

micro sintered glass crucible containing a thin layer of asbestos. After washing well with water, alcohol and ether, the crucible was dried and weighed on a semi-micro balance. The nitric acid must not be added until the compound is well charred or polynitro compounds resisting digestion will be formed. The precision of this method of analysis is $\pm 0.0\%$.

Summary

1. Three new *bis*-(4-hydroxyalkyloxyphenyl)-selenium dichlorides and their corresponding dihydroxides were prepared by the condensation of selenium oxychloride with mixed ethers.

2. The dinitro derivatives of the above compounds and the corresponding methoxy compound were obtained. These compounds were reduced catalytically to the *bis*-(3-amino-4-R-phenyl) selenide.

3. The condensation of selenium oxychloride with benzene derivatives was extended to acetanilide, resulting in *bis*-(4-acetaminophenyl)-selenium dihydroxide. On reduction, *bis*-(4-acetaminophenyl) selenide was formed. The corresponding amine also was prepared.

LINCOLN, NEBRASKA

RECEIVED MAY 8, 1939

[CONTRIBUTION FROM THE FRICK CHEMICAL LABORATORY, PRINCETON UNIVERSITY]

Studies in the Sitosterol Complex. The Structure of α_1 -Sitosterol

BY SEYMOUR BERNSTEIN¹ AND EVERETT S. WALLIS

In their experiments on α -sitosterol, Wallis and Fernholz² showed that this material first described as a compound by Anderson and his co-workers³ was in reality a mixture of at least two new sterols, α_1 - and α_2 -sitosterol.

Besides recording the physical properties of these two new compounds and their derivatives, these investigators² made the following observations. Neither α_1 - nor α_2 -sitosterol are isomers of β - and γ -sitosterol. α_1 -Sitosterol is an isomer of stigmaterol, $C_{29}H_{48}O$, and α_2 -sitosterol is in all probability a homolog, $C_{30}H_{50}O$. By titration with perbenzoic acid they showed that two double bonds are present in both sterols. They give the same Liebermann color reaction. The final color is a dark blue with a reddish tint. The Salkowski reaction for both α_1 - and α_2 -sitosterol was found to be similar to that of ergosterol, the sulfuric acid layer becomes colored, while the chloroform stays colorless. The opposite is true for cholesterol, γ -sitosterol and stigmaterol. They also noted that both α_1 - and α_2 -sitosterol are precipitated by digitonin.

In this paper we wish to report the results of further experiments on α_1 -sitosterol which to us seem pertinent to the elucidation of its structure. That the two double bonds in α_1 -sitosterol are not conjugated was shown by an absorption spectrum

study. No maxima were observed. It is also to be noted that α_1 -sitosterol does not form an addition compound with maleic anhydride. Taken together these two facts indicate that this sterol cannot have its double bonds conjugated within one ring or between two adjacent rings.

The results which we obtained from a hydrogenation study on α_1 -sitosterol are of special interest. From preliminary experiments we learned that only one of the two double bonds can be hydrogenated under ordinary conditions. If the hydrogenation, however, be carried out at a higher temperature (65 – 70°) in the presence of a small amount of concentrated hydrochloric acid, complete saturation is possible. It was further observed that the double bond originally resistant to hydrogenation can be isomerized by dry hydrogen chloride into a position which is easy to hydrogenate under ordinary conditions.

With the above facts in mind, α_1 -sitosteryl acetate (I) was hydrogenated with a platinum catalyst at room temperature and atmospheric pressure. As a result, α_1 -dihydrositosteryl acetate (II) was obtained. This acetate was then isomerized in a chloroform solution with dry hydrogen chloride at 0° . A new acetate, α_1 -isodihydrositosteryl acetate (III), was isolated and characterized. On hydrogenation at room temperature and atmospheric pressure this compound gave a completely saturated acetate, α_1 -sitostanol acetate (IV). This same substance was also obtained when α_1 -sitosteryl acetate was hydrogen-

