maximum yield the resin is soaked in methanol before use and a methanolic solution of the diester is used. After ion exchange, the solution is concentrated by rotary evaporation (~30 °C). When this solution is cooled (to ~0 °C) sodium bis(2,4-dinitrophenyl) phosphate (I) is obtained as a bright yellow solid. It is recrystallized from ethanol (yield ~85%).

Anal. Calcd for $C_{12}H_6N_4O_{12}PNa$: C, 31.9; H, 1.34; N, 12.4; P, 6.85; Na, 5.08. Found: C, 32.0; H, 1.33; N, 12.2; P, 6.68; Na, 4.87.

Sodium bis(2,4-dinitrophenyl) phosphate (I) (1 g) is rapidly dissolved in ~50 mL of aqueous 0.30 M NaOH. The resulting solution of pH ~13.5 is held at 25 ± 2 °C for 1 h (~6 half-lives), after which ~15 mL of aqueous 5 M HCl is added to lower the pH to ~0. The reaction mixture is cooled (ice bath) and the 2,4-dinitrophenol is removed by filtration. The filtrate, containing the monoester as the free acid (II), is concentrated (rotary evaporator, ~35 °C) to a thick syrup. The NaCl is now precipitated by adding dry ether. After filtration, the ether solution is stirred while 2,6-lutidine is added dropwise until the solution develops a permanent bright yellow color (~0.26 mL required). The resulting pale yellow solid is collected and washed with ether. Recrystallization from hot ethanol gives the required 2,6-lutidinium 2,4-dinitrophenyl hydrogen phosphate (III) as a white solid. A second crop may be obtained by concentrating the ethanolic solution (total yield, ~65%).

Anal. Calcd for $C_{13}H_{14}N_3O_8P$: C, 42.1; H, 3.80; N, 11.3; P, 8.34. Found: C, 41.9; H, 3.68; N, 11.2; P, 8.29.

Acknowledgment. Support of this research by the National Science Foundation is gratefully acknowledged.

Registry No. I, 76215-44-6; II, 2566-26-9; III, 6186-33-0; bis(2,4-dinitrophenyl)phosphate pyridinium salt, 76215-45-7; 2,6-lutidine, 108-48-5.

Thermal Decarboxylation of 3,17-Dioxo-4β,5-epoxy-5β-androstan-19-oic Acid and Some Transformations of the Derived Product

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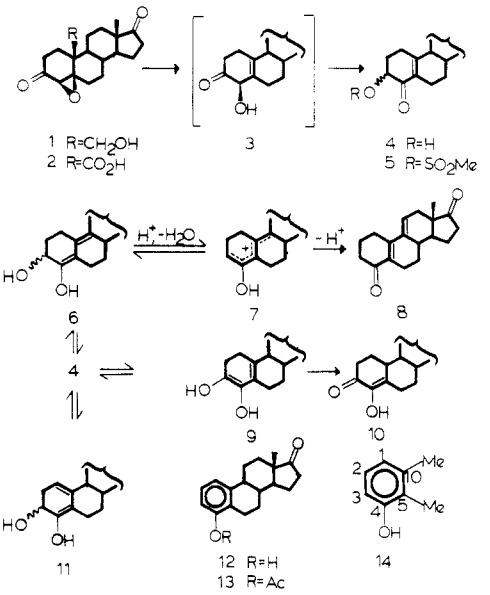
We have reported our results of reactions in which the C-10 hydroxymethyl group of 5β,6β-steroidal epoxides[1] is eliminated on epoxide opening by boron trifluoride. The observation[2] that β,γ-epoxy acids can be made to undergo thermal decarboxylative elimination under relatively mild conditions (refluxing toluene) prompted us to attempt a similar reaction with the acid **2**. It was hoped that the neutral conditions employed in this reaction would allow for the isolation of the allylic alcohol **3**, a compound of interest to us since we have postulated it as an intermediate in the enzymatic conversion[3] of the epoxide **1** to estradiol.

