4,5-CYCLO-A-HOMO-B-NORSTEROID ANDROGEN ANALOGUES*

J. JOSKA and J. FAJKOŠ

Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, 166 10 Prague 6

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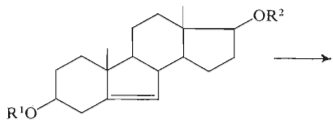
Synthesis of a series of androgen analogues with the 4α,5-cyclo-A-homo-B-nor-5α-androstane--skeleton is described.

In our previous papers[1,2] we described syntheses of 2- and 3-oxygenated, as well as 1,2-unsaturated derivatives of the 4α,5-cyclo-A-homo-B-nor-5α-cholestane. In the course of our studies of the relationship between structure and physiological activity of steroids with modified skeleton, we are dealing now with analogous derivatives in the androstane series.

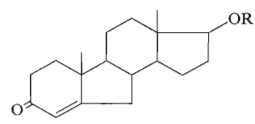
The target compounds are represented by formulae *XV*, *XVI*, *XIX*, *XXI*, *XXV*, and *XXVII* and have been prepared by standard procedures. The starting monoacetate *I* was transformed into the diester *II* which on partial hydrolysis at $C_{(3)}$ afforded the monobenzoate *III*. Oppenauer oxidation gave the benzoate *VI*, which was hydrolysed to the alcohol *IV* and acetylated to the acetate *V*. Reduction with sodium borohydride led to the unsaturated monoacetate *VIII* which was submitted to Simmons–Smith methylenation to yield the key compound *XII*. Analogous reaction sequence has also been carried out with the 17-benzoyloxy derivatives starting from the monoester *VI via* the alcohol *IX* and cyclo compound *XIII*; however, the acetates proved superior as far as the yields are concerned. The benzoates *II*, *III* and *VI* have also been prepared by Ourisson and coworkers by a different route[3].

Hydrolysis of the monoester *XII* gave the diol *XI* which on oxidation with Jones' reagent afforded the dione *XV*, the analogue of androstenedione. Oxidation of the monoacetate *XII* with the same reagent afforded the acetate *XVII* and, after hydrolysis the alcohol *XVI* which represents the analogue of testosterone. The ketone *XVII* was transformed to the bromo ketone *XVIII* with Jacques' reagent in tetrahydrofuran solution. Following dehydrobromination in collidine yielded after deacetylation at $C_{(17)}$ the analogue of dehydrotestosterone (*XIX*). Its oxidation at $C_{(17)}$ gave the dione *XXI*.

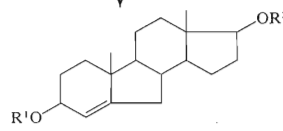
* Part CXCIII in the series On Steroids; Part CXCII: This Journal *42*, 1035 (1977).



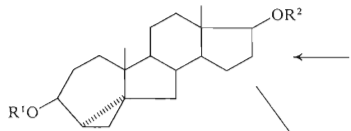
I, R^1 = Ac, R^2 = H
II, R^1 = Ac, R^2 = C_6H_5CO
III, R^1 = H, R^2 = C_6H_5CO



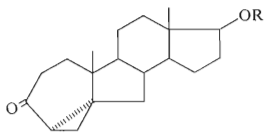
IV, R = H
V, R = Ac
VI, R = C_6H_5CO



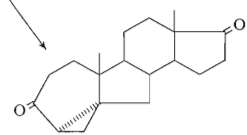
VII, R^1 = R^2 = H
VIII, R^1 = H, R^2 = Ac
IX, R^1 = H, R^2 = C_6H_5CO
X, R^1 = R^2 = Ac



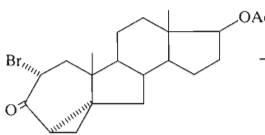
XI, R^1 = R^2 = H
XII, R^1 = H, R^2 = Ac
XIII, R^1 = H, R^2 = C_6H_5CO
XIV, R^1 = R^2 = Ac



