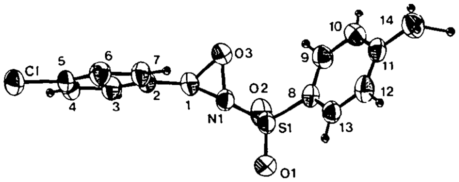

Fig. 3. *ORTEP* drawing of 2-(*p*-toluenesulfonyl)-3-(*p*-chlorophenyl)oxaziridine. Thermal ellipsoids are drawn at the 50% probability level while isotropic hydrogen thermal parameters are represented by spheres of arbitrary size.

chlorophenyl groups are *trans* (*E* configuration) with respect to the oxaziridine ring. The two sulfonyl O atoms and N(1) are symmetrically oriented with respect to the toluene ring as indicated by the torsion angles of 29 and −19° for O(2)S(1)C(8)C(9) and O(1)S(1)C(8)C(13) and 89 and −88° for N(1)S(1)-C(8)C(13) and N(1)S(1)C(8)C(9). The N lone pair lies opposite the sulfonyl O atoms which is also the conformation adopted by 2-(*p*-toluenesulfonyl)-3-(*m*-nitrophenyl)oxaziridine (Chen, Watson, Davis, Lamendola & Nadir, 1978), Table 2. In *N*,*N*,*N'*,*N'*-tetramethylsulfamide (Jordan, Smith, Lohr & Lipscomb, 1963), *p*-methoxybenzenesulfon-*p*-anisidine (Pokrywiecki, Weeks & Duax, 1973*a*) and *p*-methoxybenzenesulfon-*N*-isopropyl-*p*-anisidine (Pokrywiecki, Weeks & Duax, 1973*b*) the lone pairs of the N atoms lie between the two sulfonyl O atoms. These compounds exhibit a flattened geometry around N and a shortening of the S—N bond to an average distance of 1·628 (4) Å which was interpreted in terms of lone-pair–*d*-orbital interactions. The N—S distance in the title compound is 1·738 (4) and 1·728 (4) Å in the *m*-nitrophenyl derivative. The C(1)N(1)O(3) angle of 57·5° makes it energetically unfavorable for the N atom to achieve a pseudo-planar conformation in which the angle approaches 120°. Lone-pair–*d*-orbital interactions in these systems are not significant.

The parameters for the oxaziridine ring in the title compound and in the *m*-nitrophenyl derivative compare quite well: C—O = 1·415 (5) and 1·414 (5) Å, C—N = 1·450 (5) and 1·443 (6) Å, O—N = 1·494 (5) and 1·487 (6) Å, C—O—N = 59·7 (2) and 59·6 (3)°, O—N—C = 57·5 (2) and 57·7 (3)° and N—C—O = 62·8 (2) and 62·7 (3)°. Torsion angles and interplanar angles are compared in Table 2.

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Structure of 15β-Acetoxy-(—)-kaur-16-en-19-oic Acid (Xylopic Acid)

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Abstract. $C_{22}H_{32}O_4$, orthorhombic, $P2_12_12_1$, a = 14·966 (2), b = 11·885 (2), c = 11·074 (3) Å, V = 1969·7 Å^3, Z = 4, ρ_x = 1·27 Mg m^{-3}, μ(Cu $K\alpha$) = 0·711 mm^{-1}. The diterpene, xylopic acid, was isolated from the dried fruits of *Xylopia aethiopica*. The structure was solved by direct methods and refined to R = 0·045. One hydrogen bond is observed.

Introduction. The dried fruits of the plant *Xylopia aethiopica* are used in parts of West Africa as spices and in folklore medicine for curing bronchial infections. The diterpene, xylopic acid, is obtained upon extraction of these fruits with petroleum ether 30/40 (Ekong &

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Ogan, 1968; Mensah & Fiagbe, 1975; Owusu & Fiagbe, 1976). The isolation, chemical characterization and the microbiological screening of the title compound and of related compounds are reported separately (Mensah & Fiagbe, 1975; Boakye-Yiadom, Ayim & Fiagbe, 1978). The chemistry of the kaurenes and of the related phyllocladene class of compounds has been reviewed (Ekong & Ogan, 1968; Hanson, 1968; Ekong, 1969).

