–hexane as eluent to afford 1.43 g (51%) of the acetoxy ketone **7b** as a yellow oil: bp 140 °C (0.07 mm) (bath temperature); $[\alpha]^{25}_D$ +44.0° (c 0.315, chloroform); UV λ_{max} (EtOH) 207 nm (ϵ 5660); IR (CCl_4) 1730 (acetate C=O), 1685 cm^{-1} (C=O, cyclopropyl ketone); NMR ($CDCl_3$) δ 0.62–0.87 (m, 2 H, cyclopropyl CH_2), 1.09 (s, 3 H, angular CH_3), 1.39 (s, 6 H, $C(CH_3)_2OAc$), 1.92 (s, 3 H, CH_3CO_2); mass spectrum, *m/e* (rel intensity) 218 (36, $(M - HOAc)^+$), 203 (23), 148 (69), 123 (80), 105 (77), 79 (70), 55 (84), 43 (100).

Anal. Calcd for $C_{17}H_{26}O_3$: C, 73.34; H, 9.41. Found: C, 73.20; H, 9.40.

Synthesis of (+)-Decahydro-$\alpha,\alpha,4a\beta$-trimethyl-β-cyclopropa[*d*]naphthalene-7β-methanol (1). The cyclopropyl ketone **7b** (1.80 g, 0.0065 mol), 234 mL of diethylene glycol, 2.70 g (0.048 mol) of potassium hydroxide, and 80 mL of 95% hydrazine hydrate were placed in a 500-mL flask fitted with a variable take-off distilling head. The mixture was then heated at reflux for 2 h. The mixture was subjected to distillation until the head temperature reached 210 °C, and heating at reflux was continued for an additional 5 h. The mixture was allowed to cool to room temperature, acidified with 10% hydrochloric acid, and extracted with two 75-mL portions of pentane. The combined pentane extracts were washed with 50 mL of a saturated aqueous solution of sodium chloride and dried over anhydrous magnesium sulfate. Removal of the solvent in vacuo followed by chromatography of the residual oil (1.48 g) on silica gel using 25% ether–hexane as eluent gave 0.92 g (64%) of **1**. The product solidified on standing and was recrystallized from hexane to give pure **1**: mp 72.5–73.0 °C (lit.[3] mp 75 °C); [α] +18.2 (c 0.695, chloroform); IR (CCl_4) 3610

(13) We are grateful to Professor Robert A. Moss for copies of the NMR and IR spectra of alcohol 1.

(14) We thank Professor William Fenical for copies of the NMR and IR spectra of cycloeudesmol.

(15) Melting points and boiling points are uncorrected. Infrared spectra were taken on a Perkin-Elmer Model 457 infrared spectrophotometer. Ultraviolet spectra were taken on a Beckman DBGT recording spectrophotometer using 1-cm matched quartz cells. NMR spectra were determined at 60 MHz with a Varian T-60 spectrometer and at 100 MHz with a JEOL PFT-100 spectrometer. Mass spectra were obtained by using a Hitachi Perkin-Elmer RHU-7L or a Varian MAT 1125 spectrometer. Microanalyses were obtained by Atlantic Microlab, Inc., Atlanta, GA.

cm^{-1} (OH); NMR ($CDCl_3$, $CHCl_3$ reference) δ 0.03–0.75 (m, 3 H, cyclopropyl hydrogens), 0.96 (s, 3 H, angular CH_3), 1.11 and 1.13 (2 s, 6 H, $C(CH_3)_2OH$); mass spectrum, *m/e* (rel intensity) 222 (5) (M^+), 204 (18, $(M - H_2O)^+$), 189 (12), 149 (29), 105 (33), 59 (100), 41 (27).

The IR and NMR spectra of **1** were identical with those obtained by Moss and co-workers[2,13] and were in close agreement with those reported by Ando, Sayama, and Takase.[3]

Registry No. (+)-**1**, 71962-31-7; (−)-**4**, 18541-52-1; *cis*-**5a**, 71962-32-8; (+)-**5b**, 57605-76-2; (+)-**6**, 71911-57-4; (−)-**7a**, 71911-58-5; (+)-**7b**, 71911-59-6.

