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The Crystal Structure of Isovirescenol B, $C_{20}O_2H_{32}$

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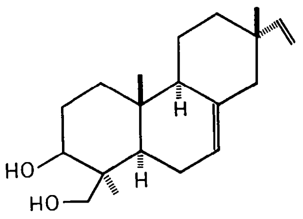
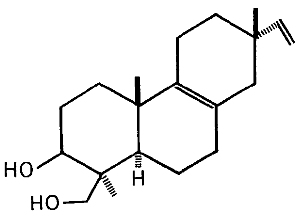
The crystal structure of isovirescenol B, $C_{20}O_2H_{32}$, has been solved and refined using 1439 intensities from data photographically recorded with Cu *Kα* radiation. The crystals are orthorhombic, space group $P2_12_12_1$, and have unit-cell dimensions $a=12{\cdot}817$ (2), $b=12{\cdot}980$ (1), $c=10{\cdot}654$ (1) Å, with four molecules per unit cell. The structure was solved by direct methods; all hydrogen atoms were located from a difference map. The positional and thermal parameters (anisotropic for non-hydrogen atoms and isotropic for hydrogen) were refined by block-diagonal least-squares calculations to an *R* of 0·066. All three rings in the molecule have a chair shape with the same orientation. The discrete molecules are bonded together by O–H···O hydrogen bonds around every other screw axis in the **c** direction. Another hydrogen bond bridges the two O's within the molecule.

Introduction

Virescenol B (I), a natural diterpene, is a metabolite of the fungus *Acremonium luzulae* (Fuckel) Gams [previously described as *Oospora virescens* (Link) Wallr.]. The compound, present in the culture media as glycoside, was first isolated by Cagnoli Bellavita, Ceccherelli, Ribaldi, Baskevitch & Polonsky (1967), and wholly characterized as isopimaradiene-3β,19-diol by Polonsky, Baskevitch, Cagnoli Bellavita & Ceccherelli (1968, 1970).



(I)



(II)

After acid hydrolysis of the glycoside it is possible to separate the isomer, isovirescenol B (II), which is different only in a double-bond position.

The X-ray analysis fully confirms the structure attributed to isovirescenol B on the basis of chemical and spectral properties, and unambiguously confirms the position of the hydroxymethylic group at C(4), excluding its alternate location at C(10).

Experimental

Crystal data

A supply of isovirescenol B was provided by Professors Cagnoli Bellavita and Ceccherelli, Istituto di Chimica Organica, Facoltà di Farmacia, Università di Perugia, Italy; crystals were grown from ethanol solution. The crystals are clear yellow and transparent, mostly in the form of slender prisms elongated in the **c** direction, optically biaxial, frequently twinned.

Preliminary oscillation and Weissenberg data indicated that the crystals have an orthorhombic lattice. As indexed, systematic absences (*h*00 when *h* odd, 0*k*0 when *k* odd, and 00*l* wnen *l* odd) uniquely determine the space group as $P2_12_12_1$. The crystal data are listed in Table 1. The lattice constants are the result of a least-squares fit to 116 observations of medium and (few) hign-angle reflexions collected from the 0*kl* and *hk*0 Weissenberg layer lines. These measurements were made at 23 (2)°C using Cu *Kα* radiation, recording also the Ag powder pattern as a standard ($a=4{\cdot}0862$ Å). The density of crystals was measured by flotation in a NaCl aqueous solution. No appreciable variation of the crystal data nor any alteration of the crystal were found after exposure to X-rays.

Table 1. *Crystal data for isovirescenol B*

Orthorhombic
$a=12{\cdot}877$ (2) Å
$b=12{\cdot}980$ (1)
$c=10{\cdot}654$ (1)
$V=1780{\cdot}72$ Å^3
Formula: $C_{20}O_2H_{32}$
F.W. 304·48

m.p. 124°C
$D_m=1{\cdot}14$ g cm^{-3}
$D_x=1{\cdot}136$ g cm^{-3}
$Z=4$
μ(Cu *Kα*) = 5·44 cm^{-1}
$F(000)=672$
Space group: $P2_12_12_1$

An untwinned crystal of approximate dimensions 0·1 × 0·1 × 0·6 mm was used both for lattice-constant determination and for intensity collection.