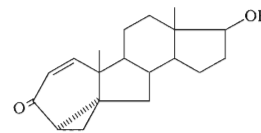
XVI, R = H
XVII, R = Ac



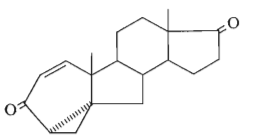
XV



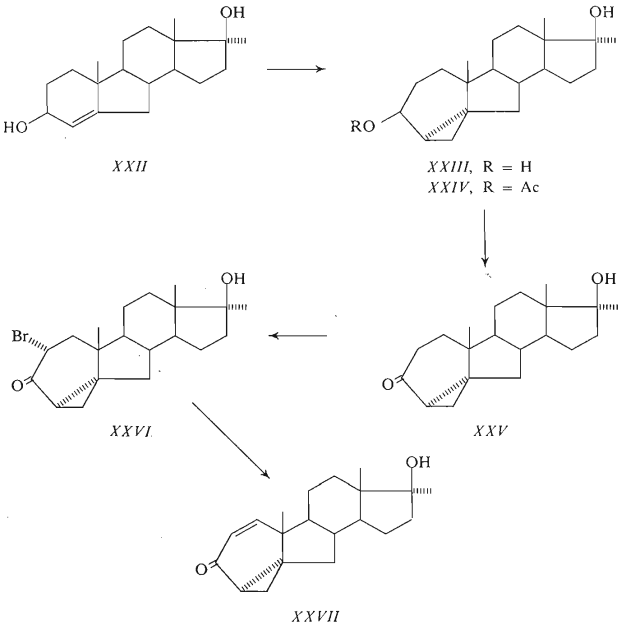
XVIII



XIX, R = H
XX, R = Ac



XXI



XXII *XXIII*, R = H; *XXIV*, R = Ac *XXV* *XXVI* *XXVII*

Our next concern were the analogues of methyltestosterone *XXV* and *XXVII*. The known[4] diol *XXII* was submitted to Simmons–Smith methylenation to yield the cyclo compound *XXIII*. Chromic acid oxidation afforded the analogue of methyltestosterone *XXV*. This ketone was transformed to the desired dehydro analogue *XXVII* *via* the bromo ketone *XXVI* by dehydrohalogenation with collidine.

The physiological properties of the compounds described in this paper are under investigation, and the results will be published elsewhere[5].

EXPERIMENTAL

Melting points were determined on a Kofler block. Analytical samples were dried at 80°C/0·2 Torr. Optical measurements were carried out in chloroform with an error of $\pm 1°$. The infrared spectra were recorded on the Zeiss UR 10 spectrometer in tetrachloromethane unless otherwise stated. The identity of samples prepared by different routes was checked by mixture melting point de-

termination, by thin-layer chromatography (TLC), and by infrared spectra. Ligroin of b.p. 40–60°C was used as solvent. Working up of an ethereal solution means extraction with 5% hydrochloric acid, water, 5% sodium hydrogen carbonate solution, water, drying with magnesium sulphate, and evaporation of the solvent.

3β-Acetoxy-17β-benzoyloxy-B-norandrost-5-ene (*II*)

A solution of the alcohol *I* (10 g) in pyridine (60 ml) was treated with benzoyl chloride (20 ml) allowed to stand at room temperature for 20 h. The mixture was decomposed with ice, diluted with water, and the product taken into ether. The ethereal solution was worked up, ether distilled off, and the residue was crystallised from methanol to yield 12·5 g of the diester *II*, m.p. 148 to 149°C, $[\alpha]_D^{20}$ −36° (*c* 1·27) in agreement with the literature[3].

17β-Benzoyloxy-B-norandrost-5-en-3β-ol (*III*)

The diester *II* (12 g) in chloroform (150 ml) and methanol (500 ml) was treated with conc. hydrochloric acid (14 ml) and allowed to stand at 35°C for 20 h. The mixture was neutralised with 10% sodium hydrogen carbonate solution, methanol removed under reduced pressure, and the residue diluted with water. The product was extracted into ether, the ethereal solution was washed with water, dried, and the product crystallised from methanol to give 9 g of the benzoate *III*, m.p. 188–189°C, $[\alpha]_D^{20}$ −47° (*c* 1·21) in agreement with the literature[3].

