A Rearrangement of 4β,5β-Epoxy-6α-hydroxyandrostan-17β-yl Acetate

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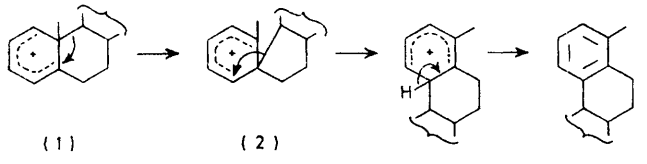
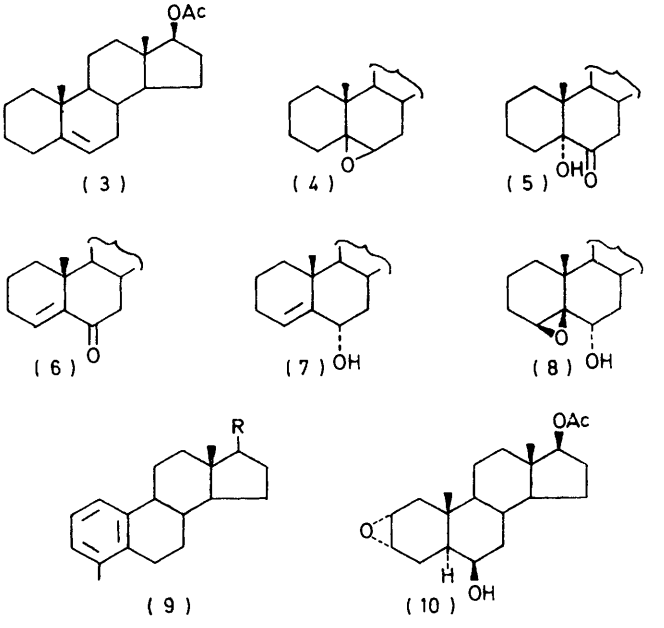
4β,5β-Epoxy-6α-hydroxy- and 2α,3α-epoxy-6β-hydroxy-androstan-17β-yl acetates have been prepared. Both gave 4-methyloestratrien-17β-yl acetate on treatment with hydrobromic acid in glacial acetic acid. The former also gave 6-oxoandrost-4-en-17β-yl acetate.

THE formation of aromatic steroids from hydroxy-epoxides on treatment with refluxing hydrobromic acid in glacial acetic acid [1] is a general class of steroidal rearrangement which is related to the dienol–benzene reaction.[2] The aromatization may proceed through a spiranic intermediate (2).[2,3] 4β,5β-Epoxy-6α-hydroxy-androstane-17β-yl acetate (8) has an interesting disposition of functionality with regard to the formation of such an intermediate. Protonation of the epoxide, which lies *trans* to the migrating C(9)–C(10) bond, could initiate the formation of spiranic intermediate prior to elimination of the 6α-hydroxy-group, thus trapping this double bond equivalent on ring B. Since elimination could occur after rearrangement, such a step would not preclude aromatization but would merely alter the order of events. On the other hand alternative reaction pathways leading, for example, to ketonic products [4] might predominate. The substrate was prepared in the following manner.



SCHEME 1



Androst-5-en-17β-yl acetate (3) [5] was treated with *m*-chloroperbenzoic acid. The 5α,6α-, and 5β,6β-epoxides (4) were separated [6,7] (α-epoxide 6-H τ 7·12, *J* 4 Hz;

