710. *Wool Wax. Part IV.* The Constitution of the Aliphatic Diols.*

By D. H. S. Horn and F. W. Hougen.

Five alkane-1 : 2-diols have been isolated from the unsaponifiable fraction of wool wax by chromatography and distillation at reduced pressure. They are identified, from infra-red studies and by degradation to fatty acids of known constitution, as *n*-hexadecane-, 16-methylheptadecane-, 18-methylnonadecane-, 20-methylheneicosane-, and 22-methyltricosane-1 : 2-diol.

Kuwata and Katuno (*J. Soc. Chem. Ind. Japan, Suppl.*, 1938, **41**, 227) isolated from the unsaponifiable fraction of wool wax a dihydric alcohol, m. p. 79—80°, for which they proposed the formula $C_{21}H_{40}(OH)_2$. During a chromatographic study of the unsaponifiable fraction of wool wax on activated alumina, we isolated a similar material from the most strongly adsorbed fraction. However, attempts to purify it revealed that it was a complex mixture of closely related compounds, differing principally in molecular weight.

Oxidation with lead tetra-acetate yielded formaldehyde, characterised as its dimedone compound, and a mixture of aliphatic aldehydes which was readily converted, on mild oxidation, into the corresponding saturated fatty acids. These preliminary investigations established the main components as alkane-1 : 2-diols (Horn and Hougen, *Chem. and Ind.*, 1951, 670). Murray and Schönfeld later (*J. Amer. Oil Chem. Soc.*, 1952, **29**, 416) also reported the isolation of some diol fractions.

Initially, the diols were prepared in 2% yield from commercial wool wax unsaponifiables (" Hartolan," Messrs. Croda Ltd.). Subsequently, they were obtained in 4—5% yield from the unsaponifiable material prepared from wool wax extracted with *iso*heptane from merino wool, kindly supplied by the Director of the Grootfontein College of Agriculture and Experiment Station. To avoid the production of artefacts (Daniel, Lederer, and Velluz, *Bull. Soc. Chim. biol.*, 1945, **27**, 218), saponification of the wax was carried out at 40°. The unsaponifiable material was separated, almost quantitatively, by a method similar to that of Barnes, Curtis, and Hatt (*Austral. J. Appl. Sci.*, 1952, **3**, 88).

After the failure of attempts to resolve the diol mixture by fractional crystallisation and chromatography, the acetylated material was fractionally distilled at reduced pressure in a spinning-band microstill. In the initial stages of the work, the acetylated mixture was distilled without a break (Fig. 1). Later, with more material available, it was more profitable to fractionate it roughly at a low reflux ratio into several arbitrary fractions, redistil each fraction more thoroughly, and combine appropriate distillates. In this way, decomposition was reduced to a minimum. Finally, the main components were separately distilled. This lengthy procedure was necessary because chromatography and crystallisation failed to remove completely the long-chain alcohols, the presence of which greatly complicated the separation of, especially, the higher components (see Fig. 1) and made it impossible to calculate accurately the relative proportions of the diols present.

The following pure diols were obtained by saponification and crystallisation of the pure fractions: *n*-hexadecane-, 16-methylheptadecane-, 18-methylnonadecane-, 20-methylheneicosane-, and 22-methyltricosane-1 : 2-diol. No other diols could be detected: if present, they occurred only in very small amounts and were lost in the intermediate fractions or did not survive the lengthy distillation.

The structures of the individual diols were determined by direct oxidation with an excess

---

* Part III, *J.*, 1953, 98.

of lead tetra-acetate in a stream of air (cf. Mendel and Coops, *Rec. Trav. chim.*, 1939, **58**, 1140) to the corresponding fatty acids having one methylene group less than the parent diols. The acids produced were compared with synthetically prepared acids of known structure.

(±)-*n*-Octadecane-1 : 2-diol required for comparison was synthesised from 1-octadecene (Wibaut, *Rec. Trav. chim.*, 1948, **67**, 113; cf. Minoru *et al.*, *J. Chem. Soc. Japan*, 1949, **52**, 50; *Chem. Abs.*, 1951, **45**, 4635) by a modification of the method of Swern, Billen, and Scanlan (*J. Amer. Chem. Soc.*, 1946, **68**, 1505).