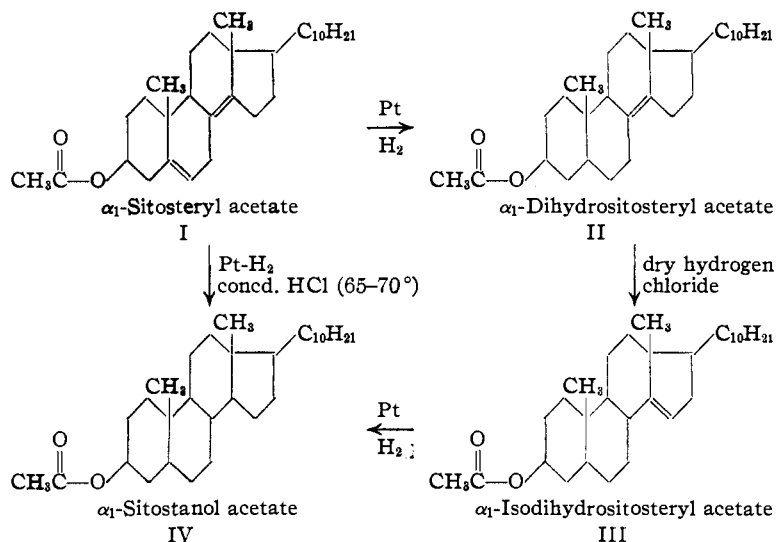
(1) Research Assistant on Special Funds from the Rockefeller Foundation.

(2) Wallis and Fernholz, *THIS JOURNAL*, **58**, 2446 (1936).

(3) Anderson, Shriner and Burr, *ibid.*, **48**, 2987 (1926); see also Anderson, *ibid.*, **46**, 1450 (1924).

ated with a platinum catalyst at 65–70°, and at atmospheric pressure in the presence of a small amount of concentrated hydrochloric acid.

These results may be explained as follows



In Table I there are also listed the properties of α_1 -sitosterol and its various hydrogenation products.

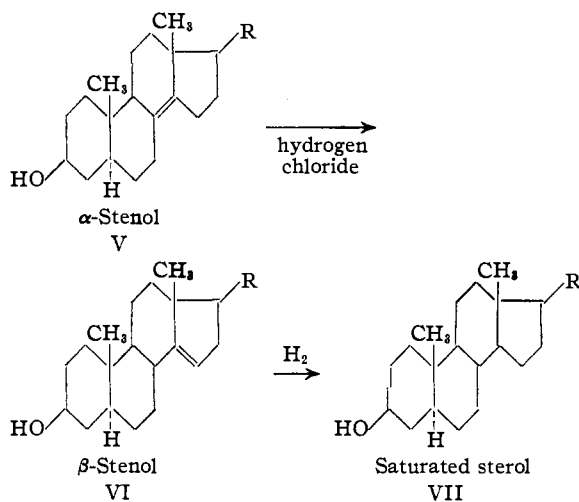
TABLE I

Compound	M. p., °C.	$[\alpha]_D(\text{CHCl}_3)$
α_1 -Sitosteryl acetate	137	+29
α_1 -Sitosterol	164–166	– 1.7
α_1 -Dihydrositosteryl acetate	108.5–110.5	+35.1
α_1 -Dihydrositosterol	152–154.5	+10.9
α_1 -Isodihydrositosteryl acetate	137.5–138.5	+42.0
α_1 -Isodihydrositosterol	152–154	+31.0
α_1 -Sitostanol acetate	147–148	+39.4
α_1 -Sitostanol	185–186	+27.0

The structures which we have proposed for this sterol and its hydrogenation products may be supported by the following arguments. All natural sterols whose structures are known have been found to have an hydroxyl at the C₃-position. That the hydroxyl group in α_1 -sitosterol is *cis* to the C₁₀ methyl group is supported by the formation of an insoluble digitonide, a characteristic property of all sterols of this type. The proposed non-conjugation of the double bonds in α_1 -sitosterol is supported by the negative Diels–Alder reaction and by the results of the absorption spectrum study. All natural sterols which are unsaturated have been found to have one double bond at the 5,6-position. It is also to be recalled that a $\Delta^{5,6}$ -sterol is hydrogenated easily. One

double bond in α_1 -sitosterol is hydrogenated easily under ordinary conditions.