Data collection and processing

X-ray diffraction intensities were recorded by means of the multiple-film technique on an integrating Weissenberg camera, with the equi-inclination method, using Ni-filtered Cu *Kα* radiation. In total 1439 independent reflexions were collected from the two settings *hk*0 to *hk*10 and 0*kl* to 3*kl*. Of these, 560 reflexions were below the observational limit, and they were assigned an intensity half the minimum one recordable on each layer line. An optical microdensitometer was used to measure the photographic densities, which were converted into intensities by application of corrections for Lp and α_1–α_2 splitting. Absorption correction was not considered warranted because of the small size of the crystal and the low absorption coefficient, the major purpose of this investigation being to determine the chemical structure of the product in question. Intensities were put on an overall relative scale using the least-squares scaling program *FILTRO* written by Coda (1973, personal communication), and on absolute scale by Wilson's (1942) method.

Though rather small: $\Delta f'=0{\cdot}047$, $\Delta f''=0{\cdot}032$ for O and $\Delta f'=0{\cdot}017$, $\Delta f''=0{\cdot}009$ for C (after Cromer & Liberman, 1970), an anomalous dispersion correction was applied at the very end of refinement.

Structure determination and refinement

A first attempt to find a solution to the structure by means of a three-dimensional Patterson synthesis proved unsuccessful, owing to the poor and contradictory indications that this method gave. A survey *a posteriori* of the Patterson map revealed that it was actually in agreement with the structure found, but only the vectors related to a few atoms (roughly one half) could be recognized in it, the remaining ones being obscured by the many spurious peaks. It was therefore necessary to rely on direct methods, the case looking favourable because of the similar weight of all heavy atoms in the molecule.

Table 2. *Initial phase assignments* (*MULTAN*)

h	*k*	*l*	$\|E\|$	Possible phases	Code*
3	11	8	3·41	45°	1
2	0	9	2·84	90	2
0	9	4	2·47	90	2
0	2	13	5·40	0, 180	3
8	13	4	4·36	±45, ±135	3
2	10	4	1·99	±45, ±135	3
4	0	10	2·52	0	4

* 1 = origin and enantiomorph definition, 2 = origin definition, 3 = starting set, 4 = known phase.

The structure was determined by the multisolution technique of Germain, Main & Woolfson (1971), using the program *MULTAN* in its fully automatic mode, with 267 reflexions having $|E| \geq 1{\cdot}20$ as input. The reflexions used to define the origin, the enantiomorph and the starting set are presented in Table 2 along with their $|E|$ values. The complete set of $|E|$ was used in the weighted tangent calculations, which yielded phasing of all input reflexions, with $2^5=32$ possible combinations for starting phases, thus giving 32 solutions. The 'absolute figures of merit' ranged in value from 1·0476 to 0·6154. The structure was derived from the Fourier *E* map obtained using solution 27 which had the highest figure of merit. This map clearly revealed the positions of all the 14 heavy atoms belonging to the rings. The structure-factor calculation for this trial structure yielded an $R=\sum||F_o|-|F_c||/\sum|F_o|$ (for reflexions with non-zero weight) of 0·49. The remaining non-hydrogen atoms were fixed from an F_o synthesis, with a resulting *R* index of 0·36. Two full-matrix cycles of least-squares calculations with the

Table 3. *Atomic coordinates and isotropic thermal parameters*

Positional e.s.d.'s for O and C are multiplied by 10^4 and for H by 10^3. *B*'s of non-hydrogen atoms are those after Hamilton (1959). *B*'s of hydrogen atoms are multiplied by 10.

