944. *Hydroaromatic Steroid Hormones. Part VI.* Some* D-*Homo-analogues Lacking Ring* B.

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The preparation of 9-methyl-6-(4-oxo*cyclo*hex-1-enyl)-*trans*-decal-1β-ol (VIII) † and **1α-ethynyl**-9-methyl-6-(4-oxo*cyclo*hex-1-enyl)-*trans*-decal-1β-ol (IX) (steroid αβ convention) in admixture with the respective αβ-unsaturated ketones is described. Addition of ethylmagnesium bromide to a 9-methyl-*trans*-decal-1-one gives a mixture containing a 1α-ethyl-9-methyl-*trans*-decal-1β-ol.

OUR previous work on relationships between structure and biological activity in non-œstrogenic steroid hormones has been concerned chiefly with (*a*) omission of the 19-methyl group and (*b*) omission of the 19- and 18-methyl groups with ring D six-membered. Clinically useful compounds have emerged.[1] Recent work has shown that anabolic activity and progestational activity are structurally less specific than was previously supposed; for example 19-nor-14β : 17α-progesterone[2] and 8α-progesterone[3] retain biological activity. We consequently deemed worthwhile the synthesis of some compounds containing incomplete ring-systems.

An obvious substance for study is the (±)-form of the ketone (I) which is an analogue of testosterone. After completion of our work the synthesis of this substance in admixture with the βγ-unsaturated isomer was reported.[4] The route employed, although similar to that described below, was rather more complex. The starting material for the present synthesis is the hydroxy-ketone (II), now more conveniently available[5] than previously.[6] In order to form rings C and D of a testosterone analogue the 1-hydroxyl group (analogous to 17α in D-homosteroids) must be *cis* to the angular methyl group. This would be expected from the method of production, but we have obtained confirmatory evidence. The ketol (II) was reduced with lithium in liquid ammonia to the saturated compound (III) which is a *trans*-decalol because it differs from the *cis*-isomer prepared earlier and converted into a known 9-methyl-*cis*-decal-1-ol.[7] The ketol (III) was oxidised with chromium trioxide in acetone to the dione (IV) which with lithium and ethanol in liquid ammonia yielded a diol (V) identical with that formed by direct reduction of the ketol (II) with the same reagent. Consequently the 1-hydroxyl group in (II) almost certainly has the

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* Part V, Birch and Smith, *J.*, 1956, 4909.

† All the substances prepared were racemic compounds, of which the formulæ represent only one form.

[1] Cf. Part V and references cited therein.

[2] Barber and Ehrenstein, *Annalen*, 1957, **603**, 89.

[3] Djerassi, Manson, and Segaloff, *J. Org. Chem.*, 1956, **21**, 490.

[4] Jaeger, *Tetrahedron*, 1958, **2**, 326.

[5] Cocker and Halsall, *J.*, 1957, 3441.

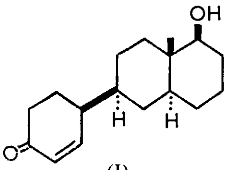
[6] Birch, Quartey, and Smith, *J.*, 1952, 327.

[7] Jaeger, Robinson, and Smith, unpublished work.

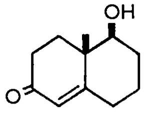
stable equatorial configuration, *cis* to the 9-methyl group. This is supported by the presence of a single band at 1240 cm.$^{-1}$ in the infrared spectrum of the acetate of the ketol (III). An axial acetoxy-compound by analogy with axial 2-, 3-, and 4-acetoxy-5α-steroids would be expected to show a complex band in the same region.[8]

The heterogeneous reaction of the ketol (III) with *p*-methoxyphenylmagnesium bromide in ether or tetrahydrofuran gave, after treatment of the product with mineral acid, the substance (VI) in yields varying from 40 to 60%. Improved and consistent yields of this compound were obtained from the homogeneous reaction of *p*-methoxyphenyl-lithium and the ketol in tetrahydrofuran. The double bond should be more stable in the 6 : 7- than in the 5 : 6-position, and the substance (VI) is formulated on this basis. Attempts to reduce the double bond with lithium in liquid ammonia or by catalytic methods were unsuccessful. Reduction of this compound (VI) with lithium and ethanol in liquid ammonia gave a decalol in which a dihydrobenzene substituent is probably attached equatorially as in formula (VII). Hydrolysis with oxalic acid then gave the ketone (VIII), converted by hot mineral acid into an equilibrium mixture of the isomers (I) and (VIII) containing, as with other 4-alkyl*cyclo*hex-2-enones,[10] an appreciable quantity (estimated as 30%) of the βγ-unsaturated ketone.

