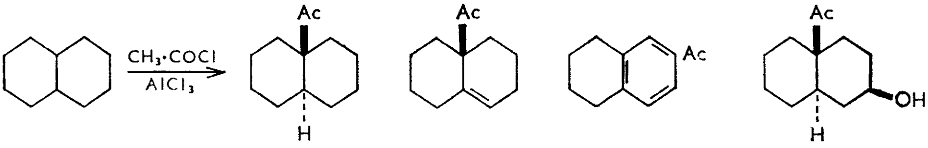
918. *The Interaction of Decalin and Friedel–Crafts Acetylating Agent. Part II.*[1]

By G. Baddeley, B. G. Heaton, and J. W. Rasburn.

The primary product of the interaction is shown to be 10β-vinyldecalin 1β,1′-oxide (V). Evidence for the *trans*-configuration of the decalin moiety will be presented in Part III. Several novel decalin derivatives are described; they include 10β-formyl-*trans*-decal-1β-ol (XIII) and the 10β-acetyl analogue (VI), which is stereospecifically reduced to 10β-1′-hydroxyethyl-*trans*-decal-1β-ol (VII) by lithium aluminium hydride, the corresponding 10β-hydroxymethyl derivative (XIV), 10-acetyl-1-decalone (XI), and 4β-hydroxy-*trans*-decalin-9β-carboxylic acid (XV) and its lactone (X).

Ozonolysis and peracid oxidation of the vinyl ether (V) are shown to give, in addition to the γ-lactone (X), the δ-lactones (IX and VIII) of 4β-hydroxy-*trans*-decalin-9β-glycollic and -9β-glyoxylic acid respectively. The latter is stereospecifically reduced to the former by potassium borohydride. The mechanism of formation of these δ-lactones is tentatively discussed.

Part I [1] was concerned with the interaction of decalin and acetyl chloride at 20—35° in the presence of an excess of aluminium chloride and showed that it gave a mixture of compounds which distilled over a wide range of temperature (115—180°/15 mm.) and included 9-acetyl-*trans*-decalin (I), 10-acetyl-$\Delta^{1(9)}$-octalin (II), 6-acetyltetralin (III), and a solid, $C_{12}H_{20}O_2$, m. p. 107—108°, which has subsequently been identified [2] as 10β-acetyl-*trans*-decal-2β-ol (IV). Recognising that the excess of aluminium chloride was probably responsible for the multiplicity of products, we have now used this reagent in amount equivalent, and sometimes less than equivalent, to that of the acid chloride. This procedure, together with a reaction temperature of 10° or lower and careful decomposition of the reaction mixture by adding it very gradually to a large excess of vigorously stirred ice–water, gave mainly an essentially pure compound, b. p. 108—112°/15 mm., which is considered to be the primary product of the interaction. It was obtained in 35—40% yield and, as shown below, is the 10β-vinyldecalin 1β,1′-oxide (V). In Part III we shall describe how the decalin moiety was shown to have the *trans*-configuration; for convenience the present formulæ anticipate this evidence.



(I) (II) (III) (IV)

(i) Analysis of the oil, b. p. 108—112°/15 mm., which hereafter will be referred to as the vinyl ether (see V), showed it to have the formula $C_{12}H_{18}O$. Unlike the acetyloctalin (II), with which it is isomeric and into which it is readily converted by the action of toluene-*p*-sulphonic acid,[2] it did not react with methylmagnesium iodide or lithium aluminium hydride; indeed, treatment with the latter reagent followed by distillation provided a ready means of separating the vinyl ether from very small amounts of the acetyloctalin (II) and 10β-acetyl-*trans*-decal-1β-ol (VI) and 10β-acetyl-*trans*-decal-2β-ol which were shown to be present by infrared analysis.