The fact that the second double bond in α_1 -sitosterol is resistant to hydrogenation but can be isomerized into a position in which it can be hydrogenated easily is a phenomenon not unknown in sterol chemistry. The naturally non-occurring sterols α -ergosterol⁴ (V) and α -cholestenol⁵ (V) have been found to be resistant to hydrogenation under ordinary conditions but can be isomerized with dry hydrogen chloride to give β -ergosterol (VI) and β -cholestenol (VI), respectively. These products then can be hydrogenated easily to give the corresponding saturated compounds (VII). The fact that the cholestanol obtained from α -cholestenol by this method is identical

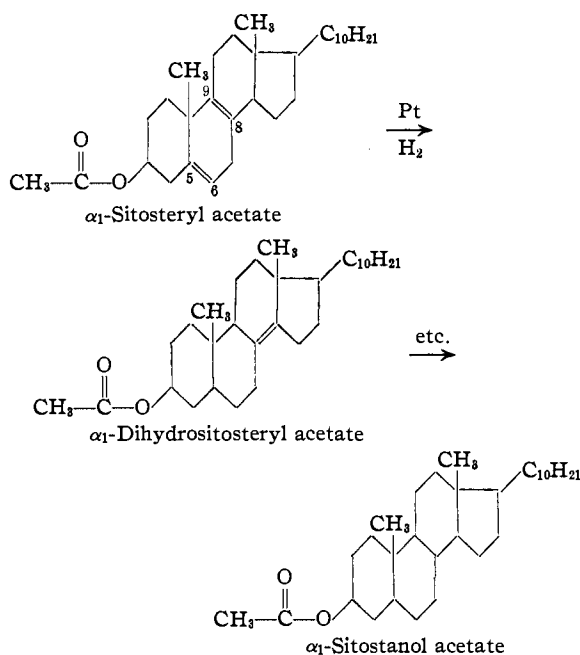


with the cholestanol obtained by hydrogenation of cholesterol shows that rearrangement with hydrogen chloride and subsequent hydrogenation do not produce epimerization at any asymmetric center. Therefore it may be argued that rearrangement of α_1 -dihydrositosterol and subsequent hydrogenation, likewise, does not produce any epimerization. If such be the case, it therefore follows that α_1 -sitostanol and stigmasterol differ solely due to isomerism of the hydrocarbon side chain.

(4) Reindel, Walter and Rauch, *Ann.*, **452**, 34 (1927); Reindel and Walter, *ibid.*, **460**, 212 (1928); Achtermann, *Z. physiol. Chem.*, **226**, 141 (1934); Laucht, *ibid.*, **237**, 236 (1935).

(5) (a) Schenck, Buchholz and Wiese, *Ber.*, **69B**, 2696 (1936). (b) Windaus, Linsert and Eckhardt, *Ann.*, **543**, 22 (1938).

In this connection it should be recalled that a δ -stenol, such as δ -cholestenol, also has been found to be resistant to hydrogenation, and that during the course of attempted hydrogenation with palladium it is converted into the α -stenol^{5b} modification. Hence the possibility presents itself that α_1 -sitosterol may be in reality unsaturated at the 5,6- and 8,9-positions. The course of hydrogenation would then become



Against this formulation may be cited our observation that in our hands α_1 -sitosteryl acetate does not give a ketone at C_7 on oxidation with chromic acid, as would be expected if its double bonds were located at the 5,6- and 8,9-positions.

TABLE II

Unsaturated at $\Delta^{5,6}$	$[\alpha]_D^{25}$ (CHCl_3)	Saturated at $\Delta^{5,6}$	$[\alpha]_D^{25}$ (CHCl_3)
Stigmasterol	-45°	Stigmastanol	+23.8
β -Sitosterol	-37°		
α_1 -Sitosterol	-1.7°	α_1 -Sitostanol	+27.0

