chloroform brought the melting point up to 149–150°. The nmr spectrum of this substance was identical in all respects with that of an authentic specimen of 4,4-dimethylcholestanol with the exception of the complete absence of a band at τ 9.04, present in the undeuterated sample; the infrared showed peaks at 3600, 3380, and 2217 cm^{-1} (w).

4β-Methyl-4α-trideuteriomethyl-5α-cholestan-3-one (VI).—A solution of 241 mg of the deuterated dimethylcholestenone IV in 25 ml of ethyl acetate containing 1 drop of 70% perchloric acid was hydrogenated at atmospheric pressure using 100 mg of platinum oxide as catalyst. The hydrogenation proceeded for 140 min. The reaction was filtered, 2 drops of pyridine was added, and the solution was taken to dryness. The crude product was dissolved in acetone (20 ml) and oxidized with 0.2 ml of a reagent prepared by dissolving 66.6 g of chromium trioxide in 57.5 ml of concentrated sulfuric acid and making the solution up to 250 ml with distilled water. Methanol was added to decompose the excess chromic acid and the mixture was filtered through Celite, evaporated, taken up in benzene, and chromatographed on a column of 15 ml of alumina in benzene. Fractions (5 ml) were taken. Fractions 3 and 4 were combined and evaporated to give 223 mg of a crystalline product, 95% pure by vpc analysis. Recrystallization from methanol–chloroform gave 173 mg of pure 4β-methyl-4α-trideuteriomethyl-5α-cholestan-3-one (VI): mp 98–100°; infrared peaks, 2230, 2065, 1708 cm^{-1}.

Deuterated 3,4-Seco-4-methyl-4-methylenecholestan-3-oic Acid (VIII).—Trideuterio-4,4-dimethylcholestanone (VI) was subjected to Baeyer–Villiger oxidation with *m*-chloroperbenzoic acid as has been described for the nondeuterated compound.[1] The lactone VII thus obtained (33 mg) was placed in a test tube which was flushed with nitrogen. The compound was heated in a Wood's metal bath at 205°. The course of the pyrolysis was followed by thin layer chromatography and the reaction was complete after 50 min. The reaction was cooled and the product dissolved in chloroform, reduced to a small volume, and taken up in a little methanol. After removal of a flocculent residue by filtration, the product, deuterated 3,4-seco-4-methyl-4-methylenecholestan-3-oic acid (VIII), crystallized slowly, mp 142–145°. The nmr spectrum of this sample was determined at 100 Mc and compared with a similar sample obtained from nondeuterated material. The shape of the signal corresponding to the methylene protons at τ 5.14 and 5.32 was identical in the two samples. There was no broadening of the τ 8.25 allylic methyl peak in the deuterated sample.

Integrating the intensity of methylene bands using the C-18 methyl band at τ 9.35 as an internal standard gave values of 60 ± 5% for the amount of $=CH_2$ methylene group present in the deuterated sample relative to the undeuterated acid. The allylic methyl group at τ 8.25 was less reliable and gave values of 35 ± 10% of allylic methyl relative to the undeuterated acid.

Treatment of Lactone VII with Strong Acid.—The deuterated lactone VII (33 mg) was dissolved in 4 ml of a solution of 10% sulfuric acid in glacial acetic acid. The reaction was shaken for 10 min and then poured into water. The sulfuric acid present was neutralized with a calculated amount of potassium bicarbonate and the product was extracted with chloroform. The organic phase was washed with saturated sodium chloride solution, dried over sodium sulfate, and evaporated. Crystallization from methanol gave 22 mg of acid VIII, mp 146–148°, whose infrared and nmr spectra were identical with those of the undeuterated acid.

Registry No.—I, 2202-04-2; IV, 14128-48-4; V, 14154-63-3; VI, 14154-64-4; VIII, 14128-49-5.