The structures of the pure diols were further confirmed by making use of Thompson and Torkington's observation (*Trans. Faraday Soc.*, 1945, **41**, 246; cf. Sobotka and Stynler, *J. Amer. Chem. Soc.*, 1950, **72**, 5139; Freeman, *ibid.*, 1952, **74**, 2526) that a doublet in the 1360—1400-cm.$^{-1}$ range of the infra-red absorption spectra of alkanes is characteristic of the *iso*propyl group. Hexadecane-1 : 2-diol showed a single absorption band, identical with that of synthetic (±)-octadecane-1 : 2-diol (Fig. 2), at 1380 cm.$^{-1}$. The other pure wool wax diols showed the characteristic splitting of the 1380 cm.$^{-1}$ band (Fig. 2), and the intensity of absorption was that expected for a single methyl branch.

FIG. 1. *Distillation curve.*

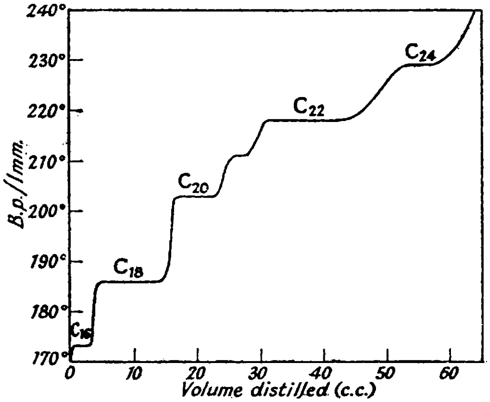
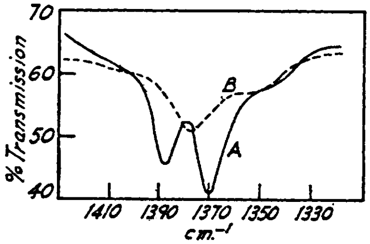


FIG. 2. *Infra-red absorption.*



A, 16-*Methylheptadecane*-1 : 2-*diol.*
B, *Octadecane*-1 : 2-*diol.*

The optical rotations of the pure diols could not be obtained with certainty because of their low solubilities. However, in contrast to (±)-octadecane-1 : 2-diol, they formed well-defined crystals and their diacetates had a slight dextrorotation. They have been correlated configurationally with the lævorotatory hydroxy-acids which occur in the wool-wax acid fraction (Horn, Hougen, and von Rudloff, *Chem. and Ind.*, 1953, 106). Thus on reduction with lithium aluminium hydride (—)-*n*-2-hydroxyhexadecanoic acid * and (—)-2-hydroxy-16-methylheptadecanoic acid yielded diols, respectively identical with *n*-hexadecane-1 : 2-diol and 16-methylheptadecane-1 : 2-diol obtained from the unsaponifiable material.

EXPERIMENTAL

Crystal long spacings were measured as described previously (Part III, *loc. cit.*). M. p. determinations were made with a heating rate of 3—4 min./degree in a "Hershberg"-type apparatus. Standardised, 8-cm., immersion thermometers were used.

The infra-red absorption spectra were obtained with the Perkin–Elmer spectrophotometer model 12C. An approx. 2% solution of the diol in carbon tetrachloride was run into a 0·5-mm. rock-salt cell warmed with an infra-red lamp. In this way, the diol could be kept in solution long enough after the lamp had been switched off to allow the absorption curve to be obtained.

Extraction of Wool Wax from Merino Fleeces.—Wool (2—3 kg.) was dried for 16 hr. at 60—70°/14—20 mm., and extracted for 7 hr. with *iso*heptane in a stainless-steel hot extractor. After storage for several days, the cold extract was decanted from the sludge and evaporated to dryness. In this way, 33·61 kg. of dried wool yielded 4·975 kg. (14·8%) of wool wax.