(ii) The vinyl ether has strong absorption bands at 1667 and 793 cm.$^{-1}$ which are typical

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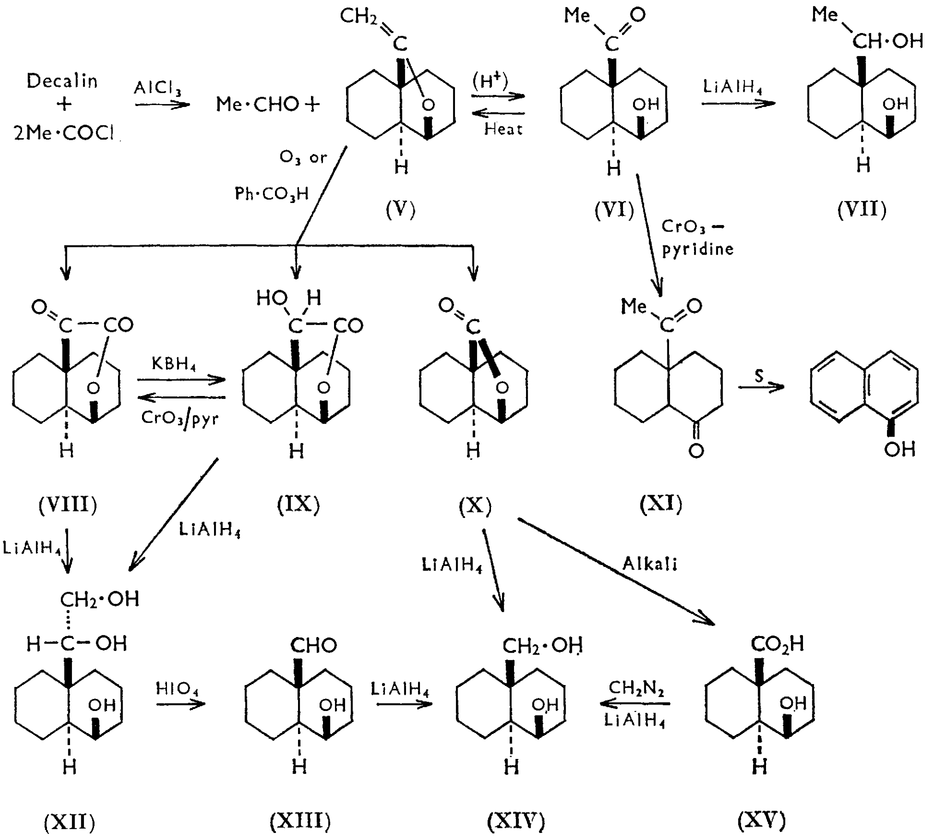
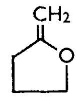
[1] Baddeley and Wrench, *J.*, 1959, 1324, is regarded as Part I.

[2] Unpublished work.

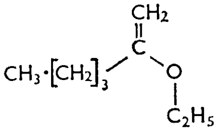
of vinyl ethers;[3] *e.g.*, these bands are present also in the spectra of tetrahydro-2-methylenefuran (XVI), 1669 and 796 cm.$^{-1}$, 2-ethoxyhex-1-ene (XVII), 1655 and 793 cm.$^{-1}$, and the product (XVIII), 1664 and 792 cm.$^{-1}$, of acid isomerisation of homolupenedione.[4]

(iii) Dehydrogenation with sulphur gave naphthalene, thereby demonstrating that the skeletal structure of decalin had been preserved.

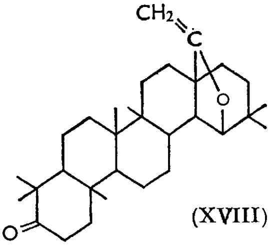




(XVI)



(XVII)



(XVIII)

(iv) In accordance with its structure, the vinyl ether was readily hydrolysed by mineral acid. Hydrolysis under very mild conditions (see Experimental section) gave a single product, $C_{12}H_{20}O_2$, m. p. 62°, which, as shown below and in Part III, is 10β-acetyl-*trans*-decal-1β-ol (VI). (*a*) It has absorption bands at 1698 cm.$^{-1}$ ($>$C=O) and 3460 cm.$^{-1}$ (–OH), and the latter is narrow, of high intensity, and independent of concentration as is required for a compound which has intramolecular hydrogen bonding. (*b*) Near 100°, or at room temperature in the presence of traces of acid, it readily evolved water and gave