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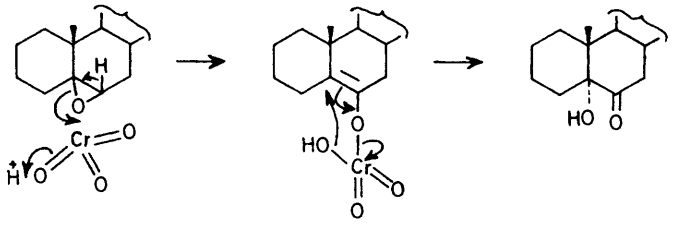
[3] J. R. Hanson, *Chem. Comm.*, 1971, 1343.
[4] D. N. Kirk and M. P. Hartshorn, 'Steroid Reaction Mechanisms,' Elsevier, London, 1968, pp. 354—355; B. N. Blackett, J. M. Coxon, M. P. Hartshorn, and K. E. Richards, *Tetrahedron*, 1969, **25**, 4999.
[5] A. Crastes de Paulet and J. Bascoul, *Bull. Soc. chim. France*, 1966, 939.
[6] K. D. Bingham, T. M. Blaiklock, R. C. B. Coleman, and G. D. Meakins, *J. Chem.Soc.* (*C*), 1970, 2330.
[7] A. D. Cross, *J. Amer. Chem. Soc.*, 1962, **84**, 3206.

19-H τ 8·94; β-epoxide 6-H τ 6·99, J 3 Hz; 19-H τ 9·00). Oxidation of both the 'α' and 'β'-epoxides with chromium trioxide [8] gave 5α-hydroxy-6-oxoandrostan-17β-yl acetate (5). The formation of the 5α-hydroxy-steroid from both epoxides suggests that insertion of the hydroxy-group proceeds through a 6-enol chromate rather than *via* diaxial opening of the epoxide (see Scheme 2). The ketol was then dehydrated



SCHEME 2

with thionyl chloride to give the known 6-oxoandrost-4-en-17β-yl acetate (6).[9] The ketone was reduced with sodium borohydride to give a C-6 alcohol (7). Saturated 6-ketones are reduced by hydride reagents to afford the 6β-alcohols.[10] However the product in the case of the unsaturated ketones was shown to be the 'α'-alcohol. Androst-5-en-17β-yl acetate was osmylated and the resulting 5α,6α-diol acetylated, and then dehydrated with thionyl chloride to afford androst-4-ene-6α,17β-diol diacetate, identical with the product of acetylation of the sodium borohydride reduction product. Epoxidation of 6α-hydroxyandrost-4-en-17β-yl acetate (7) with *m*-chloroperbenzoic acid gave a 1 : 1 mixture of the 4α,5α- and 4β,5β-epoxides (8). Surprisingly when vanadyl acetonylacetate and t-butyl hydroperoxide [11] were used for epoxidation, the major product (3 : 1) was the 4β,5β-epoxide. Epoxidation of androst-4-ene-6α,17β-diyl diacetate with *m*-chloroperbenzoic acid followed the normal pattern in which reaction occurred predominantly from the less hindered face of the molecule to afford the 4α,5α-epoxide as the major product. The stereochemistry of these hydroxy-epoxides was established by reduction of their acetates with lithium aluminium hydride and reacetylation of the triols. 4α,5α-Epoxyandrostane-6α,17β-diyl diacetate gave 5α-hydroxyandrostane-6α,17β-diyl diacetate described above, whereas the 4β,5β-epoxide gave its 5β-epimer.

Treatment of 4β,5β-epoxy-6α-hydroxyandrostan-17β-yl acetate with 48% hydrobromic acid in glacial acetic acid gave 17β-acetoxy- and 17-bromo-4-methyloestra-1,3,5(10)-triene (9; R = OAc or Br) [12] and 6-oxoandrost-4-en-17β-yl acetate (6). Thus the formation of oestratrienes is still the predominant reaction pathway; the unsaturated ketone may arise by the discharge of the C-5 carbocation through the loss of a proton from C-6.

