(Williams, Sheridan & Gordy, 1952). The P—F bond length in the present compound agrees closely with the axial P—F distance (1·57 Å) in the novel dimer $(CH_3NPF_2C_6H_5)_2$ (Cox & Corey, 1967). The C—C and C—N distances and inner ring angles are not unusual for phosphorus five-membered-ring heterocyclic compounds (Lee & Goodacre, 1971*a*,*b*; Corbridge, 1974). No unusually short internuclear separations were detected between the nonhydrogen atoms. Fig. 2 shows an [010] projection of the unit cell.

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The Crystal Structure of (—)-Avenaciolide

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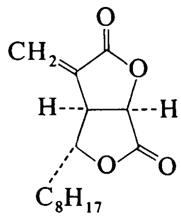
(—)-Avenaciolide, the naturally occurring antifungal metabolite of *Aspergillus avenaceus*, forms colourless, euhedral crystals which are orthorhombic, $P2_12_12_1$, with a = 7·006 (1), b = 33·475 (6), c = 6·279 (1) Å. The structure has been determined from diffractometer intensities (monochromated Mo $K\alpha$ radiation, $2\theta \leq$ 50°, 1546 unique reflections) by direct methods. On refinement to convergence, R = 0·083 for all data. In the bislactone 'head', the ring with the methylene-group substituent has a twist conformation with the two bridgehead C atoms out-of-plane. The other ring has an envelope conformation. The octyl-group 'tail' is fully extended with torsion angles at all C—C bonds *ca* 180°. The crystal consists of stacked bilayers, with bands of the bislactone 'heads' and of close-packed, parallel, hydrocarbon chains which have an orthorhombic ($O_\perp$) subcell.

Introduction

(—)-Avenaciolide is a naturally occurring antifungal metabolite of *Aspergillus avenaceus*. Its formula, (I), determined by chemical and spectroscopic means (Brookes, Tidd & Turner, 1963), shows an unusual bislactone fused-ring 'head' system and a paraffinic octyl group as 'tail'.



(I)

This compound has been of particular interest recently as an ionophore (Harris & Wimhurst, 1973), facilitating the transfer of Mg^{2+}, Ca^{2+} and K^+ ions from aqueous to organic phases, and causing the release of Ca^{2+} and Mg^{2+} from rat-liver mitochondria. However, no crystalline complexes of avenaciolide with these cations have yet been isolated, and an X-ray analysis of the free molecule was undertaken to determine the conformation and to examine the likelihood of its complex formation with cations.

Experimental

Crystals of avenaciolide are euhedral and colourless. They vary in shape from thin needles to almost square plates, but they usually have one short edge (parallel to the long b axis). The crystal mounted for examination was 0·25 × 0·10 × 0·40 mm.

Crystal data

$C_{15}H_{22}O_4$, $M_r = 266{\cdot}34$, orthorhombic, $a = 7{\cdot}006$ (1), $b = 33{\cdot}475$ (6), $c = 6{\cdot}279$ (1) Å, $U = 1472{\cdot}6$ Å^3, $D_m = 1{\cdot}185$, $Z = 4$, $D_c = 1{\cdot}206$ g cm^{-3}, $F(000) = 576$, μ(Mo $K\bar{\alpha}$) = 0·81 cm^{-1}, λ(Mo $K\bar{\alpha}$) = 0·71069 Å, space group $P2_12_12_1$, m.p. 51·0–51·5 °C (sharp; no indication of the formation of a liquid-crystal structure).

The space group and preliminary cell dimensions were found from photographs. The dimensions were refined from Guinier powder photographic measurements and later improved from settings of 24 reflections (2θ in the range 33–41°) centred automatically on an Enraf–Nonius CAD-4 diffractometer. The crystal was mounted with **c** coincident with the rotation axis, and intensities were measured on a Stoe automated two-circle diffractometer with monochromated Mo $K\alpha$ radiation; intensities of 1546 unique reflections with $2\theta \le 50°$ were corrected for Lorentz and polarization effects, but not for absorption.

Structure analysis

Normalized structure amplitudes were calculated by the K-curve method (Karle & Karle, 1965), and the E statistics tended towards the acentric value (Germain, Main & Woolfson, 1970). From 196 reflections with $|E| \ge 1{\cdot}50$, *MULTAN* produced three better-looking sets of phases, each with ABSFOM ≃ 1·02 and RESID ≃ 24·2, and an E map with one of these showed the molecule.