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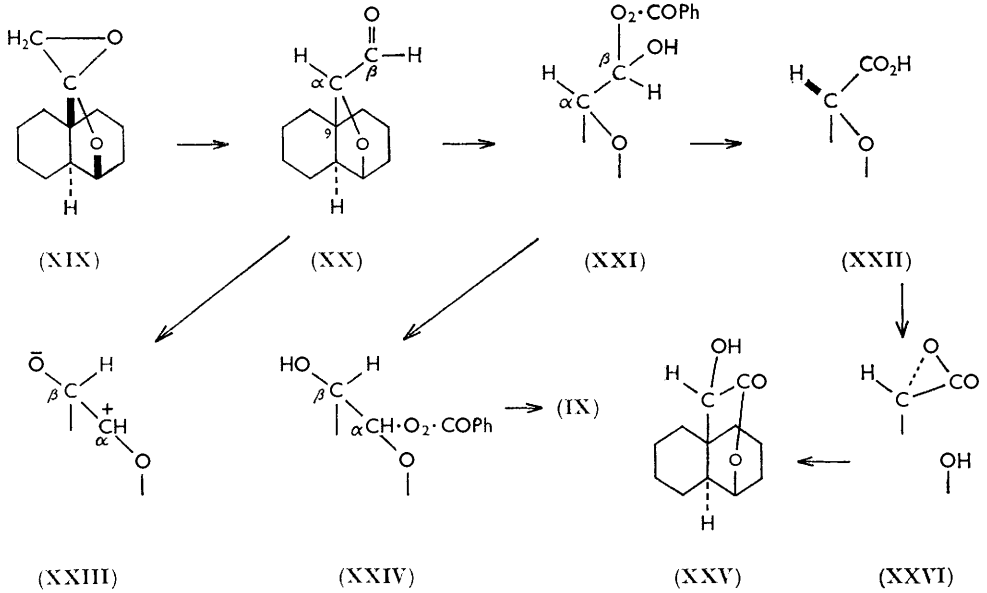
[3] Meakins, *J.*, 1953, 4170.

[4] Davy, Halsall, and Jones, *J.*, 1951, 2696.

the vinyl ether (V). (*c*) Reduction of the hydroxy-ketone (VI) with lithium aluminium hydride gave a single product, $C_{12}H_{22}O_2$, m. p. 87·5° (see VII), which (infrared spectral evidence) had free, intramolecularly hydrogen-bonded and intermolecularly hydrogen-bonded hydroxyl groups. Apparently, the reduction was stereospecific. (*d*) Oxidation of compound (VI) with chromium trioxide in pyridine gave 10-acetyl-1-decalone (XI) which has carbonyl absorption bands at 1695 cm.$^{-1}$ (acetyl) and 1713 cm.$^{-1}$ (decalone). The diketone was stable towards alkali, and gave α-naphthol when heated with sulphur.

(v) Ozonolysis of the vinyl ether in ethyl acetate or n-hexane followed by interaction with hot water gave carbon dioxide, formic acid, and three crystalline compounds, $C_{11}H_{16}O_2$, $C_{12}H_{18}O_3$, and $C_{12}H_{16}O_3$, m. p.s 27°, 120°, and 132° respectively. They have been identified as the lactones of 4β-hydroxy-*trans*-decalin-9β-carboxylic, -9β-glycollic, and -9β-glyoxylic acid (*i.e.*, X, IX, and VIII) respectively. The γ-lactone (X) was the major product. It had a strong absorption band at 1773 cm.$^{-1}$ which is typical of γ-lactones and was reduced by lithium aluminium hydride to 10β-hydroxymethyl-*trans*-decal-1β-ol (XIV) which showed strong intramolecular hydrogen bonding and gave a di-*p*-nitrobenzoate. Alkaline hydrolysis of the lactone (X) gave 4β-hydroxy-*trans*-decalin-9β-carboxylic acid (XV) which showed intramolecular hydrogen bonding, and, by melting with decomposition at 167—169°, a remarkable reluctance to lactonise. Lithium aluminium hydride did not reduce it; in this respect it resembles *trans*-decalin-9-carboxylic acid, and reduction to the diol (XIV) with this reagent was effected *via* the methyl ester which was obtained by use of diazomethane. Reactions (i)—(v) show the primary product of the interaction of decalin and acetyl chloride under Friedel–Crafts conditions to be 10β-vinyldecalin 1β,1′-oxide (V); in Part III the decalin moiety will be shown to have the *trans*-configuration. Here, further discussion is concerned only with the secondary products (VIII and IX) of the ozonolysis.