---

* Genera numbering, CO_2H = 1.

Saponification and Isolation of the Unsaponifiable Material.—To a mixture of wool wax (500 g.) and hexane (500 c.c.), at 40°, was added, with stirring, a solution of potassium hydroxide (85 g.) in ethanol (1 l. of 96%). The mixture was stirred at 40° for 24 hr. To the saponified mixture was added ethanol (660 c.c.) and water (1000 c.c.). The solution was shaken three times in a separating funnel (10 l.) with *n*-hexane. The combined hexane extracts, after being kept overnight in a tall cylindrical separating funnel, were separated from the sediment and shaken with 60% ethanol (2 × 500 c.c.). Evaporation of the hexane solution yielded extract I (131 g.).

The above alcohol solutions were combined and extracted in a continuous liquid–liquid extractor with hexane for 24 hr. The hexane extract was washed with 60% ethanol (200 c.c.) and evaporated to yield extract II (59 g.). The alcoholic wash-liquors were added to the next batch of saponified wax. The total yield of unsaponifiable material (extracts I and II) was 190 g. (38% of the wax).

Chromatographic Preparation of the Diols.—The wool-wax unsaponifiable material (500 g.), dissolved in wet ether (8 l., saturated with water at 6°), was filtered through a column of alumna (Peter Spence, type H), reactivated by 24 hr.' heating, with occasional stirring, at 400° (5 kg.; 30-cm. in height). The column was eluted with wet ether (24 l.), followed by ether (20 l.) containing ethanol (1% of 75%). The eluate was collected in 2-l. fractions. Those containing the material most strongly adsorbed were analysed by Karnovsky and Rapson's periodate titration method (*J. Soc. Chem. Ind.*, 1946, **65**, 138), and those containing considerable quantities of diols were amalgamated.

In this way, a crude diol fraction (186 g.) was obtained from the unsaponifiable material (2·2 kg.). It crystallised from ether and ethanol as a white powder (107 g.). The mother-liquors and fractions containing low diol contents (200 g.) were combined and rechromatographed. The crude diol fraction so obtained after crystallisation yielded further diol (30 g.). The total yield of crude diols (137 g.) was 6·2% of the unsaponifiable material.

To separate the diols from long-chain alcohols and other impurities, they were acetylated and distilled at 1 mm. in several batches in an electrically heated and vacuum-jacketed fractionating column (12 mm. internal diam.; 20 cm. long) packed with ⅛″ Dixon gauze rings at a reflux ratio of about 1 : 10. The fractions of similar b. p. and *n* were combined to form the four following fractions: (1) b. p. 190—200°, n_D^{30} 1·4445, 54 c.c.; (2) b. p. 220—225°, n_D^{30} 1·4510, 37 c.c.; (3) b. p. 225—250°, solid, 32 c.c.; (4) b. p. 250—290°, n_D^{30} 1·4500—1·151, 59 c.c.

Fractions 1, 2, and 3 were separately saponified and crystallised from ether. Fraction 4, which contained the bulk of the high-boiling alcohols, was saponified and rechromatographed and the diol fraction crystallised. The material, obtained from the mother-liquors after crystallisation of the diols (fractions 1, 2, 3, and 4), was chromatographed and the diol fraction was obtained by crystallisation. A total of 90 g. of purified diols was obtained (4% yield). The above five diols fractions were separately acetylated and distilled. Distillates of similar b. p. were combined and redistilled where necessary.