TABLE III

α -Stenol	$[\alpha]_D^{25}$ (CHCl_3)	β -Stenol	$[\alpha]_D^{25}$ (CHCl_3)	Satd. sterol	$[\alpha]_D^{25}$ (CHCl_3)
α -Ergosterol	+13.1	β -Ergosterol	+20.3	Ergostanol	+15.5
α -Ergostene	+11.0	β -Ergostene	+21.3	Ergostane	+19.0
α -Cholestenol	+20.7	β -Cholestenol	+34.0	Cholestanol	+29.7
α_1 -Dihydrositosterol	+10.9	α_1 -Isodihydrositosterol	+31.0	α_1 -Sitostanol	+27.0

Our assignment of the positions of the double bonds in α_1 -sitosterol and its intermediate hydrogenation products may be strengthened further by considering the relation of optical rotatory power

and constitution.⁶ It has been observed, qualitatively, that a compound unsaturated at the 5,6-position always will have a levo or a lower dextro rotation than the corresponding saturated compound which will always have an appreciable dextro-rotation. α_1 -Sitosterol fits very well into this observation, as may be seen in Table II.

That α_1 -dihydrositosterol and α_1 -isodihydrositosterol are α - and β -stenols, respectively, is likewise indicated by a qualitative interpretation of the optical activity of these compounds. An α -stenol, in general, will have a positive rotation lower in degree than the corresponding saturated compound. A β -stenol will, in general, have a positive rotation, higher in degree than the corresponding saturated compound. These two statements are borne out by Table III.

Further evidence for the proposed structure of α_1 -isodihydrositosteryl acetate (III) (which is represented as a β -stenol) has also been obtained. We have found that if α_1 -isodihydrositosteryl acetate be treated with perbenzoic acid, an oxide (VIII) is obtained. Hydrolysis of the oxide ring with glacial acetic acid and concentrated sulfuric acid gives a doubly unsaturated compound (IX), and not a triol diacetate (X).

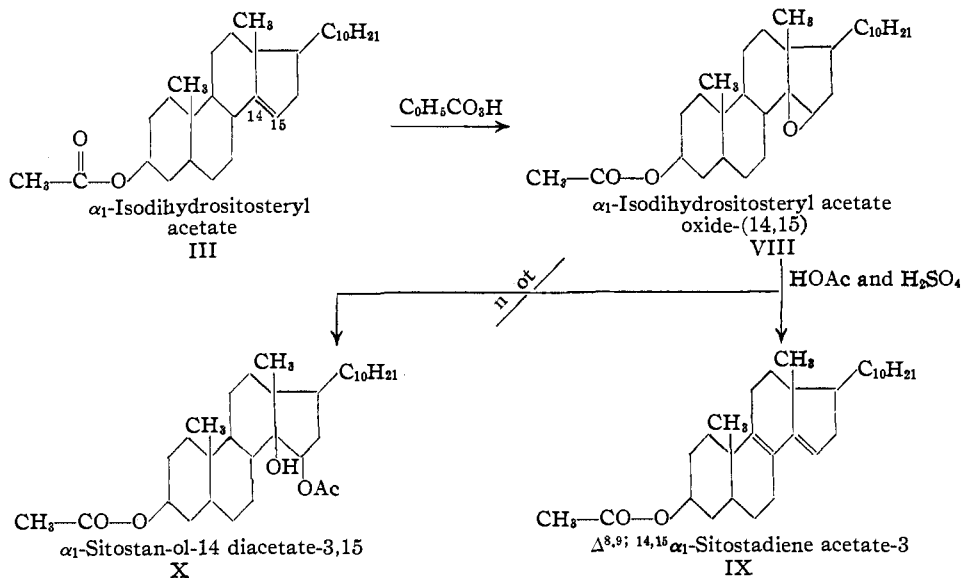
The formation of this doubly unsaturated compound from the oxide is in accordance with the experimental observations of other investigators on similar compounds. For example, Morrison and Simpson⁷ have found that when β -ergosterol oxide (XI) is dissolved in hot ethyl alcohol and treated with dilute sulfuric acid with subsequent heating for fifteen minutes on the steam-bath, a doubly unsaturated sterol, dehydroergosterol (XII), is produced. They⁸ also have reported that α -ergosterol oxide (XIII) under similar treatment gives the same dehydroergosterol. Since Callow⁹ has shown experimentally that the most probable formula for this latter doubly un-

(6) Callow and Young, *Proc. Roy. Soc. (London)*, **A157**, 194 (1936).