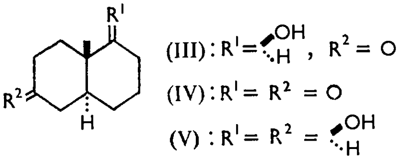
During this work it was reported that 17α-ethynyl-17-hydroxyœstr-5(10)-en-3-one shows promise in controlling menstrual disorders and as a physiological contraceptive.[11] The analogue (IX) was therefore required. The ketal (X) [from the ketone (VIII)] with chromium trioxide in pyridine gave the decalone (XI) which with lithium acetylide in refluxing tetrahydrofuran afforded the alcohol (XII). Subsequent acid hydrolysis furnished an equilibrium mixture of the βγ- and the αβ-unsatuated ketone (IX and isomer). Attempts to avoid the equilibration step by using the decalol (VII) as starting material were unsuccessful because of the failure to oxidise selectively the alcoholic to a ketonic group: Oppenauer oxidation in toluene was unsuccessful and the chromium trioxide–pyridine complex gave the ketone (XIII). The hydroxyl group in the alcohol (IX) is assigned the β-configuration by analogy with the known mode of addition of alkali-metal acetylides to 17-oxo-steroids.[12] However, it seems relevant that whereas methylmagnesium bromide with 17-oxo-steroids gives 17α-methyl-17β-alcohols, with D-homo-17a-oxo-steroids the 17aβ-methyl-17aα-ols are formed.[13] Accordingly we have sought evidence on

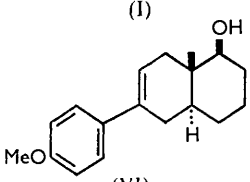


(I)

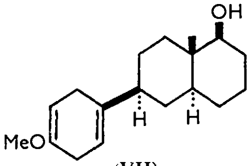


(II)

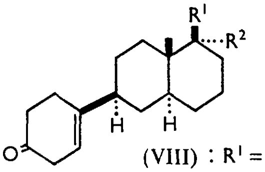




(VI)



(VII)



(VIII) : R^1 = OH, R^2 = H

(IX) : R^1 = OH, R^2 = C:CH

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[8] Cf. Braude and Waight in Klyne's " Progress in Stereochemistry," Butterworths, London, 1954, Vol. I, p. 126.

[9] Turner, Meador, and Winkler, *J. Amer. Chem. Soc.*, 1957, **79**, 4112 and references cited therein.

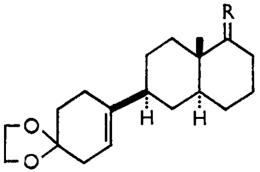
[10] Cf., *e.g.*, Birch and Mukherji, *J.*, 1949, 2531.

[11] *Chem. Eng. News.*, 1957, **35**, No. 23, p. 30.

[12] Cf. Reichstein and Meystre, *Helv. Chim. Acta*, 1939, **22**, 728; Reichstein and Gatzi, *ibid.*, 1938, **21**, 1185.

[13] Heusser, Wahba, and Winternitz, *ibid.*, 1954, **37**, 1052.

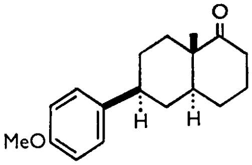
this point. Hydrogenation of the ketal (XII) gave the alcohol (XIV) in which the hydroxyl group is probably equatorial because of its resistance to dehydration by phosphorus oxychloride in pyridine, iodine in refluxing benzene, or refluxing acetic anhydride. 17β-Hydroxy-17α-alkyl-steroids and their D-ring homologues behave similarly.[13] The alcohol (XIV) was dehydrated in refluxing benzene in the presence of toluene-*p*-sulphonic acid. It was hoped that oxidation of the resulting olefin with perbenzoic acid would yield, by preferential attack at the side of the molecule remote from the 9-methyl group, an epoxide which on diaxial ring opening with lithium aluminium hydride would give an axial decalol. In fact, this sequence of reactions afforded an alcohol isomeric with (XIV) and of probable structure (XV). On the assumption that no skeletal rearrangement occurred during the acid-catalysed dehydration this result provides further evidence for the equatorial nature of the hydroxyl group in the alcohol (XIV).



