the solution was evaporated to dryness under reduced pressure. The last traces of solvent were removed by repeated addition and evaporation of methanol. Crystallization of the residual material from water gave β-ouabagenin diacetate (815 mg.) as colorless prisms melting at 192–196°. The analytical sample, prepared by recrystallization from the same solvent, melted at 193–196°, $[\alpha]^{29}D$ +1.7° (*c* 1.76, methanol).

Anal. Calcd. for $C_{27}H_{38}O_{10}\cdot H_2O$ (540.59): C, 59.98; H, 7.46; CH_3CO, 16.3. Found: C, 59.85; H, 7.75; CH_3CO, 18.6.

An anhydrous sample, m.p. 193–196°, was obtained from the monohydrate by drying under vacuum at 130° for 3 hours.

Anal. Calcd. for $C_{27}H_{38}O_{10}$ (522.57): C, 62.05; H, 7.33. Found: C, 62.23; H, 7.39.

Conversion of Ouabagenin Diacetate into Ouabagenin Monoacetonide Diacetate.—Ouabagenin diacetate (70 mg., m.p. 192–193°) was added to a suspension of 1.3 g. of anhydrous copper sulfate in 10 ml. of acetone, and the mixture was heated under reflux for 5 hours. The copper sulfate was then removed by filtration, and the filtrate was concentrated to small volume and diluted with petroleum ether. The product obtained in this manner weighed 40 mg. and melted at 269–271°, $[\alpha]^{29}D$ +42.6° (*c* 2.24, methanol). A mixed melting point with an authentic sample of ouabagenin monoacetonide diacetate showed no depression. The infrared absorption spectra of the two samples were identical.

Acetone Determinations.—The per cent. of acetone in ouabagenin monoacetonide and in "anhydroöuabagenin" was estimated under identical conditions by the following procedure. A weighed sample of the compound to be analyzed, 2.5 ml. of acetic acid and 10 ml. of water were placed in a distilling flask connected to a condenser and receiver containing a filtered solution of 100 mg. of *p*-nitrophenylhydrazine in 2.5 ml. of acetic acid and 5 ml. of water. The contents of the distilling flask were heated gently until solution took place and then slowly distilled until the volume was about 4 ml. Water (5 ml.) was added to the receiver, and after 15 min. the precipitated acetone *p*-nitrophenylhydrazone was removed by filtration and dried under vacuum at 100° for one hour. From 120 mg. of ouabagenin monoacetonide, 26.2 mg. of acetone *p*-nitrophenylhydrazone, m.p. and mixed m.p. 147.5–149.5°, was obtained, representing 53% of the theoretical amount. "Anhydroöuabagenin" (100 mg.) furnished 22.7 mg. (55%) of an identical product.

Treatment of Ouabagenin with Periodic Acid.—A solution of 17.7 mg. of ouabagenin monohydrate[9] (prepared by hydrolysis of the monoacetonide with aqueous acetic acid) in 2 ml. of water was treated with 0.50 ml. of an aqueous solution containing 12.0 mg. of periodic acid dihydrate. After standing at room temperature for 30 min., 1 ml. of 1 *N* sulfuric acid and 0.2 g. of potassium iodide were added, and the liberated iodine was titrated with 0.0193 *N* sodium thiosulfate solution to the starch end-point. In all 20.55 ml. of thiosulfate was required as compared with 20.60 ml. in a blank run.[15] The difference, 0.05 ml., corresponds to 1.2% of the theoretical value (4.2 ml.) computed on the basis of one 1,2-glycol grouping per molecule of ouabagenin. A similar result was obtained with β-ouabagenin diacetate.

(15) A stable end-point in the titration of the ouabagenin reaction was obtained only after a considerable period of time. This behavior suggests the formation of a cycle, possibly involving 1,3-hydroxyl groups, that is only slowly hydrolyzed by sulfuric acid.