The distillation was carried out in the microspinning band fractionating column of Piros and Glover (U.S. Pat. 2,608,528; *Chem. Abs.*, 1952, **46**, 9899; supplied by Messrs. H. S. Martin and Co., Illinois, U.S.A.) after modification as follows: (1) The pot heater, because of poor heat transfer, was replaced by two separately controlled heaters, one for the pot and the other for the exposed part of the column above the pot. (2) The column heater, because of uneven heating, was replaced by two evenly wound spiral heating elements. This allowed a better control over the temperature gradient in the column. (3) The partial condenser was replaced with a hollow centrally placed cold-finger condenser, which was fashioned at the end in the shape of hollow cone drawn down at one side to form a dripper. With this device it was possible, by comparison of the drop rate from this condenser with that from the dripper at the base of the column, to maintain a fine control over the column equilibrium. (4) The receiver was replaced by an electrically warmed one of the Perkin triangle type, which could be evacuated by means of a separate vacuum pump in order to avoid disturbing the column pressure. This type of still has a relatively low hold-up and low pressure drop and is thus suited to the fractionation of small quantities of not highly stable materials. The column temperatures were measured by means of iron–constantan thermocouples and recorded on a recording potentiometer (Messrs. Brown Instrument Co., Philadelphia, U.S.A.). The manostat of Birch *et al.* (*J. Soc. Chem. Ind.*, 1947, **66**, 3340) was used to maintain a pressure of 1 mm. with a maximum variation of about 0·02 mm. or a head-temperature variation of $\ngtr$ about 0·25°.

For the distillation, the column was operated at a boil-up rate of 30—40 c.c./hr., pressure drop of 1·2—1·5 mm., and rotor speed of 1200 r.p.m. Under these conditions of operation, it had an efficiency of about 15 theoretical plates when tested with Williams's mixture (*Ind. Eng. Chem.*, 1947, **39**, 779). This is much lower than that claimed by the makers for operation at atmospheric pressure (*viz.*, 100 theor. plates). It is possible that the value is low because this test mixture does not wet glass well (cf. Benner, Dinardo, and Tobin, *Ind. Eng. Chem.*, 1951, **43**, 722; and Williamson, *J. Appl. Chem.*, 1951, **1**, 33). Comparison of our distillation curves with those of Murray (*J. Amer. Oil Chem. Soc.*, 1951, **28**, 1) suggests that our column is of roughly equal efficiency.

Isolation of the Pure 1 : 2-*Diols.*—n-*Hexadecane*-1 - 2-*diol.* A portion of the material from the C_{16} plateau (b. p. 173°/1 mm.) was saponified with alcoholic potasium hydroxide. Water was added and the diol, extracted with ether, was washed with water and dried (Na_2SO_4). After removal of the ether, the remaining *diol* was crystallised to purity from hexane and methanol, forming plates, m. p. 84—84·4°, crystal long spacing 36·7 Å (Found: C, 74·4; H, 13·4%; *M*, 261. $C_{16}H_{34}O_2$ requires C, 74·4; H, 13·3%; *M*, 258).

The molecular weight was determined by the volumetric method of Karnovsky and Rapson (*loc. cit.*). The m. p. was not depressed on admixture with an authentic sample of hexadecane-1 : 2-diol, m. p. 84—84·4°, prepared from (−)-*n*-2-hydroxyhexadecanoic acid by reduction with lithium aluminium hydride (Horn, Hougen, and von Rudloff, *Chem. and Ind.*, *loc. cit.*).

n-*Pentadecanoic acid.* To hexadecane-1 : 2-diol (1 g.) in dry benzene at 50—60° was added slowly a solution of lead tetra-acetate (4 g.; Mendel, *Rec. Trav. chim.*, 1940, **59**, 720) in dry benzene (100 c.c.) and acetic acid (a few drops). A stream of air was passed through the mixture during the addition, and for 2 hr. thereafter. The mixture was then poured into an equal volume of dilute sulphuric acid, shaken, and centrifuged. The benzene layer was extracted several times with concentrated sodium hydroxide solution. The combined aqueous layers were extracted with ether, and acidified, and the liberated acid was taken up in ether. The ethereal solution was washed with water, dried (Na_2SO_4) and evaporated. The acid crystallised from pentane in plates, m. p. 52—52·2° (0·75 g.) (Found: C, 74·1; H, 12·6%; equiv., 242. Calc. for $C_{15}H_{30}O_2$: C, 74·3; H, 12·5%; equiv., 242·4). There was no m. p. depression on admixture with an authentic sample of pentadecanoic acid prepared from (±)-*n*-2-hydroxyhexadecanoic acid (Mendel and Coops, *Rec. Trav. chim.*, 1939, **58**, 1140), m. p. 52·2—52·5° (Found: C, 74·5; H, 12·7%; equiv., 243). Francis, Piper, and Malkin (*Proc. Roy. Soc.*, 1930, **128**, *A*, 214) record m. p. 52·1°.