Refinement of the non-hydrogen atom parameters (with anisotropic temperature factors) to $R = 0{\cdot}164$ was rapid. Coordinates of H atoms were calculated or located from a difference map; these and isotropic temperature factors were included in the final cycles. In the minimization function, $\sum w(|F_o| - |F_c|)^2$, *i.e.* $\sum w\Delta^2$, the weight w was adjusted to give approximately constant mean values of $w\Delta^2$ over several ranges of F_o, and was finally set to $w = 1/[\sigma_c^2(1{\cdot}401 - 0{\cdot}3924F_o + 0{\cdot}0749F_o^2)]$, where σ_c is the standard deviation derived from the measured intensity counts. Scattering factors for C and O atoms were from *International Tables for X-ray Crystallography* (1962), and for H atoms from Stewart, Davidson & Simpson (1965). At convergence, $R = 0{\cdot}083$ for all data, and $R' = 0{\cdot}081$.

It has not been possible to identify the natural enantiomer of avenaciolide by anomalous-dispersion methods. With the anomalous-scattering coefficients for O atoms (Cromer & Liberman, 1970), the calculation of structure amplitudes for the two enantio-

Table 1. *Fractional atomic coordinates* (×10^4)

E.s.d.'s are in parentheses.

	x	y	z
O(1)	3215 (5)	4639 (1)	514 (4)
O(21)	4608 (6)	4636 (1)	−2655 (4)
O(5)	3331 (5)	3973 (1)	3990 (5)
O(62)	848 (5)	4386 (1)	4388 (5)
C(2)	4761 (8)	4598 (1)	−748 (6)
C(3)	6463 (8)	4507 (1)	505 (6)
C(31)	5744 (7)	4422 (1)	2709 (6)
C(32)	8154 (10)	4513 (2)	−243 (8)
C(4)	5233 (8)	3974 (2)	3071 (7)
C(6)	2461 (8)	4326 (2)	3836 (6)
C(61)	3800 (7)	4618 (1)	2738 (6)
C(41)	6513 (10)	3740 (2)	4520 (9)
C(42)	5949 (11)	3305 (2)	4639 (11)
C(43)	6960 (11)	3064 (2)	6354 (11)
C(44)	6232 (11)	2639 (2)	6538 (10)
C(45)	7049 (12)	2407 (2)	8403 (12)
C(46)	6195 (11)	1990 (2)	8653 (10)
C(47)	6962 (13)	1769 (2)	10560 (10)
C(48)	6053 (15)	1368 (2)	10853 (15)
H(321)	8302 (70)	4587 (12)	−1861 (63)
H(322)	8998 (66)	4424 (15)	222 (62)
H(311)	6521 (59)	4509 (10)	3693 (49)
H(401)	5397 (94)	3847 (18)	1499 (80)
H(611)	3736 (59)	4887 (13)	3313 (58)
H(411)	7761 (75)	3746 (13)	4002 (64)
H(412)	6595 (60)	3874 (11)	5975 (53)
H(421)	4860 (90)	3275 (19)	5019 (91)
H(422)	6384 (89)	3174 (18)	3290 (86)
H(431)	8253 (108)	3077 (19)	6383 (97)
H(432)	6808 (84)	3213 (16)	7833 (78)
H(441)	4892 (78)	2648 (14)	6944 (64)
H(442)	6410 (87)	2493 (18)	5220 (85)
H(451)	8439 (118)	2379 (22)	8571 (105)
H(452)	6823 (121)	2597 (21)	9330 (97)
H(461)	4685 (73)	2015 (13)	8920 (61)
H(462)	6441 (100)	1862 (19)	7468 (78)
H(471)	8441 (105)	1742 (19)	10661 (93)
H(472)	6713 (108)	1959 (20)	11554 (95)
H(481)	6487 (100)	1197 (20)	11843 (90)
H(482)	4369 (102)	1376 (21)	11533 (102)
H(483)	6221 (95)	1202 (18)	9928 (73)

mers did not show any significant differences either for individual reflections or collectively (over all polar data) in R. Initially, the absolute configuration was assigned from the degradations of avenaciolide to (+)-nonylsuccinic acid (Brookes, Tidd & Turner, 1963), but more recently the synthesis of (–)-avenaciolide from D-glucose has indicated the opposite configuration (Anderson & Fraser-Reid, 1975); this latter determination is now accepted as correct and the figures and data presented in this paper represent this enantiomer.