	x/*a*	*y*/*b*	*z*/*c*	*B* (Å^2)
O(1)	0·2303 (5)	0·0136 (4)	0·5905 (5)	5·18
O(2)	0·3140 (5)	0·0402 (4)	0·3538 (5)	5·44
C(1)	0·1889 (7)	0·2923 (6)	0·6812 (6)	4·45
C(2)	0·2398 (7)	0·1844 (6)	0·6804 (6)	4·64
C(3)	0·1868 (7)	0·1155 (6)	0·5896 (7)	4·60
C(4)	0·1860 (6)	0·1527 (6)	0·4535 (6)	3·16
C(5)	0·1419 (5)	0·2715 (5)	0·4534 (6)	2·75
C(6)	0·1397 (7)	0·3183 (6)	0·3246 (6)	3·96
C(7)	0·0804 (7)	0·4198 (7)	0·3282 (7)	4·98
C(8)	0·0830 (6)	0·4787 (6)	0·4415 (6)	3·47
C(9)	0·1185 (7)	0·4427 (6)	0·5553 (7)	4·15
C(10)	0·1890 (6)	0·3437 (5)	0·5534 (6)	3·13
C(11)	0·1373 (7)	0·5149 (6)	0·6608 (6)	4·23
C(12)	0·0635 (6)	0·6054 (6)	0·6594 (6)	3·77
C(13)	0·0667 (7)	0·6626 (6)	0·5349 (7)	3·78
C(14)	0·0349 (8)	0·5828 (7)	0·4312 (8)	6·04
C(15)	−0·0228 (8)	0·7440 (7)	0·5334 (8)	6·05
C(16)	−0·0131 (10)	0·8459 (7)	0·5294 (10)	7·98
C(17)	0·1685 (7)	0·7051 (7)	0·5034 (8)	5·01
C(18)	0·1061 (7)	0·0913 (6)	0·3729 (8)	4·84
C(19)	0·2903 (7)	0·1472 (6)	0·3921 (6)	4·01
C(20)	0·3021 (7)	0·3763 (7)	0·5197 (8)	5·05
H(1)	0·247 (6)	−0·013 (6)	0·695 (6)	4·4 (14)
H(2)	0·299 (6)	0·026 (5)	0·442 (6)	5·4 (16)
H(3)	0·121 (4)	0·305 (4)	0·735 (5)	1·8 (12)
H(4)	0·224 (5)	0·334 (5)	0·738 (6)	4·8 (13)
H(5)	0·319 (4)	0·195 (4)	0·640 (5)	1·4 (9)
H(6)	0·293 (5)	0·175 (5)	0·760 (5)	3·6 (10)
H(7)	0·119 (5)	0·113 (5)	0·617 (5)	3·2 (15)
H(8)	0·068 (4)	0·266 (4)	0·500 (5)	1·9 (13)
H(9)	0·108 (4)	0·285 (4)	0·246 (4)	1·1 (20)
H(10)	0·206 (5)	0·319 (5)	0·286 (6)	4·5 (17)
H(11)	0·010 (5)	0·404 (5)	0·317 (6)	3·8 (9)
H(12)	0·105 (4)	0·457 (4)	0·236 (4)	1·0 (9)
H(13)	0·117 (5)	0·469 (5)	0·739 (5)	3·2 (9)
H(14)	0·131 (7)	0·523 (7)	0·758 (7)	8·2 (18)
H(15)	−0·003 (5)	0·604 (4)	0·700 (5)	2·5 (24)
H(16)	0·081 (4)	0·658 (4)	0·725 (5)	1·5 (18)
H(17)	−0·048 (6)	0·589 (7)	0·445 (8)	8·2 (12)
H(18)	0·057 (6)	0·616 (6)	0·332 (7)	6·6 (22)
H(19)	−0·070 (7)	0·766 (7)	0·621 (8)	9·6 (11)
H(20)	0·018 (7)	0·866 (6)	0·622 (8)	8·5 (9)
H(21)	0·028 (7)	0·856 (6)	0·445 (8)	8·4 (25)
H(22)	0·198 (7)	0·743 (7)	0·571 (8)	8·5 (22)
H(23)	0·182 (7)	0·715 (7)	0·404 (8)	8·9 (23)
H(24)	0·234 (7)	0·698 (7)	0·570 (8)	9·7 (25)
H(25)	0·024 (6)	0·093 (6)	0·400 (7)	6·2 (23)
H(26)	0·174 (7)	0·042 (7)	0·422 (8)	9·6 (22)
H(27)	0·134 (6)	0·097 (6)	0·270 (6)	5·9 (21)
H(28)	0·341 (4)	0·171 (4)	0·463 (5)	1·7 (19)
H(29)	0·292 (4)	0·171 (4)	0·295 (4)	1·3 (13)
H(30)	0·341 (5)	0·386 (5)	0·615 (6)	3·8 (9)
H(31)	0·375 (6)	0·346 (6)	0·506 (7)	7·7 (9)
H(32)	0·331 (5)	0·395 (6)	0·426 (6)	5·7 (14)

contribution of all heavy atoms and individual isotropic thermal parameters led to $R=0{\cdot}16$. Refinement was continued, still full-matrix, with introduction of anisotropic thermal parameters, varying $\frac{1}{3}$ of the total of the atoms each time. The improvement of R was only to 0·13; at this stage the H atoms were positioned on a ΔF map. The first block-diagonal cycle with their contribution (one block per atom), their positional and isotropic thermal parameters being varied, gave $R=0{\cdot}10$. Several more block-diagonal cycles (with unit weight for observed reflexions and zero weight for 'less thans') were needed to achieve convergence. During these cycles all positional and thermal parameters were varied, as well as the scale factor and the secondary extinction coefficient (Zachariasen, 1963) which reached the final value $g=4{\cdot}3\times10^{-6}$.