The acid **2** was prepared by Jones oxidation of the alcohol **1**. It was found to be stable in refluxing toluene and xylene but in decalin it reacted to give two chromatographically homogeneous products. The minor product (9%) was estrone and the major product (79%), although



tautomeric (*m/e* 288, M^+) with the desired allylic alcohol **3**, exhibited absorptions (IR 1655 and 1619 cm^{-1}; UV 247 nm) indicative of an α,β-unsaturated ketone, namely, **4**. The ^1H NMR only showed an apparent quartet for the 3-H, but the ^{13}C NMR ($CDCl_3$) indicated that it had been obtained as a mixture of epimers [the more readily assignable downfield signals were 219.92 (C-17), 182.23 and 181.58 (C-4), 159.31 and 158.26 (C-10), 129.67 C-5), 71.91 and 71.83 (C-3)].

It would appear that the allylic alcohol **3** was initially formed but isomerized to the α,β-unsaturated ketone **4** under the reaction conditions. As **4** was stable to further reflux in decalin, the estrone formed may have arisen by dehydration of the allylic alcohol.

Since this 3-hydroxy-$\Delta^{5(10)}$-4-one system is novel in steroid chemistry, we briefly examined some of its properties. With base, **4** was found to isomerize to the diosphenol **10**. This compound exhibited absorptions in the UV at 276 nm and in the IR at 1670 and 1650 cm^{-1} which are consistent with this structure.

Under acidic conditions (concentrated $HClO_4$, THF) it was found to undergo dehydration to give a compound (*m/e* 270, M^+) whose IR spectrum indicated the presence of an αβ,γδ-unsaturated ketone (1655, 1605, 1590 cm^{-1}). Since the ^1H NMR spectrum showed a single olefinic proton (δ 6.18) which was coupled with an adjacent center, it was assigned structure **8**. Although the λ_{max} (289 nm) was somewhat lower than may be expected[4] (308 nm), the relatively large extinction coefficient (23 000) is consistent with a heteroannular diene system.

The acid- or base-catalyzed reactions of **4** would be expected to proceed via the enols (or enolates) corresponding to **6**, **9**, or **11**. Under basic conditions, the enone **4** isomerizes via the enolate of **9** to the acidic diosphenol **10** which would exist as an anion in the basic solution. However, under acidic conditions, it appears that the heteroannular dienol **6** predominates or at least that the subsequent acid-catalyzed dehydration [**6** → **7** → **8**] draws any possible equilibria with the other enols, **9** or **11**, in this direction. This would mean that the enol **9**, if formed, isomerizes back to **4** more rapidly than protonation at C-10 since the diosphenol was found to be stable under these reaction conditions. The proposed dehydration sequence draws some support from the observation[5] that the 3β-acetoxy-

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(2) W. C. Still, A. J. Lewis, and D. Goldsmith, *Tetrahedron Lett.*, 1421 (1971).
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Table I. Comparison of Aromatic ^{13}C NMR Chemical Shifts of 12 with Those of 14 and 13

position	12[a]	14[b]	δ (13) − δ (12)
C-1	117.26	122.3	+5.22
C-2	126.35	125.8	+0.24
C-3	112.05	112.7	+7.95
C-4	155.04	153.3	−5.15
C-5	123.84	122.3	+5.50
C-10	142.09	137.6	+0.45

[a] Dioxane as solvent. [b] Reference 9; $CDCl_3$ as solvent; steroid numbering used.

4-methoxy-Δ^4-enol ethers undergo the loss of the allylic 3-substituent in preference to protonation at C-5 when treated with acid.