HOUSTON, TEXAS

[FROM THE CHEMO-MEDICAL RESEARCH INSTITUTE OF GEORGETOWN UNIVERSITY]

The Reaction of Saturated Steroid 3-Enol Acetates with N-Bromosuccinimide[1-3]

BY MARTIN RUBIN AND BERNARD H. ARMBRECHT

RECEIVED SEPTEMBER 8, 1952

The reaction of N-bromosuccinimide (NBS) with Δ^2-3-acetoxycholestene and Δ^3-3-acetoxycoprostene was investigated. From the Δ^2-3-acetoxycholestene reaction Δ^1- and Δ^4-cholesten-3-one, 2-bromocholestan-3-one and starting material were isolated. The amount of 2-bromocholestan-3-one, formed as a function of increasing reaction time, was found to increase at the expense of the Δ^1-cholestene-3-one. From the reaction of Δ^2-3-acetoxycoprostene with NBS, Δ^4-cholesten-3-one and crude Δ^1-coprosten-3-one were isolated.

In recent years the application of the reaction of N-bromoimides,[4] and especially N-bromosuccinimide[5] with olefins for the preparation of allyl bromides or the conjugated diene system formed from such compounds by dehydrohalogenation, has assumed increasing importance.[6] The special case in which one of the olefinic carbon atoms is also substituted by an esterified hydroxyl group (*i.e.*, the enol ester of a ketone) has received little attention. In the single previously described reaction of this type it had been observed that the reaction of NBS with the enol acetate of a 20-keto steroid, Δ^{17}-3,12,20-triacetoxypregnene, resulted in the formation of the corresponding α,β-unsaturated ketone, Δ^{16}-3,12-diacetoxypregnen-20-one.[7] We have now studied the reaction of NBS with the typical enol acetates of the 3-keto steroid ring, A/B *trans* or allo form, Δ^2-3-acetoxycholestene, and of the A/B *cis* or normal form Δ^2-3-acetoxycoprostene, XIV, in an attempt to utilize this reaction for the introduction of the Δ^4-double bond into the saturated A ring of the steroid nucleus. This step is of importance in the preparation of some steroid hormones.

In the absence of any directing influence of the 3-acetoxy group of II or of the steric arrangement of the allo molecule, we might expect that NBS substitution would result in the formation of V and VI in an equal amount. While VI might be expected to be a comparatively stable compound due to the absence of an available hydrogen atom for spontaneous dehydrohalogenation, V could lose hydrogen bromide by elimination of the bromine on C_4 and the available hydrogen on C_5. The course of the reaction appeared to follow such a hypothetical sequence. Within a minute of the typical NBS "bromination" color change, hydrogen bromide spontaneously evolved from the reaction mixture. Arrest of the reaction after one minute by cooling, followed by neutralization of the

(1) Presented at the April, 1951, Meeting of the American Chemical Society, Division of Organic Chemistry, Cleveland, Ohio.

(2) Abstracted from a thesis submitted by Bernard H. Armbrecht in partial fulfillment of the requirements for the degree of Doctor of Philosophy, June, 1951.

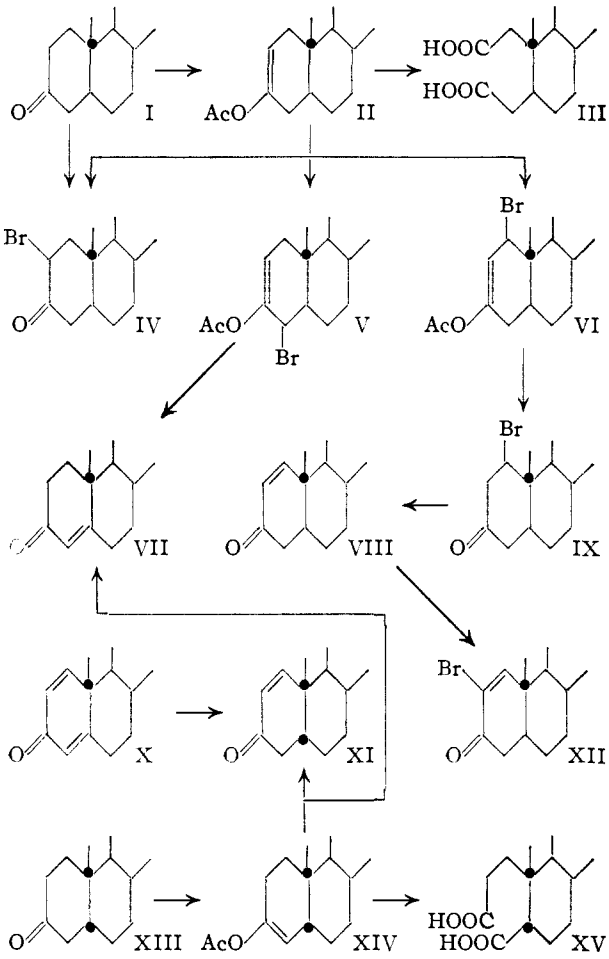
(3) Supported by research grant from the Chemical Specialties Company, Inc., New York, New York.