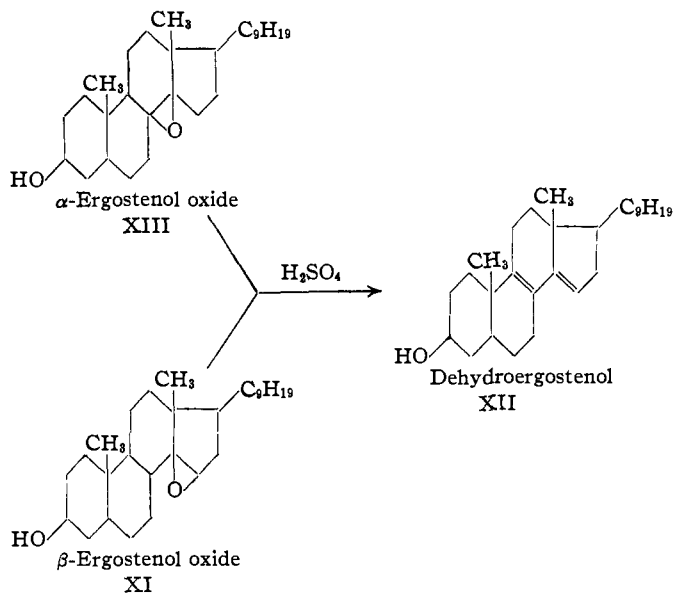
(7) Morrison and Simpson, *J. Chem. Soc.*, 1710 (1932).

(8) Ref. 7; see also Windaus and Luttringhaus, *Ann.*, **481**, 119 (1930).

(9) Callow, *J. Chem. Soc.*, 462 (1936).



saturated sterol is as indicated below, it follows that the doubly unsaturated compound obtained by us from α_1 -isodihydrositosteryl oxide is to be represented as given and therefore is, accordingly, $\Delta^{8,9; 14,15}$ α_1 -sitostadiene acetate-3.



Experimental Part

Preparation of α_1 -Sitosteryl Acetate.—Two and one-half grams of α_1 -sitosterol (m. p. 159–166°) isolated from the sitosterol complex (1627 g.) of wheat germ oil by the method of Wallis and Fernholz² was heated on the water-bath for one hour with acetic anhydride. Crystallization from methyl alcohol gave 2 g. of α_1 -sitosteryl acetate, m. p. 136–137°.

The Reaction of α_1 -Sitosteryl Acetate with Maleic Anhydride (Diels–Alder Reaction).—A mixture of 50 mg. of

α_1 -sitosteryl acetate, 20 mg. of maleic anhydride and 10 cc. of acetic anhydride was refluxed for eight hours. During this time a brown coloration appeared. The reaction mixture was evaporated to dryness *in vacuo*. The residue was crystallized from ethyl alcohol and gave a material melting at 137–138°. A mixed melting point determination with α_1 -sitosteryl acetate (136–137°) indicated identity since no depression was observed.

The Diels–Alder reaction was repeated, with xylene as the solvent. The starting material was again recovered and no indication of a positive addition reaction was observed.

An absorption spectrum study of α_1 -sitosterol (m. p. 166°) was carried out by Dr. T. J. Webb of the Research Laboratories, Merck and Company, Inc. It was found not to have any maxima in the ultraviolet region of the spectrum.

Hydrogenation of α_1 -Sitosteryl Acetate at 60° and Atmospheric Pressure.—One-tenth of a gram of α_1 -sitosteryl acetate dissolved in glacial acetic acid was hydrogenated for five hours at 60° and atmospheric pressure, with a previously reduced platinum catalyst prepared according to Adams. The catalyst was then filtered off and the acetic acid was removed by vacuum distillation. After four crystallizations from ether and methyl alcohol needles were obtained which melted at 107–108°, $[\alpha]^{25\text{D}} +36.6^\circ$ (0.0191 g. in 2 cc. of chloroform solution gave $\alpha^{25\text{D}} +0.35^\circ$). The Liebermann test was positive. The Sal-

kowski test was similar to that for ergosterol, α_1 - and α_2 -sitosterol; the chloroform layer was colorless, while the sulfuric acid layer became colored.