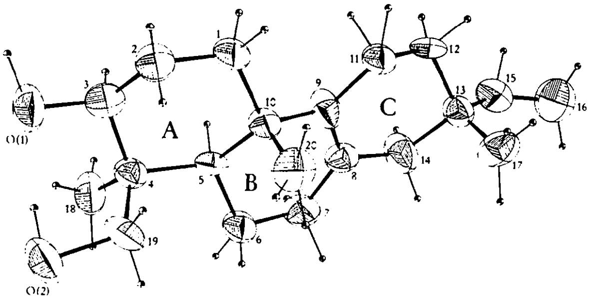


Fig. 1. The molecular structure of isovirescenol B. Carbon atoms have numbering only. The plotting program *ORTEP* (Johnson, 1965) was used both for this perspective figure and for Fig. 2. The thermal ellipsoids are scaled to include the 50% probability level. The hydrogen atoms, unlabelled, are represented by spheres with $B=1{\cdot}5$ Å^2.

At this stage it was found that two hydrogen atoms were affected by unusual and irregular shifts, especially in their B's; a survey of a new ΔF map unambiguously revealed their wrong positioning, along with two positive maxima in the correct places.

The final R is 0·066 for actually observed reflexions; $R=0{\cdot}112$ including unobserved ones. None of the shifts in the final cycle was greater than 0·05 times the standard deviation for the non-hydrogen atoms, and

Table 4. *Heavy-atom thermal parameters with e.s.d.'s* ($\times10^4$)

$$T=\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)\right].$$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(1)	119 (6)	42 (3)	107 (6)	20 (4)	−27 (5)	−1 (4)
O(2)	104 (6)	56 (4)	125 (6)	28 (4)	3 (5)	−24 (4)
C(1)	114 (8)	57 (5)	41 (6)	−1 (7)	−18 (7)	−8 (5)
C(2)	113 (9)	60 (6)	51 (6)	9 (7)	−39 (6)	3 (5)
C(3)	96 (8)	64 (6)	68 (7)	3 (7)	0 (7)	4 (6)
C(4)	56 (6)	47 (5)	57 (6)	2 (5)	−4 (5)	−7 (5)
C(5)	41 (5)	55 (5)	40 (5)	−1 (5)	3 (5)	−5 (5)
C(6)	85 (7)	56 (6)	54 (6)	0 (6)	−12 (6)	−9 (5)
C(7)	113 (9)	71 (6)	58 (7)	26 (7)	−7 (7)	13 (6)
C(8)	54 (6)	61 (5)	60 (7)	−4 (5)	−7 (5)	2 (5)
C(9)	83 (8)	45 (5)	86 (8)	11 (6)	−32 (6)	−13 (5)
C(10)	60 (6)	47 (5)	49 (6)	6 (5)	−19 (5)	0 (5)
C(11)	101 (8)	54 (5)	52 (6)	1 (6)	−8 (6)	−3 (5)
C(12)	67 (6)	71 (6)	46 (6)	1 (6)	27 (6)	−6 (5)
C(13)	73 (7)	46 (5)	75 (7)	11 (6)	2 (6)	−4 (5)
C(14)	152 (12)	55 (6)	95 (9)	21 (8)	−30 (8)	−4 (6)
C(15)	127 (10)	73 (7)	107 (10)	6 (8)	30 (8)	−15 (8)
C(16)	176 (13)	73 (7)	161 (13)	13 (10)	10 (11)	16 (9)
C(17)	56 (7)	87 (7)	120 (9)	−4 (6)	28 (7)	12 (7)
C(18)	97 (9)	44 (5)	112 (9)	6 (7)	−24 (7)	−5 (6)
C(19)	69 (7)	66 (6)	67 (7)	19 (6)	2 (6)	−23 (7)
C(20)	62 (7)	86 (7)	116 (9)	−28 (7)	−8 (7)	−10 (7)