In the 4β,5β-epoxy-6α-hydroxy-steroid, the functional groups are adjacent and one might anticipate some neighbouring group participation in the opening of the epoxide ring. However in 2α,3α-epoxy-6β-hydroxyandrostan-17β-yl acetate (10) the epoxide and hydroxy-groups are separated. 6-Oxoandrost-2-en-17β-yl acetate [13] was epoxidized with *m*-chloroperbenzoic acid and then the carbonyl group was reduced with sodium borohydride to afford the 6β-alcohol. The same product containing a 2α,3α-epoxide was obtained by epoxidation of 6β-hydroxyandrost-2-en-17β-yl acetate; thus the 6β-hydroxy-group is sufficiently distant not to direct reaction at the C-2 double bond. 4-Methyloestra-1,3,5(10)-trien-17β-yl acetate was formed by treatment of this hydroxy-epoxide with hydrobromic acid in glacial acetic acid. In this instance the carbonium ion (1) which precedes the spiranic intermediate may be readily formed by hydrolysis of the epoxide and subsequent dehydration reactions.

EXPERIMENTAL

General experimental details have been described previously.[14]

Epoxidation of Androst-5-en-17β-yl Acetate (3).—Androst-5-en-17β-yl acetate [5] (4·0 g) in benzene (75 ml) was treated with *m*-chloroperbenzoic acid (5·0 g) at room temperature for 1 h. The product was recovered and chromatographed on alumina. Elution with 5% ethyl acetate–light petroleum gave 5β,6β-*epoxyandrostan*-17β-*yl acetate* (1·0 g) as needles, m.p. 135—136° (from methanol), $[\alpha]_D^{20}$ −23° (*c* 0·2) (Found: C, 75·8; H, 9·7. $C_{21}H_{32}O_3$ requires C, 75·9; H, 9·7%), $\nu_{max.}$ 1740 cm^{-1}, τ 9·24 (3H, s, 18-H), 9·00 (3H, s, 19-H), 7·98 (3H, s, 17-Ac), 6·99 (1H, d, J 3 Hz, 6-H), and 5·42 (1H, q, J 8 and 10 Hz, 17-H). Elution with 10% ethyl acetate–light petroleum gave 5α,6α-*epoxyandrostan*-17β-*yl acetate* (2·0 g) as needles, m.p. 129—130° (from methanol), $[\alpha]_D^{20}$ −89° (*c* 0·2) (Found: C, 76·05; H, 9·5. $C_{21}H_{32}O_3$ requires C, 75·9; H, 9·7%), $\nu_{max.}$ 1740 cm^{-1}, τ 9·26 (3H, s, 18-H), 8·94 (3H, s, 19-H), 7·97 (3H, s, 17-Ac), 7·12 (1H, d, J 4 Hz, 6-H), and 5·43 (1H, q, J 8 and 10 Hz, 17-H).

5α-*Hydroxy*-6-*oxoandrostan*-17β-*yl Acetate* (5).—5α,6α-Epoxyandrostan-17β-yl acetate (2·4 g) was dissolved in ethyl methyl ketone (24 ml) at 35—40 °C and treated with aqueous 75% chromium trioxide (2·4 ml) dropwise over 20. min. The product was recovered in ethyl acetate and chromatographed on alumina. Elution with 20% ethyl acetate–light petroleum gave the 6-*ketone* (1·1 g) as needles, m.p. 162—163° (from light petroleum), $[\alpha]_D^{20}$ −78° (*c* 0·2) (Found: C, 72·3; H, 9·1. $C_{21}H_{32}O_4$ requires C, 72·4; H, 9·3%), $\nu_{max.}$ 3520, 1740, and 1720 cm^{-1}, τ 9·2 (6H, s, 18- and 19-H), 7·98 (3H, s, 17-Ac), and 5·4 (1H, q, J 8 and 10 Hz, 17-H).

5β,6β-Epoxyandrostan-17β-yl acetate (100 mg) was treated similarly to afford the same product (40 mg), m.p. 162—163°, identified by its i.r. spectrum.

6-*Oxoandrost*-4-*en*-17β-*yl Acetate* (6).—A solution of thionyl chloride (1·5 ml) in pyridine (5 ml) was added to a solution of the ketone (5) (500 mg) in pyridine (15 ml) at 0°.

