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FAJKOS, J.

CZECHOSLOVAKIA/Organic Chemistry. Natural Substances and Their Synthetic Analogues. E-3

Abs Jour: Ref Zhur - Khimiya, No. 8, 1957, 26972.

Author : Fajkoš, Jan; Šorm, František.
Inst :
Title : Steroids. XXIII. Preparation and Proof of Configuration of Both Stereoisomer 3β-Oxy-16-acetyl Derivatives of Androstane.

Orig Pub: Chem. listy, 1956, 50, No. 5, 791 - 799.

Abstract: The configuration of some 16-substitutions of androstane was established. The solution of Δ^{16}-16-cyanandrostenole-3β acetate in anisole is added at 20° to the ether solution of CH_3MgBr, heated 5 hours up to 60°, and Δ^{16}-16-acetylandrostenole-3β (I) is received by usual treatment, yield 76%, melting point 202

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CZECHOSLOVAKIA/Organic Chemistry. Natural Substances and Their Synthetic Analogues. E-3

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to 203° (from CH_3OH and sublimation), $/\alpha/^{20}D$ -46 ±1° (c 3.18). acetate (II), yield 73%, melting point 144 to 145° (from alc.), $/\alpha/^{20}D$ -57 ±1° (c 2.12). 16β-acetylandrostanole-3β (III) is received by hydrogenation of I on 5%-ual Pd/$CaCO_3$ in dioxane (of 1 mol of H_2), yield 70%, melting point 143 to 145° (from CH_3OH), $/\alpha/^{20}D$ -22 ±1° (c 2.63); acetate (IV) melting point 95 to 96° (from CH_3OH), $/\alpha/^{20}D$ -38 ±1° (c 3.8); IV is prepared also by hydrogenation of II. The known androstandiole-3β,16β diacetate was prepared by oxidation of IV with perbenzoic acid in $CHCl_3$ (7 days in darkness), yield 52%, melting point 105 to 107° (from alc.), $/\alpha/^{20}D$ -11.2 ±1° (c 2.32). The 16β-configuration of III

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and IV was proved by this, because the oxidation splitting proceeded without changing the configuration (see Gallagher T.F., Kritchevsky T.H., Amer. Chem. Soc., 1950, 72, 882). III and IV are thermodynamically unstable and epimerize easily and completely. 16 α-acétylandrostanole-3β (V) is produced by boiling IV (or III) with methanole NaOH (2 hours), yield 53%, melting point 60 to 70°, $/\alpha/^{20}D$ +9.8 ±1° (c 2.43); acetate (VI), melting point 175 to 176° (from CH_3OH), $/\alpha/^{20}D$+7.5 ±1° (c 2.15). The oxidation of VI with perbenzoic acid resulted in the known diacetate of androstandiole-3β-16α, yield 76%, melting point 174 to 175° (from alc.), $/\alpha/^{20}D$ -29.1 ±1° (c 2.2). Oxidation of I with CrO_3

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in pyridine (20°, 24 hours) produces Δ^{16}-16-acetylandrostenone-3 (VII), yield 83%, melting point 169 to 170° (from benzene), $/\alpha/^{20}D$ -21 ±1° (c 2.19). Oxidation of III with CrO_3 in CH_3COOH (20°, 20 hours) resulted in 16 -acetylandrostanone-3, yield 57%, melting point 175 to 177° (from benzene), $/\alpha/^{20}D$ -12 ±1° (c 2.06), which can be prepared also by hydrogenating VII with $Pd/CaCO_3$ in dioxane, yield 83%. Similarly, oxidation of V produces 16α-acetylandrostanone-3, yield 41%, melting point 172 to 174° (from benzene), $/\alpha/^{20}D$ +15 ±1° (c 2.43). Acetate of VIII (IX) was received by acetylizing 3β-oxyandrostanecarboxylic-(16α) acid (VIII) with $(CH_3CO)_2O$ in pyridine (16 hours, 20°), yield 85%,

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melting point 233 to 235° (from dioxane), $/\alpha/^{20}D$ -2.7 ± 1° (c 1.49); IX with $SOCl_2$ (0°, 12 hours) produced chloroanhydride of IX (X), melting point of unpurified X - 120 to 130°. Benzene solution of X is added to ether solution of $Cd(CH_3)_2$ (of CH_3MgBr and $CdCl_2$), the reaction mixture is shaken 30 min. at 20°, dissociated with ice and, after the usual treatment, the unpurified product is obtained, from which the ketone fraction is separated using Girard's agent T. VI was received after acetylization, yield 55%. Acetate of XI, melting point 192 to 194° (from benzene), $/\alpha/^{20}D$ -29.5 ± 1° (c 2.84), was obtained from 3β-oxyandrostanecarboxylic-16β acid (XI), this acetate was

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converted into chloroanhydride (XII) with $SOCl_2$, melting point of unpurified XII - 165 to 175°. Not the expected IV, but V was received from the reaction of XII with $Cd(CH_3)_2$. In view of the fact that IX and XI, as well as the chloroanhydrides X and XII are different one from the other, epimerization should take place at the interaction of XII with $Cd(CH_3)_2$. Ether solution of X is added to ether solution of an excessive amount of CH_2N_2 at -10° and left staying 12 hours at 20°, acetate of 16α-diazoacetylandrostanole-3β (XIII) is obtained, yield 70%, melting point 160 to 161° (from benzene), $/\alpha/^{20}D$ +31 ± 1° (c 2.36). Acetate of 16α-bromoacetylandrostanole-3β (XIV) is received from

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CZECHOSLOVAKIA/Organic Chemistry. Natural Substances and Their Synthetic Analogues. E-3

Abs Jour: Ref Zhur - Khimiya, No. 8, 1957, 26972.

the interaction between suspension of XIII in ether and an excessive amount of ether solution of HBr (20°, 15 min.), yield 83%, melting point 184 to 185° (from CH_3OH), $/\alpha/^{20}D$ -13 ±1° (c 3.12). Hydrogenation of XIV on 5%-ual $Pd/CaCO_3$ (24 hours, fresh catalyst was added every 8 hours) produced VI, yield 49%. Acetate of 16β-diazoacetylandrostanole-3β (XV) was obtained similarly from XII, yield 62%, melting point 158° (from benzene), $/\alpha/^{20}D$ -55 ±1° (c 1.73). From the interaction between XV with an excessive amount of ether solution of HBr, XIV was also received, yield 74%. As XIII and XV differ one from the other, the epimerization of XV occurs by the action of HBr. Epimerization could be

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avoided under following conditions. Solution of 125 mg of XV in 2 ml of dioxane and 10 ml of ether is cooled to -15°, mixed 5 min. with solution of 4.3 ml of HBr in ether (6.4 mg of HBr in 1 ml), cooled to -15°, and immediately poured out into 5%-ual solution of Na_2CO_3 at 0°, 86 mg of acetate of 16β-bromoacetylandrostanole-3β (XVI) are received, melting point 138 to 140° (from CH_3OH), $/\alpha/^{20}D$ -20 ±1° (c 1.98). Hydrogenation of XVI resulted in IV, yield 43%. In order to confirm the influence of HBr on epimerization, IV is dissolved in CH_3COOH, mixed with 40%-ual solution of HBr in CH_3COOH and left staying (12 hours, 20°). 40% of VI is separated in usual way.

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FAJKOS, J.; SOHM, F.

"Steroids. XXVIII. Synthesis of analogs of progesterone and desoxycorticosterone with the side-chain in position 16." In English.

p. 1873. (Sbornik Chekhoslovatskikh Khimicheskikh Rabot, Vol. 22, No. 6, Dec. 1957, Praha, Czechoslavakia)

Monthly index of East European Accession (EEAI) LC, Vol. 7, No. 8, August 1958

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FAJKOS, J

CZECHOSLOVAKIA/Organic Chemistry. Naturally Occurring Substances andTheir Synthetic Analogs. G-3

Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11432.

Author : Fajkos, J. and Sorm, F.
Inst :
Title : On Steroids. XXVIII. Synthesis of Analogs of Progesterone and Desoxycorticosterone with the Side Chain in the 16-Position.