(4) A. Wohl, *Ber.*, **52**, 51 (1919).

(5) K. Ziegler, A. Spaeth, E. Schaaf, W. Schumann and F. Winkelman, *Ann.*, **551**, 80 (1942).

(6) C. Djerassi, *Chem. Revs.*, **43**, 271 (1948).

(7) C. Djerassi and C. Scholz, *J. Org. Chem.*, **14**, 660 (1949).



hydrogen bromide by the addition of pyridine, permitted the isolation of unchanged II, Δ^4-cholesten-3-one, VII, and Δ^1-cholesten-3-one, VIII. The origin of these reaction products is explicable when one considers the potential thermal instability and instability to acid of the primary bromination intermediates V and VI. Spontaneous dehydrobromination of the allylic bromide V followed by acid-catalyzed cleavage of the consequent enol would yield the isolated product Δ^4-cholesten-3-one, VII. Similar ketonization of the 1-bromo enol, VI, would produce a β-bromoketone, IX, which on spontaneous dehydrobromination would yield the isolated product, Δ^1-cholesten-3-one, VIII.[7,8] The quantitative distribution of the reaction products indicated that the primary attack by NBS on II was at the C-1 position.

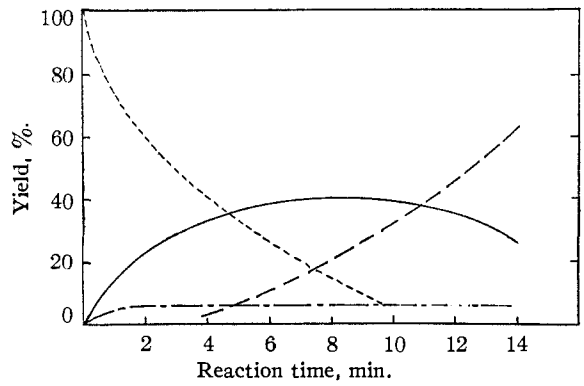


Fig. 1.—Product distribution of NBS and Δ^2-3-acetoxycholestene. Curves on graph represented by: —, Δ^1-cholesten-3-one; —·—, Δ^4-cholesten-3-one; ·····, Δ^2-3-acetoxycholestene; — —, 2-bromocholestan-3-one.

With an initial course outlined above, the reaction might be expected to become more complicated as the time increased. In addition to II, VII and VIII, as well as the intermediates leading to them, one can expect that the hydrogen bromide in the reaction mixture would catalyze the regeneration of I from II and result in the formation of free bromine by reaction with NBS. Bromine and I react to yield 2-bromocholestan-3-one, IV.[9] The same product is formed from I and NBS[10] and by the reaction of bromine and II as we have now demonstrated. Under these circumstances one may expect to find a gradual increase of 2-bromocholestan-3-one in the reaction mixture as a function of time. That this occurs is illustrated by Fig. 1. The formation of the reaction products described above has accounted for over 85% of the initial starting material. The inevitable fractionation losses and the trace nature of the other reaction products has made it difficult to establish the course of the balance of the reaction with certainty. By chromatographic separation on silica of the 2,4-dinitrophenylhydrazones of the reaction residues we have been able, however, to find evidence for the presence of Δ^1-2-bromocholesten-3-one, XII, in the mixture. Formation of XII is readily explicable as a further bromination product of VIII.[11] That it may arise at the expense of VIII in the reaction mixture is indicated by Fig. 1 which shows a decline in VIII at the later stages of the reaction at a time when XII concomitantly started to appear.