17β-Hydroxy-B-norandrost-4-en-3-one (*IV*)

The benzoate *VI* (3 g) in methanol was treated with a solution of potassium hydroxide (2·5 g) in methanol (100 ml) and refluxed for 4 h. The excess hydroxide was neutralised with acetic acid, methanol was removed under reduced pressure, and the residue treated with water. The product was taken into ether, the ethereal solution was dried, ether removed, and the residue was crystallised from ethyl acetate. Yield 2·4 g of the alcohol *IV*, m.p. 162–163°C, $[\alpha]_D^{20}$ −17° (*c* 1·34) in accordance with the literature[3,6].

17β-Acetoxy-B-norandrost-4-en-3-one (*V*)

The alcohol *IV* (5 g) was dissolved in pyridine (25 ml), treated with acetic anhydride (20 ml) and set aside for 20 h. The excess anhydride was decomposed with ice, the reaction mixture was treated with water, and the product isolated with ether. Working up of the ethereal solution and crystallisation from methanol afforded 4·8 g of the acetate *V*, m.p. 139–140°C. $[\alpha]_D^{20}$ −12° (*c* 1·18), in agreement with the literature[6].

17β-Benzoyloxy-B-norandrost-4-en-3-one (*VI*)

The alcohol *III* (9 g) was dissolved in a mixture of toluene (240 ml) and cyclohexanone (85 ml) and 50 ml of the mixture were distilled off to remove any moisture. A solution of aluminium isopropoxide (7·8 g) in toluene (40 ml) was then added, and 140 ml of solvents were distilled off in the course of 1 h. The mixture was cooled off with ice, acidified with hydrochloric acid (10%; 150 ml), and the product extracted into ether. The ethereal solution was washed with water, a sodium hydrogen carbonate solution, and the volatile constituents were removed by steam distillation. The product was taken into ether, and the solution was dried and solvent removed. The residue was chromatographed over silica gel (500 g) in benzene. The corresponding fractions

afforded after working up 8 g of a product which on crystallisation from chloroform–methanol yielded 7·5 g of the ketone *VI*, m.p. 216–217°C, $[\alpha]_D^{20}$ +46° (*c* 1·30). Literature[3] records m.p. 209–210°C, $[\alpha]_J$ +29°.

B-Norandrost-4-en-3β,17β-diol (*VII*)

A solution of the ketone *V* (1·3 g) in methanol (30 ml) and ethyl acetate (10 ml) was treated with sodium borohydride (500 mg) and stirred for 8 h. After 20 h fresh hydride (200 mg) was added and stirred for additional 8 h. The mixture was diluted with water, decomposed with 5% acetic acid, and the product was extracted with ether. The ethereal solution was washed with sodium hydrogen carbonate, water, dried, and the ether distilled off. The residue was chromatographed on a silica gel column (150 g) in benzene–ether (5 : 1). Fractions with the polar component were worked up to yield 350 mg of a product which on crystallisation from ethyl acetate–ligroin afforded 280 mg of the diol *VII*, m.p. 151–153°C, $[\alpha]_D^{20}$ −48° (*c* 1·68). For $C_{18}H_{28}O_2$ (276·4) calculated: 78·21% C, 10·21% H; found: 78·05% C, 10·05% H.

17β-Acetoxy-B-norandrost-4-en-3β-ol (*VIII*)

Fractions from the chromatography of the foregoing experiment containing the lipophilic component were combined, solvent was removed, and the residue was crystallised from ligroin to yield 590 mg of the monoester *VIII*, m.p. 124–125°C, $[\alpha]_D^{20}$ −50° (*c* 1·57). For $C_{20}H_{30}O_3$ (318·4) calculated: 75·43% C, 9·50% H; found: 75·86% C, 9·42% H.

