415. *Triterpenes of the Friedelane Series. Part II.* Hydroxy-ketones and Alcohols.*

By J. L. COURTNEY, R. M. GASCOIGNE, and A. Z. SZUMER.

Three hydroxy-ketones and two hydroxy-diketones have been isolated from the triterpene mixture from *Siphonodon australe* Benth. These compounds have been related to the ketones described in Part I and, where possible, configurations have been assigned to the hydroxyl groups. The five compounds can consequently be named *x*[eq]- and *x*[ax]-hydroxyfriedelan-3-one, *y*-hydroxyfriedelan-3-one, *x*[eq]-hydroxyfriedelane-3:*y*-dione, and *y*-hydroxyfriedelane-3 : *x*-dione.

Reduction of friedelan-*x*-one by two different methods gives two epimeric alcohols, but reduction of friedelan-*y*-one by the same two methods gives only one alcohol.

Molecular-rotation differences observed with these compounds support the absolute configuration assigned to friedelane.[1] This configuration is a partial " skeletal enantiomorph " of the 5α-steroids and the usual triterpenes; consequently reduction of 3-oxo-derivatives with sodium borohydride yields mainly axial (3β-)alcohols.

THREE hydroxy-ketones and two hydroxy-diketones have been isolated from the triterpene mixture from *Siphonodon australe* Benth. and, as described in the sequel, have been related

* Part I, preceding paper.

[1] (*a*) Corey and Ursprung, *J. Amer. Chem. Soc.*, 1955, **77**, 3667, 3668; (*b*) Brownlie, Spring, Stevenson, and Strachan, *Chem. and Ind.*, 1955, 686, 1156; (*c*) Ourisson and Takahashi, *ibid.*, p. 1155; (*d*) Dutler, Jeger, and Ruzicka, *Helv. Chim. Acta*, 1955, **38**, 1268.

to the ketones discussed in Part I.* It may be of biological significance that the nine friedelane derivatives so far isolated from this source all contain the 3-oxo-group.

To provide reference compounds for this investigation friedelan-*x*-one and friedelan-*y*-one were reduced with sodium and pentyl alcohol and also with lithium aluminium hydride. Friedelan-*x*-one yielded two different alcohols, one by each method. The alcohol obtained by reduction with sodium and pentyl alcohol must, from its method of preparation, be the more stable of the two epimers and can consequently be assumed to have the equatorial configuration. It is therefore friedelan-*x*[eq]-ol, and the alcohol obtained by reduction with lithium aluminium hydride is friedelan-*x*[ax]-ol. (It should be noted, however, that in some special cases the more stable of a pair of epimers has the axial configuration; see below.) In the case of friedelan-*y*-one by contrast, both methods of reduction gave the same alcohol, friedelan-*y*-ol. Such a result, with a hindered ketone, is unusual but an analogy is provided in the reduction of the very hindered 18α-oleanan-19-one which yielded 18α-oleanan-19β[ax]-ol by both methods of reduction;[2] in this case the axial epimer is more stable than the equatorial epimer.[2] Consequently we do not yet assign any configuration to friedelan-*y*-ol; it happens that it is not necessary to do so for present purposes.

Friedelan-*x*-one, friedelan-*y*-one, and evidently also 18α-oleanan-19-one were readily reduced by sodium and pentyl alcohol in spite of their very hindered nature. 11-Oxosteroids are also reduced by sodium and alcohols [3] and it appears that this method of reduction is not appreciably affected by steric hindrance. As is well known, the stereochemical course of the reduction is determined by thermodynamic rather than by kinetic factors.

Of the three hydroxy-ketones isolated from *Siphonodon australe* one is identical with *y*-hydroxyfriedelan-3-one, described in Part I,[4] whilst the other two on oxidation yielded friedelane-3 : *x*-dione. Since these two compounds are not identical with the epimeric 3-hydroxyfriedelan-*x*-ones described in Part I they must be the epimeric *x*-hydroxyfriedelan-3-ones. Both formed semicarbazones thus confirming the presence of the reactive 3-oxo-group. To complete the identification of these compounds it was necessary to determine which of them was the equatorial and which the axial epimer. Accordingly one of them was reduced by the Huang-Minlon procedure; it yielded friedelan-*x*[ax]-ol and consequently must be *x*[ax]-hydroxyfriedelan-3-one. The possibility of epimerisation during the reduction is eliminated since the product was the less stable of the epimeric friedelan-*x*-ols.

