cooled overnight. The resulting precipitate was recrystallized from ethanol giving colorless crystals which melted at 213–215°, indicating formation of disulfide.

Anal. Calcd. for $C_{12}H_{16}N_6S_4$: C, 38.69; H, 4.29; N, 22.57; S, 34.43. Found: C, 39.01; H, 4.60; N, 22.47; S, 34.13.

The alkaline filtrate gave a further 0.15 g. of white precipitate after standing five days at room temperature. The precipitate was recrystallized from ethanol giving a crystalline product which was found identical with the disulfide from ethanol recrystallization of the main crop.

2-Ethylthio-4-amino-5-hydroxymethylpyrimidine (XIX) was prepared from 2-mercapto-4-amino-5-carbethoxypyrimidine *via* 2-ethylthio-4-amino-5-carbethoxypyrimidine. 2-Mercapto-4-amino-5-carbethoxypyrimidine was synthesized by condensation of ethyl ethoxymethylenecyanoacetate with thiourea by the method of Ulbricht and Price,[3] and the yield of 2-ethylthio-4-amino-5-hydroxymethylpyrimidine from ethyl ethoxymethylenecyanoacetate was 48%. This was a considerable improvement over the yields obtained by other workers.[9]

2-Mercapto-4-amino-5-carbethoxypyrimidine (10 g., 0.05 mole) was dissolved in a solution of 3.1 g. (0.055 mole) of potassium hydroxide in 50 ml. of water and 8 g. (0.052 mole) of diethyl sulfate was gradually added with shaking. After stirring 3 hr., the crystals were collected, washed and dried to yield 9.4 g. (83%) of 2-ethylthio-4-amino-5-carbethoxypyrimidine. This may be recrystallized from ethanol to give pale yellow plates, m.p. 100–102°.

This material was placed in a Soxhlet extractor mounted on a flask containing a solution of 3 g. (0.079 mole) of lithium aluminum hydride in 350 ml. of dry ether. The ether was refluxed with stirring for 3 hr. After cooling, 20 ml. of ethyl acetate was added with stirring, and then 10 ml. of water. The solid precipitate was filtered after standing overnight, and then extracted three times with 100-ml. portions of boiling acetone. The acetone solutions were combined and the solvent was distilled yielding white crystalline residue. The ether was distilled from the filtrate giving pale yellow crystalline residue which was combined with the acetone extracts. The crude products were washed with acetone and benzene leaving 7.5 g. of white crystals. After recrystallizing from ethanol, 5.9 g. (74%) of crystals which melted at 154–155.5° (lit.[8] m.p. 170° and 151–152° were obtained.

Anal. Calcd. for $C_7H_{11}N_3OS$: C, 45.38; H, 5.99; N, 22.68; S, 17.31. Found: C, 45.56; H, 6.15; N, 22.59; S, 17.49.

Infrared spectra (in potassium bromide, wavelength, and % absorption): IV; 2.98 (66), 3.15 (71), 6.06 (82), 6.29 (83), 6.43 (79), 6.76 (62), 7.02 (78), 7.33 (52), 7.78 (48), 8.12 (53), 10.00 (35), 10.30 (57), 12.64 (45), 12.89 (46).

VI; 2.90 (82), 3.22 (85), 4.61 (79), 6.00 (90), 6.29 (88), 6.38 (90), 6.75 (83), 7.01 (88), 7.80 (75), 8.13 (80), 9.63 (63), 10.35 (72), 11.38 (58), 12.65 (78), 12.97 (84), 13.27 (77), 14.50 (60).

VII; 2.90 (39), 3.34 (53), 6.57 (72), 7.04 (78), 7.31 (58), 7.60 (64), 8.33 (34), 8.70 (36), 8.82 (34), 10.41 (36), 12.27 (39), 12.47 (44), 12.85 (41), 14.54 (29).

PHILADELPHIA 4, Pa.