17-Acetyl-13β-etiojerv-16-en-3β-ol *via* Performic Acid Oxidation of the Sapogenin Side Chain

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Although the Baeyer–Villiger degradation of the sapogenin side chain has been known for many years,[1] its applications have been severely limited by the absence of a route for converting the 16,20-dihydroxypregnane produced to the more tractable pregn-16-en-20-one. The problems posed in this last transformation were investigated when the classical pseudomerization and chromic acid degradation[2] failed in the 18-substituted C-nor-D-homo sapogenins.[3] The initial study reported here was made in the 18-unsubstituted derivatives.[4]

Performic acid oxidation of the rearranged sapogenin **3** followed by saponification provided as the chief product the triol **4**. The stereochemistry of the 16- and 20-hydroxyl groups of this compound is assigned by analogy to that determined in the pregnanes.[4,5] Attempts to convert this triol to the unsaturated ketone **9a** *via* selective esterification (benzoylation) led mainly to the 3,20-dibenzoate. The structure of this diester was demonstrated by oxidation to the corresponding 16-ketone (**8**-dibenzoate), which lacked an acetyl signal in the nmr; on treatment with base, β elimination of the ester group occurred, yielding an amorphous mixture of isomeric unsaturated (Δ^{17}) ketones (λ_{max} 238 mμ) in accord with the assigned structure. Selective hydrolysis of the triacetate of **4** also failed to yield the desired 20-monohydroxy compound in a reasonable yield.

The conversion of the sapogenin **3** to the unsaturated ketone **9a** was realized by use of the mixed ester obtained directly from the performic acid oxidation. The structure of this material, hitherto unexplored, was expected to be that resulting from oxidation of the tertiary carbon at position 20 adjacent to the latent 22-carbonyl, leaving a 20-valerate; the 16- and 26-hydroxyls would be esterified by the formic acid present (see **2**). Accordingly, the formate esters in the crude Baeyer–Villiger product were hydrolyzed by contact with alumina[6] and the resulting diol was etherified (presumably at 16 and 26) with dihydropyran. Saponification of the remaining ester functions (at 3, and, presumably, 20) followed by oxidation and base treatment led to a mixture lacking an appreciable acetyl signal in the nmr. This result suggested that the actual structure of the Baeyer–Villiger product was the reverse of that expected, and that in fact the valerate ester was at position 16 (**5**). The amorphous alcohol **6a**, obtained by the alumina treatment, was therefore oxidized directly; the resulting material, after base treatment, afforded the desired unsaturated ketone **9a**. The over-all yield (50%) from the sapogenin **3** compares favorably with that from the alternate pseudomerization procedure (45%).[4a]

The most probably mechanistic course of the Baeyer–Villiger oxidation is attack of peroxide at C-22 of a protonated hemiketal such as i. A subsequent bond shift would produce the symmetrical dioxolane intermediate ii[7] which is then preferentially solvolyzed at the 20-oxygen.

(1) R. E. Marker, E. Rohrmann, H. M. Crooks, E. L. Whittle, E. M. Jones, and D. L. Turner, *J. Am. Chem. Soc.*, **62**, 525 (1940). For a more recent publication modifying the original method, see K. Morita, S. Noguchi, H. Kono, and T. Miki, *Chem. Pharm. Bull.* (Tokyo), **11**, 90 (1963), and following papers. Brief mention of this reaction in the C-nor-D-homo steroids appeared here, after completion of the present work.

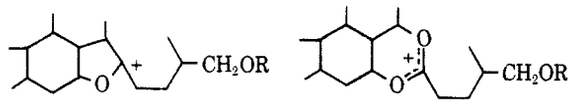
(2) See, for example, M. E. Wall and S. Serota, *Tetrahedron*, **10**, 238 (1960).

(3) Work to be published in a forthcoming communication.

(4) (a) W. F. Johns, *J. Org. Chem.*, **29**, 2545 (1964); (b) H. Mitsuhasi, K. Shibata, T. Sato, and Y. Shimizu, *Chem. Pharm. Bull.* (Tokyo), **12**, 1 (1964).

(5) C. H. Halsall, *Org. Reactions*, **9**, 79 (1957).