The two hydroxy-diketones on oxidation gave friedelane-3 : *x* : *y*-trione and both formed semicarbazones, indicating the presence of the 3-oxo-group. One of them, when reduced by the Wolff–Kishner method (anhydrous hydrazine), yielded friedelan-*x*[eq]-ol and is therefore *x*[eq]-hydroxyfriedelane-3 : *y*-dione. This conversion also provides proof for the presence of the *x*-oxo-group in friedelane-3 : *x* : *y*-trione. For the other hydroxy-diketone three possibilities remain : *x*[ax]-hydroxyfriedelane-3 : *y*-dione and the two epimers of *y*-hydroxyfriedelane-3 : *x*-dione. The first of these can be eliminated by molecular rotations : the observed $[M]_D$ is +498° whereas the calculated $[M]_D$ for *x*[ax]-hydroxyfriedelane-3 : *y*-dione is −249° {the sum of the observed $[M]_D$ of friedelane-3 : *y*-dione (−273°) and the ΔOH value for the *x*[ax]-hydroxyl group (+24°; see Table 1)}. The compound is named *y*-hydroxyfriedelane-3 : *x*-dione since its molecular rotation differences (Table 1) indicate that the hydroxyl group has the same configuration as in the other *y*-hydroxy-compounds.

The molecular-rotation differences set out in Table 1 provide supporting evidence for the names assigned to the various compounds, and further confirmation is available from the molecular-rotation contributions of the carbonyl groups (*e.g.*, the 3-oxo-group; Table 2) and from the infrared spectra which will be discussed in a later paper.

The molecular-rotation differences also support the absolute configuration (I) deduced for the friedelane structure.[1] Klyne [5] has shown that in polycyclic compounds such as

[2] Ames, Beton, Halsall, and Jones, *J.*, 1954, 1905.
[3] Heusser, Anliker, and Jeger, *Helv. Chim. Acta*, 1952, **35**, 1537; Herzog, Jevnik, and Hershberg, *J. Amer. Chem. Soc.*, 1953, **75**, 269; Barnes, Barton, Cole, Fawcett, and Thomas, *J.*, 1953, 571.
[4] Courtney and Gascoigne, preceding paper.
[5] Klyne, *J.*, 1952, 2916.

steroids and triterpenes two enantiomorphic forms of a terminal ring have rotational contributions of opposite sign, often of the same order of magnitude. On the basis of formula (I), if methyl groups are neglected (an admissible procedure [5]), friedelane is enantiomorphic with respect to ring A (also rings B and C) with androstane (II) and with

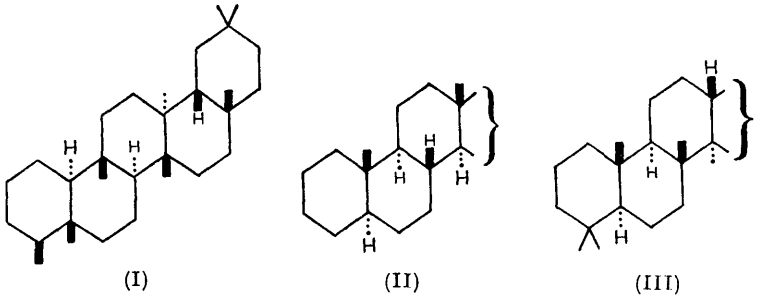
TABLE 1. *Molecular-rotation contributions of hydroxyl and acetoxyl groups.*

Symbols and conventions are as used by Klyne and Stokes (*J.*, 1954, 1979).

	$[M]_D$ CH_2 *	$[M]_D$ OH	$[M]_D$ OAc	ΔOH	ΔOAc	$[M]_D$OAc $-[M]_D$OH
3-Hydroxyl group (equatorial)						
Friedelan-3α-ol †	+91°	+69°	−99°	−22°	−190°	−168°
3α-Hydroxyfriedelan-*x*-one *	+647	+641	+552	−6	−95	−89
3-Hydroxyl group (axial)						
Friedelan-3β-ol ‡	+91	+90	+169	−1	+78	+79
3β-Hydroxyfriedelan-*x*-one *	+647	+704	+764	+57	+117	+60
3β-Hydroxyfriedelane-*x* : *y*-dione	+453	+489	+564	+36	+111	+75
3β-Hydroxyfriedelan-*y*-one	−145	−142	−97	+3	+48	+45
x-Hydroxyl group (equatorial)						
Friedelan-*x*[eq]-ol	+91	+51	+24	−40	−67	−27
x[eq]-Hydroxyfriedelan-3-one	−94	−142	−193	−48	−99	−51
x[eq]-Hydroxyfriedelane-3 : *y*-dione	−273	−328	−344	−55	−71	−16
x-Hydroxyl group (axial)						
Friedelan-*x*[ax]-ol	+91	+111	+113	+20	+22	+2
x[ax]-Hydroxyfriedelan-3-one	−94	−66	−58	+28	+36	+8
y-Hydroxyl group						
Friedelan-*y*-ol *	+91	+90	+61	−1	−30	−29
y-Hydroxyfriedelan-3-one *	−94	−89	−121	+5	−27	−32
y-Hydroxyfriedelane-3 : *x*-dione	+506	+498	+439	−8	−67	−59