The title compound was recrystallized from ether and a crystal of approximate volume 0·066 mm^3 was selected for the intensity measurements. Lattice constants were obtained from least-squares treatment of the angular coordinates of 25 reflexions measured with Cu $K\alpha$ radiation on a Philips PW 1100 diffractometer equipped with a graphite monochromator. Diffraction intensities were collected by use of a θ–2θ scan procedure for reflexions with $2\theta \leq 134°$, a scan width of 1·5° (θ) and a scan speed of 1·5° (θ) min^{-1}. Out of the 1945 reflexions recorded, 1650 were above the threshold value determined by the criterion $\sigma(I_{\text{net}})/I_{\text{net}} < 0{\cdot}30$, where $\sigma(I_{\text{net}})$ is based on counting statistics. The intensities were corrected for Lorentz and polarization effects, but not for absorption.

The structure was solved by use of the *MULTAN* direct phase-determination procedure (Main, Woolfson, Lessinger, Germain & Declercq, 1974). The E map computed with the set showing the best reliability index revealed 16 atoms. The remaining 10 non-hydrogen atoms were generated from a difference F map. The H atoms, with the exception of H(O3), were located by use of a procedure in the *SHELX* program system (Sheldrick, 1976) and from a difference Fourier map. The *SHELX* system of programs was also used for the full-matrix least-squares refinements in which the non-hydrogen atoms were included with anisotropic thermal parameters. The methyl groups were refined as rigid units and the remaining H atoms constrained at 1·08 Å from their respective C atoms, their positions being dictated by the geometry of the molecule. The hydroxyl H atom H(O3) was not included in the refinements. Individual isotropic thermal parameters were refined for the H atoms. The R value converged to 0·045 for the 1650 observed unique reflexions.* Unit weights were used throughout the refinement. The atomic scattering factors for C and O were taken from Cromer & Mann (1968) and those of H from Stewart, Davidson & Simpson (1965).

Fractional coordinates for the heavy atoms are listed in Table 1 and those for H in Table 2.

Table 1. *Positional parameters of the non-hydrogen atoms* (×10^4)

	x	y	z
C(1)	8644 (3)	−199 (4)	9130 (5)
C(2)	8048 (3)	58 (4)	8034 (5)
C(3)	8604 (3)	508 (4)	6996 (5)
C(4)	9165 (3)	1554 (4)	7319 (4)
C(5)	9746 (3)	1263 (3)	8460 (4)
C(6)	10435 (3)	2152 (4)	8836 (4)
C(7)	11121 (3)	1614 (4)	9679 (4)
C(8)	10691 (3)	1138 (3)	10819 (4)
C(9)	9894 (3)	349 (3)	10510 (4)
C(10)	9203 (3)	819 (3)	9556 (4)
C(11)	9455 (3)	−122 (4)	11683 (5)
C(12)	9412 (4)	690 (4)	12758 (6)
C(13)	10295 (3)	1336 (5)	12902 (5)
C(14)	10449 (3)	2046 (4)	11763 (4)
C(15)	11408 (3)	523 (4)	11588 (4)
C(16)	11062 (3)	499 (4)	12864 (4)
C(17)	11331 (4)	−158 (5)	13749 (6)
C(18)	9782 (4)	1822 (5)	6237 (5)
C(19)	8552 (3)	2581 (4)	7465 (4)
C(20)	8557 (3)	1708 (4)	10082 (4)
C(21)	12442 (3)	−944 (4)	11239 (4)
C(22)	12575 (4)	−2084 (4)	10691 (6)
O(1)	11615 (2)	−587 (2)	11113 (0)
O(2)	13036 (2)	−426 (3)	11721 (3)
O(3)	8987 (2)	3516 (3)	7766 (4)
O(4)	7768 (2)	2568 (3)	7277 (3)

Table 2. *Positional parameters* (×10^3) *for the hydrogen atoms*

	x	y	z
H1(C1)	910 (1)	−87 (1)	889 (1)
H2(C1)	823 (1)	−47 (1)	987 (1)
H1(C2)	755 (1)	67 (1)	828 (1)
H2(C2)	772 (1)	−71 (1)	775 (1)
H1(C3)	905 (1)	−15 (1)	670 (1)
H2(C3)	816 (1)	74 (1)	627 (1)
H(C5)	1013 (1)	56 (1)	813 (1)
H1(C6)	1010 (1)	284 (1)	929 (1)
H2(C6)	1077 (1)	247 (1)	804 (1)
H1(C7)	1160 (1)	225 (1)	994 (1)
H2(C7)	1146 (1)	94 (1)	921 (1)
H(C9)	1018 (1)	−35 (1)	1002 (1)
H1(C11)	984 (1)	−85 (1)	1196 (1)
H2(C11)	878 (1)	−38 (1)	1147 (1)
H1(C12)	888 (1)	129 (1)	1260 (1)
H2(C12)	928 (1)	22 (1)	1357 (1)
H(C13)	1027 (1)	182 (1)	1373 (1)
H1(C14)	985 (1)	249 (1)	1151 (1)
H2(C14)	1099 (1)	264 (1)	1189 (1)
H(C15)	1204 (1)	96 (1)	1156 (1)
H1(C17)	1101 (4)	−26 (5)	1458 (5)
H2(C17)	1184 (4)	−65 (5)	1369 (5)
H1(C18)	1026 (1)	116 (1)	607 (1)
H2(C18)	1013 (1)	261 (1)	637 (1)
H3(C18)	934 (1)	190 (1)	547 (1)
H1(C20)	891 (1)	249 (1)	1025 (1)
H2(C20)	833 (1)	136 (1)	1093 (1)
H3(C20)	799 (1)	187 (1)	950 (1)
H1(C22)	1320 (1)	−246 (1)	1096 (1)
H2(C22)	1253 (1)	−207 (1)	972 (1)
H3(C22)	1203 (1)	−258 (1)	1105 (1)

