

Fatty Acids. Part III. The Constitution and Properties of Santalbic Acid.*

By F. D. GUNSTONE and W. C. RUSSELL.

[Reprint Order No. 6485.]

Santalbic acid, the chief component acid in *Santalum album* (Linn.) seed oil, is shown to be *trans*-octadec-11-en-9-ynoic acid † and thus identical with ximenynic acid. Some reactions of this acid are reported.

THE evergreen tree *Santalum album* (Linn.) of the natural order Santalaceae is well known for its highly scented wood (sandalwood) which is used extensively throughout India. Distillation of the wood affords the perfume sandal oil. Much less is known about the seeds except that they are edible, rich in oil, and can be collected in quantity from sandalwood plantations. The tree varies in height from 12 to 35 feet and bears fruit twice a year (Iyer, *Analyst*, 1935, **60**, 319).

Previous investigators (Sreenivasaya and Narayana, Proc. 15th Indian Sci. Congr., 1928, p. 160; *J. Indian Inst. Sci.*, 1936, **19**, A, 1; Iyer, *loc. cit.*; Rao, *J. Annamalai Univ.*, 1937, **6**, 198; Kotasthane and Narayana, *Poona Agr. Coll. Mag.*, 1938, **29**, 126) have shown that these seeds contain an oil (50—60%) which readily polymerises. The characteristics of the oil recorded by these workers show considerable variation; the iodine value lies between 100 and 150, the saponification value between 172 and 197, and the acetyl value between 20 and 40. There is evidence of something unusual in Kotasthane and Narayana's report that "solid" and "liquid" acids have a similar high iodine value.

Madhuranath and Manjunath, in a fuller investigation (*J. Indian Chem. Soc.*, 1938, **15**, 389), reported the presence in quantity of a solid octadecatrienoic acid (santalbic acid) which appears to be non-conjugated. This view was based on the observations that stearic acid resulted from hydrogenation in which three mols. of hydrogen are absorbed, that the acid failed to react with maleic anhydride, and that it differed from punic acid, a stereoisomer of elæostearic acid. Since among naturally occurring octadecatrienoic acids the non-conjugated isomers are liquid and the conjugated isomers are solid we reinvestigated this problem.

* Part II, *J.*, 1954, 1611.

† Geneva numbering, $\text{CO}_2\text{H} = 1$.

Constitution of Santalbic Acid.—The ultraviolet spectrum of santalbic acid showed a maximum at 229 m μ (log ϵ 4.22) and an inflexion at 240 m μ (log ϵ 4.06). These values indicated immediately that the acid contains, not a conjugated triene as chromophore, but a diene or enyne system. A naturally occurring enyne acid, ximenynic acid, has been described (Ligthelm, Schwarz, and von Holdt, *J.*, 1952, 1088; Ahlers and Ligthelm, *J.*, 1952, 5039) and a comparison of the two acids and their *p*-bromophenacyl esters revealed their close similarity. Other enynes with similar ultraviolet spectra have been reported

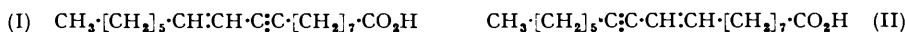
	M. p.	Ultraviolet absorption	<i>p</i> -Bromophenacyl ester (m. p.)
Ximenynic acid ...	39—40°	229 m μ (log ϵ 4.21), 240 m μ (infl.)	53.8—54.2°
Santalbic acid *.....	38.5—39.5	229 m μ (log ϵ 4.22), 240 m μ (infl.)	53—54
Santalbic acid ^b ...	38.5—39	229 m μ (log ϵ 4.20), 240 m μ (infl.)	53.5—54

* Present work. ^b Hatt and Szmer, *Chem. and Ind.*, 1954, 962 (this work will be referred to below).

(Heilbron, Jones, and Weedon, *J.*, 1944, 140 and other references quoted there) and it is known that the corresponding dienes differ chiefly in their larger values of ϵ . The identity of santalbic and ximenynic acid has now been amply confirmed by hydrogenation, oxidation, infrared spectrum, and by comparison with a synthetic specimen (Grigor, McInnes, and McLean, *J.*, 1955, 1069).