Atomic coordinates are in Table 1 and a view of the molecule, with the atomic numbering scheme, is in Fig. 1. Molecular dimensions are shown in Figs. 2 (bond lengths), 3 (valence angles) and 4 (principal torsion angles).*

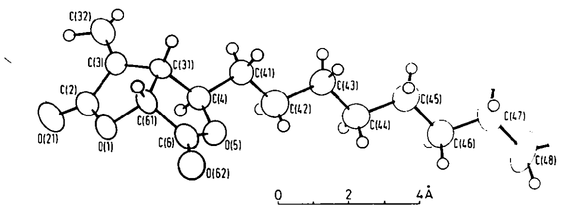


Fig. 1. View of the molecule, and atomic designations. Boundaries of thermal ellipsoids are shown for the C and O atoms; the H atoms are represented by small circles.

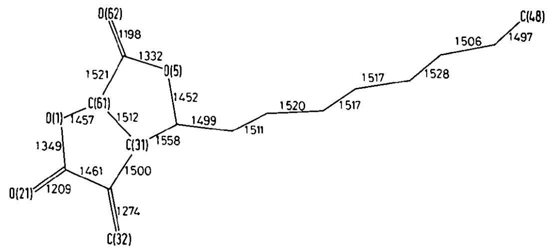


Fig. 2. Projection of the molecule, showing principal bond lengths (Å); e.s.d.'s are in the range 0·005–0·010 Å.

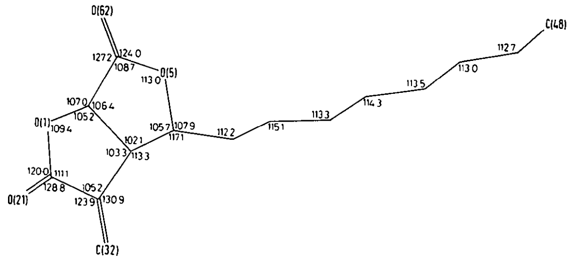


Fig. 3. Valence angles (°); e.s.d.'s are in the range 0·3–0·7°.

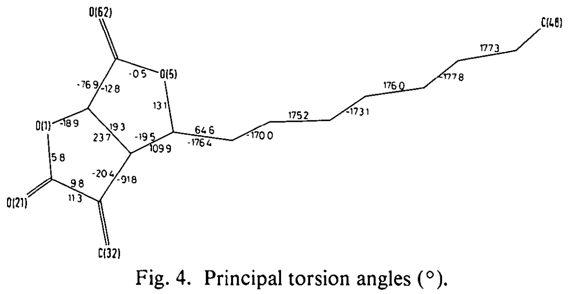


Fig. 4. Principal torsion angles (°).

Computing

MULTAN (Germain, Main & Woolfson, 1970), the full-matrix least-squares-refinement program *NUCLS* (R. J. Doedens and J. A. Ibers), the molecular-dimensions program *ORFFE* (Busing, Martin & Levy, 1964) (which also calculates statistical errors from the full-correlation matrix from *NUCLS*), and the plotting program *ORTEP* (Johnson, 1965) were adapted by Owen (1975) to run on this Station's ICL 4/70 computer. The remaining computing used our *X-RAY ARC* (1973) programs for the IBM 1130 computer.

Discussion

The conformations in the unusual furo[3,4-*b*]furan ring system may be described by the scheme of Altona,

* Lists of structure factors, thermal parameters and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33861 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Mean planes*

(*a*) Deviations (Å) of atoms from mean planes. The values marked with an asterisk denote atoms *not* used in the calculation of the plane. E.s.d.'s in these values are *ca* 0·004 Å.

Plane *A*, in the ring of O(1)
O(1) 0·0, C(2) 0·0, C(3) 0·0, C(31) –0·25,* C(61) 0·14*

Plane *B*, in the ring of O(5)
C(4) –0·002, O(5) 0·002, C(6) –0·003, C(61) 0·001, C(31) 0·33*

Plane *C*, about C(2)
O(1) 0·000, C(2) 0·002, C(3) –0·001, O(21) –0·001

Plane *D*, about C(3)
C(2) 0·001, C(3) –0·005, C(31) 0·001, C(32) 0·004

Plane *E*, about C(6)
O(5) 0·0, C(6) 0·026,* C(61) 0·0, O(62) 0·0

Plane *F*, of C(41)–C(43) of the octyl tail group
C(4) 0·24,* C(41) 0·0, C(42) 0·0, C(43) 0·0, C(44) 0·18,* C(45) 0·28,* C(46) 0·50,* C(47) 0·72,* C(48) 0·99*

(*b*) Equations of the planes, in the form $lX + mY + nZ = p$, where X, Y and Z are atomic coordinates (in Å) with respect to the crystallographic axes a, b and c.