17β-Benzoyloxy-B-norandrost-4-en-3β-ol (*IX*)

The benzoate *VI* (2·5 g) in methanol (120 ml) and ethyl acetate (30 ml) was reduced with sodium borohydride (800 mg + 400 mg) as described for the alcohol *VII*. Similar working up gave a product which was chromatographed on a silica gel column (200 g) in ether–benzene (1 : 19). Working up of the corresponding fractions and crystallisation from methanol and ligroin afforded 500 mg of alcohol *IX*, m.p. 165–166°C, $[\alpha]_D^{20}$ +22° (*c* 1·44). For $C_{25}H_{32}O_3$ (380·5) calculated: 78·91% C, 8·48% H; found: 79·12% C, 8·34% H.

3β,17β-Diacetoxy-B-norandrost-4-ene (*X*)

The alcohol *VIII* (100 mg) in pyridine (0·4 ml) was treated with acetic anhydride (0·3 ml) and after 20 h at room temperature the mixture was decomposed with ice. The product was isolated with ether, and the ethereal solution was worked up. The crude product was chromatographed over silica gel (15 g) in benzene–ligroin (1 : 2). Working up of the corresponding fractions and crystallisation from ether–methanol yielded 60 mg of the diacetate *X*, m.p. 114 to 115°C, $[\alpha]_D^{20}$ +101° (*c* 1·21). For $C_{22}H_{32}O_4$ (360·5) calculated: 73·30% C, 8·95% H; found: 73·60% C, 8·85% H.

4α,5-Cyclo-A-homo-B-nor-5α-androstan-3β,17β-diol (*XI*)

The acetate *XII* (1·5 g) in methanol (80 ml) was treated with a solution of potassium hydroxide (1·5 g) in methanol (50 ml) and allowed to stand at 50°C for 1 h. The excess alkali was removed with acetic acid, solvent was removed under reduced pressure, and the residue was diluted with water. The product was extracted with ethyl acetate, the extract was washed with sodium hydrogen carbonate, dried, and solvent removed. The residue was crystallised from ethyl acetate to afford

1·1 g of the diol *XI*, m.p. 181–182°C $[\alpha]_D^{20}$ −30° (*c* 1·14 in ethanol). For $C_{19}H_{30}O_2$ (290·4) calculated: 78·57% C, 10·41% H; found: 75·79% C, 10·47% H.

17β-Acetoxy-4α,5-cyclo-A-homo-B-nor-5α-androstan-3β-ol (*XII*)

Zinc dust (1·4 g; Baker 60–200 mesh) was added into a solution of cupric acetate monohydrate (25 mg) in acetic acid (8 ml). The mixture was shaken at 50–60°C and after decolorisation the solvent was poured off. The zinc was washed first with acetic acid (8 ml) and then decanted with eight portions of absolute ether (5 ml) each). The metal was covered with absolute ether (15 ml), iodine (20 mg), and diiodomethane (2 ml) were added, and the mixture was refluxed under stirring for 3 h. After cooling off to room temperature a solution of the monoacetate *VIII* (430 mg) in absolute ether (5 ml) was added and the mixture was stirred at room temperature for 3 h. The suspension was treated with ether, poured into 5% sodium hydrogen carbonate, and the ethereal layer was washed with 5% sodium thiosulphate, water, dried, and the solvent was distilled off. The residue was chromatographed over silica gel (30 g) in benzene–ether (19 : 1). The corresponding fractions were worked up and the residue (360 mg) was crystallised from ligroin to yield 300 mg of the cyclosteroid *XII*, m.p. 134–136°C, $[\alpha]_D^{20}$ −23° (*c* 1·28). IR: 3065 (cyclopropane), 3615 (hydroxyl), 1740, 1248, 1032 cm^{-1} (acetate). For $C_{21}H_{32}O_3$ (332·5) calculated: 75·86% C, 9·70% H; found: 75·69% C, 9·77% H.

17β-Benzoyloxy-4α,5-cyclo-A-homo-B-nor-5α-androstan-3β-ol (*XIII*)

0·5% Zn–Cu couple was prepared from zinc dust (1·4 g) and cupric acetate monohydrate (20 mg) as described in the previous experiment after similar treatment with iodine and diiodomethane a solution of the benzoate *IX* (430 mg) in absolute ether (20 ml) was added and stirred for 3 h at room temperature. Working up afforded crude product which was purified on a silica gel column (30 g) in the same solvent mixture. Crystallisation from methanol gave 180 mg of the benzoate *XIII*, m.p. 159–161°C, $[\alpha]_D^{20}$ +23° (*c* 1·36). For $C_{26}H_{34}O_3$ (394·5) calculated: 79·15% C, 8·69% H; found: 79·04% C, 8·75% H.