Orig Pub: Chem Listy, 51, No 3, 579-591 (1957) (in Czech)

Abstract: The following analogs of progesterone and desoxycorticosterone having the side chain in the 16-position have been synthesized: the action of CH_3MgBr on 3 β -acetoxy-16-cyanoandrostadiene-5,16 (II) which is selectively hydrogenated to 3β -hydroxy-16 β -

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-acetylandrostene-5 (III); the latter is readily isomerized to the 16 α -derivative (IV). III and IV after acetylation are hydrogenated to the known products 16 α -acetyl- (V) and 16 β -acetyl-3 β -acetoxyandrostane (VI). III and IV are oxidized to 3-keto-16 α -acetylandrostene-4 (VII); similarly, II gives 3-keto-16-acetylandrostadiene-4,16 (VIII). The same reaction sequence applied to 3 β -acetoxy-16-cyanoandrostadiene-5,15 (IX) gives 3 β -hydroxy-16-acetylandrostadiene-5,15 (X), 3-keto-16-acetylandrostadiene-4,15 (XI), and 3 β -acetoxy-16 β -acetylandrostene-5 (XII). On oxidation 3 β -hydroxy-16α -carboxyandrostene-4 (XIV) gives 3-keto-16 α -carboxyandrostene-5 (XIII); 3-keto-16α -acetoxyacetylandrostene-4 (XVII) is obtained from XIII via the

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also obtained by the hydrogenation (as in the case of II) of 3 β -acetoxy-16-acetylandrostadiene-5,16 (XXIV) or 3 β -acetoxy-16-acetylandrostadiene-5,15 (XXV). When a mixture of 180 mg III in 5 ml CH_3OH and 50 ml NaOH in 1 ml water is refluxed for 2 hrs, a yield of 47% IV is obtained, mp 120-121° (from benzene (bp 100-105°) and CH_3OH), $[\alpha]^{20}_D$ - 40 ± 1° (c = 2.32). IV is also obtained by the saponification of XXIII or of 3 β -acetoxy-16α -acetylandrostene-5 (XXVI) which is obtained in 60% yield by the acetylation (as in the case of II and XXII) of IV, mp 179-180° (from CH_3OH), $[\alpha]^{20}_D$ - 58 ± 1° (c = 2.43); XXVI is also obtained in 75% yield by allowing a mixture of 60 mg XXII and 0.1 gm 40% HBr

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Abs Jour: Referat Zhur-Khimiya, No 4, 1958. 11432.

acid in 4 ml CH_3COOH to stand for 18 hrs. The hydrogenation of XXIII over Pd/$CaCO_3$ in dioxane gives VI, yield 45%, mp 95° (from CH_3OH), $[\alpha]^{20}_D$ -38 ± 1° (c = 2.43); analogously XXVI gives V, yield 55%, mp 175-176° (from CH_3OH), $[\alpha]^{20}_D$ + 7.5 ± 1° (c = 2.03). The oxidation of IV (as in the case of II and VIII) gives 72% VII, mp 169-170° (from benzene), $[\alpha]^{20}_D$ + 58 ± 1° (c = 1.71). Analogously VII is obtained from III. 500 mg IX and CH_3MgBr (0.6 gm Mg) give X (procedure as for the synthesis of II from I), yield 40%, mp 171-172° (from benzene, CH_3 + benzene, and ether), $[\alpha]^{20}_D$ -166 ± 1° (c = 2.32); the latter product on acetylation (similar to that of II to XXII) gives XXV in yields of 75%, mp 187-188° (from CH_3OH), $[\alpha]^{20}_D$ -164 ± 2° (c = 1.9). The oxidation of

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X (as in the case of II to VIII) gives XI, yield

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(c = 1.77; alcohol). When a suspension of 0.3 gm XIII in 1.2 ml oxalyl chloride and 12 ml C_6H_6 is shaken for 15 min and evaporated under vacuum, 360 mg XV are obtained; the latter are dissolved in 6 ml C_6H_6 and poured at -10 to 0° into a solution of CH_2N_2 (from 5 gm nitrosomethylurea); after 1 hr the filtrate is evaporated and the XVI which is obtained (340 mg) is heated (1 hr, 90°) with 1.5 ml 5% CH_3COONa in CH_3COOH, the solution is evaporated, and the oily product is extracted with ehter; chromatography on Al_2O_3 gives 14% XVII (elution with petroleum ether : benzene = 1 : 2), mp 194-195° (from acetone), $[\alpha]_D^{20}$ + 36 ± 1° (c = 2.65). When 1 gm XX is refluxed for 1.5 hrs with 0.3 gm K_2CO_3 in 50 ml CH_3OH and 10 ml water, an 83% yield of 3 -hydroxy-17-

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Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11432.

cyanoandrostadiene-5,16 is obtained, mp 170-171° (from CH_3OH), $[\alpha]^{20}_D$ - 66 ± 1° (c = 3.18); the latter product is oxidized (as in the case of II to VIII) to 3-keto-17-cyanoandrostadiene-4,16 (XXVIII), yield 68%, mp 156-158° (from alcohol) $[\alpha]^{20}_D$ + 168 ± 2° (c = 2.08). When a mixture of 325 mg XXVIII, 2 ml ethylene glycol, 15 mg p-toluenesulfonic acid, and 10 ml C_6H_6 is refluxed for 20 hrs (removal of water), a 76% yield of 3-ethylenedihydroxy-17-cyanoandrostadiene-5,16 is obtained, mp 217-219° (from alcohol), $[\alpha]^{20}_D$ -45 ± 1° (c = 2.45) which is converted to XXI (as in the case of I to II, decomposition with a solution of NH_4Cl); after purification by chromatography on Al_2O_3 the yield

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is 27%, mp 236-239° (from benzene). The UV-spectra of I, V-IX, XI, XIII, XX, XXIII-XXVII, 3β-acetoxy-2-ketopregnanine-5, 3β-acetoxy-20-ketopregnanediene-5,16, 3,20-diketo-pregnanediene-4,16 are discussed. The molecular rotations of the substances obtained are given. For Communication XXVII see RZhKhim, 1957, 44673.

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FAJKOS, JAN

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Steroids. XXXI. Synthesis of 3β-hydroxyandrost-15-en-17-one. Jan Fajkoš (Czech. Acad. Sci., Prague). *Chem. listy* 51, 1885-93(1957); cf. *C.A.* 51, 17903*a*.—Dissolving 2 g. 3β,17-diacetoxyandrost-16-ene (I) in 100 ml. CCl_4, distg. 20 ml., adding 1.2 g. dry powd. *N*-bromosuccinimide (II), and refluxing 20 min. gave 1.4 g. 3β-acetoxy-16α-bromoandrostan-17-one (III), m. 171-2° (EtOH), $[\alpha]^{19}_D$ +36.5 ± 1°, as the sole product. In attempt to induce a free radical process the reaction was carried out under ultraviolet irradiation while boiling, the product passed over Al_2O_3 in C_6H_6 to give from 2 g. I 100 mg. nonreacted I (in the petr. ether fraction), 3β-acetoxyandrostan-17-one (IIIa), m. 104°, $[\alpha]^{19}_D$ +60 ± 1° (3:1 petr. ether), 500 mg. III (C_6H_6), and 85 mg. 3β-acetoxy-16β-bromoandrostan-17-one (IV), m. 147-8°, $[\alpha]^{19}_D$ +89 ± 1° (3:1 C_6H_6-Et_2O). IV originates by epimerization of III, since 200 mg. III yields, when left in C_6H_6-petr. ether with alk. Al_2O_3 48 hrs., 45 mg. III and 100 mg. IV. I (20 g.) was brominated as above, II filtered off, the yellow filtrate treated under stirring at −5° with 8 ml. Br in 100 ml. CCl_4, the soln. washed and evapd. below 50°, the residue stirred with hot MeOH and crystd. from $CHCl_3$ to give 7.2 g. 3β-acetoxy-15β,16α-dibromoandrostan-17-one (V), m. 220-1° ($CHCl_3$-MeOH), $[\alpha]^{19}_D$ −12.5 ± 1°. Mother liquors kept 24 hrs. with 10 g. HBr in 50 ml. MeOH, the soln. evapd., and the residue crystd. from MeOH gave 4.1 g. 3β-hydroxy-16α-bromoandrostan-17-one, m. 164-5°, $[\alpha]^{19}_D$ +52 ± 1°. Adding 10 g. HBr in 170 ml. MeOH to 1 g. V in 30 ml. $CHCl_3$, shaking 4 hrs. at 18°, evapg. the soln. to 40 ml. at 20°, dilg. with ice-H_2O, and extg. with Et_2O gave 620 mg. 3β-hydroxy-15β,16α-dibromoandrostan-17-one (VI), m. 137-40°, $[\alpha]^{19}_D$ −5.5 ± 1°. VI (670 mg.) dissolved in 67 ml. EtOH and 14 ml. AcOH