* Compounds described in Part I.[4]
† Friedelinol; the constants are given in the Experimental section.
‡ *epi*Friedelinol; our constants (Experimental) are in agreement with those of Bruun and Jefferies (*Acta Chem. Scand.*, 1954, **8**, 1948).

oleanane, ursane, and lupane (III). Consequently the rotational contribution of the 3-oxo-group in the friedelane series should be opposite in sign to the contribution of this group in the various series represented by (II) and (III). We have determined the rotational contribution of the 3-oxo-group with ten sets of friedelane derivatives [six in this paper (Table 2) and four in Part I [4]] and the average value is −170°. Klyne [5] records the



(I) (II) (III)

rotational contribution of the 3-oxo-group in several triterpenes of the oleanane, ursane, and lupane series; the values range from +64° to +144°. For steroids of the 5α-series the average value [6] is +71°. Similarly Dutler, Jeger, and Ruzicka [1d] have observed that a molecular-rotation difference for an A-nor-ketone derived from friedelin is negative whereas for an analogous compound from lanosterol it is positive.

The conclusion that, in ring A, friedelane is a "skeletal enantiomorph" of the 5α-steroids and the usual triterpenes rationalises the stereochemical course of reduction of its 3-oxo-derivatives. We have had occasion to reduce four such derivatives (friedelin,

[6] Barton and Klyne, *Chem. and Ind.*, 1948, 755.

friedelane-3 : *x*-dione, friedelane-3 : *y*-dione, and friedelane-3 : *x* : *y*-trione) with sodium borohydride and in all cases the axial (3β-)alcohol was formed in predominant amount; in the last two cases the equatorial (3α-)alcohol was not detected. It is well established that reduction of the 3-oxo-group in 5α-steroids and in triterpenes of the type (III) with

TABLE 2. *Molecular-rotation contribution of the 3-oxo-group.*

	$[M]_D$ with 3-oxo-group	$[M]_D$ without 3-oxo-group	ΔCO
x[eq]-Hydroxyfriedelan-3-one	−142°	+51°	−193°
x[eq]-Acetoxyfriedelan-3-one	−193	+24	−217
x[ax]-Hydroxyfriedelan-3-one	−66	+111	−177
x[ax]-Acetoxyfriedelan-3-one	−58	+113	−171
y-Hydroxyfriedelan-3-one	−89	+90	−179
y-Acetoxyfriedelan-3-one	−121	+61	−182

sodium borohydride (or lithium aluminium hydride) yields mainly the equatorial (3β-)-alcohol [7] and it seems likely, at least with the triterpenes, that the β-orientation of the hydroxyl group in the reaction product is determined by rear (α) attack of the reagent.[8] This would be the determining factor also in the friedelan-3-one series, again causing β-orientation in the reaction product. That friedelan-3β-ol and its congeners have the axial rather than the equatorial configuration is a consequence of the " skeletal enantiomorphic " nature of friedelane * and is presumably not relevant to the course of the reaction. Reduction of friedelan-3-one with lithium aluminium hydride [1a,b] and by hydrogenation (in acetic acid solution) [9] also yields friedelan-3β-ol.

The 3-oxo-group is somewhat more hindered in the friedelane series than in the usual triterpenes : thus 3-ketones of the latter type give a positive Zimmermann colour test [10] whereas friedelan-3-one does not; it does, however, form a semicarbazone. It is possible, but seems unlikely, that the course of reduction is not determined by rear attack in the case of the usual triterpene 3-ketones but is so determined in the case of the friedelan-3-ones.

EXPERIMENTAL

Analyses by Dr. E. Challen and Mr. D. Weedon. Infrared spectra by Mr. I. Reece.