Santalbic acid quickly absorbed hydrogen (three mols.) and gave stearic acid; oxidation with potassium permanganate in acetic acid solution gave heptanoic and azelaic acid; and the infrared spectrum was identical with that of synthetic ximenynic acid and contained, in particular, a band at 953 cm.⁻¹ indicative of a *trans*-ethylenic bond in conjugation with an acetylenic linkage.

This evidence indicated unsaturated centres in the 9 : 10- and the 11 : 12-position, one of which must be acetylenic since the compound required three mols. of hydrogen for saturation and no other unsaturated centre is present. The infrared absorption at 953 cm.⁻¹ further confirms the absence of a *trans-trans*- (988 cm.⁻¹) or *cis-trans*- (984 and 950 cm.⁻¹) diene (Ahlers, Brett, and McTaggart, *J. Appl. Chem.*, 1953, 3, 433). There are thus only two possible structures for santalbic acid, (I) or (II), of which (I) has been assigned to



ximenynic on the basis of ozonolysis to heptanal and azelaic acid (Ligthelm, Schwarz, and von Holdt; Hatt and Szmer, *loc. cit.*) and confirmed by synthesis (Grigor, McInnes, and McLean, *loc. cit.*). Before this synthetic work we had independently shown that santalbic acid has structure (I) by a method which provides additional proof of the *trans*-configuration of the double bond.

It is known that peracids react much more readily with ethylenic than with acetylenic bonds (Raphael, "Acetylenic Compounds in Organic Synthesis," Butterworths, London, 1955, p. 33) and we have applied Swern's performic acid oxidation procedure to santalbic acid (Swern, Billen, Findley, and Scanlan, *J. Amer. Chem. Soc.*, 1945, 67, 1786). A dihydroxy-acid was isolated, though in poorer yield than usual for this reaction. The product readily took up two mols. of hydrogen to give an acid (m. p. 127—128°) shown to be 11 : 12-dihydroxystearic acid by its oxidation to heptanal and 10-formyldecanoic acid with periodate and to heptanoic and 1 : 11-undecanedioic acid with potassium permanganate. Comparison of the melting point with that of the known *threo*- (m. p. 94°) and *erythro*-11 : 12-dihydroxystearic acids (m. p. 129°) (Bounds, Linstead, and Weedon, *J.*, 1954, 4219) clearly shows our product to be the *erythro*-isomer and since hydroxylation by performic acid is equivalent to *trans*-addition the double bond must have had the *trans*-configuration. Santalbic acid is therefore octadec-*trans*-11-en-9-ynoic acid (I) and identical with ximenynic acid. Dr. McLean has reported that samples of santalbic acid, its *p*-bromophenacyl ester, and 11 : 12-dihydroxystearic acid supplied by us showed no depression of melting point when mixed with their synthetic products.

Periodate oxidation of dihydroxystearic acid gave heptanal and a product considered to be 10-formyldec-9-ynoic acid. Though not very stable this compound was satisfactorily analysed, its ultraviolet absorption measured, and a 2 : 4-dinitrophenylhydrazone prepared.

This last, isolated pure only with difficulty, was yellow though ethylenic $\alpha\beta$ -unsaturated carbonyl compounds usually give orange or red dinitrophenylhydrazones [the dinitrophenylhydrazone of 11-formylundec-10-enoic acid is deep orange (Part II) but Lunt and Sondheimer (*J.*, 1950, 3361) report a yellow 2 : 4-dinitrophenylhydrazone for but-2-ynal and hept-2-ynal]. This decreased intensity of colour is probably due to the fact that the π electrons of a triple bond are more tightly bound than those of a similarly situated double bond (Raphael, *op. cit.*, p. 208). The ultraviolet spectrum of the acetylenic aldehyde is interesting in that a more intense absorption was observed for hexane solutions than for ethanol solutions; and, in addition, other maxima were noted in the former solvent which previously have not been reported. The lower absorption in alcohol may be due to hemiacetal formation, as suggested by Ashdown and Kletz (*J.*, 1948 1454; cf. Crombie, *J.*, 1955, 1011). The values obtained in this work and those obtained for similar compounds by other investigators are summarised in the Table.