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and debrominated by stirring 1.5 hrs. with 2.7 g. Zn-powder at 45°, filtering off Zn, extg. the product with Et_2O, evapg. the solvent, and recrystg. the residue gave 310 mg. 3β-hydroxyandrost-15-en-17-one (VII), m. 163–4° (Me_2CO-petr. ether), $[\alpha]_D^{19}$ −65 ± 1°. 3β-Acetoxyandrost-15-en-17-one (VIII) obtained in 400 mg. yield by debrominating 1 g. V with Zn-powder as above, or in 210 mg. yield by acetylating 200 mg. VII with 2 ml. Ac_2O in pyridine gave crystals, m. 137–8°, $[\alpha]_D^{19}$ −56 ± 1°, log $\epsilon_{max.}$ = 3.84 at 233 mμ. VIII (150 mg.) hydrogenated over Pd-$CaCO_3$ in 5 ml. pyridine and 0.3 ml. piperidine gave 125 mg. IIIa. The bromination of enol acetates of 3-oxo steroids was studied. Contrary to Rubin and Ambrecht (*C.A.* 48, 12148σ) the main product of the bromination of 2 g. 3,17β-diacetoxytestosterone (IX), when carried out with 1.2 g. II in 80 ml. CCl_4 under ultraviolet irradiation, was 720 mg. 17β-acetoxyandrost-4-en-3-one (X), m. 139–41°, $[\alpha]_D^{19}$ +87 ± 1°, besides 120 mg. 17-acetoxyandrostan-3-one (Xa), m. 140–2° (MeOH), $[\alpha]_D^{19}$ +24 ± 1°. Similar treatment of 3,17β-diacetoxyandrost-2-ene (XI) (1.8 g.) with 1.1 g. II in CCl_4 gave: 50 mg. 17β-acetoxyandrostan-3-one (XIa), m. 158–9° (MeOH), $[\alpha]_D^{19}$ +31.5 ± 1°; 85 mg. 2α-bromo-17β-acetoxyandrostan-3-one, m. 175–7° (MeOH-petr. ether), $[\alpha]_D^{19}$ +32 ± 1°; 410 mg. 17β-acetoxyandrost-2-en-3-one, m. 116–18° (Me_2CO-petr. ether), $[\alpha]_D^{19}$ +43 ± 1°; and 45 mg. X. Adding 0.04 ml. H_2SO_4 in 2 ml. CH_3:CMeCOAc to 6.5 g. Xa in 40 ml. CH_3:CMeCOAc, distg. 15 ml. of the mixt., adding further 40 ml. CH_3:CMeCOAc and 2 ml. H_2SO_4, distg. 35 ml., cooling the mixt., dilg. with 10 ml. C_5H_5N, pouring the mixt. into 1 l. petr. ether, filtering over alk. Al_2O_3, eluting with petr. ether, evapg. the solvent, and cryst. the residue from C_6H_6-petr. ether gave 4.8 g. IX, m. 102–3°, $[\alpha]_D^{19}$ +35 ± 1°. XI was obtained in 5.2-g. yield from 6 g. IIIa by similar procedure, m. 173–4°, $[\alpha]_D^{19}$ +41 ± 1°. XXXII. Synthesis and reactions of Δ^{16}-derivatives of androstane. *Ibid.* 1894–1905.—Reactions are

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given that confirm the structure and configuration of the synthesized compds. preferably under conditions excluding epimerization. Addn. of 50 mg. Br to 100 mg. VIII in CCl_4 contg. traces of pyridine at −10°, gave 115 mg. V, identical with the product obtained in 65-mg. yield by leaving 80 mg. 3β-acetoxy-15β,16α-dibromoandrostan-17β-ol (XII) 20 hrs. with 30 mg. CrO_3 in AcOH, or in 75-mg. yield by shaking 220 mg. 3β-17β-dihydroxyandrost-15-ene (XIIa) 15 hrs. with 2 g. freshly prepd. MnO_2, acetylating the product with 2 ml. Ac_2O in pyridine, and treating the resulting oil with 100 mg. Br in CCl_4. V is a configurationally stable compd. as it failed to epimerize by the action of HBr or reduction with $NaBH_4$. Both Br atoms in V thus possess trans configuration. V (360 mg.) left with 140 mg. $NaBH_4$ in dioxane-EtOH 24 hrs. at 0° gave 85 mg. XII, m. 216–17° (MeOH), $[\alpha]_D^{20}$ −13.2 ± 1°; 65 mg. XII acetylated with Ac_2O in pyridine gave 48 mg. 3β,17β-diacetoxy-15β,16α-dibromoandrostane (XIII), m. 194° (MeOH), $[\alpha]_D^{20}$ −33 ± 1°. XIII refluxed 72 hrs. with KOH in EtOH gave a yellow oily product which did not possess a CO group, thus confirming the trans configuration of V, VI, and XII. Likewise VI obtained in 105-mg. yield by treating 120 mg. VII with 70 mg. Br was identical with the product obtained by sapong. V. One of the Br atoms in the dibromo ketones is very labile; thus 600 mg. VI left 20 hrs. at 18° in pyridine soln. gave 390 mg. 3β-hydroxy-16-bromoandrost-15-en-17-one (XIV), m. 154–5°, $[\alpha]_D^{20}$ −88 ± 1°. Similarly dehydrohalogenated 300 mg. V yielded 180 mg. 3β-acetoxy-16-bromoandrost-15-en-17-one (XV), m. 144–6°, $[\alpha]_D^{20}$ −83 ± 1°, identical with the compd. obtained in 240-mg. yield by keeping 600 mg. V with 1.2 g. $KHCO_3$ in aq. EtOH, or in 720-mg. yield by leaving 1 g. VI with 4 ml. Ac_2O 20 hrs. in

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Jan Fajkos

6 ml. pyridine and evapg. the extd. product with $NaHCO_3$ soln., or in 45-mg. yield by acetylating 60 mg. XIV with 0.5 ml. Ac_2O in pyridine. XV (150 mg.) hydrogenated over Pd-$CaCO_3$ in EtOH gave 65 mg. IIIa. Reducing 160 mg. XV with 60 mg. $NaBH_4$ in abs. EtOH gave 140 mg. 3β-acetoxy-16β-bromoandrostan-17β-ol (XVI), m. 176–8° (MeOH), $[\alpha]^{22}_{D}$ −18.5 ± 1°, characterized by acetylation to 73% 3β-17β-diacetoxy-16β-bromoandrostane, m. 149–50°, $[\alpha]^{22}_{D}$ +41 ± 1°. Attempts to saponify the AcO group of dibromo ketones with $KHCO_3$ in aq. MeOH gave rise to satd. methoxy derivs. Thus, adding 4.4 g. $KHCO_3$ in 60 ml. H_2O and 220 ml. MeOH to 2.2 g. V in 133 ml. dioxane, leaving at 18° 18 hrs., distg. MeOH *in vacuo*, and extg. the product with Et_2O gave 1.05 g. 3β-acetoxy-15β-methoxy-16β-bromoandrostan-17-one (XVIa), m. 185–6° from MeOH, $[\alpha]^{22}_{D}$ +29 ± 1°, identical with the compd. obtained in 62-mg. yield by similar procedure from 70 mg. XV, or in 48-mg. yield by acetylating 60 mg. 3β-hydroxy-15-methoxy-16-bromoandrostan-17-one (XVII) with Ac_2O in pyridine, or in 66% yield by oxidizing 3β-acetoxy-15-methoxy-16β-bromoandrostan-17β-ol (XVIII) with CrO_3 in AcOH. Similarly in the 3-hydroxy series 1 g. VI treated with $KHCO_3$ in aq. MeOH gave 575 mg. XVII, m. 169–70° (MeOH), $[\alpha]^{22}_{D}$ +54 ± 1°, identical with the product obtained in 185-mg. yield by similar procedure from 200 mg. XIV, or in 58-mg. yield by leaving 80 mg. XVIa in 2.5 ml. $CHCl_3$ with 800 mg. HBr in 14 ml. MeOH 4 hrs. at 20°. Crude XVIII obtained by reducing 300 mg. XVIa with 120 mg. $NaBH_4$ in 24 ml. EtOH as an oil (266 mg.) was acetylated with 2 ml. Ac_2O in pyridine to give 135 mg. 3β,17β-diacetoxy-15-methoxy-16β-bromoandrostane (XIX), m. 160–1°, $[\alpha]^{22}_{D}$ −23 ± 1°. XIX boiled with alc. KOH 70 hrs. gave an oily product showing the absorption max. characteristic of a CO group, thus proving that XVIII and XIX have the C-16 and C-17