(9) A. Dornow and G. Petsch, *Ann.*, **588**, 45 (1954); A. Dornow and G. Petsch, German Patent **870,260** (1953); *Chem. Abstr.*, **48**, 2123 (1954); C. S. Miller, *J. Am. Chem. Soc.*, **77**, 752 (1955).

[CONTRIBUTION FROM THE EASTERN REGIONAL RESEARCH LABORATORY[1]]

Steroidal Sapogenins. XLIX. C-Ring Oxygenated Derivatives of Correllogenin[2]

EDWARD S. ROTHMAN AND MONROE E. WALL

Received May 19, 1958

In the course of conversion of natural mixtures of the 25D and 25L isomeric sapogenins gentrogenin (25D) and correllogenin (25L) to 11-keto diosgenin and 11-keto yamogenin, it was found possible to separate and characterize several intermediates which were sterically pure 25L compounds, *viz.* 3β,12β-dihydroxy-20α,22β,25L-spirost-5-en-11-one, Ia, and its diacetate, Ib; 20α,22β,25L-spirost-5-en-3β,11α-diol, IIa, and its diacetate, IIb. These new correllogenin derivatives have previously evaded isolation because of the difficulties in separating the pure parent compound. It was found that 3β,12β-diacetoxy-20α,22β,25D-spirost-5-en-11-one on treatment with calcium in liquid ammonia solution gave, in the presence of water, a high yield of 20α,22β,25D-spirost-5-en-3β,11α-diol (11α-hydroxy diosgenin).

In earlier papers of this series[3,4] we have described the isolation of gentrogenin[5] (3β-hydroxy-20α,22β,25D-spirost-5-en-12-one[5]), its 25L-diastereoisomer, correllogenin,[3] and the conversion of the former to 11-keto diosgenin.[4] In the latter paper were described the properties and reactions of a number of C-11 and C-12 oxygenated derivatives of gentrogenin. Because of the unavailability of pure correllogenin,[6] we were unable to prepare the corresponding 25L- derivatives. During the large scale conversion of a gentrogenin-correllogenin mixture to 3β-hydroxy-5,16-pregnadiene-11,20-dione,[7] we were able to isolate and characterize

(1) Eastern Utilization Research and Development Division, Agricultural Research Service, United States Department of Agriculture.

(2) Paper XLVIII, E. S. Rothman and M. E. Wall, submitted to *J. Am. Chem. Soc.*

(3) H. A. Walens, S. Serota, and M. E. Wall, *J. Org. Chem.*, **22**, 182 (1957).

(4) E. S. Rothman and M. E. Wall, *J. Am. Chem. Soc.*, **79**, 3228 (1957).

(5) For basis of formal nomenclature, particularly at C_{22}, *cf. Tentative Rules for Steroid Nomenclature*, Comptes Rendus de la Dix-Huitieme Conference, Zurich, 20–28 July, 1955, pp. 190–198.

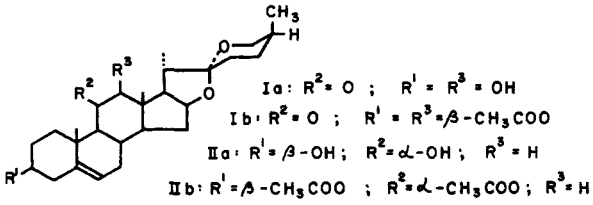
(6) Although correllogenin may constitute twenty per cent of the total ketonic fraction isolated from *D. spiculiflora*, it is difficult to separate this sapogenin from the isomeric gentrogenin[5] and consequently only minute quantities of the sterically pure 25L- form have ever been obtained.