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[11] K. B. Sharpless and R. C. Michaelson, *J. Amer. Chem. Soc.*, 1973, **95**, 6136.
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[13] H. Velgora, V. Czerny, J. Hora, L. Labler, A. Kasal, K. Slama, and F. Sorm, *Coll. Czech. Chem. Comm.*, 1968, **33**, 2226; J. S. Cochrane and J. R. Hanson, *J. Chem. Soc.* (*C*), 1971, 3730.
[14] J. R. Hanson and T. D. Organ, *J. Chem. Soc.* (*C*), 1970, 513.

After 30 min, the solution was poured into dilute hydrochloric acid (150 ml) and the Δ^4-6-ketone (200 mg) was recovered in ethyl acetate. It crystallized from methanol as plates, m.p. 160—161°, $[\alpha]_D^{20}$ +28° (*c* 0·2) (lit.,[9] m.p. 159—164°, $[\alpha]_D^{20}$ +26°) (Found: C, 76·2; H, 9·3. Calc. for $C_{21}H_{30}O_3$: C, 76·3; H, 9·15%), $\nu_{max.}$ 1750, 1690, and 1630 cm^{-1}, τ 9·19 (3H, s, 18-H), 9·04 (3H, s, 19-H), 7·96 (3H, s, 17-Ac), 5·4 (1H, q, *J* 8 and 10 Hg, 17-H), and 3·58 (1H, t, *J* 4 Hz, 4-H).

Reduction of 6-*Oxoandrost*-4-*en*-17β-*yl Acetate*.—The Δ^4-6-ketone (2·0 g) in methanol (100 ml) was treated with sodium borohydride (400 mg) for 30 min at 0°. The steroid was recovered in ethyl acetate and chromatographed on alumina. Elution with 15% ethyl acetate–light petroleum gave 6α-*hydroxyandrost*-4-*en*-17β-*yl acetate* (7) (1·2 g) as needles, m.p. 125—127° (from light petroleum), $[\alpha]_D^{20}$ +43° (*c* 0·2) (Found: C, 75·95; H, 9·6. $C_{21}H_{32}O_3$ requires C, 75·8; H, 9·7%), $\nu_{max.}$ 3580 and 1730 cm^{-1}, τ 9·19 (3H, s, 18-H), 8·99 (3H, s, 19-H), 7·95 (3H, s, 17-Ac), 5·84 (1H, m, 6-H), 5·4 (1H, q, *J* 8 and 10 Hz, 17-H), and 4·33 (1H, m, 4-H). The diacetate, prepared with acetic anhydride in pyridine, crystallized from light petroleum as needles, m.p. 106—107°, identical (t.l.c. and i.r.) with the material prepared from androst-5-en-17β-yl acetate.

5α-*Hydroxyandrostane*-6α,17β-*diyl Diacetate*.—A solution of androst-5-en-17β-yl acetate (1·2 g) and osmium tetraoxide (1·0 g) in pyridine (30 ml) was stirred at room temperature overnight. A solution of sodium disulphite (1·8 g) in water (30 ml) and pyridine (20 ml) was then added. After 30 min the steroid was recovered with dichloromethane. The residue (1·3 g) was acetylated with acetic anhydride (5 ml) in pyridine (25 ml) overnight at room temperature to give 5α-*hydroxyandrostane*-6α,17β-*diyl diacetate* (600 mg) as needles, m.p. 127—129° (from light petroleum), $[\alpha]_D^{20}$ +17° (*c* 0·2) (Found: C, 70·1; H, 9·3. $C_{23}H_{36}O_5$ requires C, 70·4; H, 9·2%), $\nu_{max.}$ 3550, 1740, and 1720 cm^{-1}, τ 9·24 (3H, s, 18-H), 9·01 (3H, s, 19-H), 7·98 (3H, s) and 7·95 (3H, s) (6- and 17-Ac), 5·4 (1H, q, *J* 8 and 10 Hz, 17-H), and 5·1 (1H, m, 6-H).