(XIX) (XX) (XXI) (XXII)

(XXIII) (XXIV) (XXV) (XXVI)

These two compounds were also obtained by oxidation of the vinyl ether with perbenzoic acid; the structures assigned to them are based on the following observations: (*a*) Both reacted with excess of lithium aluminium hydride to give the triol (XII), which gave an isopropylidene derivative and was readily oxidised with periodic acid. The product was an aldehyde and was reduced (lithium aluminium hydride) to the diol (XIV). It was, therefore, 10β-formyl-*trans*-decal-1β-ol (XIII). (*b*) Compound (IX) had an absorption band at 1736 cm.$^{-1}$ which is typical of δ-lactones, did not dissolve in aqueous sodium hydrogen carbonate solution but was readily hydrolysed by alkali; the sodium salt was isolated and re-formed the lactone (IX) on addition to dilute mineral acid. (*c*) Oxidation of compound (IX) with chromium trioxide in pyridine gave compound (VIII) from which (IX) was regenerated in almost quantitative yield by reduction with potassium borohydride. The latter reaction proceeds, therefore, stereospecifically and, since the course of the reduction is controlled kinetically and since models reveal that the line of approach of the reagent to the carbonyl group to be reduced is less hindered when it is over $C_{(1)}$ than over $C_{(6)}$ (that over $C_{(6)}$ passes between the axial hydrogen atoms of $C_{(5)}$ and $C_{(7)}$), there is little doubt that the product has the configuration shown in (IX). The configuration of the triol (XII) follows from that of compound (IX).

Should oxidation of the vinyl ether (V) with peracid follow the usual course, the resulting epoxide will, from steric considerations, have the configuration shown in (XIX). This configuration, together with the geometrical requirements of epoxide rearrangement, requires the aldehyde, if formed, to be (XX). Oxidation of the aldehyde to the corresponding carboxylic acid (XXII), *via* its addition compound (XXI) with peracid, can be expected to be followed by the rearrangements (XXII ⟶ XXVI ⟶ XXV) which give only the epimer of (IX). We suggest that the aldehyde in its preferred conformation, the carbonyl group being directed away from the decalin moiety as shown in (XX), undergoes a ring enlargement which involves migration of $C_{(9)}$ from $C_{(\alpha)}$ to $C_{(\beta)}$ (see XX ⟶ XXIII) and that subsequent interaction with peracid, or perhaps, rearrangement of the adduct (XXI), gives (XXIV) and therefrom the glycollic acid derivative (IX). The application of $CH_3\cdot{}^{14}COCl$ in the Friedel–Crafts reaction, followed by ozonolysis of the product, will show whether migration of $C_{(9)}$ from $C_{(\alpha)}$ to $C_{(\beta)}$ is involved in the formation of (IX). Our mechanism requires that carbon-14 should not be present in the diol (XIV) obtained from the triol (XII) but present, perhaps, in that from the γ-lactone (X). Ozonolysis, presumably, involves an analogous series of reactions.

EXPERIMENTAL

10β-*Vinyl*-trans-*decalin* 1β,1′-*Oxide* (V).—Acetyl chloride (940 g., 12 moles) was gradually added with stirring to a cooled (<25°) mixture of aluminium chloride (1000 g., 7·5 moles) in ethylene chloride (2250 ml.). The resulting yellow-brown solution was decanted into a flask (5 l.), cooled to <10°, and decalin (technical grade, B.D.H.; 712 g., *ca.* 5 moles) was gradually added during 3 hr. with stirring and cooling to keep the temperature of the reaction mixture below 10°. After a further 2 hr. at 10—15°, the mixture was gradually added to a vigorously stirred slurry of crushed ice (12 kg.) and water. The lower layer was separated and, together with the ethylene dichloride extracts of the aqueous layer, washed several times with ice-water and dried (K_2CO_3). The clear solution was decanted on to a little fresh potassium carbonate, and solvent was removed under reduced pressure.