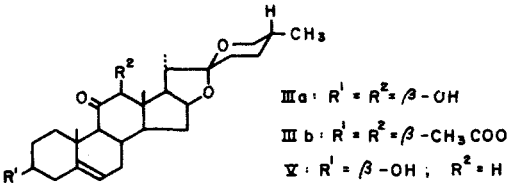
(7) Since pseudomerization followed by oxidative cleavage and alkaline hydrolysis converts the 25D- and 25L-sapogenins to the same 16-dehydro-20-keto-pregnene it is often convenient to work directly with the mixture.

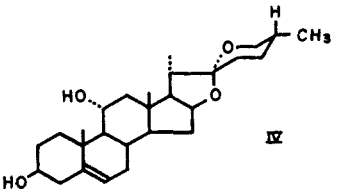
several sterically pure correllogenin derivatives. In this paper we wish to record the properties of these compounds as well as those of the corresponding 25D- series.

In the manner described previously,[4] bromination of a gentrogenin-correllogenin mixture gave the 5α,6β,11α,23-tetrabromo sapogenins which, on treatment with sodium iodide, gave the 11α,23-dibromo sapogenins of the 25D- and 25L- mixture. Alkaline equilibration in aqueous tertiary butyl alcohol[8] gave a mixture of the isomeric 25D- and 25L- derivatives of 3β,12β-diacetoxy-20α, 22β-spirosten-5-en-11-one. In experiments in the hecogenin series,[8] the English workers found that alkaline equilibration of 11α,23-dibromohecogenin acetate in methanol or ethanol gave, in addition to the desired 12β-hydroxy-11-one, some of the undesirable 11-hydroxy-12-ones. In contrast, similar equilibration in hot aqueous *t*-butyl alcohol gave only the desired 12β-hydroxy-11-one. We also[4] had previously established the absence of 12 ketone in the equilibrium mixture (by testing with Girard's reagent) when the hydrolysis step was carried out in the case of the sterically pure, brominated gentrogenin derivative. We then saponified and equilibrated a mixture of brominated sapogenins derived from a natural gentrogenin-correllogenin mixture in the tertiary-butanol system and, reasoning by analogy, felt that such treatment would lead to the desired 12β-hydroxy-11-one. After a series of crystallizations and treatment with carbon (*cf.* Experimental section) a residual fraction was saponified, resulting in the isolation of a small quantity of the very insoluble, sterically pure 3β,12β-dihydroxy-20α,22β,25L-spirost-5-en-11-one (Ia). Acetylation in hot acetic anhydride–pyridine gave 3β,12β-diacetoxy-20α,22β,25L-spirost-5-en-11-one (Ib). Because the yield of Ia was less than one per cent of the total starting material it was necessary to prove its structure. The infrared spectra of Ia and Ib showed the typical "normal" (25L) series of bands in the 800–1000 cm.$^{-1}$ region.[9] The spectrum of Ib showed the same acetate band shift from 1735 cm.$^{-1}$ to 1750 cm.$^{-1}$ observed in 11,12-ketol derivatives in the hecogenin series.[8] The carbon and hydrogen analysis of Ib confirmed the tentative structural assignments. Compound Ia did not react with Girard's reagent T eliminating from consideration ketols with the 11-hydroxy-12-one moiety. That the structure of Ia and Ib was indeed that of a 12β-hydroxy-11-one and the corresponding acetate, respectively, was shown in a clear-cut manner by the molecular rotation contribution of the 12-acetoxy group. It was shown in the hecogenin series that the Δ-M_D for a 12β-acetoxy-11-one was −237° and for the corresponding 12α-acetoxy-11-one, +212.[8] The corresponding value for Ib was −226° ± 50.[10] Hence the structure of Ia and Ib is indeed in accord with our predictions based on analogy.

The remaining crystalline fractions (after removal of sterically pure Ia) were combined, reacetylated, and reduced with calcium-ammonia[11] as described previously.[4] Chromatography of the saponified product on Florisil[12] gave first the expected mixture of 11-keto diosgenin and 11-keto yamogenin. A more tenaciously adsorbed fraction was then eluted from which, after reacetylation followed by several recrystallizations, we obtained an appreciable quantity of 3β,11α-diacetoxy-20α,22β,25L-spirost-5-ene (IIb). Saponification gave 20α,22β,25L-spirost-5-ene-3β,11α-diol (IIa).