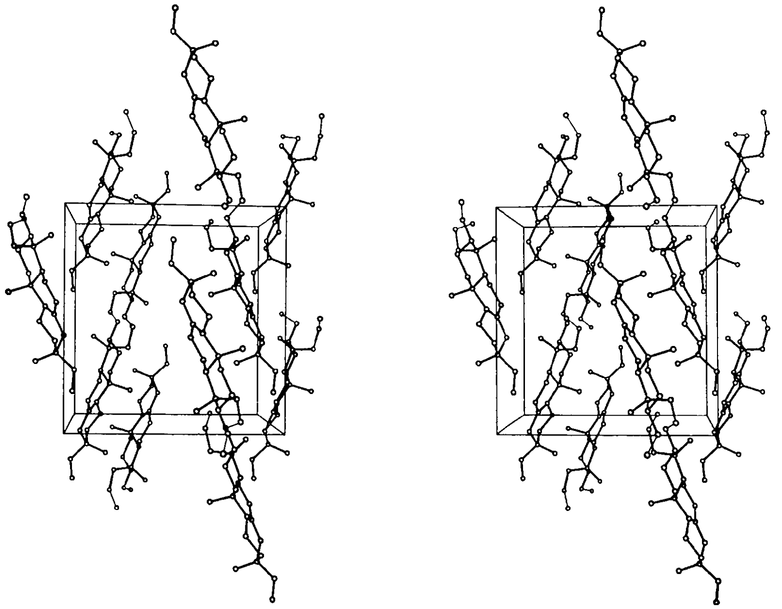


Fig. 2. Stereoscopic drawing of the crystal packing, excluding the hydrogen atoms. The unit cell is viewed in the negative z direction, with the x axis to the left and the y axis downward. The origin is in the upper right, far corner. Intermolecular hydrogen bonds are shown as thin lines.

Table 5. *Observed and calculated structure amplitudes for isovirescenol B*

An asterisk indicates a 'less than'.