The reaction of NBS and XIV yielded coprostan-3-one, Δ^4-cholesten-3-one and a product having physical characteristics ascribed to an impure sample of Δ^1-coprosten-3-one prepared by Rupe nickel reduction of $\Delta^{1,4}$-cholestadien-3-one.[12] Since the ultraviolet absorption maxima and the melting point of our sample of Δ^1-coprosten-3-one prepared either by the NBS reaction or a modified catalytic reduction of $\Delta^{1,4}$-cholestadien-3-one, was not in complete agreement with the values for the purified material,[13,14] we have examined our material for infrared absorption.[15] In addition to the anti-

(8) The referee has advanced an alternative reaction sequence for the formation of VII and VIII based on primary elimination of acetyl bromide from V and VI. To account for the hydrogen bromide evolved from the reaction mixture would require the production of hydrogen bromide from acetyl bromide under the conditions of the reaction. Direct test of this possibility was made by refluxing in carbon tetrachloride a mixture of II, VII, VIII, NBS and succinimide in the molar proportions indicated by the quantitative distribution of reaction products of Fig. 1 at one minute reaction time. Hydrogen bromide was not evolved under these conditions.

(9) A. Butenandt and A. Wolff, *Ber.*, **68**, 2091 (1935).

(10) C. Djerassi and C. R. Scholz, *Experientia*, **3**, 107 (1947).

(11) C. Djerassi and C. R. Scholz, THIS JOURNAL, **69**, 2404 (1947).

(12) H. H. Inhoffen and Huang-Minlon, *Ber.*, **71**, 1720 (1938).

(13) H. H. Inhoffen, G. Kolling, G. Koch and I. Nebel, *Ibid.*, **84**, 361 (1951).

(14) C. Djerassi and G. Rosenkranz, *Experientia*, **7**, 93 (1951).

(15) Curves for coprostan-3-one, Δ^4-cholesten-3-one, and the sample of Δ^1-coprosten-3-one prepared as described above were obtained upon a Perkin–Elmer Model 21 double beam spectrophotometer using 1-mm. cells and a concentration of 5.0 mg. sample in 0.3 ml. of carbon disulfide. We wish to thank Jonas Carol, Chemist, Food and Drug Administration, Washington 25, D. C., for these determinations.

cipated peak for an α,β-unsaturated ketone at 5.97 μ, the product exhibited some absorption at 5.84 μ, characteristic of the saturated 3-ketones such as coprostan-3-one.[16] The characteristic absorption of Δ^4-cholesten-3-one at 14.64 μ[17] and of $\Delta^{1,4}$-cholestadien-3-one at 14.25, 14.56 μ and a strong band at 11.29 μ was absent.

The isolation of VII and XI in approximately equal quantities one minute after the initiation of the NBS reaction indicated that in XIV the activation of both allylic positions (C_2 and C_5) was of the same order of magnitude. The attack at the tertiary C_5 position under the present reaction conditions was somewhat unusual in that more vigorous activation was generally believed to be required for this type of substitution by NBS.

Experimental Section[18]

Δ^2-3-Acetoxycholestene, II.—A solution of 20 g. of cholestan-3-one[19] prepared from 3β-cholestanol,[20] 40 ml. of acetyl chloride and 100 ml. of acetic anhydride was refluxed 4 hours. When cool, the solution was poured into cold 2% sodium carbonate solution, the resulting precipitate filtered and crystallized from 450 ml. of 20:10:5 methanol–ethanol–ethyl acetate and 1 ml. pyridine to obtain 20 g., m.p. 91–97°, of product. Additional crystallization from ethanol afforded a product which melted with chromophoric effects at 95–97°, $[\alpha]^{29}D$ +54.4°, 1% chloroform. Variation in the quantity of acetyl chloride, or substitution by *p*-toluenesulfonic acid resulted in decreased yield and poorer quality of product. *Anal.* Calcd. for $C_{29}H_{48}O_2$: C, 81.25; H, 11.29. Found: C, 81.38; H, 10.89. Other procedures for this preparation have yielded products of m.p. 81°[21] and 90.0–90.5°.[22] The product was ozonized in the usual manner to yield the dibasic acid, III, of m.p. 193–195°.