Fractional distillation of the residual brown oil gave decalin (203 g.), b. p. 65—70°/10 mm.; the required vinyl ether (228 g.), b. p. 105—111°/8 mm.; material, b. p. 111—145°/8 mm. (25 g.), b. p. 145—170°/10 mm. (110 g.), b. p. 170—220°/14 mm. (35 g.); and residue (109 g.).

Purification of the vinyl ether. A mixture of the fraction, b. p. 105—111°/8 mm. (228 g.), in sodium-dried ether (250 ml.) and lithium aluminium hydride (5 g.) in ether (150 ml.) was refluxed for 30 min.; excess of hydride was destroyed by cautious addition of ethyl acetate (20 ml.), and ice-cold dilute sulphuric acid (500 ml.; 0·5N) was gradually added to the cooled mixture. The ethereal layer was rapidly separated and, with the ether extracts of the aqueous layer, was washed with water and dried (K_2CO_3). Distillation afforded the pure *vinyl ether* (196 g.), b. p. 102—103°/6 mm., n_D^{25} 1·5045 (Found: C, 80·7; H, 10·2. $C_{12}H_{18}O$ requires C, 80·9; H, 10·1%), of camphor-like odour, which, unlike the vinyl ether before purification, did not develop a greenish-yellow colour on storage. It showed absorption bands at 1667 and 792 cm.$^{-1}$.

10β-*Acetyl*-trans-*decal*-1β-*ol* (VI).—A mixture of the vinyl ether (4·5 g.) in ether (30 ml.) and dilute sulphuric acid (60 ml.; N) was stirred and gently refluxed for 2·5 hr. The ether layer was separated and, with the ether extracts (2 × 30 ml.) of the aqueous layer, was dried

(K_2CO_3) and the ether was removed under reduced pressure. The residue was washed with cold light petroleum and gave the required *hydroxy-ketone* (3·4 g.) which crystallised from light petroleum (b. p. 40—60°) in prisms, m. p. 62—63·5° (Found: C, 73·7; H, 10·0. $C_{12}H_{20}O_2$ requires C, 73·5; H, 10·2%). It showed absorption bands at 1698 (>C:O) and 3460 (OH) cm.$^{-1}$; the latter was narrow, of high intensity, and independent of concentration, as is required for intramolecular hydrogen bonding. When heated at 90°, the hydroxy-ketone evolved water vapour which was collected on a cold finger and identified. The residual oil was dissolved in light petroleum, dried (K_2CO_3), and passed down an alumina column. Pure vinyl ether (V) (0·5 g.) was the first fraction eluted and was identified by its infrared absorption spectrum.

10β-1′-*Hydroxyethyl*-trans-*decal*-1β-*ol* (VII).—Hydroxy-ketone (VI) (1·4 g.) in ether (50 ml.) was added dropwise to a vigorously stirred solution of lithium aluminium hydride (1·0 g.) in ether (100 ml.) and the mixture was refluxed for a further 45 min. Ethyl acetate (10 ml.) in ether (30 ml.) and, subsequently, chilled dilute sulphuric acid (60 ml.; N) were added to the cold reaction mixture. The ether layer, with ether extracts of the aqueous layer, was washed with sodium hydrogen carbonate solution and dried (K_2CO_3). Removal of the solvent under reduced pressure left the required *diol*, m. p. 82·5—84·5° (1·1 g.) after it had been washed with a small volume of light petroleum, m. p. 86·5—87·5° after recrystallisation (Found: C, 72·8; H, 11·0. $C_{12}H_{22}O_2$ requires C, 72·7; H, 11·1%). In the region of O–H stretching vibrations, it gave absorption bands at 3636, 3559, and 3390 cm.$^{-1}$ which can be assigned to free, intramolecularly hydrogen-bonded, and intermolecularly hydrogen-bonded hydroxyl groups, respectively. Evaporation of the light petroleum washings gave a small amount of gum which could not be induced to crystallise.