General experimental conditions are given in Part I.[4] Fraction numbers refer to the chromatogram of the triterpene mixture described in Part I.

x[*ax*]-*Hydroxyfriedelan-3-one*.—By virtue of its low solubility in alcohol, x[*ax*]-*hydroxyfriedelan-3-one* (2·3 g.) was isolated from fractions 30—35. It crystallised from benzene in tetrahedra, m. p. 272—275°, $[\alpha]_D$ −15° (*c* 2·2) (Found : C, 81·5; H, 11·3. $C_{30}H_{50}O_2$ requires C, 81·4; H, 11·4%). The *acetate* formed needles (from alcohol), m. p. 265—270°, $[\alpha]_D$ −12° (*c* 2·9) (Found : C, 79·3; H, 10·8. $C_{32}H_{52}O_3$ requires C, 79·3; H, 10·8%). The *semicarbazone* formed laths (from alcohol–benzene), m. p. 322—327° (decomp.; frothing at 281—284°) (Found : C, 72·7; H, 10·9; N, 7·9. $C_{31}H_{53}O_2N_3,C_2H_5{\cdot}OH$ requires C, 72·6; H, 10·9; N, 7·7%).

When oxidised with chromium trioxide in pyridine it yielded friedelane-3 : *x*-dione, m. p. 250—252°, $[\alpha]_D$ +116° (*c* 2·5).

x[*eq*]-*Hydroxyfriedelan-3-one*.—Fractions 36—39 were combined with the alcohol-soluble material from fractions 40—43 and recrystallised repeatedly from alcohol, yielding x[*eq*]-*hydroxyfriedelan-3-one*, needles, m. p. 264—268°, $[\alpha]_D$ −32° (*c* 2·6) (Found : C, 81·2; H, 11·25%). An additional amount was obtained by chromatography of the material from the mother-liquors (total, 3·8 g.). The *acetate* formed needles (from alcohol), m. p. 236—238°, $[\alpha]_D$ −40° (*c* 2·2) (Found : C, 79·1; H, 10·8%). The *semicarbazone* formed needles (from aqueous alcohol),

* If bonds at corresponding carbon atoms in the two " skeletal enantiomorphs " are to have the same orientation (α or β) they must necessarily have different configurations (axial or equatorial). Orientation, in the sense used here, does not necessarily have any absolute significance but is defined by the direction of the exocyclic bond at $C_{(10)}$ which is α in the friedelane series [1a] and β in the series represented by structures (II) and (III).

[7] Cf. Barton, *J.*, 1953, 1027; Halsall and Jones and their co-workers, *J.*, 1953, 2548, 3019; Cooley, Ellis, and Petrow, *J.*, 1955, 2998.

[8] Cf. Cram and Elhafez, *J. Amer. Chem. Soc.*, 1952, **74**, 5828; Noyce and Denney, *ibid.*, 1950, **72**, 5743; Shoppee and Summers, *J.*, 1950, 687.

[9] Jefferies, *J.*, 1954, 473.

[10] Barton and de Mayo, *J.*, 1954, 887.

m. p. 259—262° (decomp.) (Found : C, 74·7; H, 10·7; N, 8·2. $C_{31}H_{53}O_2N_3$ requires C, 74·5; H, 10·7; N, 8·4%).

When oxidised with chromium trioxide in pyridine it yielded friedelane-3 : *x*-dione, m. p. 248—252°, $[\alpha]_D$ +114° (*c* 1·8).

y-*Hydroxyfriedelan*-3-*one*.—This compound was not detected in the present separation but was obtained in small amount in the separation of another batch of the triterpene mixture. It was eluted from alumina after *x*[eq]-hydroxyfriedelan-3-one and crystallised from benzene in laths, m. p. 305—308°, $[\alpha]_D$ −20° (*c* 0·5; 2 dm. tube). It was identical (infrared spectra and mixed m. p.) with the product prepared from friedelane-3 : *x* : *y*-trione (Part I).[4]

x[*eq*]-*Hydroxyfriedelane*-3 : y-*dione*.—By virtue of its low solubility in alcohol x[*eq*]-*hydroxyfriedelane*-3 : y-*dione* (7 g.) was isolated from fractions 40—44. It crystallised from benzene in prisms, m. p. 289—292°, $[\alpha]_D$ −72° (*c* 2·0) (Found : C, 79·0; H, 10·5. $C_{30}H_{48}O_3$ requires C, 78·9; H, 10·6%). The *acetate* formed needles (from alcohol), m. p. 276—278°, $[\alpha]_D$ −69° (*c* 1·2) (Found : C, 76·8; H, 10·0. $C_{32}H_{50}O_4$ requires C, 77·1; H, 10·1%). The *monosemicarbazone* crystallised from alcohol in plates, m. p. 282° (decomp.) (Found : C, 70·7; H, 9·95; N, 7·9. $C_{31}H_{51}O_3N_3,C_2H_5{\cdot}OH$ requires C, 70·8; H, 10·25; N, 7·5%).