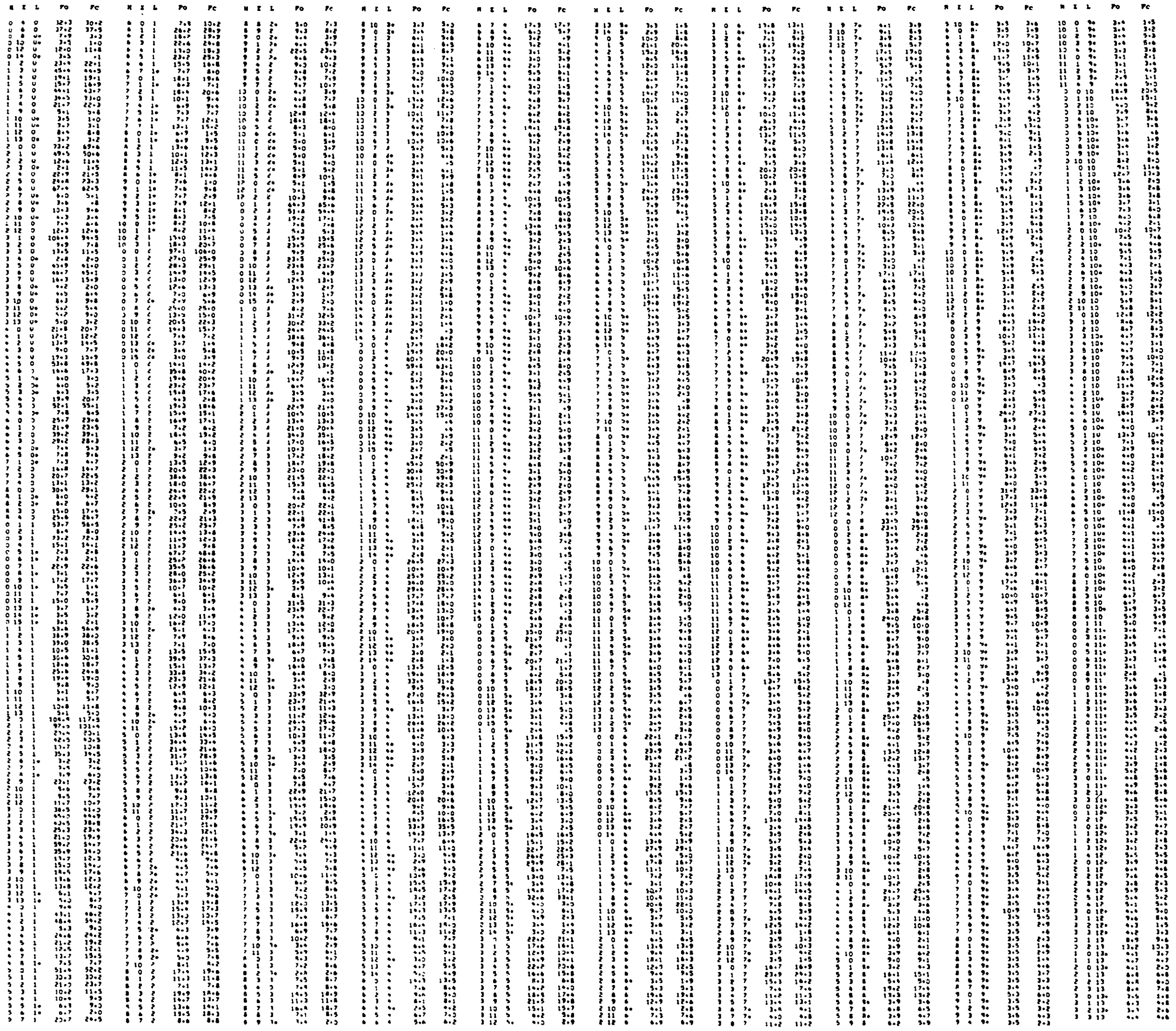


Table 6. *Bond distances* (Å)

E.s.d.'s in parentheses are multiplied by 10^3.

O(1)–C(3)	1·437 (10)	C(7)—C(8)	1·430 (10)
O(2)–C(19)	1·479 (10)	C(8)—C(9)	1·377 (10)
C(1)–C(2)	1·546 (11)	C(8)—C(14)	1·489 (12)
C(1)–C(10)	1·516 (9)	C(9)—C(10)	1·575 (11)
C(2)–C(3)	1·484 (11)	C(9)—C(11)	1·483 (10)
C(3)–C(4)	1·528 (10)	C(10)–C(20)	1·559 (12)
C(4)–C(5)	1·643 (10)	C(11)–C(12)	1·511 (12)
C(4)–C(18)	1·559 (11)	C(12)–C(13)	1·520 (10)
C(4)–C(19)	1·496 (11)	C(13)–C(14)	1·568 (11)
C(5)–C(6)	1·501 (9)	C(13)–C(15)	1·563 (13)
C(5)–C(10)	1·543 (9)	C(13)–C(17)	1·461 (12)
C(6)–C(7)	1·523 (12)	C(15)–C(16)	1·329 (13)

0·2 times that for the hydrogens. No relevant residual peak or trough was apparent in the final ΔF map. The final positional and thermal parameters and their standard deviations, as obtained from the inverse of the block-diagonal matrix, are listed in Tables 3 and 4. A list of the observed and calculated structure-factor amplitudes is given in Table 5. Atomic scattering factors for O and C were those given by Cromer & Waber (1965) and for H those of Stewart, Davidson & Simpson (1965).

Description of the structure

A general view of the molecule is shown in Fig. 1. The bond lengths, the valency angles, and the most significant planes in the molecule with the deviations of other atoms and interplanar angles, are given in Tables 6, 7 and 8 respectively. The overall shape of the molecule is determined by the *A*, *B* and C rings, all three having a chair shape with the same orientation (within 10°). As can be seen in Table 8, the torsion angles between the central rectangle in the ring and the end triangles, stretching out on opposite sides to form the flattened chair, are around 50°, except the ones where the C=C double bond is concerned; these in fact exhibit a much more remarkable flattening. The atoms adjacent to or involved in the double bond within the rings reveal anyway a noticeable displacement from planarity, namely C(9), which lies −0·243 Å from the C(7), C(10), C(11), C(14) plane and −0·223 Å from the C(8), C(10), C(11) plane. The lesser distances of C(8) from the former plane (−0·053 Å) and from the C(7), C(9), C(14) plane (0·036 Å) can be attributed to its greater distance from the perturbing methyl group, C(20), with its bonded H's, which lies only 2·545 (12) Å from C(9). An indirect indication of the above facts can also be deduced from the angular values of Table 7.

Table 7. *Valency angles (°) involving only carbon and oxygen atoms*

E.s.d.'s of the angles (×10) appear in parentheses.