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33936 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. The molecular geometry of the title compound is illustrated in Fig. 1. Bond lengths and angles involving the non-hydrogen atoms are given in Figs. 2 and 3 respectively. The average standard deviations in these bond lengths and angles are estimated to be 0·006 Å (maximum 0·007 Å) and 0·4° (maximum 0·5°) respectively. The $C(sp^3)$–$C(sp^3)$ bonds vary in the range 1·517–1·580 Å, mean value 1·545 Å; the longest bonds are connected to the crowded atoms C(4) and C(10).

The ring junction *A*/*B* is *trans* and that of *B*/*C* is *cis*. The endocyclic torsion angles are given in Fig. 4, from which it can be seen that the values for ring *A* agree well with the value of ±54° for the chair conformation given by Hendrickson (1967). In rings *B* and *C* the angles deviate from this value, but are in agreement with observations on other kaurenes (Karle, 1972; Ferguson & Marsh, 1975; Taylor & Watson, 1976). The torsion angles for rings *A* and *B* again agree well with the observation on the phyllocladene bombiccite (Foresti Serantoni, Krajewski, Mongiorgi, Riva di Sanseverino & Sheldrick, 1978) while the values for ring *C* differ slightly.

The deviations from the ideal chair conformation are also illustrated by the puckering parameters calculated according to Cremer & Pople (1975) for rings *B* and *C*: $Q_B = 0{\cdot}57$ Å, $\varphi_{2,B} = 84°$, $\theta_B = 10°$ [origin C(7)] and $Q_C = 0{\cdot}62$ Å, $\varphi_{2,C} = 42°$, $\theta_C = 23°$ [origin C(13)]. The *B* ring is hence described as a slightly distorted chair, while *C* is more distorted probably due to the fact that the atoms C(8), C(13) and C(14) are also members of the five-membered ring *D*.

Ideal conformations for rings *A*, *B* and *C* would result in distances C(20)···C(19), C(20)···C(14) and C(20)···C(12) of *ca* 2·5 Å (Karle, 1972). The deviations from ideality for bond lengths, valency angles and torsion angles, however, relieve this crowding and the cited distances are all longer than 3·4 Å.

Other conformations have been observed for the three six-membered rings of kaurenes. In the *ent*-kaurene mebadonin reported by Hirotsu, Kamikawa, Kubota, Shimada & Isobe (1973) the *A*, *B* and *C* rings are skew boat, chair and distorted chair, respectively.

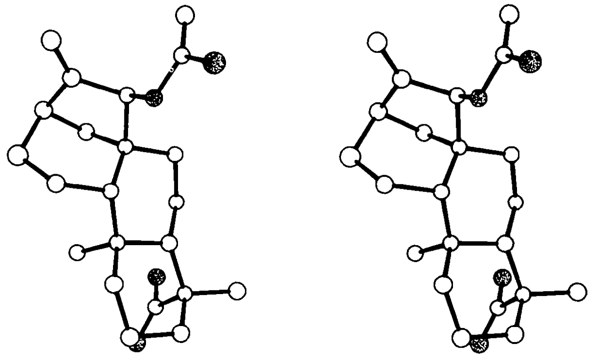

Fig. 1. Stereoscopic view of the molecule with H atoms omitted. O atoms are represented by shaded ellipsoids.

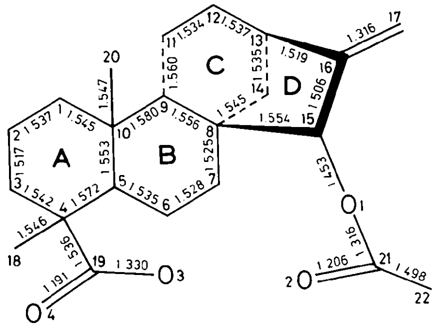

Fig. 2. Bond lengths (Å) between the non-hydrogen atoms.