The structure proof of IIa and IIb is based on the following considerations. The infrared spectrum of IIa shows the typical "normal" (25L) sapogenin fingerprint bands[9] and absence of carbonyl. Carbon and hydrogen analyses of IIa and IIb are in

(8) J. Elks, G. H. Phillipps, T. Walker, and L. J. Wyman, *J. Chem. Soc.*, 4330 (1956).

(9) M. E. Wall, C. R. Eddy, M. L. McClennan, and M. E. Klumpp, *Anal. Chem.*, 24, 1337 (1952).

(10) In order to calculate the MD contribution of the 12 acetoxy group in Ib it was necessary that the MD of the corresponding 12-desoxy steroid, 11-keto yamogenin acetate be known. Since the latter has not been prepared it was necessary to assign a hypothetical value based on the following known specific rotations:

12-keto diosgenin acetate (25D) = −56°
12-keto yamogenin acetate (25L) = −60°
11-keto diosgenin acetate (25D) = −86°
hence 11-keto yamogenin acetate (25L) = −90° ± 10°

The ±10° factor is well within the differences between all known pairs of 25D and 25L diastereoisomers.

(11) J. H. Chapman, J. Elks, G. H. Phillips, and L. J. Wyman, *J. Chem. Soc.*, 4344 (1956).

(12) Mention of commercial products does not imply endorsement by the U. S. Department of Agriculture over similar products not mentioned.

accord with the formulation of a dihydroxy or diacetoxy-sapogenin, respectively. From the method of preparation the compounds must have a 3β,11-dihydroxy function. Since IIb is a diacetate produced under mild acetylation conditions IIa and IIb must have, respectively, the 3β,11α-dihydroxy and 3β,11α-diacetoxy functions. The hitherto unknown 3β,12β-dihydroxy 20α,22β,25D-spirost-5-en-11-one (IIIa) was prepared by saponification of the known[4] 3β,12β-diacetate (IIIb).

Previously,[5] we had prepared 20α,22β,25D-spirost-5-ene-3β,11α-diol (IV) in low yield by reduction of 3β,12β-diacetoxy-20α,22β,25D-spirost-5-en-11-one (IIIb) with calcium-anhydrous liquid ammonia, the chief product being the corresponding 11-ketone (V). Reduction of IIIb with calcium–liquid ammonia in the presence of water gave IV as the sole product in good yield.

The reductive deacetoxylation of a non-olefinic 12β-acetoxy-11-ketone with calcium in ammonia *to the point of disappearance* of the blue calcium solution color *followed by* the addition of methanol was reported by Chapman, Elks, Phillipps, and Wyman.[11] Their product was 11α-hydroxy tigogenin. The behavior of a Δ5-3β-hydroxy system toward such a reactive reagent is not predictable. Birch[13] has noted that the presence of protonic substances, for example water, profoundly affects the course of such reductions. In such systems benzene forms 1,4-dihydrobenzene and allyl alcohols are converted to hydrocarbons. While Δ5-3β-ols are not true allyl alcohols, they have many properties that suggest a close interrelationship between the olefin and alcohol groups. For example, 3,5-cyclo systems form under appropriate conditions. When we reduced compound IIIb with blue calcium–liquid ammonia–water systems we did not observe any attack on the Δ5-3β-hydroxy system and obtained compound IV directly in high yield.