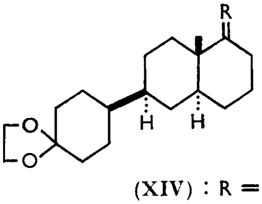
(X) : R = $^{\text{OH}}_{\text{H}}$

(XI) : R = O

(XII) : R = $^{\text{OH}}_{\text{C≡CH}}$



(XIII)



(XIV) : R = $^{\text{OH}}_{\text{Et}}$

(XV) : R = $^{\text{Et}}_{\text{OH}}$

(XVI) : R = O

Attempts to reach the same conclusion by a reaction sequence avoiding an acid-catalysed dehydration were unsuccessful. Thus, whilst the alcohol (XII) was reduced with lithium and ethanol in liquid ammonia to an olefin which should be either an ethylidenedecalin or a 1-ethyl-Δ^1-octalin, epoxidation followed by reduction with lithium aluminium hydride failed to give a crystalline product. For comparison we examined the reaction of ethylmagnesium bromide with the ketone (XVI) which was prepared from the alcohol (X) by catalytic hydrogenation followed by oxidation with the chromium trioxide–pyridine complex. The product was evidently a mixture of stereoisomers from which only a small quantity of the alcohol (XIV) could be obtained. We have confirmed the lack of androgenic activity[4] in the equilibrium mixture of the ketones (I) and (VIII). Results of other biological tests will be reported later.

EXPERIMENTAL

M. p.s were determined on a Kofler block. Ultraviolet-light absorption data refer to ethanol solutions. Infrared spectra were determined for carbon disulphide solutions unless otherwise stated. Light petroleum means the fraction of b. p. 60—80°. Florisil is a grade of Florida fuller's earth supplied by the Floridin Co., Warren, Pennsylvania.

5β-*Hydroxy*-10β-*methyl*-trans-*decal*-2-*one* (III).—5β-Hydroxy-10β-methyl-$\Delta^{1(9)}$-octal-2-one (5 g.) in tetrahydrofuran (100 c.c.) was added to lithium (0·58 g.) in liquid ammonia (250 c.c.; distilled from sodium). After 1·5 hours' stirring, sodium nitrite was added to discharge the blue colour and the ammonia was allowed to evaporate. Water (200 c.c.) was added and the product collected with ether. The gum in benzene (10 c.c.) was adsorbed on Florisil (50 g.). Elution with benzene (1 l.) gave β-*hydroxy*-10β-*methyl*-trans-*decal*-2-*one* (2·86 g.), m. p. 68—70° (from ether at −15°) (Found: C, 72·3; H, 9·85. $C_{11}H_{18}O_2$ requires C, 72·5; H, 9·95%), $\nu_{max.}$ 3635 and 1711 cm.$^{-1}$.

The *acetate* (from ether), after distillation at 75°/0·05 mm., had m. p. 49—50° (Found: C, 69·8; H, 8·9. $C_{13}H_{20}O_3$ requires C, 69·6; H, 9·0%), $\nu_{max.}$ 1740, 1710, 1240, 1030, and 1010 cm.$^{-1}$.

9β-*Methyl*-trans-*decalin*-1 : 6-*dione* (IV).—Chromium trioxide (2·67 g.) in 8·45N-sulphuric acid (1 c.c.) was added to the ketol (III) in acetone (25 c.c.). After 2 min. the green solution was poured into water (100 c.c.), and the crystalline product was collected with ether. Distillation twice at 90°/0·03 mm. gave the 9β-*methyl*-trans-*decalin*-1 : 6-*dione*, m. p. 53—55° (Found: C, 73·2; H, 9·1. $C_{11}H_{16}O_2$ requires C, 73·3; H, 8·95%), $\nu_{max.}$ 1705 and 1715 cm.$^{-1}$.

Reduction of the dione (20 mg.) with lithium (50 mg.) and ethanol (0·8 g.) in liquid ammonia (50 c.c.) and tetrahydrofuran (5 c.c.) gave the diol (V), m. p. 125—132° undepressed by the sample prepared as below and possessing an identical infrared spectrum.

9β-*Methyl*-trans-*decalin*-1β : 6β-*diol* (V).—Lithium (0·1 g.) was added to 5β-hydroxy-10β-methyl-$\Delta^{1(9)}$-octal-2-one (0·5 g.) in tetrahydrofuran (12 c.c.)–liquid ammonia (50 c.c.), and the whole was stirred for 15 min. Ethanol (1 c.c.) was rapidly run in to discharge the blue colour. After evaporation of the ammonia, water (50 c.c.) was added and the product isolated with ether. The gum was adsorbed from chloroform–ethanol (100 : 1; 5 c.c.) on Florisil. Elution with the same solvent (100 c.c.) gave 9β-*methyl*-trans-*decalin*-1β : 6β-*diol* (200 mg.), m. p. 132—133° (from ether) (Found: C, 71·4; H, 11·0. $C_{11}H_{20}O_2$ requires C, 71·7; H, 10·9%).