C(2)—C(1)–C(10)	113·2 (6)	C(8)—C(9)—C(11)	120·5 (7)
C(1)—C(2)–C(3)	110·8 (7)	C(10)–C(9)—C(11)	115·4 (6)
O(1)—C(3)–C(2)	111·8 (7)	C(1)—C(10)–C(5)	110·6 (6)
O(1)—C(3)–C(4)	107·5 (6)	C(1)—C(10)–C(9)	110·3 (6)
C(2)—C(3)–C(4)	115·5 (7)	C(1)—C(10)–C(20)	109·1 (7)
C(3)—C(4)–C(5)	107·5 (5)	C(5)—C(10)–C(9)	106·1 (6)
C(3)—C(4)–C(18)	111·4 (6)	C(5)—C(10)–C(20)	112·0 (6)
C(3)—C(4)–C(19)	113·2 (6)	C(9)—C(10)–C(20)	108·7 (6)
C(5)—C(4)–C(18)	104·6 (6)	C(9)—C(11)–C(12)	112·3 (7)
C(5)—C(4)–C(19)	110·8 (6)	C(11)–C(12)–C(13)	111·8 (6)
C(18)–C(4)–C(19)	109·1 (6)	C(12)–C(13)–C(14)	106·5 (6)
C(4)—C(5)–C(6)	112·8 (5)	C(12)–C(13)–C(15)	108·6 (6)
C(4)—C(5)–C(10)	115·7 (5)	C(12)–C(13)–C(17)	114·1 (7)
C(6)—C(5)–C(10)	113·1 (6)	C(14)–C(13)–C(15)	104·3 (7)
C(5)—C(6)–C(7)	109·7 (6)	C(14)–C(13)–C(17)	108·8 (7)
C(6)—C(7)–C(8)	118·2 (7)	C(15)–C(13)–C(17)	113·8 (7)
C(7)—C(8)–C(9)	124·7 (7)	C(8)—C(14)–C(13)	116·0 (7)
C(7)—C(8)–C(14)	114·4 (6)	C(13)–C(15)–C(16)	127·1 (9)
C(9)—C(8)–C(14)	120·7 (6)	O(2)—C(19)–C(4)	110·5 (6)
C(8)—C(9)–C(10)	117·2 (6)		

The other C(15)=C(16) double bond, being in a terminal position, does not exhibit any special feature; the C(15)–C(17) distance is 2·534 (14) Å, C(16)–C(17) 2·980 (15) Å, and the C(13)–C(15)–C(16) angle is 127·1 (9)°.

The chemically equivalent bonds are all very similar; the average values of bond distances for the various types of bonds have been calculated as 1·524 Å for C–C, 1·353 Å for C=C, and 1·458 Å for C–O; these are in agreement with expected values. All C–H distances lie within the limits given in the literature; they range from 0·91 to 1·20 Å, the mean distance being 1·06 Å.

Table 9. *Selected intramolecular distances* (Å) *and e.s.d.'s*

O(1)—O(2)	2·764 (8)	O(1)—H(2)	1·82 (7)
O(2)—C(18)	2·765 (11)	O(2)—H(26)	1·94 (9)
C(16)–C(17)	2·980 (15)	O(1)—H(26)	1·97 (8)
O(1)—C(18)	2·992 (10)		

The arrangement of the molecules in the structure is shown in Fig. 2; the most significant intramolecular distances are shown in Table 9. The plane which gives

Table 8. *Some least-squares planes through various groups of* C *atoms in the molecule*

The plane equations are of the form $PX+QY+RZ=S$ where X, Y and Z are expressed in Å relative to orthogonal axes parallel to **a**, **b** and **c**.

Ring	Plane	Atoms	P	Q	R	S	R.m.s. deviation (Å)
A	*a*	C(1), C(2), C(4), C(5)	0·8845	0·3665	−0·2887	1·4809	0·0380
	*a*1	C(2), C(3), C(4)	0·8622	−0·4787	−0·1657	0·3153	
	*a*2	C(1), C(5), C(10)	0·9072	−0·3782	−0·1844	−0·5661	
B	*b*	C(6), C(7), C(9), C(10)	0·8102	0·5272	−0·2561	2·7825	0·0373
	*b*1	C(5), C(6), C(10)	0·9074	−0·3769	−0·1857	−0·5672	
	*b*2	C(7), C(8), C(9)	0·9217	0·3156	−0·2258	1·8840	
C	*c*	C(8), C(11), C(12), C(14)	0·8244	0·5002	−0·2649	2·8399	0·1115
	*c*1	C(8), C(9), C(11)	0·9433	0·1246	−0·3076	0·3356	
	*c*2	C(12), C(13), C(14)	0·9596	−0·2570	−0·1144	−2·0374	
	d	C(1)–C(14)	0·9353	0·2791	−0·2174	1·7747	0·2575

Deviations (Å) from least-squares planes

	a	*b*	*c*	*d*
C(1)	−0·034			−0·018
C(2)	0·034			0·205
C(3)				−0·473
C(4)	−0·032			−0·032
C(5)	0·032			−0·132
C(6)		−0·032		0·309
C(7)		0·033		−0·046
C(8)			−0·097	−0·064
C(9)		−0·032		−0·030
C(10)		0·031		0·465
C(11)			0·096	0·214
C(12)			−0·096	−0·344
C(13)				0·190
C(14)			0·097	−0·242