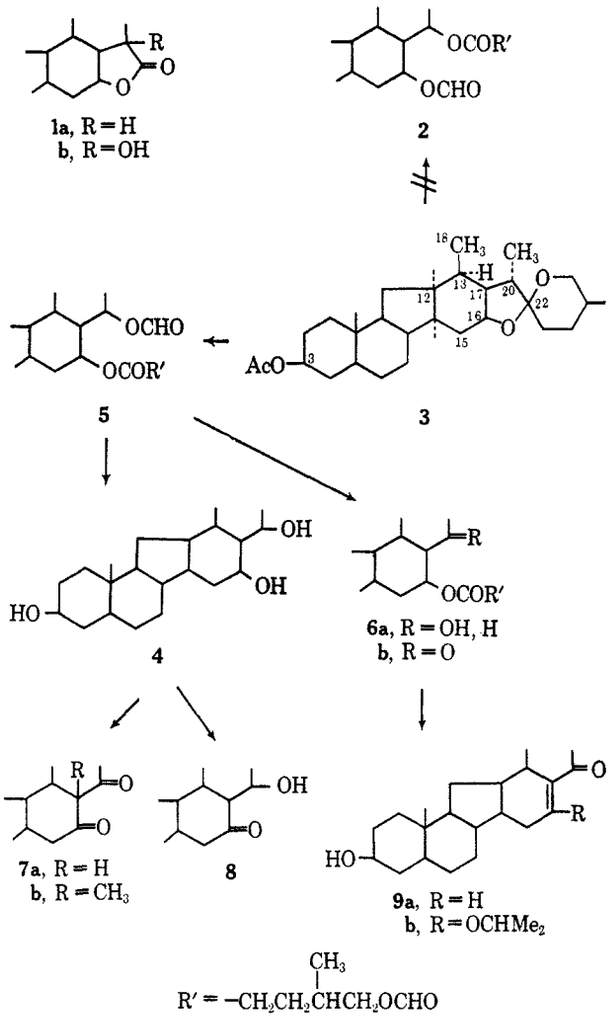
(6) F. C. Chang and R. T. Blickenstaff, *J. Am. Chem. Soc.*, **80**, 2906 (1958).



i ii

Concurrent studies directed to the conversion of the triol **4** to the ketone **9a** dealt with selective oxidation of the triol 3-monobenzoate. This compound, most readily obtained by esterification (ar position 3) of the corresponding 16,20-acetonide and subsequent hydrolysis of the acetonide group, was oxidized to yield predominantly the 20-hydroxy-16-ketone **8**, a result reflecting the axial nature of the 16-hydroxyl.[8,9] The structure of the hydroxy ketone **8** is clear from its lack of acetyl absorption in the nmr, from its dissimilarity to the known 16-hydroxy-20-ketone,[4a] and from its benzoylation to the same 3,20-dibenzoate (of **8**) prepared above. The ORD curve of **8** showed an unexpected negative Cotton effect, best rationalized by a D-ring twist conformation with the prows (C-13,15) above the plane of the ring.[10]

Although none of the 16-hydroxy-20-ketone was found in any of the oxidation studies, the diketone **7a** was an appreciable component even when limited amounts of chromic acid were used. Alkylation of the diketone with isopropyl iodide gave predominantly the enol ether **9b**, whereas methyl iodide gave the C-alkylated material **7b**. Thus none of the major oxidation products or their simple transformation products could be easily used in the preparation of the unsaturated ketone **9a**.