Androst-4-*ene*-6α,17β-*diyl Diacetate*.—A solution of thionyl chloride (1·2 ml) in pyridine (4 ml) was cooled to −20 °C and added to a solution of the foregoing 5α-alcohol (400 mg) in pyridine (12 ml) at −20 °C. After 30 min the solution was poured into dilute hydrochloric acid (120 ml) and the steroid was recovered in ethyl acetate to yield the *olefin* (200 mg) as needles, m.p. 106—107° (from aqueous methanol), $[\alpha]_D^{20}$ +77° (*c* 0·2) (Found: C, 73·8; H, 9·2. $C_{23}H_{34}O_4$ requires C, 73·8; H, 9·15%), $\nu_{max.}$ 1740 cm^{-1}, τ 9·20 (3H, s, 18-H), 8·95 (3H, s, 19-H), 7·97 (3H, s) and 7·91 (3H, s) (6- and 17-Ac), 5·42 (1H, q, *J* 8 and 10 Hz, 17-H), and 4·4 (1H, m, 4-H), identical with the product of acetylation of 6α-hydroxyandrost-4-en-17β-yl acetate.

Epoxidation of 6α-*Hydroxyandrost*-4-*en*-17β-*yl Acetate*.—t-Butyl hydroperoxide (300 mg) was added to a solution of 6α-hydroxyandrost-4-en-17β-yl acetate (1·0 g) and vanadyl acetylacetonate (50 mg) in refluxing benzene (50 ml). After 30 min the solution was cooled and diluted with ethyl acetate. It was washed thoroughly with aqueous iron(II) sulphate, dilute hydrochloric acid, aqueous sodium hydrogen carbonate, and water, dried, and evaporated to a gum. The n.m.r. signals at τ 6·85 and 6·58 showed this to be a 1 : 3 mixture of the 4α,5α- and 4β,5β-epoxides. 4β,5β-*Epoxy*-6α-*hydroxyandrostan*-17β-*yl acetate* (300 mg) crystallized from light petroleum as needles, m.p. 143—145°, $[\alpha]_D$ −20° (*c* 0·1) (Found: C, 72·4; H, 9·3. $C_{21}H_{32}O_4$ requires C, 72·4; H, 9·3%), $\nu_{max.}$ 3530 and 1720 cm^{-1}, τ 9·20 (3H, s, 18-H), 9·01 (3H, s, 19-H), 7·98 (3H, s, 17-Ac), 6·58 (1H, q, *J* 2 and 4 Hz, 4-H), 5·96 (1H, q, *J* 5 and 12 Hz, 6-H), and 5·38 (1H, q, *J* 8 and 10 Hz, 17-H).

Epoxidation with *m*-chloroperbenzoic acid in chloroform gave a 1 : 1 mixture (n.m.r. signals at τ 6·85 and 6·58), of the 4α,5α- and 4β,5β-epoxides. 4β,5β-*Epoxyandrostane*-6α,17β-*diol diacetate*, prepared with acetic anhydride in pyridine, crystallized from light petroleum as needles, m.p. 152—153°, $[\alpha]_D^{20}$ +6° (*c* 0·2) (Found: C, 70·75; H, 8·6. $C_{23}H_{34}O_5$ requires C, 70·7; H, 8·8%), $\nu_{max.}$ 1735 cm^{-1}, τ 9·20 (3H, s, 18-H), 8·95 (3H, s, 19-H), 8·01 (3H, s) and 7·96 (3H, s) (6- and 17-Ac), 6·68 (1H, m, 4-H), 5·38 (1H, t, *J* 9 Hz, 17-H), and 4·75 (1H, q, *J* 6 and 12 Hz, 6-H).