Δ^2-3-Acetoxycholestene Bromination with Bromine. (a) In the Presence of Sodium Acetate.—A mixture of 2.50 g. of Δ^2-3-acetoxycholestene, 0.53 g. of sodium acetate and 135 ml. acetic acid was warmed to about 35° to dissolve the solids and then cooled to 20°. To this solution 6.38 ml. of 0.915 *M* bromine in acetic acid was added. Initially the bromine decolorized rapidly but slowed as an increasing amount of the bromine solution was added. After standing 3/4 hour, the mixture was chilled and filtered to obtain 0.72 g., m.p. 164–168°, of product. Dilution of the mother liquors with water gave 1.79 g. of additional material, m.p. 150–162°. Several crystallizations of the combined fractions from ethanol–chloroform afforded 2-bromocholestan-3-one, as fine long white needles, m.p. 170°, $[\alpha]^{29}D$ +47.3°, 1% chloroform. **(B) In the Absence of Sodium Acetate.**—A mixture of 5.00 g. of Δ^2-3-acetoxycholestene and 225 ml. of acetic acid at 20° was treated with 10 ml. of 0.915 *M* bromine in acetic acid. Solution of the solid was followed by the formation of a precipitate. Fifteen minutes after addition of the bromine solution the mixture was poured into water, and the resulting solid filtered and washed. The cake was dissolved in 200 ml. of hot ethanol–chloroform, cooled and filtered to obtain 2.73 g., m.p. 163–164°, of crude 2-bromocholestan-3-one. A second recrystallization afforded a product identical with the 2-bromocholestan-3-one obtained in A.

Δ^2-3-Acetoxycholestene Bromination with N-Bromosuccinimide. General Procedure.—From a solution of 5.00 g. of Δ^2-3-acetoxycholestene and 120 ml. of carbon tetrachloride about 15 ml. of solvent was distilled. The condenser was set for reflux and 2.47 g. of NBS was added all at once using about 25 ml. of solvent to rinse remaining crystals into the reaction vessel. Following a 1 to 2 minute induction period at reflux temperature the solution turned to a brown color which remained for about 1 to 1.5 minutes and then suddenly faded. At this time hydrogen bromide was evolved from the upper outlet of the reflux condenser and the succinimide came to the surface of the solution.

This reaction was studied under varying time conditions, calculating the time from the point where the NBS dissociated. Each run was arrested at the appropriate time by cooling. About 2 ml. of pyridine was then added to neutralize the hydrogen bromide.

The reaction mixture was filtered to remove insoluble salts and the carbon tetrachloride solution was washed with 2% hydrochloric acid, and saturated sodium chloride solution. After drying the carbon tetrachloride layer over magnesium sulfate, the solvent was removed by concentration in vacuum. The residue was dissolved in 250 ml. of petroleum ether, 30–60°, adsorbed on 40 g. of alumina (according to Brockman) and eluted with like volumes of 1:1 petroleum ether–benzene, ethyl ether, methanol and acetone. The illustrative workup of an eight-minute reaction time run is summarized in Table I. In the reactions of less than three minutes duration some starting material could be recovered from the petroleum ether eluate of the chromatographic separation of the products.