1a, R = H
b, R = OH

2

5

3

4

6a, R = OH, H
b, R = O

7a, R = H
b, R = CH_3

8

9a, R = H
b, R = $OCHMe_2$

R′ = $-CH_2CH_2\underset{\displaystyle CH_3}{\underset{|}{C}}HCH_2OCHO$

Experimental Section[11,12]

17β-Ethyl-13β-etiojervan-3β,16β,20α-triol (4).—Five grams of well-pulverized sapogenin **3**[13] was stirred in 100 ml of 85% formic acid and 10 ml of 30% aqueous hydrogen peroxide at 70–75° for 1 hr. After an inital heating period, the reaction became slightly exothermic and the compound dissolved. After 1 hr the solution was cooled, diluted with water, and extracted with benzene, yielding 5.2 g of an oil (preparation A). This product was heated at reflux in 100 ml of methanol and 20 ml of 5% aqueous potassium hydroxide for 1 hr. The solution, on dilution with water, afforded 3.4 g of triol **4**, mp 195–205°. The product was recrystallized from acetone to give 2.41 g of the pure material, mp 212–214°, λ_{max}^{KBr} 2.98 μ, $[\alpha]D$ 50° (EtOH).

Anal. Calcd for $C_{21}H_{36}O_3$: C, 74.95; H, 10.78. Found: C, 74.92; H, 10.70.

The two known[13] lactones **1a** and **1b** were found in the mother liquors of this reaction (less than 10% combined yield)

17β-Ethyl-13β-etiojervan-3β,16β,20α-triol triacetate, prepared from **4** with acetic anhydride–pyridine, was purified by recrystallization from acetone to give the pure sample, mp 214–215°; λ_{max} 5.79 μ.

Anal. Calcd for $C_{27}H_{42}O_6$: C, 70.10; H, 9.15. Found: C, 70.38; H, 9.29.

Attempts at selective hydrolysis of this material led to mixtures containing little of the desired 3,16-diacetate (of **4**).

17β-Ethyl-13β-etiojervan-3β,16β,20α-triol 3,20-Dibenzoate and 17β-Ethyl-13β-etiojervan-3β,16β,20α-triol 3-Benzoate.—A solution of 1.1 g of the triol **4** in 40 ml of pyridine containing 0.75 ml of benzoyl chloride (2.0 mole equiv) was allowed to stand at room temperature for 20 hr. The solution was diluted with 5 ml of water and heated at 100° for 20 min. The product was extracted with benzene and chromatographed: elution with 1% ethyl acetate–benzene yielded first 0.31 g of material which was recrystallized from methylene chloride–methanol to give 0.12 g of the 3,20-dibenzoate (of **4**), mp 159–160°, λ_{max} 2.72 and 5.82 μ.

Anal. Calcd for $C_{35}H_{44}O_5$: C, 77.01; H, 8.14. Found: C, 76.82; H, 8.36.

Elution with 20% ethyl acetate–benzene gave material which was recrystallized from acetone to yield the pure 3-monobenzoate (of **4**), mp 193–196°, λ_{max} 2.75, 5.82 μ.

Anal. Calcd for $C_{28}H_{38}O_4$: C, 76.67; H, 8.63. Found: C, 76.96; H, 8.72.

Attempts to obtain the tribenzoate led to an amorphous material which lacked hydroxyl absorption in the infrared and did not afford a satisfactory elemental analysis.

3β,20α-Dihydroxy-17β-ethyl-13β-etiojervan-16-one 3,20-Dibenzoate.—A solution of 90 mg of the 3,20-dibenzoate (of **4**) in 10 ml of acetone was treated with 0.5 ml of Jones reagent[14] with

(7) This ion is analogous to Winstein's postulated intermediate in solvolyses with neighboring group participation; see G. Schneider and L. K. Láng, *Chem. Commun.*, 13 (1967), and references cited therein.

(8) E. L. Eliel, N. L. Allinger, S. J. Angyal, and G. A. Morrison, "Conformational Analysis," Interscience Publishers, Inc., New York, N. Y., 1965, p 271.

(9) Selective oxidation of pregna-16β,20α-diols affords a similar product; see S. Noguchi, M. Imanishi, and K. Morita, *Chem. Pharm. Bull.* (Tokyo), **12**, 1184 (1964).

(10) C. Djerassi and W. Klyne, *J. Chem. Soc.*, 2390 (1963); *Proc. Natl. Acad. Sci., U. S.*, **48**, 1093 (1962).