When oxidised with chromium trioxide in pyridine it yielded friedelane-3 : *x* : *y*-trione, m. p. 300—303°, $[\alpha]_D$ +70° (*c* 1·2).

y-*Hydroxyfriedelane*-3 : x-*dione*.—This compound could not be obtained pure in the present separation although it was present in considerable amount in fractions 46—53. From chromatography of a previous batch of the triterpene mixture (which had been extracted from the bark with light petroleum and probably did not contain the polyhydroxy-triterpenes which interfere with the purification) a fraction was obtained with $[\alpha]_D$ +94° (corresponding in position to fractions 46—53 in the present chromatogram). Rechromatography of this material (1·4 g.) over alumina (100 g.) and recrystallisation of the higher fractions from alcohol yielded y-*hydroxyfriedelane*-3 : x-*dione* in needles, m. p. 304—310° (decomp.), $[\alpha]_D$ +109° (*c* 2·4) (Found : C, 78·9; H, 10·6%). The *acetate* formed rods (from methyl alcohol), m. p. 205—207°, $[\alpha]_D$ +88° (*c* 1·6) (Found : C, 77·1; H, 9·8%). The *monosemicarbazone* was obtained microcrystalline from aqueous alcohol, with m. p. 270° (decomp.) (Found : C, 70·5; H, 10·1; N, 7·9. $C_{31}H_{51}O_3N_3,C_2H_5{\cdot}OH$ requires C, 70·8; H, 10·25; N, 7·5%).

When oxidised with chromium trioxide in pyridine it yielded friedelane-3 : *x* : *y*-trione, m. p. 300—303°, $[\alpha]_D$ +65° (*c* 0·7; 2 dm. tube).

Reduction of Friedelan-x-*one*.—(*a*) Sodium (2 g.) was added to a boiling solution of friedelan-*x*-one (0·5 g.) in pentyl alcohol (25 ml.); when the reaction was complete the alcohol was removed with steam. The product was chromatographed over alumina (50 g.) from solution in 3 : 1 light petroleum–benzene. Elution with benzene yielded *friedelan*-x[*eq*]-*ol* (0·4 g.), needles (from *cyclo*hexane), m. p. 261—264°, $[\alpha]_D$ +12° (*c* 0·8; 2 dm. tube) [Found (after vacuum-sublimation) : C, 84·3; H, 12·3. $C_{30}H_{52}O$ requires C, 84·05; H, 12·2%]. The *acetate* formed prisms (from alcohol–chloroform), m. p. 239—242°, $[\alpha]_D$ +5° (*c* 1·1) (Found : C, 81·8; H, 11·6. $C_{32}H_{54}O_2$ requires C, 81·6; H, 11·6%).

(*b*) A solution of friedelan-*x*-one (0·7 g.) in benzene (25 ml.) was added to a suspension of lithium aluminium hydride (2 g.) in ether (60 ml.), and the boiling mixture was stirred under reflux for 8 hr. and then poured into ice-cold 3N-sulphuric acid. Recrystallisation of the product from alcohol–benzene yielded *friedelan*-x[*ax*]-*ol*, plates, m. p. 276—279°, $[\alpha]_D$ +26° (*c* 1·4) (Found : C, 83·8; H, 12·0%). The *acetate* formed plates, m. p. 258—261°, $[\alpha]_D$ +24° (*c* 0·6; 2 dm. tube), from alcohol–benzene (Found : C, 81·9; H, 11·7%).

Reduction of Friedelan-y-*one*.—(*a*) Reduction of friedelan-*y*-one (1 g.) with sodium and pentyl alcohol and purification of the product as described above yielded friedelan-*y*-ol (0·75 g.), m. p. 224—226°, $[\alpha]_D$ +23° (*c* 1·4).

(*b*) Reduction with lithium aluminium hydride as described above also yielded friedelan-*y*-ol, m. p. 224—226°, $[\alpha]_D$ +21° (*c* 1·6).