Epoxidation of Androst-4-*ene*-6α,17β-*diol Diacetate*.—The diacetate (250 mg) in chloroform (40 ml) was treated with *m*-chloroperbenzoic acid (300 mg) at room temperature overnight. The steroid was recovered. The n.m.r. spectrum of the residue showed that it was a 3 : 1 mixture of 4α,5α- and 4β,5β-epoxides. 4α,5α-*Epoxyandrostane*-6α,17β-*diol diacetate* (100 mg) crystallized from light petroleum as needles, m.p. 120—121°, $[\alpha]_D^{20}$ +45° (*c* 0·2) (Found: C, 70·7; H, 8·6. $C_{23}H_{34}O_5$ requires C, 70·7; H, 8·8%), $\nu_{max.}$ 1740 cm^{-1}, τ 9·20 (3H, s, 18-H), 8·90 (3H, s, 19-H), 7·99 (6H, s, 6- and 17-Ac), 6·85 (1H, t, *J* 2 Hz, 4-H), 5·4 (1H, q, *J* 8 and 10 Hz, 17-H), and 4·78 (1H, q, *J* 4 and 12 Hz, 6-H).

Reduction of the Epoxides.—(*a*) 4β,5β-Epoxyandrostane-6α,17β-diol diacetate (150 mg) in dry ether (20 ml) was treated with lithium aluminium hydride (80 mg) at room temperature for 30 min. The steroid was recovered and acetylated with acetic anhydride (2 ml) in pyridine (4 ml) overnight to give 5β-*hydroxyandrostane*-6α,17β-*diol diacetate* (80 mg) as needles, m.p. 133—134° (from aqueous methanol), $[\alpha]_D^{20}$ +23° (*c* 0·1) (Found: C, 70·3; H, 9·5. $C_{23}H_{36}O_5$ requires C, 70·4; H, 9·2%), $\nu_{max.}$ 3520 and 1730 cm^{-1}, τ 9·24 (3H, s, 18-H), 9·08 (3H, s, 19-H), 7·98 (3H, s) and 7·96 (3H, s) (6- and 17-Ac), 5·4 (1H, q, *J* 8 and 10 Hz, 17-H), and 4·96 (1H, q, *J* 5 and 12 Hz, 6-H).

(*b*) 4α,5α-Epoxyandrostane-6α,17β-diol diacetate, when treated similarly, gave the 5α-alcohol, identical (m.p., t.l.c., and i.r.) with the material prepared from androst-5-en-17β-yl acetate (see above).

Reaction of 4β,5β-*Epoxy*-6α-*hydroxyandrostan*-17β-*yl Acetate with Hydrobromic Acid*.—The steroid (250 mg) in 48% hydrobromic acid (0·5 ml) and glacial acetic acid (2·0 ml) was heated under reflux for 15 min. The product (150 mg) was recovered in ethyl acetate and chromatographed on alumina. Elution with 5% ether–light petroleum gave 17-bromo-4-methyloestra-1,3,5(10)-triene (10 mg) (mass and n.m.r. spectra) as an oil. Elution with 15% ether–light petroleum gave 4-methyloestra-1,3,5(10)-trien-17β-yl acetate (30 mg), m.p. 184° (lit.,[12] 188°), identified by its i.r. spectrum. Elution with 50% ether–light petroleum gave 6-oxoandrost-4-en-17β-yl acetate (10 mg) as needles, m.p. 160—161° (from methanol) (lit.,[9] 159—164°), identified by its i.r. spectrum.