(11) We wish to thank Dr. R. T. Dillon and staff for the analyses, spectra, and rotations reported here. The infrared spectra were determined in chloroform, the ultraviolet spectra were determined in methanol, and the rotations were determined in chloroform (1%). The nmr spectra were run in deuteriochloroform on a Varian Model A-60 spectrometer at 60 Mc, with tetramethylsilane as an internal standard; $\Delta\nu$ = 0 cps.

(12) We are indebted to Dr. E. G. Daskalakis and staff for performing all of the chromatographic analyses described in this paper; these were routinely run on silica gel (60 × the weight of the adsorbed compound).

(13) See ref 4a. The nomenclature and numbering of these compounds are described in this paper.

(14) C. Djerassi, R. R. Engle, and A. Bowers, *J. Org. Chem.*, **21**, 1547 (1956).

stirring at room temperature for 10 min. The solution was diluted with water and 1 ml of methanol. The resulting precipitate was separated and recrystallized from acetone yielding 62 mg of the 3,20-dibenzoate (of **8**), mp 212–215°; λ_{max}^{KBr} 5.82 μ; $\Delta\nu$ 82 and 88 (21-CH_3) cps; the nmr showed no $COCH_3$ signal.

Anal. Calcd for $C_{35}H_{42}O_5$: C, 77.46; H, 7.80. Found: C, 77.29; H, 7.68.

Treatment of this ketone in refluxing *t*-butyl alcohol with aqueous base gave an amorphous unsaturated ketone mixture, λ_{max} 238 (6130) mμ; λ_{max} 2.75 and 5.91 μ; $\Delta\nu$ 317 (C=C*H*) cps. The nmr further indicated that both benzoate and acetyl groups were absent.

17β-Ethyl-13β-etiojervan-3β,16β,20α-triol 16,20-Acetonide.—*p*-Toluenesulfonic acid (0.40 g) was added to a slurry of 4.71 g of the triol **4** in 40 ml of acetone with stirring, effecting rapid solution of starting material. After 40 min the solution was diluted with 200 ml of aqueous sodium bicarbonate. The product, 4.9 g, mp 187–197°, was separated and recrystallized from acetone–hexane to afford the pure acetonide, mp 202–204°, λ_{max}^{KBr} 2.82 μ.

Anal. Calcd for $C_{24}H_{40}O_3$: C, 76.55; H, 10.71. Found: C, 76.38; H, 10.56.

17β-Ethyl-13β-etiojervan-3β,16β,20α-triol 3-benzoate 16,20-acetonide was prepared by treatment of the acetonide with benzoyl chloride–pyridine yielding the 3-monobenzoate, mp 125–128°, λ_{max} 5.82 μ (s), $[\alpha]_D$ 5°.

Anal. Calcd for $C_{31}H_{44}O_4$: C, 77.46; H, 8.85. Found: C, 77.54; H, 9.08.

Heating an acidic methanol solution of the acetonide provided the 3-monobenzoate 16,20-diol in high yield.

3β-Hydroxy-13β-etiojerv-16-en-20-one (9a). A. Baeyer–Villiger Oxidation.—A portion of preparation A (see first experiment) was chromatographed (on silica gel), yielding first a crystalline material eluted with 3% ethyl acetate–benzene. Recrystallization of this material from acetone–hexane gave pure 13β-etiojervan-3β,16β,20α-triol 3-acetate 16,20-diformate, mp 170–175°, λ_{max} 5.79 μ, $\Delta\nu$ 479 (OC*H*O) cps.

Anal. Calcd for $C_{25}H_{38}O_6$: C, 69.09; H, 8.81. Found: C, 68.99; H, 9.10.

Later fractions yielded the amorphous, mixed ester **5** (contaminated with some of the isomer **2**).