1 : 2-*Diacetoxy*-16-*methylheptadecane.* The *diacetate* from the C_{18} plateau (b. p. 186·5°/1 mm.) crystallised from methanol at −20° as needles, m. p. 16—17°, $[\alpha]_D^{20} = +0{\cdot}012°$ (no solvent) (Found: C, 71·2; H, 11·6; Ac, 23·1. $C_{22}H_{42}O_4$ requires C, 71·3; H, 11·4; 2Ac, 23·2%).

16-*Methylheptadecane*-1 : 2-*diol.* 1 : 2-Diacetoxy-16-methylheptadecane was saponified and the *diol* crystallised from methanol and hexane as plates m. p. 75—75·4°, crystal long spacing 40·6 Å (Found: C, 75·8; H, 13·4%; *M*, 288. $C_{18}H_{38}O_2$ requires C, 75·5; H, 13·4%; *M*, 287). It showed no m. p. depression on admixture with 16-methylheptadecane-1 : 2-diol obtained by lithium aluminium anhydride reduction of (−)-16-methyl-2-hydroxyheptadecanoic acid (Horn, Hougen, and von Rudloff, unpublished work).

15-*Methylhexadecanoic acid.* 16-Methylheptadecane-1 : 2-diol was oxidised with lead tetra-acetate as described above. The acid was crystallised to purity from acetone, hexane, and methanol, forming plates, m. p. 60·1—60·5°, crystal long spacing 33·3 Å (Ställberg, Stenhagen, *et al.*, *Arkiv Kemi, Min., Geol.*, 1948, **26**, *A*, No. 19, give 33·35 Å) (Found: C, 75·6; H, 12·7%; equiv., 271. Calc. for $C_{17}H_{34}O_2$: C, 75·5; H, 12·7%; equiv., 270). The m. p. showed no depression on admixture with an authentic specimen of 15-methylhexadecanoic acid, m. p. 60·3—60·5° (*idem, ibid.*).

1 : 2-*Diacetoxy*-18-*methylnonadecane.* The *diacetate* from the C_{20} plateau (b. p. 203°/1 mm.) crystallised from methanol as needles, m. p. 26—27° (Found: C, 72·6; H, 11·7; Ac, 21·9. $C_{24}H_{46}O_4$ requires C, 72·3; H, 11·6; Ac, 21·6%).

18-*Methylnonadecane*-1 : 2-*diol.* The foregoing diacetate was saponified. The *diol* crystallised from hexane and methanol as plates, m. p. 80·8—81·1°, crystal long spacing 44·5 Å (Found: C, 76·6; H, 13·6%; *M*, 322. $C_{20}H_{42}O_2$ requires C, 76·4; H, 13·5%; *M*, 315).

17-*Methyloctadecanoic acid.* 18-Methylnonadecane-1 : 2-diol was oxidised with lead tetra-acetate. The acid so obtained was crystallised to purity from hexane and acetone, giving plates, m. p. 66·6—66·8°, crystal long spacing 36·8 Å (Velick, *J. Amer. Chem. Soc.*, 1947, **69**, 2317, gives 36·2 Å) (Found: C, 76·5; H, 13·0%; equiv., 295. Calc. for $C_{19}H_{38}O_2$: C, 76·5; H, 12·8%; equiv., 298). The m. p. showed no depression on admixture with 17-methyloctadecanoic acid, m. p. 66·9—67·3°, kindly supplied by Prof. Cason.

1 : 2-*Diacetoxy*-20-*methylheneicosane.* The *diacetate*, b. p. 217—219°/1 mm., crystallised

from methanol as needles, m. p. 33·3—33·8° (Found : C, 73·2; H, 11·9; Ac, 20·4. $C_{26}H_{50}O_4$ requires C, 73·2; H, 11·8; Ac, 20·2%).