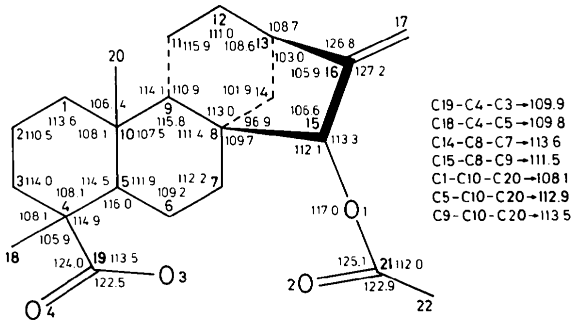

Fig. 3. Bond angles (°) involving the non-hydrogen atoms.

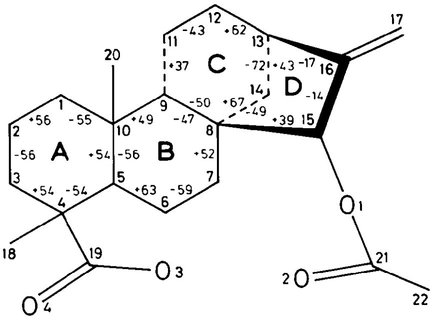

Fig. 4. Endocyclic torsion angles (°).

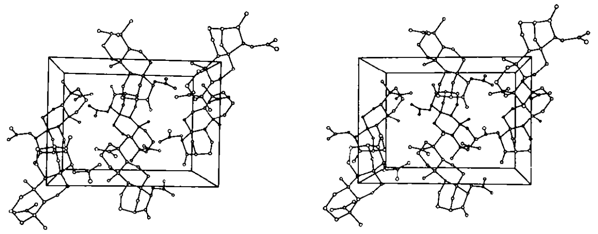

Fig. 5. A stereoscopic view of the packing arrangement in the unit cell. The origin is at the lower left-hand corner of the unit cell, *a* is directed to the right, *c* upwards and *b* downwards perpendicular to the plane of the paper.

Ring *D* is best described as having a twist conformation with the approximate C_2 axis through C(16). Puckering parameters for this ring are: $Q = 0{\cdot}50$ Å, $\varphi = 92°$.

The molecular packing is shown in Fig. 5. Although the position of H(O3) could not be determined, it is most probable that the short O(3)—O($2^{\rm i}$) contact (2·74 Å) [(i) $x - \frac{1}{2}, \frac{1}{2} - y, 2 - z$] really represents a hydrogen bond. Only two other intermolecular distances shorter than 3·4 Å are observed. These are C(15)—O($4^{\rm ii}$) = 3·30 Å [(ii) $x + \frac{1}{2}, \frac{1}{2} - y, 2 - z$] and C(19)—O($4^{\rm iii}$) = 3·35 Å [(iii) $2 - x, y - \frac{1}{2}, 1\frac{1}{2} - z$].

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2*e*-(4-Nitrophenoxy)-*trans*-1-oxadecalin

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Abstract. $C_{15}H_{19}NO_4$, $M_r = 277{\cdot}32$, monoclinic, $P2_1/c$, $a = 11{\cdot}360\,(3)$, $b = 5{\cdot}369\,(3)$, $c = 22{\cdot}880\,(6)$ Å, $\beta = 100{\cdot}05\,(2)°$, $U = 1374$ Å^3, $Z = 4$, $D_x = 1{\cdot}340$ Mg m^{-3}, μ(Mo $K\alpha$) = 0·06 mm^{-1}. The structure was refined to $R = 0{\cdot}057$ for 1766 reflexions. The C—O lengths of the acetal group do not differ significantly either from each other, or from the corresponding equatorial phenoxy compound.

Introduction. Our examination of the effects of ground-state structure on the reactivity of acetals (Jones, Kennard, Kirby & Osborne, 1978; Jones, Kennard, Kirby, Osborne & Malik, 1978) has revealed significant differences in the bond lengths and angles of the axial and equatorial isomers of 2-phenoxy-*trans*-1-oxadecalin [(I), R = Ph] (Jones, Kennard, Chandrasekhar & Kirby, 1978*a*,*b*). In the axial isomer the exocyclic C—OR bond is longer than the endocyclic C—O(2) by 0·03 Å, though the lengths of these bonds in the equatorial isomer are not significantly different. These differences [which are in the opposite direction to

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