Hydrogenation of α_1 -Sitosteryl Acetate at 65–70° in the Presence of Concentrated Hydrochloric Acid.—Two-tenths of a gram of α_1 -sitosteryl acetate was dissolved in glacial acetic acid to which was added 0.5 cc. of concd. hydrochloric acid. It was then hydrogenated at 65–70° and atmospheric pressure for five and one-half hours with a platinum catalyst prepared according to Adams. The

catalyst was reduced in the presence of the sterol acetate. The hydrogenation mixture took up the calculated amount for the complete saturation of the two double bonds. The catalyst was filtered off, and the reaction mixture was evaporated to dryness *in vacuo*. The residue gave a negative Liebermann test. After several crystallizations from dilute alcohol needles were obtained which melted at 147–148°, $[\alpha]^{19D} + 39.4$ (0.0208 g. in 2 cc. of chloroform solution gave $\alpha^{19D} + 0.41^\circ$).

Anal. Calcd. for $C_{31}H_{54}O_2$: C, 81.16; H, 11.87. Found: C, 80.89; H, 11.71.

Preparation of α_1 -Sitostanol.— α_1 -Sitostanol acetate was hydrolyzed by refluxing it for one hour with a 5% alcoholic solution of potassium hydroxide. The sterol was then worked up in ether in the usual manner. Recrystallization from dilute alcohol gave needles which melted at 185–186°, $[\alpha]^{25D} + 27.0$ (10.4 mg. in 2 cc. of chloroform solution gave $\alpha^{25D} + 0.14$).

Anal. Calcd. for $C_{29}H_{52}O$: C, 83.58; H, 12.58. Found: C, 83.71; H, 12.48.

Preparation of α_1 -Dihydrositosteryl Acetate.—Nine hundred and sixty milligrams of α_1 -sitosteryl acetate dissolved in glacial acetic acid was hydrogenated at room temperature and atmospheric pressure with 0.14 g. of a platinum catalyst prepared according to Adams. The catalyst was reduced in the presence of the sterol acetate. Only one mole of hydrogen was taken up, and the hydrogenation was complete in fifteen minutes. The catalyst was filtered off, and the solvent was evaporated *in vacuo*, leaving an oily residue which crystallized on cooling. Recrystallization from ethyl alcohol gave needles of m. p. 108.5–110.5° ($[\alpha]^{20D} + 35.1$ (0.0442 g. in 5 cc. of chloroform solution gave $\alpha^{20D} + 0.31^\circ$). The Liebermann test was positive. This material is identical with that obtained by hydrogenation of α_1 -sitosteryl acetate at 60° and atmospheric pressure. A melting point determination showed no depression.

Anal. Calcd. for $C_{31}H_{52}O_2$: C, 81.52; H, 11.48. Found: C, 81.30; H, 11.33.

Preparation of α_1 -Dihydrositosterol.—One hundred and two milligrams of α_1 -dihydrositosteryl acetate was hydrolyzed by refluxing it on the water-bath with 5% alcoholic potash solution. The sterol was precipitated with water and filtered. Purification was obtained by crystallization from ethyl acetate and methyl alcohol; needles m. p. 152–154°, $[\alpha]^{20D} + 10.9^\circ$ (0.0238 g. in 2 cc. of chloroform solution gave $\alpha^{20D} + 0.13^\circ$).

Anal. Calcd. for $C_{29}H_{50}O$: C, 83.98; H, 12.15. Found: C, 83.9; H, 11.9.

Acetylation of α_1 -dihydrositosterol with acetic anhydride gave back α_1 -dihydrositosteryl acetate, m. p. 108°. The melting point was not depressed by α_1 -dihydrositosteryl acetate previously prepared by hydrogenation of α_1 -sitosteryl acetate.

Preparation of α_1 -Isodihydrositosteryl Acetate.—Dry hydrogen chloride was passed for two hours into a chloroform solution of α_1 -dihydrositosteryl acetate kept at 0°. The reaction mixture was then evaporated to dryness *in vacuo*. The oily residue gave a negative Beilstein test. Since the residue had a slight coloration it was taken up in ether and decolorized with animal charcoal. Recrystalli-

zation from ethyl alcohol gave pure α_1 -isodihydrositosteryl acetate in the form of plates m. p. 137.5–138.5°, $[\alpha]^{20D} + 42.0^\circ$ (0.0572 g. in 5 cc. of chloroform solution gave $\alpha^{20D} + 0.48^\circ$).