2α,3α-*Epoxy*-6-*oxoandrostan*-17β-*yl Acetate*.—6-Oxoandrost-2-en-17β-yl acetate (1·0 g) [13] in benzene (50 ml) was treated with *m*-chloroperbenzoic acid (1·3 g) at room temperature overnight. The steroid was recovered and chromatographed on alumina. Elution with 10% ethyl acetate–light petroleum gave the 2α,3α-*epoxide* (800 mg) as needles, m.p. 193—194° (from light petroleum), $[\alpha]_D^{20}$ −4° (*c* 0·2) (Found: C, 72·8; H, 9·0. $C_{21}H_{30}O_4$ requires C,

72·8; H, 8·7%), $\nu_{max.}$ 1735 and 1705 cm^{-1}, τ 9·27 (3H, s, 18-H), 9·21 (3H, s, 19-H), 7·96 (3H, s, 17-Ac), 6·8br (2H, m, 2- and 3-H), and 5·4 (1H, q, *J* 8 and 10 Hz, 17-H).

Reduction of 2α,3α-*Epoxy*-6-*oxoandrostan*-17β-*yl Acetate*.—The steroid (680 mg) in methanol (60 ml) was treated with sodium borohydride (250 mg) at 0 °C for 2 h. A few drops of acetic acid were added and the product was recovered in ethyl acetate to give 2α,3α-*epoxy*-6β-*hydroxyandrostan*-17β-*yl acetate* (500 mg) as needles, m.p. 129—130° (from light petroleum), $[\alpha]_D^{20}$ +2° (*c* 0·2) (Found: C, 72·5; H, 9·4. $C_{21}H_{32}O_4$ requires C, 72·4; H, 9·3%), $\nu_{max.}$ 3480, 3420, and 1740 cm^{-1}, τ 9·20 (3H, s, 18-H), 9·03 (3H, s, 19-H), 7·97 (3H, s, 17-Ac), 6·8br (2H, m, 2- and 3-H), 6·24 (1H, m, 6-H), and 5·37 (1H, q, *J* 8 and 10 Hz, 17-H).

Reduction of 6-*Oxoandrost*-2-*en*-17β-*yl Acetate*.—The steroid (300 mg) in methanol (40 ml) was treated with sodium borohydride (80 mg) at 0 °C for 2 h. A few drops of acetic acid were added and the product was recovered to afford 6β-*hydroxyandrost*-2-*en*-17β-*yl acetate* (200 mg) as needles, m.p. 130—131° (from light petroleum), $[\alpha]_D^{20}$ +29° (*c* 0·2) (Found: C, 75·95; H, 9·6. $C_{21}H_{32}O_3$ requires C, 75·9; H, 9·7%), $\nu_{max.}$ 3560, 3530, and 1725 cm^{-1}, τ 9·17 (3H, s, 18-H), 9·02 (3H, s, 19-H), 7·97 (3H, s, 17-Ac), 6·12 (1H, m, 6-H), 5·35 (1H, q, *J* 8 and 10 Hz, 17-H), and 4·35 (2H, m, 2- and 3-H).

Epoxidation of 6β-*Hydroxyandrost*-2-*en*-17β-*yl Acetate*.—The steroid (150 mg) in benzene (30 ml) was treated with *m*-chloroperbenzoic acid (200 mg) at room temperature overnight to give the 2α,3α-epoxide (100 mg) as needles, m.p. 129—130° (from light petroleum), identical (t.l.c. and i.r.) with the material described above.

Reaction of 2α,3α-*Epoxy*-6β-*hydroxyandrostan*-17β-*yl Acetate with Hydrobromic Acid*.—The steroid (800 mg) in glacial acetic acid (10 ml) containing 48% hydrobromic acid (2·5 ml), was heated under reflux for 15 min. The steroids were recovered in ether and chromatographed on alumina. Elution with 4% ether–light petroleum gave 17-bromo-4-methyloestratriene (100 mg), *m/e* 332 and 334. Elution with 20% ether–light petroleum gave 4-methyloestra-1,3,5(10)-trien-17β-yl acetate (215 mg) as needles, m.p. 185—186° (from light petroleum) (lit.,[12] 188°), identified by its i.r. spectrum. No other crystalline product was obtained.

We thank Schering A.G. (Berlin) for financial support.

[4/2409 *Received*, 18*th November*, 1974]