Discussion. In both structures the ethylene groups are disordered. This disorder is evident in many ET-based structures (Leung *et al.*, 1986; Psycharis *et al.*, 1988; Terzis, Psycharis *et al.*, 1988). The difference maps showed two positions for the ethylene group, *A* and *B*. The site occupancies for these two positions were refined, keeping the sum of the two constant at one. Occupancies for *A* refined to 0·598 (3) (I) and 0·653 (2) and 0·601 (4) for the cations (1) and (2) of (II). Bond lengths and angles of the cations are given in Table 2. The anion in (II) is disordered as well. The difference map shows a plethora of peaks around the position of the boron. We accepted two models for BF_4^- with site occupancies equal to 0·5. At this stage a ΔF synthesis showed one weak and two strong peaks, the weaker one being at a center of symmetry. We assumed this to be a solvent molecule, CH_2Cl_2, disordered so that the average structure appears centrosymmetric. It was included in the refinement with site occupancy of 0·5. The bond distances and angles of the anion and the solvent molecule were not satisfactory and the *U*'s were very large.

In both structures the donors pack side by side in the plane of the molecule with strong S···S and weak S···N contacts (Fig. 2). Stacking of the donors is observed (Figs. 3 and 4), with weak intrastack S···S contacts between 3·64 and 3·80 Å. This mode of packing generates the 'sheet networks' (Fig. 4), of donor molecules that have been observed in all other salts of this donor that we have studied (Psycharis *et al.*, 1988; Terzis, Psycharis, *et al.*, 1988). This mode of packing apparently does not afford the superconducting state and these salts undergo a metal-to-semiconductor (I) (Terzis, Hountas *et al.*, 1988) and metal-to-insulator (II) (Papavasiliou, Underhill, Kaye, Geserich, Terzis & Yiannopoulos, 1987) transition at 280 K (I) and 180 K (II).

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Structure of a Tetraquinane Intermediate in the Synthesis of the Antibiotic Crinipellin A

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Abstract. 3αH,7αH-12α-Isopropyl-8β,11α-dimethyltetracyclo[6.6.0.$0^{1,11}$.$0^{3,7}$]tetradecane-6,9-dione, $C_{19}H_{28}O_2$, $M_r = 288{\cdot}43$, monoclinic, $P2_1/n$, $a = 9{\cdot}001$ (1), $b = 8{\cdot}106$ (1), $c = 22{\cdot}106$ (4) Å, $\beta = 96{\cdot}14$ (1)°, $V = 1603{\cdot}7$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}195$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5{\cdot}5$ cm^{-1}, $F(000) = 632$, $T = 295$ K, $R = 0{\cdot}062$, $wR = 0{\cdot}067$ for 2457 observed reflections, $I \geq 3{\cdot}0\sigma(I)$. The ring skeleton consists of four fused five-membered rings. Rings *A* and *C* are in the envelope conformation while rings *B* and *D* are half chairs. The isopropyl group is in the 12α-orientation. Crystal packing is solely dictated by van der Waals forces.

Introduction. Polyquinanes, particularly those formed by linear or angular fusion of three five-

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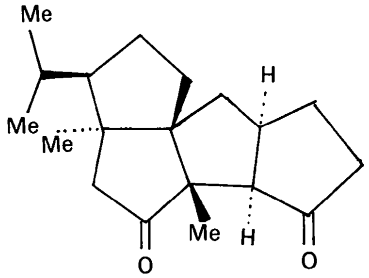
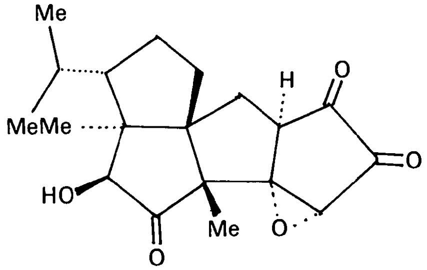
membered rings, are well known (Paquette & Doherty, 1987). However, polyquinane natural products with four fused five-membered rings (tetraquinanes) have remained unknown until the recently reported structure determination of crinipellin diterpenes (Anke, Heim, Knoch, Mocek, Steffan & Steglich, 1985). The title compound (I) is an intermediate obtained during the synthesis of the complex antibiotic crinipellin A [(II): Mehta, Srinivasa Rao & Sreenivas Reddy (1988)]. This X-ray analysis was performed to establish the stereochemistry of the isopropyl group.