α_1 -Isodihydrositosteryl acetate gives a positive Liebermann test. It gives a depression of the melting point when intimately mixed with α_1 -sitosteryl acetate (m. p. 136–137°).

Anal. Calcd. for $C_{31}H_{52}O_2$: C, 81.52; H, 11.48. Found: C, 81.4; H, 11.6.

Preparation of α_1 -Isodihydrositosterol.—One hundred and thirty milligrams of α_1 -isodihydrositosteryl acetate was hydrolyzed by refluxing it for one hour with a 5% alcoholic potash solution. The sterol was worked up in ether. Recrystallization from ethyl alcohol gave needles melting at 152–154°, $[\alpha]^{20D} + 31.0^\circ$ (0.0452 g. in 5 cc. of chloroform solution gave $\alpha^{20D} + 0.28^\circ$). The melting point is depressed by α_1 -dihydrositosterol.

Anal. Calcd. for $C_{29}H_{50}O$: C, 83.98; H, 12.15. Found: C, 83.7; H, 11.9.

Acetylation of a sample of α_1 -isodihydrositosterol with acetic anhydride again gave back α_1 -isodihydrositosteryl acetate, m. p. 137°.

Hydrogenation of α_1 -Isodihydrositosteryl Acetate.— α_1 -Isodihydrositosteryl acetate dissolved in glacial acetic acid was hydrogenated at room temperature and atmospheric pressure with a platinum catalyst prepared according to Adams. The catalyst was reduced in the presence of the sterol acetate. α_1 -Sitostanol acetate was again obtained, m. p. 147°.

Preparation of α_1 -Isodihydrositosteryl Acetate Oxide (14,15).—Sixty-five milligrams of α_1 -isodihydrositosteryl acetate (m. p. 137.5–139) was treated for ten days at room temperature with a large excess of a chloroform solution of perbenzoic acid. The reaction mixture was washed once with water, twice with dilute sodium hydroxide, and then with water until neutral. The chloroform solution was dried with anhydrous sodium sulfate, and evaporated to dryness *in vacuo*. The residue was crystallized from methyl alcohol. Needles were obtained which melted at 152–154°.

Anal. Calcd. for $C_{31}H_{52}O_3$: C, 78.76; H, 11.09. Found: C, 78.71; H, 10.97.

α_1 -Isodihydrositosteryl acetate oxide was dissolved in dry pyridine and treated with acetic anhydride for one-half hour on the water-bath. The reaction mixture was worked up in ether in the usual manner. Recrystallization from methyl alcohol gave back the starting material, m. p. 152°.

Preparation of $\Delta^{8,9,14,15}$ - α_1 -Sitostadiene Acetate-3.— α_1 -Isodihydrositosteryl acetate oxide was treated with 1 cc. of glacial acetic acid and one small drop of concentrated sulfuric acid. The reaction mixture was warmed on the water-bath for a very short time to dissolve completely the oxide. The reaction mixture was allowed to stand at room temperature for twenty-four hours. Small needles separated. The product was taken up in ether. The ether solution was washed successively with water, sodium bicarbonate, and water, and dried with anhydrous sodium sulfate. The ether solution was decolorized with animal charcoal. Evaporation of the ether left an oil which was crystallized from petroleum ether (30–60°) and methyl

alcohol, and methyl alcohol. Needles were obtained which melted at 121.5–122°.

Anal. Calcd. for $C_{31}H_{50}O_2$: C, 81.88; H, 11.08. Found: C, 81.62; H, 10.90.

We wish to take this opportunity to express our thanks to the Rockefeller Foundation for a grant-in-aid for this work, to Merck & Co., Inc., of Rahway, New Jersey, for the analytical and absorption spectrum data and to Dr. O. H. Emerson of the University of California for kindly furnishing part of the crude sterol from wheat germ oil from which the α_1 -sitosterol used in these experiments was isolated.