Plane	l	m	n	p
A	0·1895	0·9775	0·0917	15·6374
B	0·3803	0·2647	0·8861	6·6277
C	0·1894	0·9779	0·0881	15·6418
D	0·0867	0·9679	0·2357	15·0771
E	0·3465	0·2740	0·8971	6·7009
F	–0·7249	0·2301	0·6492	1·4160

Geise & Romers (1968). The angle of pseudorotation, Δ (which marks the position of the pucker in the ring), is measured for each ring in a clockwise manner from the midpoint of C(31)–C(61); the maximum torsion angle φ_m is a measure of the amplitude of puckering. Results for the O(1) ring are: $\Delta = 8{\cdot}6$ and $\varphi_m = 23{\cdot}8°$, indicating a C(31)/C(61) twist arrangement; for the O(5) ring: $\Delta = -37{\cdot}7$ and $\varphi_m = 20{\cdot}4°$, an almost perfect C(31) envelope conformation. These conformations can also be derived from the ring torsion angles (Fig. 4) and the mean-planes data of Table 2.

Torsion angles about C(31)–C(61) show twisting of *ca* 20° from the eclipsed position.

The substituent groups about C(2) and C(3) each form good planes (Table 2, planes *C* and *D*), but are rotated about C(2)–C(3) by 10·3° from coplanarity: the short C(2)–C(3) bond indicates delocalization of π electrons between the two double-bond substituents.

At the other carbonyl group, C(6) is 0·025 Å (5σ) from the plane of its three neighbours; *i.e.* there is an umbrella arrangement about this atom (plane *E*).

The lone pairs of the O atoms in this fairly rigid bislactone ring structure are not suitably oriented for quadridentate complex formation; chelation through O(1) and O(62) appears feasible. However, attempts to form complexes of avenaciolide by reaction with Mg and Ca thiocyanates in methanol gave rapid production of syrups which were thought to be polymeric complexes; crystalline products have not been achieved (Truter, 1976).

In the paraffinic tail at C(4), C(41)–C(42) is *trans* to C(4)–C(31), and the chain has an extended conformation with all torsion angles *ca* 180°. There is slight bowing of the plane of the C atoms of the tail (plane *F*).

The packing is shown in Fig. 5. There are bands, normal to **b**, of the bislactone 'heads' and of the paraffinic 'tails', an arrangement very similar to the bilayer systems of decyl α-D-glucopyranoside (Moews & Knox, 1976) and glucosylphytosphingosine hydrochloride (Abrahamsson, Dahlén & Pascher, 1977). The octyl chains, although short in comparison with those of many long-chain fatty acids and their ester derivatives of steroids, form close hydrocarbon packing. The idealized subcell of this chain packing has dimensions: $a_s = 7{\cdot}0$, $b_s = 5{\cdot}4$, $c_s = 2{\cdot}52$ Å, and the volume per methylene group is 24·0 Å^3; its symmetry is 'common orthorhombic': $O_\perp$ in the nomenclature of Abrahamsson (1959) and Larsson (1965), and O_a in the comprehensive system of Segerman (1965). There are several normal van der Waals interactions between neighbouring chains, and between the end methyl group and the surroundings of its pocket.

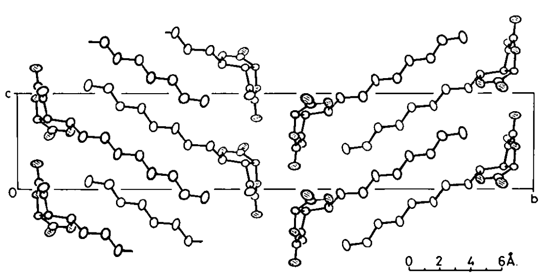


Fig. 5. The packing arrangement viewed down **a**.