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Jan Rajkos

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substituents in cis configuration. Boiling crude XVIII (from 500 mg. XVIa) with 2.5 g. powd. Zn in 20 ml. EtOH 3 hrs. under reflux gave 85 mg. 3β-acetoxy-15-methoxyandrost-16-ene (XX), m. 101-2° (MeOH), $[\alpha]_D^{20}$ −107 ± 1°. Leaving a soln. of 100 mg. XX 20 hrs. with 84 mg. BzO_2H in $CHCl_3$ at −4° gave 165 mg. 3β-acetoxy-15-methoxy-16α,17α-epoxyandrostane, m. 121-2° (MeOH), $[\alpha]_D^{20}$ −56 ± 1°. VIII (200 mg.) treated with $KHCO_3$ in aq. MeOH as above gave 135 mg. 3β-acetoxy-15ξ-methoxyandrostan-17-one, m. 148-9° (ligroine), $[\alpha]_D^{20}$ +19 ± 1°, identical with the product obtained in 105-mg. yield by acetylating 120 mg. 3β-hydroxy-15-methoxyandrostan-17-one (XXI) with Ac_2O in pyridine. VII (100 mg.) treated with $KHCO_3$ in aq. MeOH gave 78 mg. XXI, m. 165-8° (aq. Me_2CO), $[\alpha]_D^{20}$ +31 ± 1°. Reducing 250 mg. VIII with $LiAlH_4$ in abs. Et_2O and acetylating the crude product with Ac_2O in pyridine gave 170 mg. 3β,17β-diacetoxyandrost-15-ene (XXII), m. 142-3°, $[\alpha]_D^{20}$ −45 ± 1°, identical with the compd. obtained in 400-mg. yield by stirring at 50° 800 mg. 3β,17β-diacetoxy-15β,16α-dibromoandrostane with 3.4 g. powd. Zn in 80 ml. EtOH and 15 ml. AcOH, or in 37-mg. yield by acetylating 50 mg. XIIa with Ac_2O in pyridine. Shaking a suspension of 200 mg. XXII in 16 ml. MeOH with 200 mg. NaOH in 30 ml. MeOH 18 hrs. at 27° gave XIIa, m. 196-7°, $[\alpha]_D^{20}$ −27.2 ± 1°. Reducing 500 mg. VIII with 150 mg. $NaBH_4$ in EtOH and acetylating the crude product with Ac_2O in pyridine gave 370 mg. 3β,17β-diacetoxyandrostane, m. 126-8° (MeOH), $[\alpha]_D^{20}$ −6.3 ± 1°, identical with the compd. obtained in 42-mg. yield by shaking 100 mg. XIII with 200 mg. 5% Pd-CaO_2 6 hrs. in H atm., or in 35-mg. yield by hydrogenating 100 mg. XXII over PtO_2 in AcOH. Infrared spectra of VII, XIV, and XXI, and ultraviolet spectra of IIIa, VIII, XV, and XVIa are charted and discussed.

L. J. Urbánek

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FAJKOS, JAN

CZECHOSLOVAKIA/Organic Chemistry. Natural Substances and Their Synthetic Analogs. G

Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64602.

Author : Fajkos Jan
Inst :
Title : On Steroids. XXXII. Synthesis and Reactions of Derived Androstans.

Orig Pub: Chem licty, 1957, 51, No 10, 1894-1905.

Abstract: The configuration of substitutions upon the C(15) and C(16) of the acetate of 15beta, 16alpha-dibromandrostanol-3beta-one-17 (I), the results of which have been described earlier (see prev. ref), flow from the following reactions: (I), m.p. 215° (in CH_2Cl-acetone), $[\alpha]^{20}_D$ D-9° (w 2.28), produced in brominated acetate Δ^{15}-androstenol-3beta-one-17, in $CHCl_3$ in the presence

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Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64602

of a trace of pyridine, at a temperature of -10°. Upon bromination, the bromine atoms assume the trans-position; HBr in CH_3COOH does not cause a change in (I). Upon reducing (I) with the aid of $NaBH_4$, mixed in alcohol and dioxane (24 hours, at 0°), there is produced the 3-acetate of 15beta,16alpha dibromandrostandiole-3beta,17-beta (III), yield 24%, m.p. 216-217° (in CH_3OH), $[\alpha]^{20}$ D-13.2° (w 1.62). This latter is acetylized by $(CH_3CO)_2O$ in pyridine to give the diacetate of 15 beta, 16 alpha dibromandrostandiole-3beta, 17beta (IV), m.p. 195°, $[\alpha]^{20}$ D-34 (w 2.83). Oxidizing (III) with CH_3COOH in the presence of CrO_3 (24 hours, 20°) leads to (I) (80%). From these reactions, it is evident that (I)

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Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64602.

has a stable configuration. Upon boiling (IV) for 72 hours with an alcohol solution of KOH, there are produced oily substances free of bromine. These, according to their UV-spectra, do not contain carboxylic groups. This suggests that the bromine of the C (15) and C (16) are to be found in the trans-position. The reductive dehalogenation of (IV) on 5% Pd/$CaCO_3$ in alcohol with repeated additions of fresh catalyst leads to the known diacetate of androstandiole-beta, 17beta (V), yield 60%, m.p. 127-128° (in CH_3OH), $[\alpha]^{20}$ D-5° (w 2.18). Upon the bromination of Δ^{15}-androstenol-3beta-one-17 (VI), there is produced 15beta,16alpha-dibromandrostanol-3beta-one-17 (VII), m.p. 138-139° (in CH_3OH), $[\alpha]^{20}$ D-6.2° (w 2.82).

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Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64602.

(VII) is acetylized with $(CH_3CO)_2O$ in pyridine (20 hours, 20°) and simultaneous dehydrohalogenation and gives the acetate of Δ^{15}-16beta-bromandrostenol-3beta-one-17 (VIII), yield 79%, m.p. 147-148° (in CH_3OH), $[\alpha]^{20}$ D-79.5°. (VIII) is likewise produced from (I) by letting it stand for 20 hours in pyridine solution at 20° (yield 72%), or by letting it stand for 15 hours with a mixture of dioxane-alcohol-aqueous solution of $KHCO_3$. Upon hydrogenation on 5% $Pd/CaCO_3$ in ether, (VIII) produces the acetate of androstanol-3beta-one-17, yield 78%, m.p. 104°, $[\alpha]^{20}$ D + 71° (w 1.95). The reduction of (VIII) with the aid of $NaBH_4$ (18 hours, at 0° in alcohol) leads to the known 3-acetate of 16beta-

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bromandrostandiole-3beta,17beta, m.p. 176-178° (in CH_3OH), $[\alpha]^{20}_D$ -18.5° (w 2.35) diacetate, m.p. 149-150° (in CH_3OH), $[\alpha]^{20}_D$ + 41° (w 2.32). By letting (I) stand with a dioxane-CH_3OH-aqueous solution of $KHCO_3$ for 18 hours at 18°, there occurs, in addition to dehydrohalogenation, the junction of CH_3OH to the resulting acetate 15-methoxy-16betabromandrostanol-3beta-one-17 (IX), yield 55%, m.p. 187-188° (in CH_3OH), $[\alpha]^{20}_D$ + 28.5° (w 1.75). The production of (IX) from (I) by way of (VIII) as an intermediary product is supported by the fact that (VIII), after remaining for 18 hours in a dioxane-CH_3OH-aqueous solution of $KHCO_3$, gives (IX) (82%). (VII), after standing in pyridine for 20 hours at 18°, produces Δ^{15}-16 beta-bromandrostenol-3beta-one-17 (X), m.p. 154-155° (in CH_3OH), $[\alpha]^{20}_D$ -88° (w 2.02), from which acetylization with $(CH_3CO)_2O$ in pyri-

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Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64602.

dine yields (VIII), yield 67%. The union of CH_3OH with (VII) produces 15-methoxy-16beta-bromandrostanol-3beta-one-17 (XI), yield 63%, m.p. 169-170° (upon 100% change from crystalline form), $[\alpha]^{20}D + 54°$ (w 1.75). (XI) was derived in similar fashion with a yield of 80%, by letting (IX) stand for 4 hours in a mixture of $CHCl_3$ and methanol solution of Hbr at 20°. Upon acetylization of (XI) with $(CH_3CO)_2O$ in pyridine, (IX) is obtained (73%). Upon reducing (IX) with the aid of $NaBH_4$ (15 hours, at 0° in alcohol), and acetylization of the resulting fatty bromchydrine (XII) with $(CH_3CO)_2O$ in pyridine, there is produced the diacetate of 15-methoxy-16beta-bromandrostandiole-3beta, 17beta (XIII), yield

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Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64602.

acid at 4° and during 20 hours on (XIV) in CHCl gives the acetate of 15ξ -methoxy-16alpha,17alpha-epoxyandrostanole-3beta, yield 82%, m.p. 121-122° (in CH_3OH), $[\alpha]^{20}D$-56° (w 1.60). Upon standing for 20 hours in a mixed solution of CH_3OH-aqueous K_2CO_3, (II) produces the acetate of 15ξ -methoxyandrostanol-3beta-one-17 (XV), yield 61%, m.p. 148-149°, $[\alpha]^{20}D$ + 19° (w 2.55). Similarly, from (VI) there is produced 15ξ -methoxyandrostanol-3beta-one-17, yield 70%, m.p. 165-168° (in aqueous acetone), $[\alpha]^{20}D$ + 31° (w 2.15). This, upon acetylization, gives (XV). Upon reduction of (II) with the aid of $NaBH_4$ (18 hours at 0° in alcohol), there is produced a saturated dioxy product which, after acetylization with $(CH_3CO)_2O$ in pyridine gives (V), yield 65%. Reduction of (II) with the

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Abs Jour: Ref. Zhur-Khimiya, No 19, 1958, 64602.