Aldehyde	$\lambda_{\max.}$ ($m\mu$)	EtOH	ϵ <i>n</i> -Hexane	Aldehyde	$\lambda_{\max.}$ ($m\mu$)	EtOH	ϵ <i>n</i> -Hexane
$\text{CH}_3\text{:C:C}\cdot\text{CHO}^a$	225	2800	—	$\text{HO}_2\text{C}\cdot[\text{CH}_2]_7\text{:C:C}\cdot\text{CHO}$	208	—	7800
$\text{CH}_3[\text{CH}_2]_3\text{:C:C}\cdot\text{CHO}^a$	227	3200	—		222	—	6500
$\text{CH}_3\cdot[\text{CH}_2]_4\text{:C:C}\cdot\text{CHO}^b$	227	—	8000		231	4000	6000

^a Lunt and Sondheimer, *loc. cit.* ^b Crombie, *loc. cit.*

Catalytic Reduction of Santalbic Acid.—Ligthelm *et al.* (*loc. cit.*) failed to convert the enynic into the corresponding dienoic acid, but, using the poisoned catalyst of Lindlar (*Helv. Chim. Acta*, 1952, **35**, 446), we have now succeeded in preparing octadeca-*cis*-9-*trans*-11-dienoic acid. In ethanol solution there was no break in hydrogen absorption and the reaction was stopped after the required amount of hydrogen had been absorbed; in light petroleum solution there appeared to be no reduction. Our product is similar to that obtained from linoleic acid by alkali-isomerisation (Nichols, Herb, and Riemen-schneider, *J. Amer. Chem. Soc.*, 1951, **73**, 247). In the presence of iodine it readily isomerised to the familiar *trans-trans*-dienoic acid (Mangold, *Monatsh.*, 1894, **15**, 309), characterised as its maleic anhydride adduct. It is interesting to compare the ultraviolet spectra of the enynic and of the two dienoic acids. The *trans-cis*-diene was not purified and the inflexion at 240 $m\mu$ may be due to traces of santalbic acid; this is less likely in the *trans*-

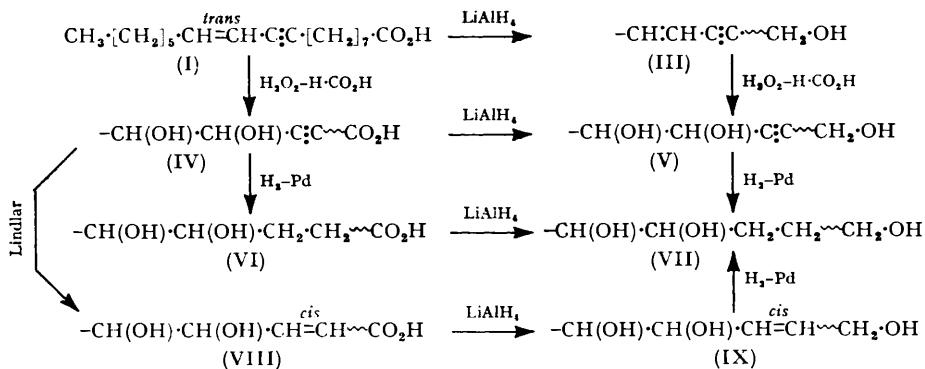
	$\lambda_{\max.}$ (ϵ)	Inflexion
<i>trans</i> -11-en-9-yn-	229 $m\mu$ (16,600)	240 $m\mu$
<i>trans</i> -11- <i>cis</i> -9-dien-	231 $m\mu$ (24,000)	240 $m\mu$
<i>trans</i> -11- <i>trans</i> -9-dien-	231 $m\mu$ (33,300)	227, 239 $m\mu$

trans-diene which shows inflexions on both sides of the maximum. The maxima agree well with these previously reported (Nichols *et al.*, *loc. cit.*; Kass, "Protective and Decorative Coatings," ed. Mattiello, J. Wiley & Sons, 1944, Vol. IV, p. 362; van der Hulst, *Rec. Trav. chim.*, 1935, **54**, 639, 644).