3β,17β-Diacetoxy-4α,5-cyclo-A-homo-B-nor-5α-androstane (*XIV*)

The monoacetate *XII* (50 mg) in pyridine (1 ml) was acetylated with acetic anhydride (0·7 ml) for 20 h at room temperature. The excess anhydride was decomposed with ice, the product taken into ether, and the ethereal solution was worked up. Crystallisation from methanol yielded 35 mg of the diacetate *XIV*, m.p. 160–161°C, $[\alpha]_D^{20}$ −57° (*c* 1·50). For $C_{23}H_{34}O_4$ (374·5) calculated: 73·76% C, 9·15% H; found: 74·05% C, 9·23% H.

4α,5-Cyclo-A-homo-B-nor-5α-androstan-3,17-dione (*XV*)

A solution of the diol *XI* (900 mg) in acetone (100 ml) was treated with Jones' reagent and stirred at room temperature for 10 minutes. The excess oxidising agent was removed with methanol (5 ml), and the product was precipitated with water, collected, and dissolved in ether. The ethereal solution was washed with sodium hydrogen carbonate, water, dried and ether distilled off. The residue was crystallised from methanol to yield 480 mg of the dione *XV*, m.p. 182–183°C, $[\alpha]_D^{20}$ +35° (*c* 1·37). For $C_{19}H_{26}O_2$ (286·4) calculated: 79·68% C, 9·15% H; found: 79·93% C, 8·98% H.

17β-Hydroxy-4α,5-cyclo-A-homo-B-nor-5α-androstan-3-one (*XVI*)

A solution of potassium hydroxide (100 mg) in methanol (5 ml) was added to a solution of the acetate *XVII* (100 mg) in methanol and allowed to stand at 50°C for 1 h. The alkali was removed with acetic acid and the solvent was distilled off under reduced pressure. The residue was diluted with water, and the product isolated with ether. The ethereal extract was washed with sodium hydrogen carbonate, water, dried, and evaporated. The residue was crystallised from methanol–water to yield 55 mg of the alcohol *XVI*, m.p. 211—212°C, $[\alpha]_D^{20}$ −41·5° (*c* 1·49). IR: 3080, 1691 (cyclopropane), 3615, 1063, 1047 cm^{-1} (hydroxyl). For $C_{19}H_{28}O_2$ (288·4) calculated: 79·12% C, 9·78% H; found: 79·40% C, 9·83% H.

17β-Acetoxy-4α,5-cyclo-A-homo-B-nor-5α-androstan-3-one (*XVII*)

The alcohol *XII* (2·1 g) in acetone (150 ml) was treated with excess Jones' reagent and the reaction mixture was stirred at room temperature for 10 minutes. The oxidising agent was destroyed with methanol, the product was precipitated with water, and collected by succion. The crystals were dissolved in ether, the ethereal solution was washed with sodium hydrogen carbonate, water, dried, and solvent removed. The residue was chromatographed on a silica gel column (200 g) in benzen–ether (5 : 1). Working up of the corresponding fractions and crystallisation from methanol gave 1·34 g of the ketone *XVII*, m.p. 151—152°C, $[\alpha]_D^{20}$ −31° (*c* 1·42). For $C_{21}H_{30}O_3$ (330·5) calculated: 76·32% C, 9·15% H; found: 75·99% C, 9·07% H.