6-p-*Methoxyphenyl*-9β-*methyl*-trans-Δ^6-*octalin*-1β-*ol* (VI).—(*a*) 5β-Hydroxy-10β-methyl-*trans*-decal-2-one (1·5 g.) in ether (45 c.c.) was added with stirring to *p*-methoxyphenyl-lithium [from butyl-lithium (2·03 g.) and *p*-bromoanisole (6·6 g.)] in ether (100 c.c.) at −30° under nitrogen. Tetrahydrofuran was added to dissolve the precipitate, and the solution was stirred at −15° for 30 min., warmed to 0° during 15 min., and poured on ice. The product was isolated with ether and heated at 100°/0·1 mm. to remove anisole and *p*-bromoanisole. The gum was refluxed for 30 min. in benzene (100 c.c.) containing a crystal of iodine (Dean–Stark water-separator). The cooled solution was washed with aqueous sodium hydrogen sulphite, dried ($MgSO_4$), and evaporated. The residue was adsorbed from benzene (20 c.c.) on Florisil (50 g.). Elution with benzene and crystallisation of the solid from benzene gave 6-p-*methoxyphenyl*-9β-*methyl*-trans-Δ^6-*octalin*-1β-*ol* (1·68 g., 74%), m. p. 113—115° (Found: C, 79·1; H, 9·1. $C_{18}H_{24}O_2$ requires C, 79·4; H, 8·9%), $\nu_{max.}$ 3615, 802, and 837 cm.$^{-1}$, $\lambda_{max.}$ 255, 256, and 258 mμ (ε 10,100, 10,100, and 9900 respectively).

(*b*) The ketol (III) (1 g.) in tetrahydrofuran (100 c.c.) was added to a boiling solution of *p*-methoxyphenylmagnesium bromide (from the metal, 0·526 g.) in tetrahydrofuran (120 c.c.) under nitrogen. The suspension was refluxed for 1·5 hr., and on cooling was poured into ice-cold 4N-sulphuric acid (500 c.c.). After 30 min. the product was extracted with ether. It was adsorbed from the saturated solution in benzene on Florisil. Elution with light petroleum–benzene (9 : 1) gave 4 : 4′-dimethoxydiphenyl, m. p. 173—175°, and elution with light petroleum benzene (1 : 1) and benzene gave (from benzene) the octalin (VI) (0·61 g.), m. p. 113—115°, identical with that prepared as in (*a*).

6β-(2 : 5-*Dihydro*-4-*methoxyphenyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (VII).—The octalinol (VI) (2 g.) in tetrahydrofuran (100 c.c.) was added with stirring to lithium (1·5 g.) in liquid ammonia (400 c.c.). After 5 min. ethanol was added during 10 min. After discharge of the blue colour, water (200 c.c.) was added and the product collected with ether. Crystallisation of the gum from ether–light petroleum gave 6β-(2 : 5-*dihydro*-4-*methoxyphenyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (1·18 g.), m. p. 100—110°, raised to 111—113° by recrystallisation from di*iso*propyl ether (Found: C, 77·95; H, 10·4. $C_{18}H_{28}O_2$ requires C, 78·2; H, 10·2%), $\nu_{max.}$ 3620, 1660, and 1690 cm.$^{-1}$.

6β-(4-*Oxo*cyclo*hex*-1-*enyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (VIII).—Oxalic acid dihydrate (0·375 g.) in water (5 c.c.) was added to the decalol (VII) in methanol (25 c.c.) and the cloudy solution kept at 25° for 40 min. Saturated aqueous sodium hydrogen carbonate was added and the product was extracted with ether. The gum was distilled at 200° (bath-temp.)/0·02 mm. to give 6β-(4-*oxo*cyclo*hex*-1-*enyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (236 mg.) (Found: C, 77·5; H, 10·2. $C_{17}H_{26}O_2$ requires C, 77·8; H, 10·0%), $\nu_{max.}$ 3620 and 1725 cm.$^{-1}$.

The 2 : 4-*dinitrophenylhydrazone* (from ethanol) had m. p. 100—110° (Found: C, 62·3; H, 7·0. $C_{23}H_{30}O_5N_4$ requires C, 62·5; H, 6·9%), $\lambda_{max.}$ 228, 229, and 360 mμ (ε 17,700, 17,700, and 23,500 respectively).