B. Formate Hydrolysis.—The crude ester (preparation A) was adsorbed on 400 g of Merck alumina. The column was eluted with benzene and then with ethyl acetate, the combined fractions yielding 0.90 g of a mixture of starting material **3** and lactones **1a** and **1b**. Elution with methanol afforded 10.5 g of a stiff foam, **6a**, which was decolorized in ether with Darco. The resulting material exhibited λ_{max} 2.78 and 5.79 μ; $\Delta\nu$ 69 and 76 (21-CH_3), 122 (3-OAc), 208 and 213 (CH_2OH) cps.

Anal. Calcd for $C_{29}H_{48}O_6$: C, 70.69; H, 9.82. Found: C, 71.12; H, 9.95.

The unesterified hydroxyl groups at positions 20 and 26 were converted to their pyranyl ethers. Subsequent saponification of the ester functions at positions 3 and 16 followed by chromic acid oxidation and base treatment afforded a mixture containing no appreciable amount of the desired unsaturated ketone **9a**.

C. Chromic Acid Oxidation.—A solution of 4.5 g of the ester **6a** in 50 ml of acetone at 5° was treated over a 10-min period with 12 ml of Jones reagent.[14] After an additional 10 min, the solution was diluted with 5 ml of methanol and with water. The product **6b**, isolated by extraction with methylene chloride, was 4.3 g of an amorphous solid, $\Delta\nu$ 127 cps ($COCH_3$).

D. Side-Chain Elimination.—A solution of 4.2 g of the ketone **6b** in 200 ml of *t*-butyl alcohol and 40 ml of 5% aqueous potassium hydroxide was heated at reflux with stirring under an atmosphere of nitrogen for 1 hr. The diluted solution was concentrated and then extracted with ether (dilute base wash). An ultraviolet spectrum of the product (2.3 g) implied 55% of the material was the unsaturated ketone **9a** [λ_{max} 237 (8000) mμ]. Chromatography yielded fractions (1.8 g), eluted with 4% ethyl acetate–benzene, which were recrystallized from ether–hexane to yield 0.88 g of the unsaturated ketone **9a**, mp 158–162°, λ_{max} 236 mμ (11,100), identical in the infrared with an authentic sample.[4]

3β,20α-Dihydroxy-17β-ethyl-13β-etiojervan-16-one 3-Benzoate.—N-Bromosuccinimide (0.25 g) was added to a stirred solution of 0.55 g of the 3-monobenzoate (of **4**) in 20 ml of dioxane and 2 ml of water. After 18 hr the solution was diluted with water and the resulting precipitate, 0.51 g, was separated and recrystallized from ether to yield 0.10 g of the 3-monobenzoate of ketone **8**, mp 155–158°. Recrystallization from acetone gave the pure sample as a different crystalline form, mp 191–193°; λ_{max} 2.82, 5.81, and 5.89 μ; $\Delta\nu$ 47 and 54 (18-CH_3), 53 (19-CH_3), 68 and 75 (21-CH_3) cps. ORD (dioxane) gave $[\phi]_{320}$ −5740°, $[\phi]_{298}$ 0°; a = −87.[15]

Anal. Calcd for $C_{28}H_{38}O_4$: C, 76.67; H, 8.73. Found: C, 76.91; H, 8.75.

Esterification of this hydroxy ketone with benzoyl chloride in pyridine provided the known 3,20-dibenzoate (of **8**) in good yield. Chromium trioxide–pyridine oxidation (0°, 1 hr) of the 16,20-diol afforded an alternate, but less satisfactory, route to prepare the hydroxy ketone **8**; no 16,20-dione, **7a**, was formed with either of these reagents.