It was noted that, if the homoannular dienol 11 underwent a similar dehydration, the 4-hydroxy isomer of estrone (12) would have been obtained. This somewhat obscure compound[6] can be derived from 4 by refluxing its 3-mesylate 5 in collidine. The ^{13}C NMR assignments for the aliphatic portion of this molecule were made by comparison with those for estrone.[7] The only major difference found was an upfield shift of ca. 6 ppm for C-6. This shielding would be expected to arise from the ortho interaction[8] of C-4 substituent. The aromatic carbons were assigned by comparison with those of 2,3-dimethyl phenol (14 Table I). They were also found to exhibit the usual type of acetylation shifts,[10] namely, downfield for the ortho and para carbons and upfield for the acetate-bearing carbon.

We are continuing to explore alternative approaches for the preparation of the allylic alcohol 3.

Experimental Section

Melting points were taken on a Hoover Uni-Melt apparatus and are uncorrected. Optical rotations are for chloroform solutions, using a Perkin-Elmer 141 polarimeter. IR spectra were recorded on a Beckman IR-8 spectrophotometer, using chloroform solutions. UV spectra were obtained on a Bausch and Lomb Spectronic 600 instrument. ^1H NMR spectra were obtained on a Varian T-60 or H-100 instrument with Me_4Si as internal standard. ^{13}C NMR spectra were obtained on a Varian FT-80 instrument with ca. 0.2 M solutions of the steroid in dioxane, using an internal coaxially placed D_2O lock. The chemical shifts are relative to the internal standard, Me_4Si, and those assignments quoted in parentheses may be interchanged. Mass spectra were obtained on an AEI MS9025 spectrometer. Microanalyses were carried out by Galbraith Laboratories, Knoxville, TN.

3,17-Dioxo-4β,5-epoxy-5β-androstan-19-oic Acid (2). To a cooled solution (ca. 5 °C) of 1 (500 mg, 1.57 mmol) in acetone (50 mL) was added a solution of Jones reagent[11] (1.75 mL). After 4 h, methanol (2 mL) was added and, after an additional 15 min, it was diluted with water (50 mL). It was extracted with ether and the extracts were washed with water and brine and then dried (Na_2SO_4). Removal of the solvents followed by crystallization from ether–petroleum ether afforded 413 mg (80%) of 2: mp 184–186 °C dec.; $[\alpha]_D$ +189° (c 8.9); IR 3100 (OH), 1720 cm^{-1} (3-CO, 17-CO, and 19-CO_2H); ^1H NMR ($CDCl_3$) δ 0.97 (s, 3 H, 18-CH_3), 3.08 (s, 1 H, 4α-H); mass spectrum, m/e 322 (M^+). Anal. Calcd for $C_{19}H_{24}O_5$: C, 68.65; H, 7.28. Found: C, 68.56; H, 7.31.

Decarboxylation of 2. A suspension of 2 (800 mg, 2.48 mmol) in decalin (100 mL) was heated at reflux under nitrogen for 2.5 h. After the mixture cooled, the solvent was removed under reduced pressure. The residual semisolid was chromatographed on silica gel (50 g, eluting with mixtures of ethyl acetate–hexane varying from 1:4 to 7:13) to afford estrone (58 mg, 9%) (a sample coverted to the 3-acetate was idential (TLC, ^1H NMR) with authentic material) and 541 mg (79%) of 4: mp 159–161 °C (from methanol–ether); UV (methanol) λ_{max} 247 nm (ε 11 200); IR 3460 (OH), 1735 (17-CO), 1655 and 1619 cm^{-1} (α,β-unsaturated ketone); ^1H NMR ($CDCl_3$) δ 0.92 (s, 3 H, 18-CH_3), 4.09 (apparent q, 1 H, J = 5 and 13 Hz); mass spectrum, m/e 288 (M^+). Anal. Calcd for $C_{18}H_{24}O_3$: C, 74.97; H, 8.39. Found: C, 74.78; H, 8.43.