The foregoing decalol was refluxed in ethanol (5 c.c.) containing 2N-hydrochloric acid (2 c.c.) for 5 min., the solution added to saturated aqueous sodium hydrogen carbonate, and the product collected in ether. The infrared spectrum of the gum, which is probably a mixture of the ketones (I) and (VIII), showed bands at 3620, 1718 (strong), and 1680 cm.$^{-1}$ (medium).

6β-(4 : 4-*Ethylenedioxy*cyclo*hex*-1-*enyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (X).—The ketol (VIII)

(1·83 g.) was refluxed for 12 hr. in benzene containing ethylene glycol (6 c.c.) and toluene-*p*-sulphonic acid (*ca.* 40 mg.) (Dean–Stark head). The cooled solution was poured into saturated aqueous potassium hydrogen carbonate (100 c.c.), and the product was extracted with ether. The gum was adsorbed from benzene (20 c.c.) on Florisil (150 g.). Benzene (1·1 l.) eluted a gum which was distilled, to give 6β-(4 : 4-*ethylenedioxy*cyclo*hex*-1-*enyl*)-9β-*methyl*-trans-*decal*-1β-*ol* as a gum, b. p. 200° (bath temp.)/0·2 mm. (Found: C, 74·75; H, 10·1. $C_{19}H_{30}O_3$ requires C, 74·75; H, 9·85%), $\nu_{max.}$ 3620, 1115, 1060, 1025, and 860 cm.$^{-1}$.

6β-(4 : 4-*Ethylenedioxy*cyclo*hex*-1-*enyl*)-9β-*methyl*-trans-*decal*-1-*one* (XI).—The decalol (X) (2 g.) in pyridine (20 c.c.) was added to chromium trioxide (2 g.) in pyridine (20 c.c.). After 20 hr. at room temperature the mixture was poured into 5% aqueous sodium hydroxide (1 l.) and extracted with ether (4 × 200 c.c.). The product was adsorbed from light petroleum (50 c.c.) on Florisil (200 g.), and the column was washed with light petroleum (1 l.). Elution with light petroleum–benzene (1 : 1; 1 l.), benzene (4 l.), and benzene–ether (100 : 1; 2 l.) gave fractions which were combined and distilled, to give 6β-(4 : 4-*ethylenedioxy*cyclo*hex*-1-*enyl*)-9β-*methyl*-trans-*decal*-1-*one* as a gum, b. p. 200° (bath-temp.)/0·5 mm. (Found: C, 75·2; H, 9·5. $C_{19}H_{28}O_3$ requires C, 74·95; H, 9·25%), $\nu_{max.}$ (liquid film) 1700, 1115, 1060, 1020, and 865 cm.$^{-1}$.

6β-(4-*Methoxyphenyl*)-9β-*methyl*-trans-*decal*-1-*one* (XIII).—The decalol (VII) (120 mg.) in pyridine (1 c.c.) was added to chromium trioxide (0·4 g.) in pyridine (4 c.c.). Next day the mixture was poured into water (100 c.c.), and the product collected with ether. Recrystallisation from di*iso*propyl ether–light petroleum gave 6β-(4-*methoxyphenyl*)-9β-*methyl*-trans-*decal*-1-*one* (100 mg.), m. p. 73—75° (Found: C, 79·05; H, 9·0. $C_{18}H_{24}O_2$ requires C, 79·35; H, 8·9%), $\nu_{max.}$ 1710 and 830 cm.$^{-1}$, $\lambda_{max.}$ 223, 224, 276, and 283 mμ (ε 11,800, 11,800, 2500, and 1900 respectively).

1α-*Ethynyl*-6β-(4 : 4-*ethylenedioxy*cyclo*hex*-1-*enyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (XII).—The decalone (XI) (1·5 g.) was stirred with a suspension of lithium acetylide (from the metal, 6 g.) in tetrahydrofuran (150 c.c.) in a stream of acetylene. After 1 hr. the acetylene was replaced by nitrogen. The mixture was stirred overnight at room temperature, refluxed for 1·5 hr., and cooled in ice, then ice-water (500 c.c.) was cautiously added. The product was collected with ether. The gummy crystals were adsorbed from benzene (20 c.c.) on alumina (150 g.). Elution with benzene–ether (5 : 1, 4 l.; 1 : 1, 1 l.) gave (from light petroleum–ether) 1α-*ethynyl*-6β-(4 : 4-*ethylenedioxy*cyclo*hex*-1-*enyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (1·04 g.), m. p. 140—142°, raised to 143—144° by sublimation at 120°/0·03 mm. (Found: C, 76·1; H, 9·2. $C_{21}H_{30}O_3$ requires C, 76·3; H, 9·15%), $\nu_{max.}$ (Nujol mull) 3400, 3220, 2110, 1115, 1060, 1020, and 865 cm.$^{-1}$.