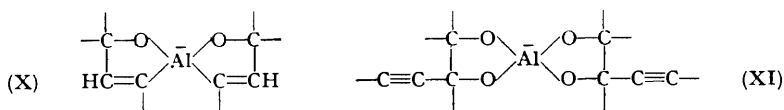
Lithium Aluminium Hydride Reduction of Santalbic Acid and its Derivatives.—Reduction of santalbic acid affords the corresponding alcohol (III) as reported by Ligthelm, von Rudloff, and Sutton (*J.*, 1950, 3187). Acetylenic bonds are unaffected by lithium aluminium hydride except when connected to a carbon atom carrying a hydroxyl group; they are then stereospecifically reduced to *trans*-ethylenic bonds (Raphael, *op. cit.*, p. 30 and references there cited). By this reaction it was hoped to convert dihydroxystearolic acid into 1 : 11 : 12-trihydroxyoctadec-*trans*-9-ene but despite several attempts only the acetylenic triol (V) was obtained. This also resulted from per-acid oxidation of the enyne alcohol (III). Dihydroxyleic acid (VIII) however was produced from dihydroxystearolic acid by catalytic reduction (Lindlar) and this, with lithium aluminium hydride, afforded the *cis*-ethylenic triol (IX). The saturated triol (VII) was obtained by lithium aluminium hydride reduction of dihydroxystearic acid (VI) or by catalytic reduction of either the acetylenic triol (V) or the *cis*-ethylenic triol (IX).

Attenburrow, Cameron, Chapman, Evans, Hems, Jansen, and Walker (*J.*, 1952, 1101) have suggested that stereospecific reduction of an α -hydroxy-acetylenic compound by

lithium aluminium hydride involves formation of an intermediate aluminium complex of type (X), and the only reported failure of this reaction has been attributed to steric hindrance (Raphael, *op. cit.*). This is unlikely in the present case and we suggest that the



glycol preferentially forms a complex of type (XI) in which the triple bond is not involved and hence not reduced. This constitutes a limitation of this reaction in that it is not applicable to acetylenic glycols of the type $-\text{C}(\text{OH})\text{-C}(\text{OH})\text{-C}\equiv\text{C}-$. Glycols of the type $-\text{C}(\text{OH})\text{-C}\equiv\text{C}\text{-C}(\text{OH})-$ have been reduced (unpublished results of the Manchester school quoted by Raphael, *op. cit.*, p. 114) but in this case a complex of type (XI) involving four hydroxyl groups is unlikely on spatial grounds.



Santalum album Seed Oil.—Spectroscopic data show that the mixed acids (excluding unsaponifiable material) contain 95% of santalbic acid along with 5% of other unidentified acids. The enynic acid has now been recognised by Ligthelm, Horn, Schwartz, and von Holdt (*J. Sci. Food Agric.*, 1954, **5**, 281) in three South African *Ximenia* species (N. O. Olacaceae) [*X. caffra* (24.3%), *X. caffra* var. *natalensis* (22.0%), *X. americana* var. *microphylla* (21.9%)], and by Hatt and Szumer (*Chem. and Ind.*, 1954, 962) in two species of the *Santalum* genus (N. O. Santalaceae) [*S. acuminatus* (D.C.) (the sweet Quandong) and *S. Murrayana* (F. v. M.) (the Bitter Quandong) (40–43%)], in addition to the present source (88%). These two genera belong to closely related families grouped in the Santalales and apart from two analyses (Puntambekar and Krishna, *J. Indian Chem. Soc.*, 1937, **14**, 268; Boekenoogen *Fette u. Seifen*, 1939, **46**, 717) of *X. Americana* seed oil which may be in error (cf. Ligthelm, Horn, Schwarz, and von Holdt, *loc. cit.*) each species of these two genera yet examined contains this enynic acid.

Ligthelm (*Chem. and Ind.*, 1954, 249) has reported that in *X. caffra* seed oil the enynic acid is accompanied by a small amount of hydroxy-acid, probably 8-hydroxyoctadec-11-en-9-yenoic acid, but we have found no evidence of such an acid in sandal wood oil.