EXPERIMENTAL

3β,12β-Dihydroxy-20α,22β,25L-spirost-5-en-11-one (Ia). The isolation of Ia was fortuitous. We had been working up mother liquors from a debromination experiment leading to 160 g. of the isomeric mixture of 3β,12β-diacetoxy-20α,22β-spirost-5-en-11-one compounds.[4] The first crop from 300 ml. of ethanol was 69 g. of 25D and 25L mixed product. The filtrate was diluted with 300 ml. of benzene and then with 1700 ml. of petroleum ether, b.p. 89–98°. The clear supernatant liquor was decanted from 64 g. of a tarry residue (which later yielded an additional 21 g. of crystalline mixed product) and the solution was boiled to expel benzene. The cooled filtrate was treated with 100 g. of Darco G-60 carbon which was well washed with petroleum ether. These washings were concentrated to a small volume whereupon 8.2 g. of crystalline mixed isomers separated. Further washing of the carbon with methylene chloride and with ethanol gave, after evaporation, 17 g. of a glassy residue. Saponification of the residue gave 1.03 g. of a very insoluble crystallizate, m.p. 244–247°, insoluble in methylene chloride and in ethanol but soluble in a 1:1 mixture of these solvents. The product recrystallized from this mixture in hexagonal prisms, $[\alpha]_D^{25}$ −112°, and melted from 247 to 248° with characteristic transition to smaller, bladed forms only above 240°. The infrared spectrum (KBr disk) showed a strong hydroxyl band envelope at 3400 to 3500 cm.$^{-1}$, ketone at 1716 cm.$^{-1}$ and sapogenin bands at 846, 900, 917, and 985 cm.$^{-1}$ of the type characteristic of 25L sapogenins. The fingerprint spectrum was highly complex showing forty-five well-defined bands. In chloroform solution the ketone band occurred at 1707 cm.$^{-1}$

3β,12β-Diacetoxy-20α,22β,25L-spirost-5-en-11-one (Ib). The dihydroxy ketone of the above preparation was acetylated by refluxing for 2 hr. in 1:1 acetic anhydride–pyridine mixture (forcing conditions). After cooling and diluting with water, the steroid was collected by filtration and dried. Its solution in methylene chloride was freed of brownish coloration by passing through a pad of Florisil.[12] After evaporation to dryness the colorless residue was crystallized from ethanol to give felted microneedles, $[\alpha]_D^{25}$ −120°, m.p. 194.3–196.8°, after slight sweating at 178°.

Anal. Calcd. for $C_{31}H_{44}O_7$: C, 70.43; H, 8.39. Found: C, 70.48; H, 8.66.

3β,11α-Diacetoxy-20α,22β,25L-spirost-5-en (IIb). Ninety grams of a C-25 diastereoisomeric mixture of 3β,12β-diacetoxy-20α,22β-spirost-5-en-11-one was deacetoxylated at C-12 by reduction with calcium in liquid ammonia.[4] Chromatography of the saponified reduction product on Florisil[12] gave, on elution with 20% chloroform in benzene, the expected mixture of 11-keto diosgenin and 11-keto yamogenin. Further elution with chloroform gave a dihydroxy fraction only partly soluble in benzene. The benzene suspension was filtered and the filtrate, which was richer in the 25L-component, was evaporated to dryness. The residue was crystallized from ether and acetylated to give 10 g. of the nearly sterically pure isomer 3β,11α-diacetoxy-20α,22β,25L-spirost-5-ene (IIb). A single recrystallization from methanol gave a sterically pure sample. Five recrystallizations from methanol did not alter the melting point, *viz.* 190–191.5°, $[\alpha]_D^{25}$ −123°, feathery, felted microneedles.

Anal. Calcd. for $C_{31}H_{46}O_6$: C, 72.34; H, 9.01. Found: C, 72.62; H, 9.07.

20α,22β,25L-Spirost-5-en-3β,11α-diol (IIa). A sample of the diacetate of the preceding preparation was saponified in methanolic 5% potassium hydroxide. Crystallization from methylene chloride–hexane gave hexagonal prisms with pyramidal caps $[\alpha]_D^{25}$ −123°, m.p. 247.2–248.2° after transition over 243° to wedges.