1α-*Ethyl*-6β-(4 : 4-*ethylenedioxy*cyclo*hexyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (XIV).—(*a*) The alcohol (XII) (50 mg.) in ethyl acetate (20 c.c.) was shaken with hydrogen over platinum (from platinum dioxide monohydrate, 50 mg.) for 8 hr. The infrared absorption spectrum of the crystalline product showed no band in the 860—870 cm.$^{-1}$ region. It was adsorbed from light petroleum on Florisil (5 g.). Elution with light petroleum–benzene (1 : 1) gave (from light petroleum) 1α-*ethyl*-6β-(4 : 4-*ethylenedioxy*cyclo*hexyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (40 mg.), m. p. 114—116° (Found: C, 75·1; H, 10·7. $C_{21}H_{36}O_3$ requires C, 74·95; H, 10·8%). (*b*) The alcohol (XI) (100 mg.) in ethyl acetate (20 c.c.) was shaken in hydrogen with finely divided platinum (from the dioxide hydrate, 50 mg.) for 12 hr. The product, unlike all compounds in this series which contain a 4 : 4-ethylenedioxy*cyclo*hex-1-enyl residue, showed no band in the infrared 860—870 cm.$^{-1}$ region. It was dissolved in pyridine (1 c.c.) and added to chromium trioxide in pyridine (3 c.c.). After two days the product was obtained in the usual manner and adsorbed from light petroleum on Florisil (5 g.). Elution with benzene gave a ketonic gum (infrared band at 1705 cm.$^{-1}$; no band in 3300—3400 cm.$^{-1}$ region) which was treated with ethylmagnesium bromide in tetrahydrofuran solution in the standard fashion. The resulting gum (infrared band at 3450 cm.$^{-1}$) was chromatographed on Florisil in ether, to give the alcohol (XIV) (2 mg.), m. p. 113—115° undepressed with the material obtained as under (*a*), as the only recognisable product.

1β-*Ethyl*-6β-(4 : 4-*ethylenedioxy*cyclo*hexyl*)-9β-*methyl*-trans-*decal*-1α-*ol* (XV).—The decalol (XII) (45 mg.) was refluxed for 12 hr. in benzene (50 c.c.) containing ethylene glycol (1 c.c.) and toluene-*p*-sulphonic acid (20 mg.) (Dean–Stark water-separator). The cooled solution was poured into sodium hydrogen carbonate solution, and the product was collected with ether and adsorbed from light petroleum on activated alumina. Elution with light petroleum–benzene (9 : 1) gave a gum (40 mg.), the infrared absorption spectrum of which showed a band at 830 cm.$^{-1}$ but no band in the 3600 cm.$^{-1}$ region. The gum was treated with perbenzoic acid

(44 mg.) in chloroform (5 c.c.) at 0°. After 1 hr. ether was added and the solution was washed successively with aqueous ferrous sulphate, water, aqueous sodium hydrogen carbonate, and water and dried ($MgSO_4$). The product was adsorbed from light petroleum on alumina (5 g.). Elution with light petroleum–benzene (9 : 1) gave a gum (36 mg.) which was dissolved in tetrahydrofuran (5 c.c.) and treated with lithium aluminium hydride (200 mg.). After 1 hr. at room temperature the mixture was heated at 50° for 45 min. The product, isolated in the known manner, was adsorbed from light petroleum on Florisil. Elution with light petroleum–benzene (4 : 1) yielded a crystalline fraction which after crystallisation from light petroleum gave 1-β-*ethyl*-6β-(4 : 4-*ethylenedioxy*cyclo*hexyl*)-9β-*methyl*-trans-*decal*-1β-*ol* (34 mg.), m. p. 140—143°, depressed to 80—135° on admixture with the foregoing 1β-alcohol (XII) (Found: C, 74·8; H, 10·5. $C_{21}H_{36}O_3$ requires C, 74·95; H, 10·8%). The infrared absorption spectra of the alcohols (XIV) and (XV) were different.

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