Summary

1. Certain experimental results on the determination of the structure of α_1 -sitosterol are described.

2. An absorption spectrum study, and the non-formation of an addition compound with maleic anhydride, indicate that the two double bonds in α_1 -sitosterol are not conjugated.

3. Only one of the double bonds in α_1 -sitosteryl acetate can be hydrogenated under ordinary conditions. α_1 -Dihydrositosterol and its acetate

have been prepared and characterized. Evidence is offered to show that α_1 -dihydrositosterol is an α -stenol.

4. The second double bond which is resistant to hydrogenation can be isomerized by dry hydrogen chloride into a position which is easy to hydrogenate. α_1 -Isodihydrositosterol and its acetate have been prepared and characterized. Evidence is offered to show that α_1 -isodihydrositosterol is a β -stenol.

5. Hydrogenation of α_1 -isodihydrositosteryl acetate gives α_1 -sitostanol acetate. Certain characteristic properties of this latter compound have also been described and we have observed that it may be obtained also by the direct complete hydrogenation of α_1 -sitosteryl acetate at 65–70° in the presence of a small amount of concentrated hydrochloric acid.

6. α_1 -Sitostanol and stigmastanol are not identical. This difference is probably the result of isomerization in the hydrocarbon side chain.

7. A structural formula for α_1 -sitosterol has been proposed.

PRINCETON, N. J.

RECEIVED JUNE 19, 1939

[CONTRIBUTION FROM THE EXPERIMENT STATION OF THE HAWAIIAN SUGAR PLANTERS' ASSOCIATION]

Electrolytes and the Viscosity of Pectin Solutions¹

By HUGO P. KORTSCHAK

Introduction

That the addition of electrolytes to certain colloidal solutions will cause marked changes in the viscosity is well known, but no explanation has been offered that seems completely satisfactory for all cases. This paper describes the effect of electrolytes on pectin sols, and shows that the colloidal character of the solutions is not a determining factor.

Experimental.—The pectin used was Eastman Citrus, practical. This was not the same sample used in the previous paper;² solutions prepared from it had a somewhat higher viscosity, indicating greater purity. The equivalent weight was 1500–2000. Other reagents were c. p. grade. Ordinary distilled water was used. No precautions were taken to prevent contamination by atmospheric carbon dioxide, except for boiling before use with substances containing calcium.

(1) Experimental work reported in this paper was done as part of Project ST-P-5 of the H.S.P.A. Experiment Station.

(2) H. P. Kortschak, *THIS JOURNAL*, **61**, 681 (1939).

Since it is difficult to reproduce pectin sols exactly, a correction factor (always less than 2%) was applied to values at 0.1% pectin, to make them more easily comparable. Correction was made for density at salt concentrations above 0.1 *N* but no other correction factors were applied. For other experimental details see previous paper.² Viscosities reported are relative to water at the temperature of measurement, 27.5 ± 0.5°.

TABLE I
VISCOSITY OF 0.1% PECTIN SOLS

<i>N</i> added base	NaOH		Ca(OH) ₂		KOH	NaOH + 0.001 <i>N</i> NaCl	
	η	<i>pH</i>	η	<i>pH</i>	η	η	<i>pH</i>
0	1.414	3.83	1.414	3.83	1.414	1.309	3.87
5 × 10 ⁻⁵	1.424	3.93	1.419	3.82	1.434	1.323	4.00
1 × 10 ⁻⁴	1.445	4.03	1.421	3.84	1.457	1.333	4.13
2 × 10 ⁻⁴	1.474	4.32	1.428	3.91	1.504	1.339	4.52
5 × 10 ⁻⁴	1.513	7.15	1.423	4.09	1.484	1.300	8.50
1 × 10 ^{-3a}	1.43	10.2	1.31	4.56	1.41	1.29	10.3
2 × 10 ^{-3a}	1.34	11.2	1.30	1.19	11.1
5 × 10 ^{-3a}	1.29	12.0	1.22	1.17	11.7

^a Pectin hydrolyzes rapidly in alkaline solution; viscosities at these concentrations are therefore inaccurate.