It is apparent from the figures quoted that sandal seeds provide the best source of this enynic acid which is readily isolated in high yield by crystallisation of the mixed acids. Starting with the less satisfactory sources the South African and Australian workers have had to use distillation, chromatography, or urea complex formation before crystallisation and have isolated the acid in much lower yield.

EXPERIMENTAL

Absorption spectra were determined with a Unicam quartz spectrophotometer, ethanol being the solvent except where otherwise stated. Light petroleum refers to the fraction of b. p. 40–60°.

S. album Seed Oil.—The dried seeds were spherical, about $\frac{1}{4}$ " in diameter, and brown, and had an average weight of 0.15 g. When coarsely ground in a mortar and extracted with light petroleum the oil (53.5%) was obtained as a viscous, pale greenish-yellow liquid of iodine value 152.5, sap. equiv. 332.8, containing 6.9% of unsaponifiable material, and having absorption max. at 229 μ ($E_{1\text{cm}}^{1\%}$, 497) in hexane. For the mixed acids from which unsaponifiable material had been removed this value was 570. These values indicate that the oil contains 88% of santalbic glyceride and the mixed acids (excluding unsaponifiable material) 95% of santalbic acid.

Santalbic Acid: Isolation, Hydrogenation, and Oxidation.—The oil (131 g.) was hydrolysed by boiling alcoholic potassium hydroxide for 1 hr., during which a gum separated; this was rejected before acidification and ether extraction of the mixed acids (103 g.). Crude santalbic acid (77 g.; m. p. 36—38°) was obtained by crystallisation from light petroleum. Recrystallisation and working up of mother-liquors gave pure acid (68 g.; m. p. 38.5—39.5°) as white plates and a further quantity (10 g.) of crude acid; the absorption max. was at 229 μ ($\log \epsilon$ 4.22; $E_{1\text{cm}}^{1\%}$, 596) and an inflexion was at about 240 μ . The *p*-bromophenacyl ester had m. p. 53—54° (Found: C, 65.6; H, 7.6; Br, 17.1. Calc. for $C_{24}H_{35}O_3Br$: C, 65.7; H, 7.4; Br, 16.8%).

Alternatively the acid was obtained directly from the seeds without separate extraction of the oil. The crushed seeds (30 g.) were refluxed with alcoholic potassium hydroxide for 1 hr., the solution filtered, and the filtrate diluted with water, acidified, and extracted (ether). The product (10.6 g.) gave santalbic acid (5.8 g.; m. p. 37—39°) when crystallised from light petroleum. Troublesome emulsions were encountered in the extraction.

Santalbic acid (0.5 g.), hydrogenated in the presence of palladium-charcoal, took up hydrogen equiv. to 3.0 mols. The product (0.53 g.) afforded stearic acid on crystallisation (m. p. 69—70°) (*p*-bromophenacyl ester, m. p. 90°) (both m. p.s were unchanged on admixture with authentic samples).

A portion of the acid (2.0 g.), oxidised by potassium permanganate in acetic acid as previously described (Part II), gave a volatile acid (0.36 g.) shown to be heptanoic acid (*p*-bromophenacyl ester, m. p. and mixed m. p. 68—69°). This was accompanied by azelaic acid (0.8 g.), m. p. and mixed m. p. 104—105°.

erythro-11:12-Dihydroxystearic Acid (IV): Preparation, Hydrogenation, and Oxidation.—Hydrogen peroxide (30%; 12 ml.) was added to a solution of santalbic acid (22 g.) in formic acid (98—100%; 240 ml.), and the mixture kept at 40° with stirring for 2½ hr. The product after recovery in the usual way (Swern *et al.*, *loc. cit.*) was crystallised from ether-light petroleum (1:1; 200 ml.). The crude dihydroxystearic acid (8.4 g.) was purified by crystallisation from ethyl acetate and from alcohol, with charcoal if necessary. The pure acid (4.8 g.) melted at 88—89° (Grigor *et al.*, *loc. cit.*, give 89—90°) (Found: C, 69.5; H, 10.2. Calc. for $C_{18}H_{32}O_4$: C, 69.2; H, 10.3%).