(I)



(II)

Experimental. Colourless needle (m.p. 388 K) 0·5 × 0·4 × 0·3 mm from hexane. Three-dimensional intensity data collected on an Enraf–Nonius CAD-4 automatic diffractometer, $\omega/2\theta$ scan mode with $2 \leq 2\theta \leq 130°$, Cu $K\alpha$ radiation, data corrected for direct-beam polarization and Lorentz factor and for absorption (the average transmission factor is 0·818 with maximum and minimum values of 0·975 and 0·661), $0 \leq h \leq 10$, $0 \leq k \leq 9$, $-25 \leq l \leq 25$, 2760 measured data, 2457 unique and 2364 observed with $I \geq 3{\cdot}0\sigma(I)$, cell constants from 24 reflections with $24 \leq 2\theta \leq 84°$, three standard reflections monitored every 100 reflections showed no significant variation in intensity, scan width = (0·8 + 0·14tanθ)°, aperture width = (4·0 + 2tanθ) mm. The maximum time spent on any reflection measurement was 30 s and the background count was half the scan time. Structure solution by direct methods, H atoms from $\Delta\rho$ synthesis, full-matrix least-squares refinement on F, non-H atoms anisotropic and H atoms isotropic to a final $R = 0{\cdot}062$ and $wR = 0{\cdot}067$ with individual weights based on counting statistics: $w = 4(F_o)^2/\sigma^2(F_o)^2$, where $\sigma^2(F_o)^2 = [\sigma^2(I) + p^2(F_o^2)^2]^{1/2}/\mathrm{Lp}$ where p (=0·02) is the instrumental instability factor, $(\Delta/\sigma)_{\max} = 0{\cdot}03$, $(\Delta/\sigma)_{\rm mean} = 0{\cdot}02$, $S = 8{\cdot}29$ for 302 parameters, final $\Delta\rho$ variations within $\pm 0{\cdot}35$ e Å^{-3}. All calculations on a VAX 11/730 computing system using the *SDP* package (Frenz, 1978). Atomic scattering factors from Cromer & Waber (1974).

Table 1. *Positional and equivalent isotropic thermal parameters for non-*H *atoms and their e.s.d.'s*

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as: $B_{\rm eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

	x	y	x	$B_{\rm eq}$(Å^2)
C1	1·0014 (3)	0·0520 (4)	0·8297 (1)	3·57 (6)
C2	0·8562 (3)	−0·0445 (4)	0·8328 (1)	4·02 (7)
C3	0·7378 (3)	0·0897 (4)	0·8294 (1)	3·87 (6)
C4	0·8052 (3)	0·2349 (4)	0·8676 (1)	3·16 (6)
C5	0·9842 (3)	0·2072 (4)	0·8693 (1)	3·52 (6)
C6	1·0365 (3)	0·1731 (5)	0·9373 (1)	4·44 (7)
C7	0·9247 (3)	0·2501 (4)	0·9729 (1)	3·69 (6)
C8	0·7704 (3)	0·2422 (4)	0·9359 (1)	3·08 (5)
C9	0·6895 (3)	0·4067 (4)	0·9437 (1)	3·42 (6)
C10	0·7317 (3)	0·5197 (4)	0·8932 (1)	3·88 (6)
C11	0·7434 (3)	0·3985 (4)	0·8411 (1)	3·94 (6)
C12	0·5167 (3)	0·4078 (4)	0·9358 (1)	4·04 (6)
C13	0·4626 (4)	0·5447 (5)	0·8927 (2)	4·85 (8)
C14	0·6019 (4)	0·6445 (4)	0·8815 (2)	4·83 (8)
C15	0·6942 (3)	0·0896 (4)	0·9605 (1)	4·04 (7)
C16	1·0644 (4)	0·3575 (5)	0·8482 (2)	5·34 (8)
C17	1·1412 (3)	−0·0583 (4)	0·8423 (1)	4·31 (7)
C18	1·1387 (4)	−0·1956 (5)	0·7955 (2)	6·33 (9)
C19	1·2900 (4)	0·0310 (5)	0·8465 (2)	6·00 (9)
O1	0·9467 (2)	0·3004 (3)	1·02420 (9)	5·22 (5)
O2	0·4384 (2)	0·3179 (3)	0·9619 (1)	5·21 (5)