Synthesis of 14β,17β(H)-Cholest-5-en-3β-ol

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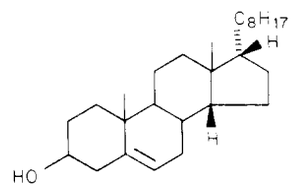
Sterols with a modified stereochemistry with respect to that of natural compounds appear to exhibit different noticeable activities on the enzyme systems which catalyze both the biosynthesis and catabolism of cholesterol. The first example is represented by the inhibitory effect on the cholesterol biosynthesis of the triterpenoid euphol, which differs from lanosterol only in the stereochemistry.[1] These observations prompted us to synthesize 5α,14β-cholest-7-en-3β-ol[2] and 14β-cholest-5-en-3β-ol,[3] both containing a cis C/D ring junction due to the β configuration of the hydrogen at C-14. Studies on in vitro catabolism of these compounds showed that rat liver enzymes are able to transform the former[4] into 5α-cholest-7-en-3β-ol, a cholesterol precursor, but leave unaffected the latter which, however, significantly modifies the lecithin cholesterol acyl transferase activity in plasma.[5] In addition Schroepfer et al.[6] were able to demonstrate that two 14β steroids (i.e., 5α,14β-cholest-7-en-3β,15β-diol and 5α,14β-cholest-7-en-3β,15α-diol) are inhibitors of sterol biosynthesis in L cells and in primary cultures of liver cells. More recently Koreeda and Koizumi[7] reported that (20*S*)-cholesterol exhibits a significant in vitro inhibitory activity in the conversion of cholesterol to pregnenolone and that side chain cleaving enzymes appear to be fastidious in their steric requirement with respect to side chain stereochemistry.

In continuation of our work we now report a stereochemically controlled synthesis of 14β,17β(H)-cholest-5-en-3β-ol (**1**), which differs from cholesterol in side chain configuration and in C/D ring junction, in order to check its influence on cholesterol biosynthesis and/or catabolism. A simple method to obtain 17β(H) steroids from 17α(H) steroids was proposed recently by us[8,9] and Caspi et al.[10,11] simultaneously. The side chain inversion is effected on a 7-, 8(14)- and 14-ene steroid by hydrogen chloride at controlled temperature to yield a 14β-chloro,17β(H) steroid which is transformed by either triethylamine treatment or chromatography on silica into the 14-ene,17β(H) compound. Catalytic hydrogenation of the Δ^{14} double bond of a 17β(H) steroid gives a 14β,17β(H) compound. The cis geometry of C/D rings was established by Caspi et al.[10] on the basis of ^{13}C NMR evidence and by Brunke et al.[12] by X-ray diffraction analysis. With this in mind, the key compound chosen for the synthesis of **1** was 5α-cholest-



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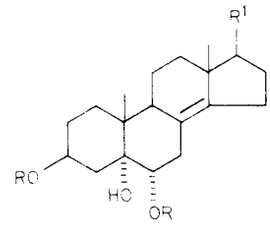
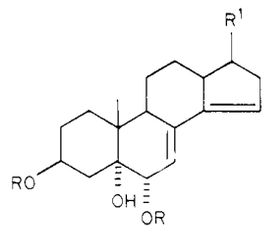
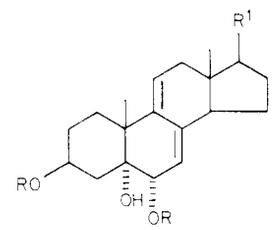


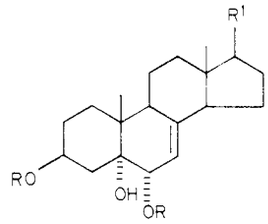
2a, R = H; R^1 = C_8H_{17}
b, R = H; R^1 = C_9H_{19}
c, R = Ac, R^1 = C_8H_{17}



3a, R = H; R^1 = C_8H_{17}
b, R = H; R^1 = C_9H_{17}
c, R = Ac; R^1 = C_8H_{17}



4a, R = H; R^1 = C_8H_{17}
b, R = H; R^1 = C_9H_{17}
c, R = Ac; R^1 = C_8H_{17}



5a, R = H; R^1 = C_8H_{17}
b, R = H; R^1 = C_9H_{17}
c, R = Ac; R^1 = C_8H_{17}

8(14)-ene-3β,5,6α-triol 3,6-diacetate (**2c**) containing a $\Delta^{8(14)}$ double bond suitable for the side chain inversion and a masked 5α,6α-diol system useful for the introduction of a Δ^5 double bond. The diacetate (**2c**) and the parent triol (**2a**) are unknown. However, their homologues with a C_9H_{19} side chain were obtained by M. Fieser et al.[13] by hydrogenation of a material considered to be 5α-ergosta-7,14,22-triene-3β,5,6α-triol (**3b**) derived from permanganate oxidation of ergosterol. Actually the supposed **3b** was shown[14] to be a mixture of three different compounds: 5α-ergosta-7,9(11),22-triene-3β,5,6α-triol (**4b**), 5α-ergost-

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