$(CH_3CO)_2O$, and the resulting liquid acetate brominated in CCl_4 at 0°, giving (I), yield 20%. Added are data on the UVspectra of (IX), (X) and the UV-curve of (X), and the IR-spectra of (VI), (X) and (XIL). $[\alpha]^{20}_D$ were determined in $CHCl_3$, excluding those specified.

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Abstract : See Ref Zhur-Khimiya, 1958, 64601

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

COUNTRY : CZECHOSLOVAKIA
CATEGORY : Organic Chemistry. Natural Substances and Their Synthetic Analogs
ABS. JOUR. : RZKhim., No. 23 1959, No. 82,14

AUTHOR : Labler, L.; Cerny, V.; Fajkos, J.; Sorm, F.
INST. : -
TITLE : On Steroids. XXXIII. Holarrhidine, a New Alkaloid from Holarrhena antidysenterica Wall. Labler, L., Cerny, V. XXXIV. The Structure*
ORIG. PUB. : Collect. Czechosl. Chem. Communs, 1959, 24, No 2, 370-377. 378-383; No 3, 766-768

ABSTRACT : No abstract.
See RZKhim., 1954, No 24, Nos 81782, 81783; 1959, No 9, No 31506.

*of Holarrhidine. Cerny, V., Labler, L., Sorm, F. XXXV. The Synthesis of Epimeric 16-Bromo Derivatives of Androsterone and Testosterone. Fajkos, J., Sorm, F.

CARD: 1/1

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Fajkos, Jan

CZECHOSLOVAKIA/Organic Chemistry. Natural Compounds and Their Synthetic Analogues. G-3

Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

Author : Fajkos, Jan; Sorm, Frantisek
Inst : -
Title : On Steroids. XXXV. Synthesis of Epimeric 16-Bromoderivatives of Androsterone and Testosterone.

Orig Pub : Chem. listy, 1958, 52, No 3, 505-522

Abstract : By the interaction of androsterone acetate with isoproponylacetate in the presence of H_2SO_4, diacetate of Δ^{16}-androstendiol-3α-17 (I) is obtained, yield 63 percent, melt. p. 169-170° (from benz.-petr. ether), $[\alpha]^{20}D$ = +35.6° (c = 2.91). 16-α-bromoandro-

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Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

stanol-3 α-one-17 acetate (II) is produced by bromination of I in CCl_4 at -5°, yield 80 percent, melt. p. 187-188° (from alc.), $[\alpha]^{20}D$ = +55° (c = 1.59). The solution of II in $CHCl_3$ is allowed to stand with methanol solution of HBr for 20 hours, and 16 α-bromoandrostanol-3 α-one-17 (III) is separated, yield 69 percent, melt. p. 205-207°, $[\alpha]^{20}D$ = +53° (c = 3.05); II is obtained again by acetylation of III. 16 β-bromoandrostanediol-3α-,17β triacetate is formed, with a simultaneous epimerization at $C_{(16)}$, by the reduction of II with $NaBH_4$ (at 5° in alcohol, 20 hours); the yield

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is 43 percent, melt. p. 171-172° (from CH_3OH), $[\alpha]^{20}D$ = +8° (c = 2.53). IV produces 16 β-bromoandrostanediol-3 α, 17β diacetate [sic] (V) by acetylation with $(CH_3CO)_2$) in pyridine, yield 85 percent, melt. p. 173-174° (from CH_3OH), $[\alpha]^{20}D$ = +61° (c = 2.03). By the saponification of IV or V (analogously to II), 16 β-bromoandrostanediol-3 α, 17β was obtained, yield 64 percent and 63 percent, melt. p. 177-178° (from aqueous CH_3OH), $[\alpha]^{20}D$ = +12° (c = 1.85), which being acetylated with $(CH_3CO)_2O$ in pyridine converts into V with a yield of 63 percent. Androsterone is prepared

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Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

by boiling IV with methanol solution of KOH (48 hours), yield 70 percent, melt. p. 181-182° (from acetone-petr. ether), $[\alpha]^{20}D = +94°$ (c = 2.42; alc.), which confirms the 16 β configuration of Br. The configuration of OH at $C_{(17)}$ in IV and V was established by hydrogenation of V (5 percent Pd / $CaCO_3$ in alcohol) into the known androstanediol-3 α, 17β diacetate, melt. p. 161-162° (from CH_3OH), $[\alpha]^{20}D = +119.4°$ (c = 1.23). Oxidation of IV with CrO_3 in CH_3COOH (20°, 20 hours) produces 16 β-bromoandrostanol-3 α-one-17 acetate, melt. p. 103-105° (from CH_3OH), $[\alpha]^{20}D = +107°$ (c = 1.49), which when

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CZECHOSLOVAKIA/Organic Chemistry. Natural Compounds and Their Synthetic Analogues. G-3

Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31500

saponified (analogously to II) produces 16 β -bromoandrostanol-3 α-one-17 (VI), yield 62 percent, melt. p. 228-230° (from CH_3OH), subliming at above 200°, $[\alpha]^{20}D$ = = +112° (c = 1.37). Similarly, 16 α-bromoandrostanedione-3,17 (VII) is produced by oxidation of III, yield 47 percent, melt. p. 148-149° (from CH_3OH) or 192-193°, $[\alpha]^{20}D$ = = +50° (c = 1.82), and from VI, 16 β -bromoandrostanedione-3,17 (VIII) is produced, yield 43 percent, melt. p. 144-145° (from CH_3OH), $[\alpha]^{20}D$ = +108° (c = 3.15). No epimerization takes place at $C_{(16)}$ in the reduction of II with $LiAlH_4$ in ether at 0° for 6.5

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Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

hours. The oily product obtained, when acetylated with $(CH_3CO)_2$ in pyridine, produces 16 α-bromoandrostanediol-3 α, 17 β diacetate (II), melt. p. 171-172° (from CH_3OH), $[\alpha]^{20}D = -29°$ (c = 2.07). The 16 α configuration of Br in IX is confrimed by dehydrobromination (boiling with methanol KOH for 72 hours) into 16 β, 17 β -epoxyandrostanol-3 α, yield 82 percent, melt. p. 190-191° (after crystallization from CH_3OH and sublimation at 180° / 0.1 mm), $[\alpha]^{20}D = +35.5°$ (c = 1.69); acetate, melt. p. 185-188° (from CH_3OH), $[\alpha]^{20}D = +289$ (c = 1.85). By the saponi-

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CZECHOSLOVAKIA/Organic Chemistry. Natural Compounds and Their Synthetic Analogues. G-3

Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

fication of 16 α -bromoandrostanol-3 β -one-17 acetate (X), 16 α -bromoandrostanol-3 β -one-17 (XI) was obtained, yield 80 percent, melt. p. 164-165° (from CH_3OH), $[\alpha]^{20}D = +52°$ (c = 2.38). No epimerization takes place at $C_{(16)}$ in the saponification, because in the acetylation with $(CH_3CO)_2O$ in pyridine, XI produces X again, melt. p. 173° (from CH_3OH), $[\alpha]^{20}D = +36°$ (c = 1.93). By the oxidation with CrO_3 or by Oppenauer's method, XI produces VII in both cases, yield 52 and 59 percent. By the saponification of 16 β -bromoandrostanol-3 β -one-17 acetate, 16 β -bromoandrostanol-3 β -one-17, melt. p.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

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203° (from CH_3OH), $[\alpha]^{20}D$ = +113° (c = = 2.39) is obtained, which when oxidized with CrO_3 in CH_3COOH, produce VIII, yield 50 percent. Analogous conversions were carried out with dehydroepiandrosterous acetate. $\Delta^{5,16}$-androstadienediol-3 β -17 diacetate is obtained from the latter (similarlyto I), yield 48 percent, melt. p. 147-148° (from benz.-petr. ether), $[\alpha]^{20}D$ = -49.7° (c = = 3.82); when brominated in CCl_4 at -10°, this discetate produces Δ 5-16 α-bromo-androstenol-3 β -one-17 acetate (XII), yield 77 percent, melt. p. 181-183° (from alc.), $[\alpha]^{20}D$ = -23.7° (c = 3.77). By the

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Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