17β-Acetoxy-2α-bromo-4α,5-cyclo-A-homo-B-nor-5α-androstan-3-one (*XVIII*)

The ketone *XVII* (3·3 g) in tetrahydrofuran (80 ml) was treated with Jacques' reagent (5 g) and set aside for 30 minutes. The mixture was diluted with water, the product taken into ether, and the ethereal solution was washed with sodium hydrogen carbonate, sodium thiosulphate, water, dried, and ether distilled off. The residue was chromatographed on a silica gel column (150 g) in benzen–ether (9 : 1). Fractions with the polar component (some starting ketone was present) were worked up, solvent removed, and the residue was crystallised from methanol–ether to yield 1·3 g of the bromo ketone *XVIII*, m.p. 170—172°C, $[\alpha]_D^{20}$ −22° (*c* 1·37). IR: 3080 (cyclopropane), 1741, 1249, 1045 (acetate), 1712 cm^{-1} (carbonyl). For $C_{21}H_{29}BrO_3$ (409·4) calculated: 61·61% C, 7·14% H, 19·52% Br; found: 61·81% C, 7·19% H, 20·01% Br.

17β-Hydroxy-4α,5-cyclo-A-homo-B-nor-5α-androst-1-en-3-one (*XIX*)

The acetate *XX* (50 mg) in methanol (3 ml) was treated with a solution of potassium hydroxide (50 mg) in the same solvent (1 ml) and allowed to stand at 37°C for 2 h. The mixture was acidified with acetic acid, diluted with water, and the product extracted with ether. The ethereal solution was washed with sodium hydrogen carbonate, water, dried, and ether removed. The residue was crystallised from methanol–ligroin to yield 32 mg of the alcohol *XIX*, m.p. 180—181°C, $[\alpha]_D^{20}$ +238° (*c* 1·25). For $C_{19}H_{26}O_2$ (286·4) calculated: 79·68% C, 9·15% H; found: 79·82% C, 9·31% H.

17β-Acetoxy-4α,5-cyclo-A-homo-B-nor-5α-androst-1-en-3-one (*XX*)

A solution of the bromo ketone *XVIII* (1·05 g) in *sym*-collidine was refluxed for 8 h. Collidine was distilled off under reduced pressure, the residue was dissolved in ether, the solution was worked up, and the residue was chromatographed over silica gel (60 g) in benzene–ether (9 : 1).

Crystallisation from methanol–ligroin gave 587 mg of the unsaturated ketone *XX*, m.p. 112 to 113°C, $[\alpha]_D^{20}$ +234° (*c* 1·47). IR: 1665, 1621 (conjugated ketone), 1740, 1246, 1047, 1036 cm^{-1} (acetate). For $C_{21}H_{28}O_3$ (328·4) calculated: 76·79% C, 8·59% H; found: 77·03% C, 8·57% H.

4α,5-Cyclo-A-homo-B-nor-5α-androst-1-en-3,17-dione (*XXI*)

The alcohol *XIX* (80 mg) in acetone (6 ml) was oxidised with excess Jones' reagent under stirring for 10 minutes at room temperature. The excess reagent was removed with methanol, the reaction mixture was diluted with water, the product was taken into ether, and worked up. The residue was crystallised from acetone–water to yield 45 mg of the dione *XXI*, m.p. 172—173°C, $[\alpha]_D^{20}$ +308° (*c* 1·18). IR: 1744 (17-oxo), 1666 (3-oxo), 3075, 3035 cm^{-1} (cyclopropane and double bond). For $C_{19}H_{24}O_2$ (284·4) calculated: 80·24% C, 8·51% H; found: 80·11% C, 8·40% H.

17α-Methyl-4α,5-cyclo-A-homo-B-nor-5α-androstan-3β, 17β-diol (*XXIII*)

0·5% Zn—Cu couple was prepared from zinc dust (11·2 g) and cupric acetate monohydrate (200 g) as decribed for the preparation of the monoacetate *XII*. The couple was then refluxed for 3 h with iodine (170 mg) and diiodomethane (16 ml) in absolute ether (50 ml). The complex was treated with a solution of the olefin[3] *XXII* (3·9 g) in absolute tetrahydrofuran (50 ml) and stirred at room temperature for 3 h. The mixture was worked up as described above, and the product was chromatographed over silica gel (100 g) in benzene–ether (2 : 1). Working up of the corresponding fractions and crystallisation from acetone gave 3·2 g of the diol *XXIII*, m.p. 218 to 220°C, $[\alpha]_D^{20}$ −46° (*c* 1·23 in ethanol). For $C_{20}H_{32}O_2$ (304·5) calculated: 78·89% C, 10·60% H; found: 78·63% C, 10·41% H.