Isomerization of 4 with Base. A solution of 4 (60 mg, 0.21 mmol) in methanolic sodium methoxide (3 mL, 0.55 M) was heated at reflux for 2 h under nitrogen. The solution was acidified with 5% HCl and then diluted with water. It was extracted with ethyl acetate and the extracts were washed with water and brine and dried (Na_2SO_4). Removal of the solvents followed by chromatography on silica gel (15 g; eluting with mixtures of ethyl acetate–hexane varying from 1:4 to 2:3) afforded 42 mg (70%) of 10 as prisms (acetone): mp 200–202 °C; $[\alpha]_D$ +120° (c 4.9); UV (methanol) λ_{max} 276 nm (ε 11 000); IR 3460 (OH), 1740 (17-CO), 1670 and 1650 cm^{-1} (COCOH=CR_2); mass spectrum, m/e 288 (M^+). Anal. Calcd for $C_{18}H_{24}O_3$: C, 74.97; H, 8.39. Found: C, 75.03; H, 8.36.

Acid-Catalyzed Dehydration of 4. A solution of 4 (33 mg, 0.11 mmol) in tetrahydrofuran (3 mL) containing perchloric acid (0.5 mL, 70% was allowed to stand for 3 days under nitrogen. It was then diluted with water and extracted with methylene chloride. The extracts were washed with water and brine and dried (Na_2SO_4). After removal of the solvents, the residual oil was chromatographed on silica gel [7 g; eluting with ethyl acetate–hexane (1:3)] to afford 18 mg (61%) of 8 as a crystalline solid: mp 130–132 °C; $[\alpha]_D$ +412° (c 1.5); UV (methanol) λ_{max} 289 nm (ε 23 000); IR 1740 (17-CO), 1655, 1605, 1590 cm^{-1} (αβ,γδ-unsaturated ketone); ^1H NMR ($CDCl_3$) δ 0.92 (s, 3 H, 18-CH_3), 6.18 (m, 1 H, w/2 = ca. 6 Hz, 11-H); mass spectrum, m/e 270 (M^+). Anal. Calcd for $C_{18}H_{22}O_2$: C, 79.96; H 8.20. Found: C, 79.79; H, 8.29.

Similar treatment of the diosphenol 10 (30 mg) afforded, after workup and chromatography, 25 mg of unreacted starting material.

Conversion of 4 to 4-Hydroxyestra-1,3,5(10)-trien-17-one (12). To a cooled solution (ca. 5 °C) of 4 (100 mg, 0.35 mmol) in pyridine (3 mL) was added methanesulfonyl chloride (0.1 mL). After 2 h, the solution was poured into water and extracted with methylene chloride. The extracts were washed with 5% HCl, 5% $NaHCO_3$, water, and brine and then dried (Na_2SO_4). Removal of the solvents afforded crude 5 as an oil: IR 1740 (17-CO), 1690 and 1625 (α,β-unsaturated ketone), 1180 cm^{-1} (OSO_2Me); ^1H NMR ($CDCl_3$) 0.90 (s, 3 H, 18-CH_3), 3.23 (s, 3 H, OSO_2Me), 5.00 (m, 1 H, 3-H). The oil was taken up in 2,4,6-collidine (3 mL) and heated at reflux for 20h. After cooling the solution was poured into 5% HCl and extracted with ethyl acetate. The extracts were washed with 5% HCl, 5% $NaHCO_3$, water, and brine and dried (Na_2SO_4). After removal of the solvents, the crude 12 was acetylated (acetic anhydride–pyridine) and chromatographed on silica gel [15 g; eluting with ethyl acetate–hexane (3:7)] to afford 65 mg (60%) of 13 as needles (from ethanol–water): mp 173–174 °C; $[\alpha]_D$ +145° (c 3.5); IR 1740 (17-CO and OAc), 1610, 1580 cm^{-1} (aromatic); ^1H NMR (acetone-d_6) 0.87 (s, 3 H, 18-CH_3), 2.24 (s, 3 H, OAc), 6.7–7.2 (higher order m, 3 H, aromatic); ^{13}C NMR 123.48 (C-1), 126.59 (C-2), 120.00 (C-3), 149.89 (C-4), 129.34 (C-5), 23.87 (C-6) (26.36, C-7), 38.10 (C-8), 45.18 (C-9), 142.54 (C-10) (26.27, C-11), 32.30 (C-12), 47.94 (C-13), 50.85 (C-14), 21.90 (C-15), 35.66 (C-16), 223.14 (C-17), 13.62 (C-18, 20.27 and 168.87 (OAc); mass spectrum, m/e 312 (M^+). Anal. Calcd for $C_{20}H_{24}O_3$: C, 76.89; H, 7.74. Found: C, 76.99; H, 7.87.