The diol (0·5 g.), in dry benzene (25 ml.), was treated with *p*-nitrobenzoyl chloride (1·5 g.) and dry pyridine (5 ml.) for several days at room temperature and was then heated on the steam-bath for 2 hr. The mixture was diluted with benzene (40 ml.) and ether (60 ml.) and shaken with 2N-sulphuric acid (100 ml.). The organic layer was separated, washed with 5% sodium carbonate solution and with water, and dried ($MgSO_4$). After removal of solvent, the residue, along with some solid which had separated on the first addition of dilute acid, was crystallised from ethanol and then from benzene and gave the *di*-p-*nitrobenzoate* as needles (*ca.* 0·5 g.), m. p. 215—215·5° (Found: C, 62·8; H, 5·8; N, 5·7. $C_{26}H_{28}N_2O_8$ requires C, 62·9; H, 5·7; N, 5·7%).

Dehydrogenation of the vinyl ether. This was effected by refluxing it (1·75 g.) with sulphur (1·3 g.) for 4 hr. A volatile acid was evolved and distillation of the residue with steam gave naphthalene (identified by its picrate, m. p. and mixed m. p. 148—149°).

10-*Acetyl*-1-*decalone* (XI).—A solution of 10β-acetyl-*trans*-decal-1β-ol (0·96 g.) in pyridine (6 ml.) was added dropwise to a cooled slurry of chromium trioxide (1·14 g.) in pyridine (10 ml.). After 24 hr. at room temperature, water (20 ml.) was added and excess of oxidising agent was destroyed with sulphur dioxide. The mixture was extracted with ether (3 × 30 ml.) and the combined extracts were washed with dilute mineral acid and sodium hydrogen carbonate solution and dried (K_2CO_3). Removal of the solvent gave the required *diketone*, which crystallised from light petroleum (b. p. 40—60°) in plates (0·48 g.), m. p. 67—69° (Found: C, 74·3; H, 9·3. $C_{12}H_{18}O_2$ requires C, 74·2; H, 9·3%). It showed carbonyl absorption bands at 1695 and 1713 cm.$^{-1}$.

Dehydrogenation of 10-*Acetyl*-1-*decalone*.—This was effected by refluxing it (0·35 g.) with sulphur (0·26 g.) for 3 hr. A small quantity of a very pungent liquid distilled from the mixture, and the residue afforded α-naphthol, m. p. and mixed m. p. 91—92° after crystallisation from light petroleum.

Ozonolysis of the Vinyl Ether.—This compound (14 g.) in hexane (80 ml.) at 5—10° absorbed ozone and deposited an oil. Removal of the solvent under reduced pressure left a green, viscous oil which slowly evolved carbon dioxide. This oil was shaken for 5 hr. with water (20 ml.) and gave formic acid, identified by conventional methods. The mixture was extracted with chloroform and the extract dried (Na_2SO_4). Removal of the solvent followed by fractional distillation of the dark red residue gave mainly two fractions: (i) b. p. 146—151°/15 mm. (6 g.), $n_D^{25·5}$ 1·4949, and (ii) b. p. 120—140°/0·15 mm. (2·5 g.). Ozonolysis of the vinyl ether (20 g.) in ice-cold ethyl acetate (140 ml.) gave a greenish-yellow solution which was shaken with sodium hydroxide solution (5 ml.; 40%), and the solvent was removed at room temperature and under reduced pressure. The residual oil was mixed with sodium hydroxide solution (30 ml.; N) and heated on the steam-bath for 30 min. The mixture was cooled, acidified with dilute sulphuric acid, and set aside overnight. The oil was extracted with chloroform and dried ($MgSO_4$), and the solvent removed. Fractional distillation of the residue gave: (i) b. p. 146—151°/15 mm. (10 g.), $n_D^{25\cdot4}$ 1·4949, and (ii) b. p. 180—186°/7 mm. (6 g.).