3β-Acetoxy-17α-methyl-4α,5-cyclo-A-homo-B-nor-5α-androstan-17β-ol (*XXIV*)

The diol *XXIII* (100 mg) in pyridine (1 ml) was acetylated with acetic anhydride (0·5 ml) at room temperature for 20 h. The mixture was decomposed with ice, the product isolated with ether, and the ethereal solution was worked up. The residue was crystallised from methanol–water to yield 65 mg of the monoacetate *XXIV*, m.p. 134—135°C, $[\alpha]_D^{20}$ −80° (*c* 1·30). For $C_{22}H_{34}O_3$ (346·5) calculated: 76·26% C, 9·89% H; found: 75·99% C, 9·90% H.

17β-Hydroxy-17α-methyl-4α,5-cyclo-A-homo-B-nor-5α-androstan-3-one (*XXV*)

The alcohol *XXIII* (3·1 g) in acetone (500 ml) was oxidised with excess Jones' reagent at room temperature under stirring for 10 minutes. Methanol was then added to destroy the agent, acetone was partly removed under reduced pressure and the residue was treated with water. The product was collected by succion and dissolved in ether–ethyl acetate. The solution was worked up, and the residue was crystallised from ethyl acetate to yield 2·7 g of the ketone *XXV*, m.p. 185—186°C, $[\alpha]_D^{20}$ −84° (*c* 1·39). IR: 3615 (hydroxyl), 3085 (cyclopropane), 1682 cm^{-1} (3-oxo). For $C_{20}H_{30}O_2$ (302·4) calculated: 79·42% C, 10·00% H; found: 79·16% C, 9·90% H.

2α-Bromo-17β-hydroxy-17α-methyl-4α,5-cyclo-A-homo-B-nor-5α-androstan-3-one (*XXVI*)

A solution of the ketone *XXV* (1 g) in tetrahydrofuran (30 ml) was treated with Jacques' reagent and set aside for 30 minutes. The mixture was poured into 10% sodium hydrogen carbonate, the product taken into ether, and the solution was worked up. The residue was chromatographed on a silica gel column (60 g) in benzene–ether (4 : 1). Working up of the corresponding fractions

and crystallisation from methanol afforded 550 mg of the bromo ketone *XXVI*, m.p. 181—182°C, $[\alpha]_D^{20}$ −37° (*c* 1·31). IR: 3620 (hydroxyl), 3080 (cyclopropane), 1702 cm^{-1} (3-oxo). For $C_{20}H_{29}BrO_2$ (381·4) calculated: 62·99% C, 7·67% H, 20·97% Br; found: 63·40% C, 7·89% H, 20·50% Br.

17β-Hydroxy-17α-methyl-4α,5-cyclo-A-homo-B-nor-5α-androst-1-en-3-one (*XXVII*)

The bromo ketone *XXVI* (500 mg) in *sym*-collidine was refluxed for 8 h. Solvent was removed under reduced pressure, the residue was dissolved in ether, and the ethereal solution was worked up. The product was chromatographed on a silica gel column (30 g) in benzen–ether (4 : 1), and the residue after working up of the corresponding fractions was crystallised from acetone–water to yield 320 mg of the unsaturated ketone *XXVII*, m.p. 179—180°C, $[\alpha]_D^{20}$ +204° (*c* 1·16). IR: 3615 (hydroxyl), 1620, 1653 cm^{-1} (3-oxo). For $C_{20}H_{28}O_2$ (300·4) calculated: 79·95% C, 9·39% H; found: 80·10% C, 9·34% H.

The analyses were carried out in the Analytical Laboratory of this Institute by Mr V. Štěrba and Mrs E. Sýkorová under the direction of Dr J. Horáček. The infra red spectra were recorded by Mr P. Formánek under the direction of Dr J. Smolíková.

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