20-*Methylheneicosane*-1 : 2-*diol*. The foregoing diacetate was saponified. The *diol* crystallised from hexane and methanol as plates, m. p. 84·0—84·4°, crystal long spacing 48·6 Å (Found : C, 77·1; H, 13·6%; *M*, 342. $C_{22}H_{46}O_2$ requires C, 77·1; H, 13·5%; *M*, 343).

Synthesis of 19-*methyleicosanoic acid*. 18-Methylnonadecanoic acid (Part III, *loc. cit.*), reduced with lithium aluminium hydride in the usual way, yielded 18-methylnonadecanol, plates (from acetone and hexane), m. p. 50—50·3° (Found : C, 80·5; H, 14·4. Calc. for $C_{20}H_{42}O$: C, 80·5; H, 14·2%). Murray and Schoenfeld (*J. Amer. Oil Chem. Soc.*, 1952, **29**, 416) record m. p. 50·3° for 18-methylnonadecanol obtained from wool wax.

18-Methylnonadecanol (4 g.), iodine (2 g.), and red phosphorus (0·16 g.) were heated for 5 hr. at 145—150° with occasional shaking. An ethereal extract of the mixture was then washed free from acid, dried ($CaCl_2$), and evaporated, to yield 18-methylnonadecyl iodide (5·3 g.), plates (from acetone), m. p. 41—43°. A solution of this (4·8 g.) and potassium cyanide (1·6 g.) in 90% ethanol (80 c.c.) was refluxed for 24 hr. Potassium hydroxide (10 g.) in water (10 c.c.) was then added and the solution refluxed for 100 hr. The mixture, after dilution with water, was extracted at 50° with hexane. The alcoholic layer was acidified with hydrochloric acid, boiled for 10 min. and then extracted with three portions of hexane at 50°. The combined hexane extracts were washed with hot water, dried (Na_2SO_4), and concentrated to 50 c.c. The 19-*methyleicosanoic acid* then crystallised as plates (2·9 g.). Recrystallised from acetone, it had m. p. 73—73·3°, crystal long spacing 40·2 Å (Found : C, 77·2; H, 13·2%; equiv., 326. $C_{21}H_{42}O_2$ requires C, 77·2; H, 13·0%; equiv., 327).

The acid obtained from 20-methylheneicosane-1 : 2-diol by oxidation with lead tetra-acetate, crystallised to purity from acetone and hexane, formed plates, m. p. and mixed m. p. 73—73·4°, crystal long spacing 40·4 Å (Found : C, 77·3; H, 12·9%; equiv., 325).

1 : 2-*Diacetoxy*-22-*methyltricosane*. The *diacetate*, b. p. 228—230°/1 mm., crystallised from methanol as needles, m. p. 37·5—38·5° (Found: C, 74·1; H, 12·0; Ac, 19. $C_{28}H_{54}O_4$ requires C, 74·0; H, 12·0; Ac, 18·9%).

22-*Methyltricosane*-1 : 2-*diol*. 1 : 2-Diacetoxy-22-methyltricosane was saponified and the *diol* crystallised from hexane and then methanol formed plates, m. p. 86·6—97°, crystal long spacing 52·3 Å (Found : C, 78·1; H, 13·6%; *M*, 360. $C_{24}H_{50}O_2$ requires C, 77·8; H, 13·6%; *M*, 371).

Synthesis of 21-*methyldocosanoic acid*. 18-Methylnonadecanoic acid (5·5 g., 0·0175 mole), ethyl hydrogen glutarate (4·4 g., 0·027 mole), and sodium (0·02 g., 0·0008 g. atom) were electrolysed, to yield 21-*methyldocosanoic acid* (0·75 g.), plates (from acetone and ethanol), m. p. 77·9—78·1°, crystal long spacing 43·9 Å (Found : C, 78·0; H, 13·2%; equiv., 353. $C_{23}H_{46}O_2$ requires C, 77·9; H, 13·1%; equiv., 355).