The Lactone (X) *of* 4β-*Hydroxy*-trans-*decalin*-9β-*carboxylic Acid.*—Fractions (i) from the ozonolyses solidified at −40° and recrystallised from light petroleum (b. p. 40—60°) in prisms, m. p. 25—27° (Found: C, 73·2; H, 9·1. $C_{11}H_{16}O_2$ requires C, 73·2; H, 8·9%). It was the required *lactone* (see below for confirmation of structure) and had a strong absorption band at 1773 cm.$^{-1}$ (γ-lactone). Ozonolysis of the vinyl ether (132 g.) gave this lactone (74 g.).

Lactones of 4β-*Hydroxy*-trans-*decalin*-9β-*glycollic and* -9β-*glyoxylic Acids* (IX and VIII, respectively).—Fractions (ii) from the ozonolyses readily solidified and were mixtures of these two δ-lactones. Crystallisation of the mixture from light petroleum (b. p. 60—80°) readily afforded the *lactone* of the glycollic acid derivative, the less soluble component, as needles, m. p. 118—119° (Found: C, 68·6; H, 8·6. $C_{12}H_{18}O_3$ requires C, 68·6; H, 8·6%). It showed absorption bands (CCl_4 solution) at 3534 (free OH), 3460 (bonded OH), and 1736 cm.$^{-1}$ (δ-lactone). It was obtained from the vinyl ether in 17·5% yield. A mixture of this lactone (0·40 g.) and sodium hydroxide solution (0·75 ml.; 5N) in ethanol (10 ml.) was heated on the steam-bath for 10 min. and, on cooling, deposited the *sodium salt* of the glycollic acid as cubes (0·30 g.) after washing with alcohol and a little water (Found: C, 57·7; H, 7·6; Na, 9·2. $C_{12}H_{19}O_4Na$ requires C, 57·6; H, 7·6; Na, 9·2%). This regenerated the lactone when added to dilute mineral acid. After separation of this lactone from the products of the ozonolysis, evaporation of the filtrates gave a solid, m. p. 90—105°; it was boiled with N-sodium hydroxide solution for a few minutes and undissolved material was separated; acidification of the alkaline solution gave a solid which crystallised from aqueous ethanol in plates, m. p. 131—132° (Found: C, 69·1; H, 8·0. $C_{12}H_{16}O_3$ requires C, 69·2; H, 7·7%). This *lactone* of 4β-hydroxy-*trans*-decalin-9β-glyoxylic acid was reduced by potassium borohydride to the lactone of the corresponding glycollic acid (80% of theory), m. p. and mixed m. p. 118—119°, which regenerated the lactone of the glyoxylic acid when oxidised with chromium trioxide in pyridine. Ozonolysis of the vinyl ether gave the keto-lactone (VIII) in about 2% yield.

4β-*Hydroxy*-trans-*decalin*-9β-*carboxylic Acid* (XV).—The corresponding lactone (X) (2·5 g.), provided by ozonolysis of the vinyl ether, was refluxed for 9 hr. with ethanol (10 ml.) in which sodium (0·32 g.) had been dissolved. The mixture was cooled, diluted with water (10 ml.), and neutralised by careful addition of dilute sulphuric acid (6·9 ml.; 2N). The precipitate (2·1 g.) was separated and its solution in sodium hydrogen carbonate solution was filtered and extracted with ether. Acidification of the aqueous layer gave the required *hydroxy-acid* (1·5 g.) which crystallised from aqueous ethanol in needles, m. p. 167—169° (decomp.) (Found: C, 66·1; H, 9·2%; equiv., 198·3. $C_{11}H_{18}O_3$ requires C, 66·7; H, 9·1%; equiv., 198·2). Its absorption spectrum showed strong intramolecular hydrogen bonding. This compound was not reduced by lithium aluminium hydride in boiling ether, but its methyl ester, obtained by means of diazomethane, was readily reduced to the diol (XIV) (see below), m. p. and mixed m. p. 110—110·5°.