Hydrolysis (methanol, 10% NaOH, reflux, 4 h) afforded 12 in an essentially quantitative yield: mp 269–271 °C (needles from methanol) (lit.[6a] mp 270–275 °C); $[\alpha]_D$ +152° (c 1.1); UV (dioxane) λ_{max} 275 nm (ε 1900); ^{13}C NMR δ 117.26 (C-1), 126.35 (C-2), 112.05 (C-3), 155.04 (C-4), 123.84 (C-5), 23.93 (C-6), (26.74) (C-7), 38.32 (C-8), 45.31 (C-9), 142.09 (C-10) (26.44, C-11), 32.36 (C-12), 47.98

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(C-13), 50.98 (C-14), 21.95 (C-15), 35.73 (C-16), 223.13 (C-17), 13.68 (C-18).

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Registry No. 1, 53875-00-6; 2, 76251-10-0; 4 (epimer 1), 76251-11-1; 4 (epimer 2), 76251-12-2; 5 (epimer 1), 76251-13-3; 5 (epimer 2), 76251-14-4; 8, 76251-15-5; 10, 76251-16-6; 12, 38522-00-8; 13, 76251-17-7; estrone, 53-16-7.

Preparation of (*R*)-(+)- and (*S*)-(-)-4-Iodo-1,2-epoxybutane [(*R*)- and (*S*)-(2-Iodoethyl)oxirane], Useful Chiral Synthons

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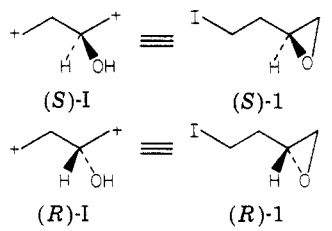
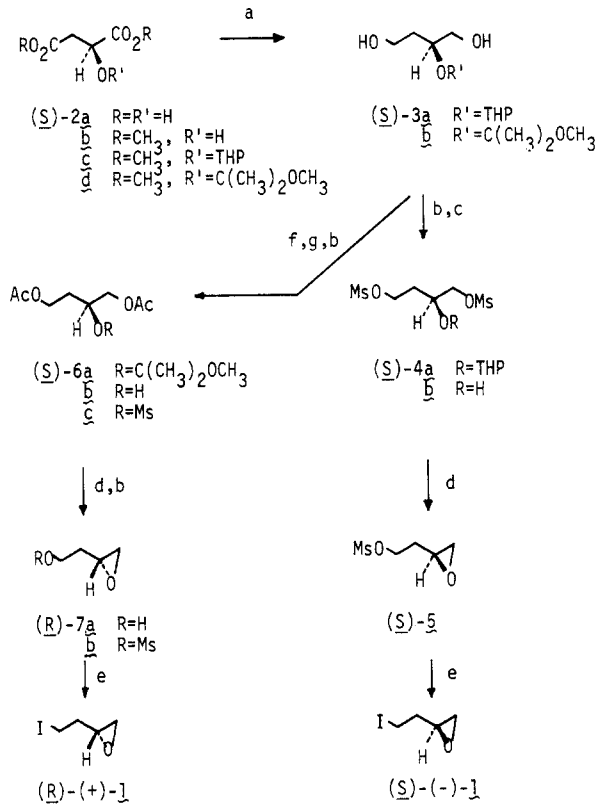
In the course of studies involving the total synthesis and conclusive determination of the absolute configuration of certain optically active natural products, we required synthetic equivalents of the chiral 4-carbon synthons (*R*)-I and (*S*)-I possessing *different* electrophilic centers at each