$[\alpha]^{20}D$ = +9° (c = 2.22). By the saponification of XV, Δ^{5}-16β-bromo-androstenediol-3β,17β is obtained, melt. p. 175-176° (from CH_3OH), $[\alpha]^{20}D$ = -41.5° (c = 187). The dehydrobromination of VY (methanol solution of KOH), 20°, 48 hours) results in dehydroepiandrosterone (XVI), yield 67 percent, melt. p. 147-148° (from CH_3OH), $[\alpha]^{20}D$ = +2.8° (c = 1.75), which confirms the 16β-configuration of Br. By the oxidation of VI with CrO_3 in pyridine, Δ^{5}-16β-bromoandrostenol-3β-one-17 acetate (XVII) is produced, yield 69 percent, melt. p. 173-174° (from CH_3OH),

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$[\alpha]^{20}D$ = +36° (c = 2.22). Then saponified, XVII produces Δ^{5}-16β-bromoandrostenol-3β-one-17 (XVIII), melt. p. 176-177° (from CH_3OH), $[\alpha]^{20}D$ = +59° (c = 2.6). XVII is formed by acetylation of XVIII, and the oxidation of XVIII according to Oppenauer results in Δ^{4}-16β-bromoandrostenedione-3,17 (XIX), yield 66 percent, melt. p. 181-182° (from CH_3OH), $[\alpha]^{20}D$ = +188° (c = 1.52). A mixture of 17-hydroxyepimers is obtained by the reduction of XII with $LiAlH_4$ (similarly as in the reduction of II). Δ^{5}-16α-bromoandrostenodiol-3β,17β diacetate (XX) and its 17-epimer (XXI) are

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CZECHOSLOVAKIA/Organic Chemistry. Natural Compounds and Their Synthetic Analogues. G-3

Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

separated from this mixture after acetylation with $(CH_3CO)_2O$ in pyridine and cristallization from CH_3OH. XX: yield 38 percent, melt. p. 186-188°, $[\alpha]^{20}D$ = -94° (c = 2.56). XXI: yield 15 percent, melt p. 203-205°, $[\alpha]^{20}D$ = -64° (c = 1.34). The dehydrobromination of XXI results in XVI, yield 62 percent, and from XX Δ^5-16β,17β-epoxyandrostenol-3β is formed, melt. p. 143-144° (from CH_3OH), [ ]^{20}D = -25° (c = 1.84); acetate, melt. p. 168-169° (from CH_3OH), $[\alpha]^{20}D$ = -68.5° (c = 1.11). XIV and XIX diketones were used to obtain epimeric 16-bromotestosterones. The reduction

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Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

of XIV with $LiAlH_4$ at 0° produces crude Δ^4-16α-bromoandrostenediol-3β,17β (XXII), which is oxidized in $CHCl_3$ with freshly prepared MnO_2 (20°, 18 hours) into Δ^4-16α-bromoandrostenol-17β-one-3 (XXIII), yield 21 percent, melt. p. 172-174° (from chlorof.-petr. ether), $[\alpha]^{20}D$ = +53.8° (c = 2.6); acetate, melt. p. 216-217° (from CH_3OH), $[\alpha]^{20}D$ = +29.8° (c = 1.41). Δ^4-16β-bromoandrostenol-17β-one-3 (XXV) is synthesized analogously to XXIII from XIX through the crude 16-epimer of XXII (XXIV), yield 26 percent, melt. p. 187-189° (from acetone), $[\alpha]^{20}D$ = +73° (c = 2.14°);

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Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

acetate, melt. p. 158-159° (from CH_3OH), $[\alpha]^{20}D = +126°$ (c = 2.41). The configuration of Br in XXIII and XXV is confirmed by their oxydation with CrO_3 in pyridine into XIV, yield 65 percent, and XIX, yield 67 percent respectively. The dehydrobromination of XXII (boiling with methanol KOH for 48 hours) resulted in a product, the infrared spectrum of which did not show the CO group; on the contrary, the product of dehydrobromination of XXIV contained the CO group, which confirms the configuration of OH in the initial compounds. XXV was synthesyzed also in another way. XVIII formate,

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CZECHOSLOVAKIA/Organic Chemistry. Natural Compounds and Their Synthetic Analogues. G-3

Abs Jour : Ref Zhur-Khimiya, No 9, 1959, 31508

p. 200-201° (from chlrof.-CH_3OH), $[\alpha]^{20}D$ = = +5.2° (c = 2.58). The latter produces XXV acetate by the oxidation with Al isopropylate in toluene in the presence of cyclohexanone, and the XXV acetate produces XXV by saponification. Data concerning the ultraviolet spectra of XIV, XIX, XXIII and XXV are presented. All the $[\alpha]$Ds were determined in $CHCl_3$, the exceptions are mentioned. See Ref Zhur-Khimiya, 1958, 81783, for report XXXIV. -- Antonin Emr.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

AUTHORS: Fajkoš, J. and Šorm, F. CZECH/8-52-11-15/30

TITLE: On Steroids (O steroidech) XXXIX. Epimeric 2- and 4-bromo Derivatives of 3-ketoandrostane (XXXIX. Epimerní 2-brom a 4-bromderiváty 3-ketoandrostanu)

PERIODICAL: Chemické Listy, 1958, Vol 52, Nr 11, pp 2115 - 2133 (Czechoslovakia)

ABSTRACT: The preparation of the epimeric 2- and 4-bromoderivatives of androsterone, 3α, 17β-dihydroxyandrostane, 3,17-diketoandrostane and dihydrotestosterone are described. The original bromohydrins were prepared by the decomposition of the related epoxides and reactions were carried out to prove the structure of the stated materials. The IR. spectra and optical rotation of the epimeric 2- and 4-bromo-3-ketoderivative are discussed. A previous report (Ref 1) has described the preparation of a series of steroidal hormone analogues with a bromo-substituent in the 16 position. The 2- and 4-bromo-derivatives were studied to show the effect of substitution on physiological activity of the hormones. It was considered at the same time that these substances would be of interest from the

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CZECH/8-52-11-15/30

On Steroids XXXIX. Epimeric 2- and 4-bromo Derivatives of 3-ketoandrostane

sterochemical and spectroscopic viewpoint for the displacement of the carboxyl maxima in bromoketones for it is possible to infer the configuration of the bromine atom and even, in certain cases, the conformation of the related ring. The structures of the various compounds are discussed in considerable detail. The following preparations are given: 2α,3α-epoxy-17-keto androstane; 2β-bromo-3α-hydroxy-17-keto androstane; 2β-bromo-3α-acetoxy-17-keto androstane; 2β-bromo-3α-acetoxy-17β-hydroxyandrostane; 2β-bromo-3α,17β-diacetoxyandrostane; 2β-bromo-3α,17β-dihydroxyandrostane; 3α-acetoxy-17-ketoandrostane; 3α,17β-diacetoxyandrostane; 2β-bromo-3α-hydroxy-17β-acetoxyandrostane; 17β-acetoxyandrostane (2); 2α,3α-epoxy-17β-acetoxyandrostane; 3α,4α-epoxy-17β-acetoxyandrostane; 2α,3α-epoxy-17β-hydroxyandrostane; 2β-acetoxy-3α-hydroxy-17-ketoandrostane; 2β,3α-diacetoxy-17-keto androstane; 2β-acetoxy-3α-mesyloxy-17-keto-androstane; 2β,3β-epoxy-17-ketoandrostane; 2β,3β-epoxy-17β-hydroandrostane; 2β-bromo-3,17,diketoandro-

Card2/4 stane; 2β-bromo-3β,17β-dehydroxyandrostane; 2β-bromo-3β,

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On Steroids XXXIX. Epimeric 2- and 4-bromo Derivatives of 3-ketoandrostane

17β-diacetoxyandrostane; 3-keto-17β-hydroxyandrostane; 3β,17β-diacetoxyandrostane; 2α-bromo-3,17-diketoandrostane; 2α-bromo-3β,17β-dihydroxyandrostane; 2α-bromo-3β-17β-diacetoxyandrostane; 2β-bromo-3-keto-17β-acetoxyandrostane; 2α-bromo-3 keto-17β-acetoxyandrostane; 2β-bromo-3β-hydroxy-17β-acetoxyandrostane; 2α-bromo-3β-hydroxy-17β-acetoxyandrostane; 3α,4α-epoxy-17-ketandrostane; 3α-hydroxy-4β-bromo-17-ketoandrostane; 3α-hydroxy-4β-bromo-17β-acetoxyandrostane; 3α-acetoxy-4β-bromo-17-keto-androstane; 3,17-diketo-4β-bromoandrostane; 3,17-diketo-4α-bromoandrostane; 3α-acetoxy-4β-bromo-17β-acetoxyandrostane; 3α,17β-diacetoxy-4β-bromoandrostane; 3 keto-4β-bromo-17β-acetoxyandrostane; 3 keto-4α-bromo-17β-acetoxy-androstane; 3 keto-17β-acetoxyandrostane (4).
There are 1 figure, 1 table and 23 references, 2 of which are Czech, 15 English, 2 Swiss and 4 German.