21-Methyldocosanoic acid, obtained from 22-methyltricosane-1 : 2-diol by oxidation with lead tetra-acetate and crystallised to purity from acetone and hexane, formed plates, m. p. and mixed m. p. 76—76·6°, crystal long spacing 43·9 Å (Found : C, 77·9; H, 13·0%; equiv., 354).

Octadec-1-*ene*. Octadecyl alcohol (27 g., 0·1 mole) and palmitic acid (30 g., 0·114 mole) were heated together at 240—260° until evolution of water had ceased (*ca.* 1 hr.), and then distilled (b. p. 350—360°/600 mm.). The crude octadecene was separated from the bulk of the palmitic acid by distillation at atmospheric pressure through a " Towers 10-plate " fractionating column, then fractionated in the spinning-band column, and the material of constant b. p. and refractive index was collected (8·8 g.; b. p. 129°/1 mm., n_D^{30} 1·4408 ± 0·0001. Cf. Wibaut, *Rec. Trav. chim.*, 1948, **67**, 113).

(±)-n-1 : 2-*Diacetoxyoctadecane*. The method of Swern, Billen, and Scanlan (*loc. cit.*) was modified as follows : To a slowly stirred mixture of octadec-1-ene (16·3 g.) and formic acid (155 c.c.; 98—100%) at room temperature was added hydrogen peroxide (9·9 g. of 30% hydrogen peroxide, *i.e.*, 30% excess). The mixture was heated at 40° with slow stirring for 20 hr. The excess of formic acid was taken off under reduced pressure and the residue refluxed with potassium hydroxide (25 g.) in ethanol (200 c.c.) for 1 hr. The mixture was evaporated to small volume under reduced pressure, diluted with water, and extracted with ether. The ethereal layer was washed with water, dried (Na_2SO_4), and evaporated. The residue crystallised from pentane, to yield the crude diol (14·3 g.; m. p. 78·5—80·5°). It was dissolved in acetic anhydride (50 c.c.) and heated on a water-bath for 5 hr. The excess of acetic anhydride was removed and the residue fractionated at 1 mm. in the spinning-band column. The material of constant b. p. and refractive index (b. p. 191·5° ± 0·5°, n_D^{30} 1·4419 ± 0·0001) crystallised from methanol in needles, m. p. 39·5—40°, after resolidification m. p. 41·8—42·8° (Found : C, 71·5; H, 11·6; Ac, 23·3. Calc. for $C_{22}H_{42}O_4$: C, 71·3; H, 11·4; Ac, 23·2%). Niemann and Wagner (*J. Org. Chem.*, 1942, **7**, 227) record m. p. 40·0° for (±)-1 : 2-diacetoxyoctadecane.

(±)-*Octadecane*-1 : 2-*diol.* Pure (±)-1 : 2-diacetoxyoctadecane was saponified and the diol crystallised from methanol as a colourless powder, m. p. 81—81·8°, crystal long spacing 40·4 Å (Found: C, 75·6; H, 13·5%; *M*, 285. Calc. for $C_{18}H_{38}O_2$: C, 75·5; H, 13·4%; *M*, 287). Swern *et al.* (*J. Amer. Chem. Soc.*, 1946, **68**, 1505) record m. p. 80—81° and Niemann and Wagner (*loc. cit.*), m. p. 79·0—79·5° for (±)-octadecane-1 : 2-diol.

The authors thank Drs. W. S. Rapson and D. A. Sutton for many helpful discussions, Mr. F. W. G. Schöning for the microanalyses, Dr. J. N. van Niekerk and Mr. F. R. L. Schöning for the *X*-ray measurements, Mr. C. A. J. Hoeve for the infra-red absorption spectra, and Prof. Cason for a sample of 17-methyloctadecanoic acid. This paper is published by permission of the South African Council for Scientific and Industrial Research. Acknowledgment is made of support from the South African Wool Board.

National Chemical Research Laboratory,
South African Council for Scientific and Industrial Research,
Pretoria, South Africa.

[*Received, June* 15*th*, 1953.]