10β-*Hydroxymethyl*-trans-*decal*-1β-*ol* (XIV).—The γ-lactone (X) (1·3 g.) was refluxed for 3 hr. with a solution of lithium aluminium hydride (3 g.) in ether (150 ml.). The reaction mixture was worked up in the usual manner and gave the required *diol*, m. p. 110—110·5° (Found: C, 71·8; H, 10·9. $C_{11}H_{20}O_2$ requires C, 71·7; H, 10·9%) after crystallisation from ethyl acetate–light petroleum. The absorption spectra of its solutions in carbon disulphide showed intramolecular hydrogen bonding. A mixture of the diol (0·10 g.), *p*-nitrobenzoyl chloride (0·4 g.), and pyridine (2·5 ml.) in benzene (10 ml.) was heated for 10 min. on the steam-bath. Dilution with benzene (20 ml.) and ether (30 ml.) and successive extraction with water, 2N-sodium carbonate solution, water, 2N-hydrochloric acid, and water, followed by drying and removal of solvent, gave the *di*-p-*nitrobenzoate* which crystallised from ethanol in needles, m. p. 173—174° (Found: C, 62·3; H, 5·5; N, 6·2. $C_{25}H_{26}N_2O_8$ requires C, 62·2; H, 5·4; N, 5·8%).

10β-1′,2′-*Dihydroxyethyl*-trans-*decal*-1β-*ol* (XII).—This *triol* was obtained in good yield by reduction ($LiAlH_4$) of each of the δ-lactones (VIII) and (IX). It crystallised from ethyl acetate in plates, m. p. 156° (Found: C, 67·4; H, 10·5. $C_{12}H_{22}O_3$ requires C, 67·3; H, 10·4%), and readily reacted with acetone in the presence of anhydrous copper sulphate to give the *isopropylidene derivative*, which crystallised from light petroleum in prisms, m. p. 120—121° (Found: C, 70·9; H, 10·3. $C_{15}H_{26}O_3$ requires C, 70·9 H, 10·2%).

10β-*Formyl*-trans-*decal*-1β-*ol* (XIII).—A solution of the above triol (0·107 g.) in a mixture of ethanol (6 ml.), water (1·5 ml.), and aqueous periodic acid solution (2·6 ml.; 0·206M) was kept in a stoppered flask at room temperature for 40 hr. Formaldehyde was shown to be formed. Excess of saturated sodium hydrogen carbonate solution was added and the mixture was extracted repeatedly with ether. The extracts were dried ($MgSO_4$) and gave the required hydroxy-aldehyde as an oil of strong ginger-like odour. The *semicarbazone* crystallised from ethyl acetate–light petroleum in needles, m. p. 224—225° (Found: C, 60·5; H, 8·8; N, 17·7. $C_{12}H_{21}N_3O_2$ requires C, 60·3; H, 8·8; N, 17·6%). The 2,4-*dinitrophenylhydrazone* separated from ethanol in orange-yellow needles, m. p. 153—154° (Found: C, 56·2;H, 6·2; N, 15·2. $C_{17}H_{22}N_4O_5$ requires C, 56·4; H, 6·1; N, 15·5%). When reduced with lithium aluminium hydride, the hydroxy-aldehyde gave 10β-hydroxymethyl-*trans*-decal-1β-ol (XVI), m. p. and mixed m. p. 110—110·5°.

Reaction of the Vinyl Ether with Perbenzoic Acid in Chloroform.—One mol. of the peracid was immediately (<15 min.) consumed and the lactone (IX) was isolated from the reaction mixture. When 3 mol. of peracid were used, 2 mol. were consumed in 24 hr. and the mixture gave the lactones (VIII) and (IX) and a residual oil which, with lithium aluminium hydride, gave the diol (XIV), m. p. and mixed m. p. 110—110·5°; apparently, some of the γ-lactone (X) had been formed during the oxidation process.

THE MANCHESTER COLLEGE OF SCIENCE AND TECHNOLOGY,
MANCHESTER, 1.

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