Huang-Minlon Reduction of x[*ax*]-*Hydroxyfriedelan*-3-*one*.—A solution of *x*[ax]-hydroxyfriedelan-3-one (2·15 g.), potassium hydroxide (1·4 g.), and hydrazine hydrate (4 ml. of 100%) in diethylene glycol (20 ml.) was refluxed for 1½ hr. Hydrazine hydrate was then removed by distillation and the mixture was kept at 200° for 4 hr. The product, isolated from the acidified mixture with chloroform, was dissolved in 3 : 1 light petroleum–benzene and chromatographed over alumina (60 g.). Elution with the same solvent gave friedelan-*x*[ax]-ol (1·7 g.), m. p. 276—279°, $[\alpha]_D$ +26° (*c* 1·1).

Wolff–Kishner Reduction of x[*eq*]-*Hydroxyfriedelane*-3 : y-*dione*.—A mixture of *x*[eq]-hydroxyfriedelane-3 : *y*-dione (2 g.), anhydrous hydrazine (13 ml.), and a solution of sodium

(4·9 g.) in methyl alcohol (70 ml.) was heated at 200° for 16 hr. The product, isolated with chloroform after the mixture had been acidified, was chromatographed in 3 : 1 light petroleum–benzene over alumina (75 g.). Elution with benzene yielded friedelan-*x*[eq]-ol (1·7 g.), m. p. 260—262°, $[\alpha]_D$ +10° (*c* 1·2).

Reduction of Friedelin with Sodium Borohydride.—Friedelin (7 g.) was dissolved in hot pyridine (250 ml.), and the cooled solution was added to a solution of sodium borohydride (2·1 g.) in methyl alcohol (150 ml.) containing N-sodium hydroxide (1 ml.). The mixture was left for 2 days and then acidified. The product, isolated with chloroform, was chromatographed in benzene over alumina (700 g.) to give 8 fractions. Fractions 1 and 2 (2·0 g.) consisted entirely, and fractions 3 and 4 (1·15 g.) mainly, of friedelin. The infrared spectra of fractions 5—7 were identical; they were combined (2·64 g.) and recrystallised from benzene, yielding *epi*friedelinol, plates, m. p. 280—282°, $[\alpha]_D$ +21° (*c* 0·7; 2 dm. tube). The acetate crystallised from alcohol–benzene in plates, m. p. 290—293°, $[\alpha]_D$ +36° (*c* 1·1). The infrared spectrum of fraction 8 (0·94 g.) indicated that it consisted mainly of friedelinol, an authentic specimen of which was prepared by reduction of friedelin with sodium and pentyl alcohol, having m. p. 303—306°, $[\alpha]_D$ +16° (*c* 0·4; 2 dm. tube). The acetate had m. p. 316—318°, $[\alpha]_D$ −21° (*c* 1·1). Drake and Campbell [11] record m. p. 301—304° and for the acetate m. p. 315—316°.

Reduction of Friedelane-3 : y-dione with Sodium Borohydride.—Reduction of friedelane-3 : *y*-dione (5 g.) and chromatography of the product (8 fractions) was carried out as described for friedelin. Fractions 1—4 (2·0 g.) consisted mainly of starting material. Fractions 4—8 (2·2 g.) all had the same infrared spectra and were combined and recrystallised from alcohol, yielding 3β-*hydroxyfriedelan*-y-*one*, prisms, m. p. 319—322°, $[\alpha]_D$ −32° (*c* 1·1) (Found: C, 81·2; H, 11·3%). The *acetate* formed needles, m. p. 329—333°, $[\alpha]_D$ −20° (*c* 1·1), from alcohol–benzene (Found: C, 79·6; H, 10·9%).

Reduction of Friedelane-3 : x : y-trione with Sodium Borohydride.—The reduction and the chromatography of the product were carried out as described for friedelin. Chromatographic separation was not successful, consequently the product was fractionally crystallised from benzene and then from alcohol, yielding 3β-*hydroxyfriedelane*-x : y-*dione*, prisms, m. p. 314—318°, $[\alpha]_D$ +107° (*c* 1·1) (Found: C, 78·8; H, 10·7%). The *acetate* formed plates (from alcohol), m. p. 321—325°, $[\alpha]_D$ +113° (*c* 1·1) (Found: C, 76·9; H, 10·0%).

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[11] Drake and Campbell, *J. Amer. Chem. Soc.*, 1936, **58**, 1681.