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CZECH/8-52-11-15/30

On Steroids XXXIX. Epimeric 2- and 4-bromo Derivatives of 3-ketoandrostane

ASSOCIATION: Oddělení přírodních látek, Chemický ústav, Československá akademie věd, Praha (Division of Natural Products, Institute of Chemistry, Czechoslovak Ac.Sc., Prague)

SUBMITTED: March 12, 1958

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APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

AUTHOR: Fajkoš, J. CZECH/8-52-11-16/30

TITLE: On Steroids (O steroidech) XL. Reduction of Steroid Ketones with Lithium Tri-terc-butoxyaluminohydride (XL. Redukce steroidních ketonů tri-terc-butoxyaluminohydridem lithným)

PERIODICAL: Chemické Listy, 1958, Vol 52, Nr 11, pp 2134 - 2139 (Czechoslovakia)

ABSTRACT: Lithium tri-terc-butoxyaluminohydride reduction of various types of steroid ketones has been studied as well as their steric course and velocity of reduction in comparison with the course of reduction by other hydride reagents. It was found that the new reagent (introduced by Brown and McFarlin), in the majority of cases, reduces with ease and with greater stereo-specificity than lithium aluminium hydride or sodium borohydride. The following conversions were brought about: 3β-formyloxy-17-keto-androstene (5) to 3β-formyloxy-17β-hydroxyandrostene (5), 3β-acetoxy-17-ketandrostene-(5) to 3β-acetoxy-17β-hydroxy-androstene-(5); 3β-hydroxy-17-ketoandrostane; 2α,3α-epoxy-17-ketoandrostane to 2α,3α-epoxy-17β-hydroxyandrostane; 2β-bromo-3α-acetoxy-17 ketoandrostane to 2β-bromo-3α-acetoxy-17β-hydroxyandrostane; 4β-bromo-3α-acetoxy-17β-hydroxyandrostane; 2β-bromo-3,

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CZECH/8-52-11-16/30

On Steroids. XL. Reduction of Steroid Ketones with Lithium Tri-terc-butoxyaluminohydride

17-diketoandrostane to 2β-bromo-3β-17β-dihydroxyandrostane; 3β-acetoxy-16α-bromo-17-ketoandrostane to 3β-17β-17α-diacetoxy-16α-bromoandrostane; 3β-acetoxy-16β-bromo-17-ketoandrostane to 3β-acetoxy-17β-hydroxy-16β-bromoandrostane; 3β-formyloxy-16β-bromo-17-ketoandrostene-(5) to 3β-formyloxy--16β-bromo-17β-hydroxyandrostene-(5); 3,17-diketo androstene-(4) to 3-keto-17β-acetoxyandrostene (4); 3β-acetoxy-7,17-diketoandrostene-(5) to 3β,17β diacetoxy-7-ketoandrostene-(5) (in these last two cases the crude product was acetylated and recrystallised from methanol); 3β-acetoxy-16-bromo-17-ketoandrostene-(15) to 3β-acetoxy--16 bromo-17β-hydroxyandrostene-(15) (in this case the product was acetylated with acetic anhydride in pyridine and recrystallised from methanol); 2α-bromo-4-ketocholestane to 2α-bromo-3β-hydroxycholestane; 2β-bromo-3-ketocholestane to 2β-bromo-3β-hydroxycholestane; 3β-acetoxy-7-ketocholestane to 3β-acetoxy-7α + 7β hydroxycholestane. The yields of the β-epimer were, in the main, between 80% and 100% and proved, on the whole, to give higher yields than

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On Steroids XL. Reduction of Steroid Ketones with Lithium Tri-terc-butoxyaluminohydride

the other hydrides. Apart from the marked stereo-specificity, the reagent has a number of other advantages over the other hydrides. During reduction no degradation of the ester groups was noted; the epoxide bond was undisturbed. In the reduction of conjugated ketones, the unsaturated system was maintained and where it is necessary to use more energetic conditions for the reduction of carbonyl the double bond remains unlike the case of sodium borohydride and again unlike this latter reagent no epimerisation results with the bromine atom in the unstable bromo ketones and thus the reagent has significance in the study of the configuration of these bromo ketones. Reduction of Steroidal Ketones. Lithium tri-terc butoxyaluminohydride (Brown and McFarlin - Ref 1). The substances to be reduced (500 mg) were dried by distillation with benzene, dissolved in dry tetrahydrofuran (5 ml.) and cooled to 0 °C. This solution was added to the hydride solution (1 g - calculated on 1 ketogroup to be reduced in 5 ml. tetrahydrofuran), cooled to 0 °C and left to stand

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

CZECHOSLOVAKIA/Optics - Spectroscopy. K

Abs Jour : Ref Zhur Fizika, No 4, 1960, 9951

Author : Horak, M., Fajkos, J.
Inst : -
Title : On Steroids. XLII. Infrared Spectra and Conformation of Steroid Bromhydrines.

Orig Pub : Collect. Czechosl. Chem. Communs, 1959, 24, No 5, 1515-1519

Abstract : The authors investigated the effect of the halogen on the frequency of the hydroxyl maximum in steroid bromhycrines. The results are discussed from the point of view of stereo chemistry of the investigated compounds.

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APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

FAJKOS, J.; SORM, F.

Steroids XXXIX. Epimeric 2- and 4-bromo derivatives of androstan-3-one. In English. Coll.Cz.Chem. 24 no.9:3115-3135 S '59.
(MRAI 9:5)

1. Department of Natural Products, Institute of Chemistry, Czechoslovak Academy of Science, Prague.
(Steroids) (Adrostanone) (Bromine)

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

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FAJKOS, J.

Steroids. XXXVI. Catalytic hydrogenation of steroidal enol acetates; a new synthesis of testosterone and oestradiol. Coll Cz Chem 25 no.4: 1078-1085 Ap '60. (EEAI 9:12)

1. Department of Natural Products, Institute of Chemistry, Czechoslovak Academy of Science, Prague.
(Hydrogenation) (Steroids) (Enols) (Testosterone) (Estradiol) (Catalysis) (Acetates)

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

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JOSKA, J.; FAJKOS, J.; SORM, F.

Steroids. L. Analogues of androgens with the B-norsteroid skeleton.
Coll Cz Chem 25 no.4:1086-1090 Ap '60. (EEAI 9:12)

1. Department of Natural Products, Institute of Chemistry, Czechoslovak Academy of Science, Prague.
(Steriods) (Androgens) (Norsteroids)

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

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MALUNOWICZ, I.; FAJKOS, J.; SORM, F.

On steroids. Part 41: Epimeric 2-bromo and 4-bromo derivates of cholestan-3-one. Coll Cz Chem 25 no.5:1359-1370 My '60.

1. Department of Natural Products, Institute of Chemistry, Czechoslovak Academy of Sciences, Prague. 2. On leave of absence from the "Wyzsza szkola rolnicza, Katedra chemii ogolnej," Wroclaw (for Malunowicz).

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

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Steroids. LI. Derivatives of 5α- and 5β-b-norandrostane. Coll Cz Chem 25 no.9:2341-2357 S '60. (EEAI 10:9)

1. Department of Natural Products, Institute of Chemistry, Czechoslovak Academy of Science, Prague.

(Steroids) (Norandrostane)

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

FAJKOS, J.; JOSKA, J.

Steroids. LV. Bromination of 3β-acetoxy-5α-androstan-16-one.
Coll Cz Chem 25 no.11:2863-2877 N '60. (EEAI 10:6)

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Science, Prague.
(Steroids) (Bromination) (Acetoxy group)
(Androstanone)

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

FAJKOS, J.; JOSKA, J.

On Steroids. Part 57: Infrared spectra of the epimeric 16-Bromo-17-Ketones and alcohols of the androstane series; conformation of the D-ring. Coll Cz Chem 26 no.4:1118-1136 Ap '61.

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague.

(Steroids) (Ketone) (Androstane)

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

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On steroids. LVIII. Some analogues of androgens substituted in position 7. Coll Cz chem 26 no.6: 1646-1657 Je '61.

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Science.

(Androgens)

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

JOSKA, J.; ACHREM, A.A.; FAJKOS, J.; SORM, F.

On steroids. Part 61: Some B-nor steroid hormone analogues. Coll Cz Chem 26 no.8:2050-2057 '61.

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague. 2. On leave of absence from the Institute of Organic Chemistry, Academy of Science, Moscow, USSR. (for Achrem).