Angles (°) between planes

	a	*b*	*c*	*d*
a				7·1
*a*1	50·6			44·9
*a*2	44·2			38·5
b	10·3			16·1
*b*1		54·3		38·4
*b*2		13·8		2·3
c	8·5	1·8		14·5
*c*1			22·9	10·3
*c*2			46·1	31·7

Angles (°) between two-heavy-atom substituents and ring planes

	A	*C*
O(2)—C(19)	80·1	
C(15)–C(16)		66·7

the best fit to the 14 ring atoms was determined by least-squares calculation to be:

$$0·9353x+0·2791y-0·2174z=1·7747\ .$$

An interesting feature of the molecule is that the two OH groups are hydrogen-bonded within the molecule, the O(1)–O(2) intramolecular distance being 2·764 (8) Å, O(2)–H(2) being 0·98 (7) Å, and the O(1)–H(2) distance 1·82 (7) Å; the O(1)···H(2)–O(2) angle is 162 (6)°. The other hydroxyl hydrogen atom provides the O–O intermolecular connexion (see below). In Table 10 the torsion angles within the rings are given.

Table 10. *Selected torsional angles* (°)

Ring *A*	
C(1)—C(2)—C(3)—C(4)	58·8
C(2)—C(3)—C(4)—C(5)	51·5
C(3)—C(4)—C(5)—C(10)	46·2
C(4)—C(5)—C(10)–C(1)	47·8
C(5)—C(10)–C(1)—C(2)	52·3
C(10)–C(1)—C(2)—C(3)	57·6
Ring *B*	
C(5)—C(6)—C(7)—C(8)	30·6
C(6)—C(7)—C(8)—C(9)	12·8
C(7)—C(8)—C(9)—C(10)	18·2
C(8)—C(9)—C(10)–C(5)	39·5
C(9)—C(10)–C(5)—C(6)	60·2
C(10)–C(5)—C(6)—C(7)	56·6
Ring *C*	
C(8)—C(9)—C(11)–C(12)	32·2
C(9)—C(11)–C(12)–C(13)	55·2
C(11)–C(12)–C(13)–C(14)	59·8
C(12)–C(13)–C(14)–C(8)	43·9
C(13)–C(14)–C(8)—C(9)	24·0
C(14)–C(8)—C(9)—C(11)	17·3

None of the intermolecular contacts are abnormal; the closest approaches between carbon atoms of different molecules are 3·715 (12) Å [C(14)–C(18)] and 3·770 (12) Å [C(1)–C(15)]; O(2) has three neighbours: C(3), C(2) and C(12) at 3·465 (9), 3·520 (9), 3·730 (10) Å respectively. The C–H intermolecular distances are from 2·63 Å upward. The crystals contain discrete molecules linked into chains along **c** by O···O hydrogen bonds. It is evident from Fig. 2 that a molecule is bonded *via* an O(1)–H(1)···O(2) bond to its $(\frac{1}{2}-x, -y, \frac{1}{2}+z)$ neighbour, to make helices every second screw axis. The intermolecular O(1)–O(2) distance is 2·946 (8) Å, O(1)–H(1) is 1·18 (6) Å, and the H(1)···O(2) distance is 1·90 (6) Å, probably influenced by the normal shortening of O···H bond lengths in X-ray diffraction. The O(1)–H(1)···O(2) angle is 145 (5)°, and the C(3)–O(1)–H(1) angle is 110 (3)°, fairly close to the tetrahedral angle.

Each of the above infinite zigzag chains shows the molecules alternatively stretching out on opposite sides in the **b** direction at approximately a right angle to the chain axis, and at approximately 17° to **b** in the **ab** plane, in a fern-like alternate pinnate arrangement. The molecules, connected in this way, are interlayered with their neighbours in the **a** and **b** directions through overlapping; this is obvious if one looks at their packing along the **a** direction. The contacts between the chains are provided by van der Waals interactions, the greater number of them taking place in the **ab** plane. Besides the H(1) hydrogen bond which produces the chain, quite a few additional van der Waals contacts are found in the **c** direction.

Beside the *FILTRO* program and other local programs for the CII 10070 computer, extensive use was made of *ORFLS* (Busing, Martin & Levy, 1962) with a block-diagonal modification, of *ORTEP* (Johnson, 1965), and of *BONDLA* and *LSQPL* from the X-RAY System (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).

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