Fractionation of 2,4-Dinitrophenylhydrazones.—The hydrazones were fractionated by chromatography over 25 g. of 25–200 mesh silica gel.[23] The elution with increasing quantities of ether in benzene was followed by observation of the characteristic ultraviolet spectra.[24] In this way it was possible to fractionate the 2,4-dinitrophenylhydrazone, (DNPH), of Δ^4-cholesten-3-one $\lambda_{max}^{chloroform}$ 392 mμ, and Δ^1-2-bromocholesten-2-one $\lambda_{max}^{chloroform}$ 376 mμ. The apparent yield of Δ^4-cholesten-3-one DNPH thus obtained was corrected for any of the compound that may have formed by spontaneous dehydrobromination under the conditions of DNPH formation[25] of the hypothetically present 4-bromo cholestan-3-one. The maximal quantity of this bromide was calculated from the bromine content of the crude fraction prior to DNPH formation. Because of these corrections the yield of Δ^4-cholesten-3-one plotted in Fig. 1 is the minimum quantity formed in the reaction.

Δ^3-3-Acetoxycoprostene, XIV.—The procedure was the same as that described above for the preparation of Δ^2-3-acetoxycholestene. Reaction of 14.35 g. of coprostan-3-one,[26,27] m.p. 60–61°, $[\alpha]^{25}D$ +37.5°, 0.5% in chloroform, 80 ml. each of acetic anhydride and acetyl chloride yielded 19.2 g. of oil. The oil, dissolved in 150 ml. of petroleum ether, was adsorbed on 190 g. of alumina and eluted with 1:1 petroleum ether–benzene. Upon concentration the residue could not be crystallized. $[\alpha]^{25}D$ +42.8°, 0.53% in chloroform. *Anal.* Calcd. for $C_{29}H_{48}O_2$: C, 81.25; H, 11.29. Found: C, 81.64; H, 11.37.

Since this product has been reported as a crystalline solid, m.p. 77–79°,[22] the oil obtained above was ozonized in the usual manner. The acidic fraction, m.p. 230–232°, on recrystallization from petroleum ether afforded the 3//4 diacid, m.p. 242–244°. The melting point was not depressed on admixture with an authentic sample.[28]

Δ^3-3-Acetoxycoprostene Bromination with N-Bromosuccinimide.—This bromination was conducted in the manner described for Δ^2-3-acetoxycholestene. The induction period for the reaction was shorter and the spontaneous evolution of hydrogen bromide more rapid than in the corresponding reaction with Δ^2-3-acetoxycholestene. The crude product was chromatographed as previously described and the results are summarized in Table II. Reaction runs of longer duration than one minute yielded intractable oils.

Partial Hydrogenation of $\Delta^{1,4}$-Cholestadien-3-one.[28,29]—The study of the selective reduction of $\Delta^{1,4}$-cholestadien-3-

(16) R. N. Jones, V. Z. Williams, M. S. Wahlen and K. Dobriner, THIS JOURNAL, **70**, 2024 (1948); and R. F. Furchgott, H. Rosenkrantz and E. Shorr, *J. Biol. Chem.*, **167**, 627 (1947), and earlier papers.

(17) This band does not seem to have been previously commented upon.

(18) Melting points are uncorrected.

(19) W. F. Bruce, *Org. Syntheses*, **17**, 43 (1937).

(20) E. B. Hershberg, E. Oliveto, M. Rubin, H. Staeudle and L. Kuhlen, THIS JOURNAL, **73**, 1144 (1951).

(21) L. Ruzicka and W. Fisher, U. S. Patent 2,248,438 (July 8, 1941).

(22) W. G. Dauben, R. A. Micheli and J. F. Eastham, THIS JOURNAL, **74**, 3852 (1952).

(23) Davison Chemical Corporation, Baltimore 3, Md., type 12-08-K 1926.

(24) C. Djerassi and E. Ryan, THIS JOURNAL, **71**, 1000 (1949).

(25) V. R. Mattox and E. C. Kendall, *ibid.*, **70**, 882 (1948).

(26) H. Grasshof, *Z. physiol. Chem.*, **223**, 249 (1934).

(27) H. S. Anker and K. Bloch, THIS JOURNAL, **66**, 1782 (1944).

(28) H. H. Inhoffen, G. Stoeck, G. Kolling and U. Stoeck, *Ann.*, **568**, 181 (1950).

(29) A. Butenandt, L. Mamoli, H. Dannenberg, L. Masch and J. Paland, *Ber.*, **72**, 1617 (1939).