Discussion. Final atomic parameters are given in Table 1.* Fig. 1 shows a stereoview of the molecule (Motherwell & Clegg, 1978). The bond lengths, bond angles and selected torsion angles are given in Table 2. It has been reported that the Csp^3—Csp^3 bond lengths between tetrasubstituted C atoms vary from 1·566 to 1·610 Å with a mean of 1·588 (25) Å and that Csp^3—Csp^2 bond lengths vary from 1·494 to 1·510 Å with a mean of 1·502 (13) Å (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). In (I) the Csp^3—Csp^3 bond C(4)—C(5) at 1·623 (2) Å and the Csp^3—Csp^2 bond C(6)—C(7) at 1·480 (4) Å deviate significantly from these standard values.

Most of the bond angles observed in (I) also deviate from expected values and all deviations may

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles, least-squares planes and bond distances < 3·5 Å have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51844 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å), *angles* (°) *and intra-annular torsion angles* (°) *for the ring skeleton* (*four fused five-membered rings*)

Numbers in parentheses are e.s.d.'s in the least significant digits.

C1	C2	1·530 (4)	C7	O1	1·200 (3)
C1	C5	1·550 (4)	C8	C9	1·538 (4)
C1	C17	1·545 (4)	C8	C15	1·541 (4)
C2	C3	1·519 (4)	C9	C10	1·523 (4)
C3	C4	1·536 (4)	C9	C12	1·547 (4)
C4	C5	1·623 (4)	C10	C11	1·526 (4)
C4	C8	1·575 (4)	C10	C14	1·547 (4)
C4	C11	1·530 (4)	C12	C13	1·508 (5)
C5	C6	1·552 (4)	C12	O2	1·203 (4)
C5	C16	1·516 (5)	C13	C14	1·534 (5)
C6	C7	1·480 (4)	C17	C18	1·518 (5)
C7	C8	1·536 (4)	C17	C19	1·516 (5)

C2	C1	C5	104·7 (2)	C4	C8	C7	104·5 (2)
C2	C1	C17	112·3 (3)	C4	C8	C9	106·6 (2)
C5	C1	C17	119·8 (2)	C4	C8	C15	116·7 (2)
C1	C2	C3	103·3 (2)	C7	C8	C9	108·4 (2)
C2	C3	C4	106·8 (2)	C7	C8	C15	104·9 (2)
C3	C4	C5	103·8 (2)	C9	C8	C15	115·0 (2)
C3	C4	C8	116·7 (2)	C8	C9	C10	106·3 (2)
C3	C4	C11	110·4 (2)	C8	C9	C12	118·3 (2)
C5	C4	C8	106·4 (2)	C10	C9	C12	103·9 (2)
C5	C4	C11	116·6 (2)	C9	C10	C11	102·1 (2)
C8	C4	C11	103·5 (2)	C9	C10	C14	105·9 (2)
C1	C5	C4	105·0 (2)	C11	C10	C14	114·0 (2)
C1	C5	C6	111·4 (2)	C4	C11	C10	108·7 (2)
C1	C5	C16	113·5 (3)	C9	C12	C13	109·2 (3)
C4	C5	C6	104·1 (2)	C9	C12	O2	125·1 (3)
C4	C5	C16	112·7 (2)	C13	C12	O2	125·6 (3)
C6	C5	C16	109·7 (3)	C12	C13	C14	106·0 (2)
C5	C6	C7	106·7 (2)	C10	C14	C13	104·1 (3)
C6	C7	C8	109·0 (2)	C1	C17	C18	110·5 (3)
C6	C7	O1	126·3 (3)	C1	C17	C19	115·6 (3)
C8	C7	O1	124·4 (3)	C18	C17	C19	109·9 (3)