Anal. Calcd. for $C_{27}H_{42}O_4$: C, 75.31; H, 9.83. Found: C, 75.05; H, 9.91.

3β,12β-Dihydroxy-20α,22β,25D-spirost-5-en-11-one (IIIa). A sample of the diacetate[4] was saponified by refluxing in methanolic 5% potassium hydroxide for 4 hr. The mixture was poured into water and the steroid collected. The dried product was dissolved in ether, diluted with hexane, and the solution was freed of ether by volume reduction to yield a crystalline residue which was further recrystallized from acetone to give short, hexagonal microprisms, $[\alpha]_D^{25}$ −95°, melting from 236–240° and giving a pink melt. Incomplete transition to branched filaments was observed beyond 223°.

Reduction of IIIb to 20α,22β,25D-spirost-5-en-3β,11α-diol (11α-hydroxydiosgenin) (IV). A sample of 3β,12β-diacetoxy-20α,22β,25D-spirost-5-en-11-one, 85 g., in 900 ml. of toluene was added to a solution of 67 g. of calcium metal in 4 liters of liquid ammonia during an addition time of 20 min. The mixture was mechanically stirred during the addition and during the subsequent reaction time of 10 min. Water was added cautiously in a thin stream until the blue color of the reaction mixture was discharged, an excess of water doing no harm. The mixture was evaporated in an open vessel to a white solid residue. This residue was shaken with ether and dilute aqueous hydrochloric acid until all solids were in solution. The organic layer was separated, washed with water and with saturated saline solution, and evaporated to dryness. To insure complete saponification of the 3-acetate it

(13) A. J. Birch, *Quart. Revs. (London)*, **4**, 69 (1950).

was occasionally necessary to carry out a saponification step with 5% methanolic caustic. The product, m.p. 228–233°, was very soluble in hexane and ether but gave thick, hexagonal prism-like forms on recrystallization. The analytical sample thus obtained melted from 233–235° after undergoing crystal transition above 228° to whips, $[\alpha]_D^{25}$ −116° ($CHCl_3$). This compound is 11α hydroxy diosgenin.

Acknowledgments. The authors wish to thank S. Serota for optical rotation measurements, C. R. Eddy and C. Leander for infrared spectra, and C. L. Ogg and associates for semimicroanalyses.

PHILADELPHIA 18, PA.

[CONTRIBUTION FROM THE RESEARCH LABORATORIES OF SYNTEX, S.A.]

Steroids. XCVIII.[1] Synthesis of Some 10β-Hydroxy-19-norsteroids

J. PEREZ RUELAS, J. IRIARTE, F. KINCL, AND CARL DJERASSI

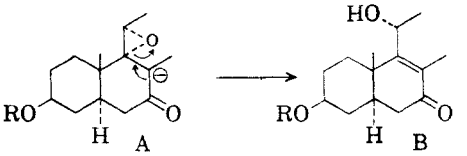
Received May 29, 1958

The direction and stereochemistry of the acid- and base-catalyzed opening of 5,10-epoxides of certain 19-norsteroids is discussed and the synthesis of several 10β-hydroxy-19-norsteroids is reported.

The removal of the angular methyl group at C-10 of certain steroids such as progesterone[2,3] or 17α-ethinyltestosterone[4] has led to a marked increase in biological activity. This is particularly noteworthy in the latter compound, 19-nor-17α-ethinyltestosterone (Ib)[4] whose high hormonal activity[1,4] by the oral route has led to the introduction of this compound (Norlutin) into medical practice. It was felt that it might be of interest to examine the effect of other angular substituents upon biological potency and the present paper is concerned with certain 10β-hydroxy-19-norsteroids.