terminus.[1] The different level of reactivity exhibited by primary iodides and terminal oxiranes (epoxides) suggested that the use of the iodo epoxides (*R*)- and (*S*)-4-iodo-1,2-epoxybutane (1) [(*R*)- and (*S*)-(2-iodoethyl)oxirane] would allow sufficient selectivity for the introduction of two different, though similarly reactive, nucleophiles. Herein, we describe convenient and high-yielding preparations of both (*R*)- and (*S*)-I from readily available (*S*)-(-)-malic acid (**2a**, natural form), Scheme I.[2]

Scheme I[a]



[a] Reagents and conditions: a, $LiAlH_4$, THF, reflux; b, CH_3SO_2Cl, Et_3N, CH_2Cl_2, -20 to -15 °C; c, cat. CH_3SO_3H, EtOH, 50 °C; d, K_2CO_3, MeOH-THF, 25 °C; e, NaI, acetone, 25 °C; f, Ac_2O, pyridine, THF, cat. DMAP, 25 °C; g, 5% aqueous HCl wash.

Lithium aluminum hydride reduction of the THP-protected dimethyl ester of (*S*)-(-)-malic acid [(*S*)-**2c**] to give (*S*)-**3a**[1c] followed by immediate mesylation[3] afforded (*S*)-**4a** (65–70% overall).[1c] Acid-catalyzed hydrolysis of the THP ether (74%) and subsequent mild base treatment of the crystalline dimesylate alcohol (*S*)-**4b** afforded (*S*)-**5** (98%). Treatment of (*S*)-**5** with sodium iodide (2.0 equiv, acetone, 25 °C, 48 h, 81%)[4] gave (*S*)-(-)-4-iodo-1,2-epoxybutane [(*S*)-(-)-**1**], $[\alpha]^{24}_D$ -13.52° (*c* 5.00, CH_2Cl_2).[4]

The preparation of (*R*)-(+)-**1** from (*S*)-(-)-malic acid (**2a**) requires inversion of the chirality about the hydroxyl center and is detailed below. Lithium aluminum hydride reduction of (*S*)-**2d** as described previously gave the labile, protected triol (*S*)-**3b**.[1d] Immediate acetylation in the

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(2) (*R*)-, (*S*)-, and racemic 4-iodo-1,2-epoxybutane [(2-iodoethyl)oxirane] have not been previously described. Racemic and (*S*)-4-bromo-1,2-epoxybutane [(2-bromoethyl)oxirane] and their use have been described; see ref 1b and: Cruickshank, P. A.; Fishman, M. *J. Org. Chem.* **1969**, *34*, 4060.

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(4) The conditions for the mesylate displacement employing sodium iodide–acetone represent optimized conditions. The use of less sodium iodide (1.0 equiv) or shorter reaction times (24 h) results in significant amounts of recovered starting material, whereas the use of more sodium iodide (4.0 equiv) but not longer reaction times affords varying amounts of 1,4-diiodo-2-butanol. The optical rotations of impure samples of (*R*)- and (*S*)-4-iodo-1,2-epoxybutane were *higher* than those of purified material. The chiral purity of each enantiomer could be determined by ^{1}H NMR, using the chiral shift reagent tris[3-[(trifluoromethyl)hydroxymethylene]-*d*-camphorato]europium(III) [Eu(tfc)$_3$]. In the presence of 0.1–0.3 molar equiv of [Eu(tfc)$_3$], (*S*)-**1** and (*R*)-**1** were determined to be 99 ± 1% and 98 ± 2% enantiomerically pure, respectively; cf. ref 1b and 1g.

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