C5—C1—C2—C3	39·8 (3)	C5—C6—C7—C8	32·2 (3)
C2—C1—C5—C4	−25·5 (3)	C6—C7—C8—C4	−25·9 (3)
C1—C2—C3—C4	−39·2 (3)	C4—C8—C9—C10	−22·4 (3)
C2—C3—C4—C5	22·6 (3)	C8—C9—C10—C11	34·5 (3)
C3—C4—C5—C1	2·0 (3)	C12—C9—C10—C14	28·5 (3)
C8—C4—C5—C6	8·6 (3)	C10—C9—C12—C13	−12·6 (3)
C5—C4—C8—C7	9·6 (3)	C9—C10—C11—C4	−34·9 (3)
C11—C4—C8—C9	0·9 (3)	C9—C10—C14—C13	−33·9 (3)
C8—C4—C11—C10	21·2 (3)	C9—C12—C13—C14	− 8·3 (4)
C4—C5—C6—C7	−24·4 (3)	C12—C13—C14—C10	25·5 (3)

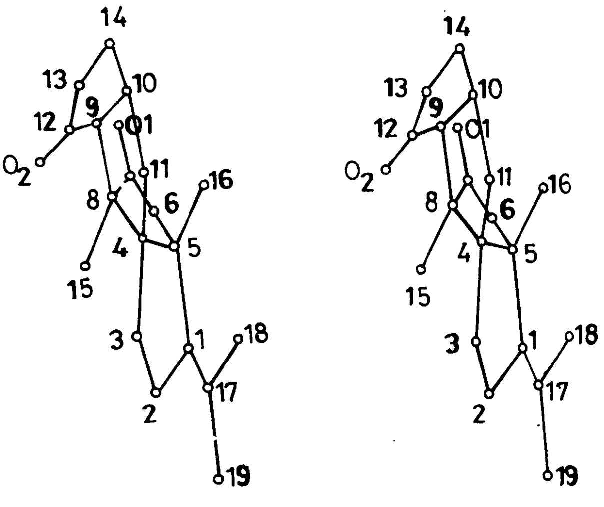

Fig. 1. Stereoview of the molecule.

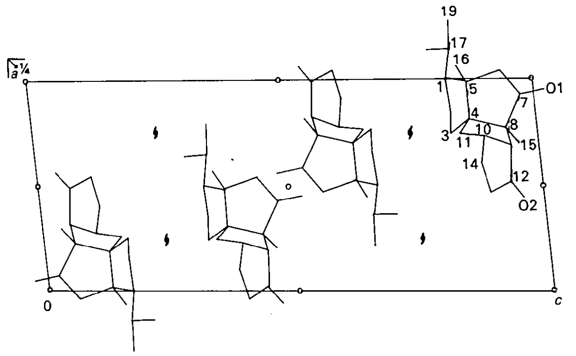

Fig. 2. Packing diagram of the molecule in the unit cell projected down the *b* axis.

be ascribed to the strain caused by ring fusion. Rings *A* and *C* are in the envelope conformation while rings *B* and *D* adopt half-chair conformations. The isopropyl group is in the α-orientation and the torsion angle C(2)—C(1)—C(17)—C(18) = 60·4 (3) and C(2)— C(1)—C(17)—C(19) = −174·0 (3)°, whereas crinipellin A has a β-isopropyl group.

The intramolecular contacts C(15)···O(2) 2·956 (4), C(9)···O(1) 2·895 (3) and C(15)···O(1) 3·063 (4) Å are less than the sum of the van der Waals radii (3·25 Å, Taylor & Kennard, 1982). In the unit cell, packing of the molecules (Fig. 2) is purely by van der Waals forces.

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