Table 3. *Shorter intermolecular contacts* (Å)

a or a_i	b or b_j	$a \cdots b_j$ or $b \cdots a_i$	j	i
O(21)	C(6)	2·863	(I)	(II)
O(21)	C(61)	2·949	(I)	(II)
O(21)	C(31)	3·102	(I)	(II)
O(1)	C(61)	3·348	(III)	(IV)
C(32)	C(48)	3·600	(V)	(VI)
C(2)	C(61)	3·742	(III)	(IV)
C(32)	C(61)	3·825	(VII)	(VIII)
C(43)	C(48)	3·862	(IX)	(X)
C(31)	C(32)	3·869	(VIII)	(VII)
C(2)	C(6)	3·872	(I)	(II)
C(6)	C(48)	3·879	(VI)	(V)
O(1)	H(611)	2·51	(III)	(IV)
O(21)	H(311)	2·69	(I)	(II)
O(21)	H(611)	2·74	(I)	(II)
C(43)	H(482)	2·85	(IX)	(X)
C(2)	H(611)	3·05	(III)	(IV)
C(6)	H(483)	3·08	(VI)	(V)
C(48)	H(322)	3·09	(VI)	(V)
C(32)	H(611)	3·10	(VII)	(VIII)
C(43)	H(471)	3·16	(X)	(IX)
C(45)	H(461)	3·16	(IX)	(X)
C(32)	H(482)	3·20	(V)	(VI)
C(41)	H(482)	3·21	(IX)	(X)
C(48)	H(431)	3·21	(X)	(IX)
C(32)	H(483)	3·22	(V)	(VI)
C(47)	H(441)	3·24	(IX)	(X)
C(45)	H(451)	3·24	(X)	(IX)
C(47)	H(431)	3·27	(X)	(IX)
H(432)	H(482)	2·30	(IX)	(X)
H(431)	H(482)	2·38	(IX)	(X)
H(432)	H(471)	2·55	(X)	(IX)

Symmetry code

(I)	x,	y,	$-1+z$	(II)	x,	y,	$1+z$
(III)	$\frac{1}{2}-x$,	$1-y$,	$-\frac{1}{2}+z$	(IV)	$\frac{1}{2}-x$,	$1-y$,	$\frac{1}{2}+z$
(V)	$\frac{1}{2}+x$,	$\frac{1}{2}-y$,	$1-z$	(VI)	$-\frac{1}{2}+x$,	$\frac{1}{2}-y$,	$1-z$
(VII)	$\frac{3}{2}-x$,	$1-y$,	$-\frac{1}{2}+z$	(VIII)	$\frac{3}{2}-x$,	$1-y$,	$\frac{1}{2}+z$
(IX)	$\frac{1}{2}+x$,	$\frac{1}{2}-y$,	$2-z$	(X)	$-\frac{1}{2}+x$,	$\frac{1}{2}-y$,	$2-z$

There are also close contacts between the bislactone 'heads', around the screw axis parallel to **c** and between molecules translated in the **c** direction. O(21) lies particularly close to C(31), C(61) and C(6) of the molecule at $x, y, z-1$. These and other short contacts are in Table 3.

I thank Dr W. B. Turner (ICI Ltd, Pharmaceuticals Division) for a sample of the natural compound, Dr I. W. Nowell (Sheffield City Polytechnic) for the measurement of diffraction intensities, Dr M. R. Truter for advice and encouragement, and the Royal Society for some equipment.

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The Crystal and Molecular Structure of Decaphenylcyclopentasilane, $C_{60}H_{50}Si_5$

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$C_{60}H_{50}Si_5$ is monoclinic, space group $C2/c$, with $a = 39{\cdot}721$ (8), $b = 12{\cdot}862$ (3), $c = 22{\cdot}701$ (4) Å, $\beta = 99{\cdot}62$ (2)°, $Z = 8$. The structure was refined to $R = 0{\cdot}113$ for 5504 non-zero reflections. The nucleus of the molecule is a five-membered Si ring puckered in a form intermediate between C_s and C_2 symmetry. The Si–Si lengths vary between 2·371 and 2·413 Å (average 2·396 Å) and the Si–Si–Si angles between 102·7 and 106·7° (average 104·5°). The Si–C lengths range from 1·869 to 1·917 Å (average 1·895 Å). The planes of the Si–C bond pairs of the $SiPh_2$ groups are almost perpendicular to the adjoining Si–Si–Si plane with a maximum deviation of 10·5°.

Introduction

The nucleus of decaphenylcyclopentasilane (DPHCPSI) is a five-membered Si ring similar to gaseous cyclopentasilane, Si_5H_{10}, which has been studied by electron diffraction (Smith, Seip, Hengge & Bauer, 1976). An analysis of the decaphenyl derivative was carried out to determine the conformation of the cyclopentasilane ring and the Si–Si lengths in a system where ring strain is likely to be greater than in cyclopentasilane but smaller than in octaphenylcyclotetrasilane (OPHCTSI: Párkányi, Sasvári & Barta, 1978).

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