Dihydroxystearic acid (2.0 g.) when reduced (Pd-C; H_2 equiv. to 2.0 mols.) gave *erythro-11:12*-dihydroxystearic acid, m. p. 127—128° (from ethanol) (lit., 129°; Bounds, Linstead, and Weedon, *loc. cit.*) depressed on admixture with *erythro-9:10*-dihydroxystearic acid (m. p. 129—131°) (Found: C, 68.3; H, 11.1. Calc. for $C_{18}H_{36}O_4$: C, 68.3; H, 11.5%). Oxidised with potassium permanganate in acetic acid solution (see Part II), this acid (0.6 g.) gave heptanoic acid (0.15 g.) (*p*-bromophenacyl ester, m. p. and mixed m. p. 68—70°) and 1:11-undecanedioic acid (0.3 g.), m. p. 108.5—109.5° (identical with a specimen described below). Periodate oxidation of the dihydroxystearic acid (2.0 g.) (see Part II) afforded heptaldehyde which was identified as the 2:4-dinitrophenylhydrazone, m. p. and mixed m. p. 103—104° (Found: C, 53.3; H, 5.9. Calc. for $C_{13}H_{18}O_4N_4$: C, 53.0; H, 6.2%). This was accompanied by 10 *formyldecanoic acid*, m. p. 47—48° [from light petroleum (b. p. 60—80°)] (Found: C, 65.7; H, 9.9. $C_{11}H_{20}O_3$ requires C, 65.9; H, 10.1%), oxidised to 1:11-undecanedioic acid, m. p. 109—110° (lit., 110°) (Found: C, 60.9; H, 9.6. Calc. for $C_{11}H_{20}O_4$: C, 61.1; H, 9.3%). The 2:4-*dinitrophenylhydrazone* of ethyl 10-formyldecanoate resulted from an attempt to prepare the derivative of the acid in ethanol solution, and had m. p. 59—60° (Found: C, 55.7; H, 6.7; N, 13.9. $C_{19}H_{28}O_6N_4$ requires C, 55.9; H, 6.9; N, 13.7%).

Periodate oxidation of dihydroxystearic acid (2 g.) gave heptanal (0.5 g.), characterised as the 2:4-dinitrophenylhydrazone, m. p. and mixed m. p. 105—105.5° (Found: C, 53.2; H, 6.0; N, 18.8. Calc. for $C_{13}H_{18}O_4N_4$: C, 53.0; H, 6.2; N, 19.0%), and as the semicarbazone, m. p. and mixed m. p. 108—108.5°. The non-volatile fission product was 10-*formyldec-9-ynoic acid*, m. p. 25.5—26.5° (Found: 67.3; H, 7.9. $C_{11}H_{16}O_3$ requires C, 67.3; H, 8.2%), absorption max. see p. 3784. The 2:4-*dinitrophenylhydrazone* prepared in acetic acid solution and purified

by passage through a column of silica gel was obtained as a deep yellow powder, m. p. 105.5—106.5° (Found : C, 53.9; H, 5.3; N, 14.7. $C_{17}H_{20}O_6N_4$ requires C, 54.2; H, 5.4; N, 14.9%).

Reductions with Lindlar's Catalyst.—Santalbic acid (2 g.) in ethanol (30 ml.) containing Lindlar's catalyst (200 mg.) and quinoline (80 mg.) was shaken with hydrogen which was steadily absorbed. The reaction was stopped after 200 ml. had been taken up (175 ml., 2 mol.). The product which was solid at 0° but liquid at room temperature was octadeca-*cis*-9 : *trans*-11-dienoic acid.