Pederson and collaborators[5] reported recently that microbiological hydroxylation of 19-nortestosterone (Ia)[6] led in poor yield to a 10-hydroxy derivative, whose structure was confirmed by osmium tetroxide hydroxylation[7] of the β,γ-unsaturated precursor IIa[8] of 19-nortestosterone (Ia) followed by dehydration of the intermediate glycol. The stereochemistry of the introduced 10-hydroxyl group was not established by the Upjohn group[5] but conclusive evidence in favor of the 10β-orientation could be provided[9] by noting the coincidence of the rotatory dispersion curve of 10-hydroxy-19-nortestosterone (IVa) with that[10] of 19-nortestosterone (Ia), where the 10β-orientation is established. If the hydroxylation product had been the 10α-isomer VIa, then the rotatory dispersion curve would have been of an antipodal type.[11] Consequently, 10β-hydroxy-19-nortestosterone (IVa) can now be employed as the key reference compound for stereochemical considerations in this series.

Since we were interested in preparing 10β-hydroxy-19-norsteroids which might also bear substituents at C-5 (*vide infra*), the most attractive synthesis of 10β-hydroxy-19-norsteroids might well proceed *via* the 5,10-epoxide (*e.g.*, III) of a 5,10-unsaturated 19-nor-3-ketosteroid (II). In fact, earlier work from this laboratory[12] had demonstrated the facile conversion of the epoxy ketone A by alkaline treatment to the unsaturated hydroxy ketone B and the structural situation should be completely analogous in a 5,10-epoxy-3-ketone (III). Nevertheless, there exists a patent claim[13] that epoxidation of IIa leads to a sharp-melting epoxide (IIIa or Va) which upon exposure to alkali furnishes both C-10 epimeric hydroxy-19-nortestosterones (IVa and VIa). The mechanistic unlikeliness of such a reaction—assuming the epoxide to be homogeneous[14]—prompted us to re-examine the epoxidation of IIa and to establish



(1) Paper XCVII, D. A. McGinty and C. Djerassi, *Ann. N. Y. Acad. Sci.*, **71**, 500 (1958).

(2) C. Djerassi, L. Miramontes, and G. Rosenkranz, *J. Am. Chem. Soc.*, **75**, 4440 (1953).

(3) G. W. Barber and M. Ehrenstein, *Ann.*, **603**, 89 (1957).

(4) C. Djerassi, L. Miramontes, G. Rosenkranz, and F. Sondheimer, *J. Am. Chem. Soc.*, **76**, 4092 (1954).

(5) R. L. Pederson, J. A. Campbell, J. C. Babcock, S. H. Eppstein, H. C. Murray, A. Weintraub, R. C. Meeks, P. D. Meister, L. M. Reineke, and D. H. Peterson, *J. Am. Chem. Soc.*, **78**, 1512 (1956).

(6) A. J. Birch, *J. Chem. Soc.*, 367 (1950); A. L. Wilds and N. A. Nelson, *J. Am. Chem. Soc.*, **75**, 5366 (1953); J. A. Hartman, A. J. Tomasewski, and A. S. Dreiding, *J. Am. Chem. Soc.*, **78**, 5662 (1956).

(7) R. L. Pederson and J. C. Babcock, U.S. Patent **2,806,862**.

(8) A. J. Birch and S. M. Mukherji, *J. Chem. Soc.*, 2531 (1949).

(9) C. Djerassi, R. Riniker, and B. Riniker, *J. Am. Chem. Soc.*, **78**, 6377 (1956).

(10) C. Djerassi, R. Riniker, and B. Riniker, *J. Am. Chem. Soc.*, **78**, 6362 (1956).

(11) See C. Djerassi, M. Ehrenstein, and G. W. Barber, *Ann.*, **612**, 93 (1958).

(12) C. Djerassi, O. Mancera, J. Romo, and G. Rosenkranz, *J. Am. Chem. Soc.*, **75**, 3505 (1953).

(13) F. B. Colton, U.S. Patent **2,729,654**.

(14) The physical constants of this epoxide are in reasonable agreement with those found in our laboratory for a homogeneous specimen.