17β-Acetyl-3β-hydroxy-13β-etiojervan-16-one 3-Benzoate (7a, 3-Benzoate).—A solution of 10.6 ml of Jones reagent[14] was added dropwise over a 10-min period to a solution of 7.0 g of the 3-monobenzoate of **4** in 350 ml of tetrahydrofuran at −40°. The solution was kept at −40° for 1 hr and then allowed to warm to 0°. After 18 hr the product was isolated from the diluted reaction mixture by extraction with ethyl acetate, affording 5.8 g of an amorphous solid, λ_{max} (0.1 *N* KOH–MeOH) 308 (11,400) mμ, corresponding to 68% of the pure diketone **7a**. Chromatography yielded material (2.69 g) eluted with 1% ethyl acetate–benzene which was crystallized from acetone–hexane and recrystallized from methylene chloride–methanol to yield 1.58 g of **7a**, mp 173–175°; λ_{max} 5.82 and 6.22 μ; $\Delta\nu$ 53 (19-CH_3), 60 and 67 (18-CH_3), 126 ($COCH_3$), and 962 (OH) cps. The intensity of the ultraviolet maximum varied depending on the degree of enolization; a more consistent value was obtained in 0.1 *N* KOH–MeOH, λ_{max} 308 mμ (16,800), although this maximum disappeared in less than 24 hr in the basic solution.

Anal. Calcd for $C_{28}H_{36}O_4$: C, 77.03; H, 8.31. Found: C, 77.02; H, 8.29.

When the amount of chromium trioxide used was reduced to 1.1 mole equiv the product consisted of 25% starting material, 35% diketone **7a**, and 25% hydroxy ketone **8** (chromatographic analysis).

17-Acetyl-13β-etiojerv-16-en-3β,16-diol 3-Benzoate 16-Isopropyl Ether (9b).—Potassium carbonate (2 g) and 0.45 g of the diketone **7a** were stirred in 50 ml of acetone and 2 ml of 2-propyl iodide at reflux under an atmosphere of nitrogen for 22 hr. The mixture was diluted with water and the product, 0.53 g of a foam, was isolated by extraction with methylene chloride. The material crystallized from ether and was recrystallized from methylene chloride–methanol to yield 0.10 g of the pure enol ether **9b**, mp 175–178°; λ_{max} 5.82 and 6.24 μ; λ_{max} 228 mμ (14,500) and 272 (12,600); $\Delta\nu$ 144 ($COCH_3$) cps.

Anal. Calcd for $C_{31}H_{42}O_4$: C, 77.78; H, 8.84. Found: C, 77.56; H, 8.69.

17-Acetyl-3β-hydroxy-17-methyl-13β-etiojervan-16-one 3-Benzoate (7b).—A mixture of 0.50 g of the diketone **7a** and 2.5 g of potassium carbonate in 50 ml of acetone and 2 ml of methyl iodide were stirred at reflux under an atmosphere of nitrogen for 6 hr. The mixture was diluted with water and extracted with benzene. Recrystallization of the product from acetone–hexane (Darco) yielded 0.40 g of the pure diketone **7b**, mp 181–184°; λ_{mox} 5.81 (s) μ; $\Delta\nu$ 53 (19-CH_3), 60 and 67 (18-CH_3), 83 (17-CH_3), and 132 ($COCH_3$) cps.

Anal. Calcd for $C_{29}H_{38}O_4$: C, 77.30; H, 8.57. Found: C, 77.36; H, 8.57.

Registry No.—**4**, 14194-97-9; **4**-triacetate, 14320-25-3; 3,20-dibenzoate of **4**, 14194-98-0; 3-monobenzoate of **4**, 14320-26-4; **6a**, 14194-99-1; **7a**-3-benzoate, 14195-00-7; **7b**-3-benzoate, 14195-01-8; 3,20-dibenzoate of **8**, 14195-02-9; 3-monobenzoate of ketone **8**, 14195-03-0; **9a**, 14195-04-1; **9b**-3-benzoate, 14195-05-2; 17β-ethyl-13β-etiojervan-3β,16β,20α-triol 16,20-acetonide, 14195-06-3; 17β-ethyl-13β-etiojervan-3β,16β,20α-triol 3-benzoate 16,20-acetonide, 14195-07-4; 13β-etiojervan-3β,16β,20α-triol 3-acetate 16,20-diformate, 14195-08-5.

Acknowledgment.—The author wishes to acknowledge the capable assistance of Mr. Ivar Laos.

(15) This ORD spectrum was kindly provided by Dr. N. L. McNiven.