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

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PROCHAZKA, Z.; FAJKOS, J.; JOSKA, J.; SORM, F.

On steroids. Part 60: Microbial oxygenation of B-nor steroids. Coll Cz Chem 26 no.8:2068-2071 '61.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

FAJKOS, J.

SURNAME, Given Names

Country: Czechoslovakia

Academic Degrees: [not given]

Affiliation: Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague

Source: Prague, Collection of Czechoslovak Chemical Communications, Vol 26, No 11, November 1961, pp 2734-2748

Data: "On Steroids. LXIII. Some Analogues of Androgens Substituted in Position 16."

Authors:

SANDA, V
FAJKOS, J

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

FAJKOS, J.; JOSKA, J.

On steroids. Part 67 : 16-bromoderivates in the 5β-B-norandrostane series. Coll Cz Chem 27 no.8:1849-1855 Ag '62.

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague.

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On steroids. Part 68 : Synthesis of the epimeric 15,16-epoxides in the androstane series. Coll Cz Chem 27 no.8:1856-1860 Ag '62.

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APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

CSSR

JOSKA, J.; FAJKOS, J.; SORM, F.

no academic degrees indicated

Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Science, Prague

Prague, Collection of Czechoslovak Chemical Communications, No 1, 1963, pp 82-100.

"On Steroids, LXXII. Fission of the 5α,6α-Epoxyderivatives in the B-Norsteroid Series"

(3)

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CSSR

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Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague

Prague, Collection of Czechoslovak Chemical Communications, No 3, 1963, pp 621-628.

"On Steroids LXIV. 5α,6α-Epoxides in the B-Norsteroid Series"

(2)

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

CZECHOSLOVAK

FAJKOS, J; JOSKA, J; PITHA, J; SORM, F.

Institute of Organic Chemistry and Biochemistry of the Czechoslovak Academy of Sciences, Prague (for all)

Prague, Collection of Czechoslovak Chemical Communications, No 9, 1963, pp 2337-2343

"On Steroids. LXXX. Intramolecular Hydrogen Bonding in 3,6-Disubstituted 5-Beta-B-Norsteroids: Conformation of Ring A."

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

FAJKOS, J.; JOSKA, J.; SORM, F.

On steroids. Pts. 74-75. Coll Cz Chem 28 no.3:605-628 Mr '63.

1. Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague.

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On steroids. Pts. 82-83. Coll Cz Chem 28 no.10:2605-2617 O '63.

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APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

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FAJKOS, J.; JOSKA, J.; SORM, F.

On steroids. Pt. 85. Coll Cz Chem 29 no. 3:652-671 Mr '64.

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"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

FAJKOS, J.

CZECHOSLOVAKIA

JOSKA, J; FAJKOS, J; SOHM, F

Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences, Prague - (for all)

Prague, Collection of Czechoslovak Chemical Communications, No 1, January [illegible], pp [illegible]

"On steroids. Part 95: 4,5-epoxides in the b-norsteroid series."

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

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JOSKA, J; FAJKOS, J; SOHM, F

Institute of Organic Chemistry and Biochemistry, Czechoslovak Academy of Sciences - (for all)

Prague, Collection of Czechoslovak Chemical Communications, No 7, July 1966, pp 2745-2758

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APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

"APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7

CZECHOSLOVAKIA

FAJKOS, J; JOSKA, J; SORM, F

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Medical establishments and problems of their construction. Cesk. nemoc 22 no.3-4:50-55 My '54.

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(HOSPITALS,
*construction)

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(HOSPITAL ADMINISTRATION,
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(MEDICINE, PREVENTIVE,
in Czech.)

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(NEOPLASMS, statist.
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(NEOPLASMS, prev. & control
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Previous experiences with dispensary treatment and screening of precancerous conditions in the northern Moravian region. Cesk. zdrav. 11 no.12:508-513 D'63.

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FAJMON, Josef, inz.; ADAM, Jan, inz.; KRACMER Dusan, inz.

Thorough preparation of investments is always worthwhile. Inz stavby 12 no.5:214-216 My '64.

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Use of vibration ramming engines in pipe duct broaching. Inz stavby 11 no.11;Supplement: Mechanizace no.11:173-175 N'63.

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[illegible], [illegible].

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1. Prirodovedecka fakulta Karlovy university; Geologicky ustav, Ceskoslovenska akademie ved.

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FAJST, M; FREJVALD, M.

Natural Sciences Faculty of Charles University (Prirodovedecka fakulta Karlovy university), Prague (for both)

Prague, Casopis pro mineralogii a geologii, No 1, 1964, pp 99-101

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Hearing after mastoidectomy with protective coagulum and primary suture. Cesk. otolar. 5 no.1:44-49 Feb 56.

1. Z Otolaryngologicke Kliniky VLA J. Ev. Purkyne v Hradci Kralove.

(MASTOID, surg.
radical excis. with drainage, eff. on hearing, comparison with mastoidectomy with protective coagulum & primary suture. (Cz))

(HEARING,
postop., after mastoidectomy with drainage & mastoidectomy with protective coagulum & primary suture, comparison. (Cz))

APPROVED FOR RELEASE: 03/13/2001 CIA-RDP86-00513R000412410002-7"

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Certain cytological aspects of nasal secretion. Cesk. otolar. 7 no.6: 344-349 Dec 58.

1. Otolaryngologicka klin. VLA J. Ev. Purkyne v Hradci Kralove, predn. prof. MUDr. Jan Hybasek. J. F., ORL klin. KU, Hradec Kralove.
(NASAL CAVITY,
secretions, cytol. aspects (Cz))

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CZECHOSLOVAKIA

Major Mojmir SALEMY Graduate Physician (promovani lekar) and Major Jaroslav FAJSTAVR CSc MD; Department of Otorhinolaryngology of the Central Military Hospital (OEL oddeleni Ustredni vojenske nemocnice) Head (naceinik) Col Docent Edwin CERNY MD; Prague.

"Role of Allergy in Acute Inflammations of the Maxillary Sinus."

Prague, Vojenske Zdravotnicke Listy, Vol 31, No 6, Dec 62; pp 281-285.

Abstract [English summary modified]: Authors saw acute sinusitis in 21 from among 1000 soldiers in a year. They analyze 84 patients seen Jul 60 - Jun 61; very thorough clinical and bacteriologic data are given about their symptoms, etiology, bacteriologic tests done. Allergy was responsible for about one third of the maxillary sinusitis cases; mostly bacterial allergy. Five Western references, Czech thesis by junior author.

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FAJSTAVR, J.

Neurinoma of the nasal septum. Cesk. otolaryng. 12 no.2:122-124 Mr '63.

1. Otolaryngologicke oddeleni UVN v Praze, vedouci doc. dr. E. Cerny.

(NOSE NEOPLASMS) (NEURILEMMOMA)
(NASAL SEPTUM)

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FAJSTAVR, J.

Sluder's syndrome. Cesk. otolaryng. 12 no.5:283-285 O '63.

1. Otorinolaryngologicke oddeleni UVN v Praze, nacelnik doc. dr. E. Cerny.
(FACIAL NEURALGIA) (DIAGNOSIS, DIFFERENTIAL)
(ALCOHOL, ETHYL)

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ZELENY, M.; FAJSTAVR, J.

The role of allergy in chronic inflammations of the maxillary sinuses. Cesk. otolaryng. 14 no.2:78-83 Ap'65.

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DIAGNOSIS

UDC 616.21-002-022.78.75-07

CZECHOSLOVAKIA

FAJSTAVR, Jaroslav: Otolaryngological Department of the Central Military Hospital (Otolaryngologicke Oddeleni Ustredni Vojenske Nemocnice), Prague, Head (Nacelnik) Dr ERVIN CERNY.

"Early Diagnosis of Influenza Catarrh of the Upper Airways."

Prague, Vojenske Zdravotnicke Listy, Vol 35, No 4, Aug 66, pp 156 - 158

Abstract: An analysis of cytological findings in nasal secretion during an epidemic of influenza of the type A_2 is presented. The nature of the disease was verified virologically and serologically. Some degenerative changes in ciliated epithelial cells appear to be typical for influenza type infections. The data were obtained by examination of 25 boys aged 16. 2 Figures, 6 Western, 4 Czech, 2 Russian, 1 Japanese reference.

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FAJT, A.

No affiliation given

Bratislava, Farmaceuticky obzor, No 10 [October] 1966, p 477

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KALAB, Z.; FAJT, M.; STEINEROVA, H.

Familial recurrent amyotrophic neuralgia in children. Cesk. pediat. 20 no.10:883-886 O '65.

1. Vyzkumny ustav pediatricky v Brne a Ortopedicke a neurologicke oddeleni Fakultni detske nemocnice v Brne.

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