Exposed to ultraviolet light or sunlight in the presence of iodine in light petroleum solution this *cis-trans*-diene was isomerised to octadeca *trans*-9-*trans*-11-dienoic acid (1 g.), m. p. 48—50° which after crystallisation from alcohol melted at 53—54° (lit., 54°) (Found : C, 76.9; H, 11.5; Calc. for $C_{18}H_{32}O_2$: C, 77.1; H, 11.5%). For absorption max. see p. 3784. This *trans-trans*-diene reacted with maleic anhydride at 75° to form the diene addition product, m. p. 92.5—93.5 [lit., 88° (Böeseken and Hoevers, *Rec. Trav. chim.*, 1930, **49**, 1165), 94.5° (Kaufmann and Baltes, *Fette u. Seifen*, 1936, **43**, 93), 78—96° (Schmid and Lehmann, *Helv. Chim. Acta*, 1950, **33**, 1494)] (Found : C, 69.8; H, 8.8. Calc. for $C_{22}H_{34}O_5$: C, 69.8; H, 9.1%).

Partial reduction of dihydroxystearic acid (1 g.) by the same method gave erythro-10 : 11-*dihydroxyoleic acid*, m. p. 68—68.5° after repeated crystallisation from light petroleum and finally from nitromethane (Found : C, 68.6; H, 10.8; microhydrogenation shows 1.1 C:C. $C_{18}H_{34}O_4$ requires C, 68.7; H, 10.9%; 1.0 C:C).

Reductions with Lithium Aluminium Hydride.—A solution of santalbic acid (1.6 g.) in dry ether (25 ml.) was slowly added to a suspension of lithium aluminium hydride (0.3 g., 5 mols.) in dry ether (25 ml.) and then refluxed for 1 hr. Next morning ethyl acetate (5 ml.) and cold 3*N*-hydrochloric acid (25 ml.) were added and the resulting solution was extracted with ether and washed with alkali. The product (0.9 g.), crystallised from light petroleum (b. p. 80—100°), gave octadec-*trans*-11-en-9-ynol, m. p. 23—25° (lit., 30—31°) [α -naphthylurethane, m. p. 56—57° (lit., 57—57.5°)].

Dihydroxystearic acid (1 g.), similarly reduced (7.5 mols. of lithium aluminium hydride; refluxing for 3 hr.), gave 1 : 11 : 12-*trihydroxyoctadec-9-yne*, m. p. 79—80° (Found : C, 72.1; H, 11.5. $C_{18}H_{34}O_3$ requires C, 72.4; H, 11.5%; microhydrogenation indicated 1.8 C:C or 0.9 C:C). Increased amounts of lithium aluminium hydride and different reflux periods did not change the nature of the product which was also obtained (m. p. 76—77°; mixed m.p. 77—79°) by performic acid oxidation of octadec-*trans*-11-en-9-ynol.

Reduction of dihydroxystearic acid gave 1 : 11 : 12-*trihydroxyoctadecane*, m. p. 126—126.5° (from ethanol) (Found : C, 71.6; H, 12.5. $C_{18}H_{38}O_3$ requires C, 71.5; H, 12.7%). The same compound was obtained by catalytic reduction of 1 : 11 : 12-*trihydroxyoctadec-9-yne*.

Dihydroxyoleic acid, reduced with lithium aluminium hydride, gave a small quantity of solid, m. p. 51—52°, which was probably 1 : 11 : 12-*trihydroxyoctadec-cis*-9-ene since microhydrogenation (H_2 uptake, 0.94 double bond) gave a product (m. p. 122—124°) identical with the 1 : 11 : 12-*trihydroxyoctadecane* described above.

The authors thank Dr. S. Krishna (Scientific Adviser to the High Commissioner of India in the U.K., and Scientific Liaison Officer) for supplying the seeds used in this investigation, the D.S.I.R. for a Maintenance Allowance (to W. C. R.), Dr. J. McLean for making the mixed m. p. determinations referred to in the text, Mr. M. A. McGee for assistance with some early experiments, Dr. G. D. Meakins (Manchester) for the infrared measurements, and Mr. J. M. L. Cameron and Miss M. W. Christie for the microanalyses.

CHEMISTRY DEPARTMENT, UNIVERSITY OF ST. ANDREWS (F. D. G.).

CHEMISTRY DEPARTMENT, UNIVERSITY OF GLASGOW (W. C. R.).

[Received, June 6th, 1955.]