TABLE I

EIGHT-MINUTE REACTION OF NBS AND Δ^2-3-ACETOXYCHOLESTENE CHROMATOGRAPHIC FRACTIONATION SUMMARY

Eluent	Crude wt., g.	λ_{max}^{alc} mμ	M.p., °C.	Remarks
Petroleum ether	3.34	228–230 Weak absorption		Fractionated as described in footnote[a]
1:1 Petroleum ether–benzene	1.30	230–233	99–101[b]	$[\alpha]^{25}D$ +88.2°, 1% in chloroform. M.p. not depressed on admixture with authentic sample of Δ^1-cholesten-3-one
Benzene	0.09	237–244		Converted to 2,4-DNPH-C
Ether	0.15	237–238		Converted to 2,4-DNPH-C

[a] Slurry with methanol dissolved the product responsible for the ultraviolet absorption. The methanol insoluble residue on crystallization from hexane afforded 2-bromocholestan-3-one, m.p. 169–170°. The melting point was not depressed on admixture with an authentic sample of 2-bromocholestan-3-one. The methanol solution was concentrated and the residue dissolved in petroleum ether. Filtration of the solution through 15 g. of alumina followed by concentration yielded crude Δ^1-cholesten-3-one, m.p. 94–96°, not depressed on mixture with an authentic sample of Δ^1-cholesten-3-one. [b] From methanol.

TABLE II

REACTION OF NBS WITH Δ^3-3-ACETOXYCOPROSTENE

Reaction Time, min.	Eluent	Crude wt., g.	λ_{max}^{alc} mμ	Product wt., g.	M.p., °C.	Remarks
One	20% Petroleum ether–benzene	0.698	None	0.562[a]	58–60	Coprostanone
	Benzene	0.751	Weak 230–232	An oil		Triturated with methanol to yield coprostan-3-one and an unworkable oil
	1:1 Benzene–ether	1.031	234–238	0.484[a]	84–86	Δ^1-coprosten-3-one[b]
	Ether	0.462	242	An oil		Combined and rechromatographed to obtain 20 mg. Δ^4-cholesten-(3-one,[c] m.p. 78–80°)
	Ethanol	0.658	240	An oil		
	Acetone	0.048	240–244	An oil		

[a] Recrystallized from ethanol. [b] This product $[\alpha]^{25}D$ +106.8°, 0.5% in chloroform λ_{max}^{alc} 232 mμ semicarbazone, m.p. 208–211°, λ_{max}^{alc} 272 mμ, did not depress the m.p. of the crude Δ^1-coprosten-3-one obtained by partial hydrogenation of $\Delta^{1,4}$-cholestadien-3-one (preparation below), but the mixed m.p. with Δ^1-cholesten-3-one was depressed to 85–91°. [c] The Δ^4-cholesten-3-one was obtained from the benzene eluate. Several crystallizations afforded the product whose m.p. was not depressed when mixed with authentic material.

one was facilitated by the characteristic ultraviolet response and optical rotation of the chromatographic fractions of the products. The optimal conditions found for the preparation of crude Δ^1-coprosten-3-one follow: A solution of 5.00 g. of X in 200 cc. of ethanol, 500 mg. of palladized calcium carbonate[30] and 500 mg. of quinoline absorbed 1 mole of hydrogen in two hours and 40 minutes at room temperature and 15 lb. pressure. There was no further uptake of hydrogen. Chromatography of the reaction product over alumina yielded 2.54 g. of crude Δ^1-coprosten-3-one in the 7:3 petroleum ether–benzene eluate. The analytical sample obtained by recrystallization from ethanol had a m.p. of 85–87°, $[\alpha]^{25}D$ +104.8°, 0.5% in chloroform. *Anal.* Calcd. for $C_{27}H_{44}O$: C, 84.51; H, 11.53. Found: C, 84.28; H, 11.18. The semicarbazone had the same m.p. 207°, as previously described.[28] As indicated by the infrared data and the low melting point, this material was not completely pure.

(30) Kindly supplied by Dr. F. B. LaForge.

WASHINGTON 7, D. C.