The plane of the carboxyl group [C(2), C(7), O(1), O(2)] is tilted at an angle of 4·1 (3)° to the least-squares plane of the six atoms of the pyridine ring. The pyridine ring shows marked, but barely significant, deviations from planarity: deviations from the plane through N(1), C(2), C(3), C(4), C(5) and C(6) are 0·002 (3), −0·005 (3), 0·004 (3), −0·002 (3), −0·008 (3) and 0·009 (3) Å respectively. This distortion probably results from the strain imposed on the system in opening the exocyclic angles at C(2) and C(3) to accommodate the intramolecular hydrogen bond.

The C(3)—S(3) bond length lies between that expected for a single bond associated with a conjugated system, e.g. 1·783 (4) Å in 3,3'-dithiodi-2-pyridinecarboxylic acid (Chaikum & Taylor, 1980), and that expected for a double bond. This fact, and the other bond lengths in the molecule, suggest that (I) is a significant contributor to the resonance hybrid. (II) and (III) are also important with the negative charge shown on C(2) probably being delocalized over the carboxyl group O atoms.

(I)   (II)   (III)
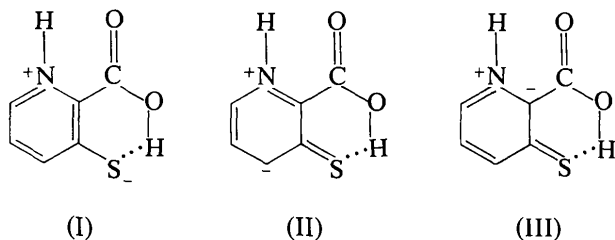
The molecular structure is, therefore, similar to those of the 2,4- (Declercq, Schanck, Germain, Dereppe & Van Meerssche, 1977) and the 2,6-isomers (Germain,

Declercq, Schanck, Dereppe & Van Meerssche, 1977) in that the N atom of the pyridine ring is protonated, but different in that in the 2-isomers the adjacent N atom constrains the C—S bond to have more double-bond character. Canonical forms like (I) make smaller contributions to these 2-isomers.

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Structure of Funicin,* Antimicrobial Substance from Aspergillus funiculosus, $C_{17}H_{18}O_5$

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Abstract. $M_r = 302\cdot4$, triclinic, $P\bar{1}$, $a = 11\cdot147$ (5), $b = 7\cdot388$ (4), $c = 10\cdot551$ (4) Å, $\alpha = 106\cdot01$ (6), $\beta = 107\cdot77$ (6), $\gamma = 101\cdot27$ (6)°, $V = 757\cdot4$ Å^3, $Z = 2$, $D_c = 1\cdot33$ Mg m^{-3}, $\lambda(Cu\,K\alpha) = 1\cdot5418$ Å, $\mu = 0\cdot82$ mm^{-1}, $T = 293$ K. Final $R = 0\cdot049$ for 1844 unique reflections. The dihedral angle between the two phenyl rings is 64°. An intramolecular hydrogen bond $O(4)—H(17)\cdots O(1)$ is formed between hydroxyl and

carbonyl groups. A pair of molecules is linked by an intermolecular hydrogen bond at the center of symmetry.

Introduction. Funicin (I) was isolated from *Aspergillus funiculosus* and showed inhibitory activity towards bacteria and fungi (Hamasaki, Kimura, Hatsuda & Sugawara, 1980). The structure was shown to be ethyl 2-hydroxy-4-(3-hydroxy-5-methylphenoxy)-6-methyl-benzoate based on spectroscopic and chemical data. The present analysis was undertaken to establish the conformation of the molecule as well as to confirm the spectroscopic assignment.

(I)
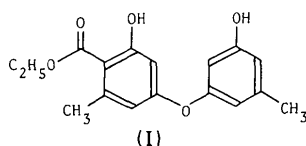
Experimental. Crystallized from *n*-hexane/acetone solution, $0.2 \times 0.2 \times 0.5$ mm, microcomputer-controlled four-circle diffractometer developed in this laboratory (Katsube, 1982); unit-cell dimensions derived from least-squares fit to the observed values of $\pm\theta$ for 27 reflections; maximum $2\theta = 120°$, θ–2θ scan technique, Ni-filtered Cu $K\alpha$, 2098 independent, 1846 with $I > 2\sigma(I)$; a periodically monitored reflection showed no significant change in intensity, Lp correction; Patterson function followed by successive Fourier syntheses, positional and anisotropic temperature factors refined by block-diagonal least-squares which minimized $\sum w(|F_o| - |F_c|)^2$ (Ashida, 1973); all H (located in difference map) isotropic, $R_w = 0.055$, $w = 1.0$ for $F_o \leq 20$ and $w = [1.0 + 0.25(F_o - 20)]^{-1}$ for $F_o > 20$, $F(000) = 320$; atomic scattering factors taken from *International Tables for X-ray Crystallography* (1974).

Discussion. The final atomic parameters are given in Table 1.* A perspective view of funicin is shown in Fig. 1 together with the atomic-numbering scheme used in this paper. Bond lengths and angles for non-H atoms are listed in Table 2. The present analysis has confirmed the previous spectroscopic and chemical assignments (Hamasaki *et al.*, 1980). An intramolecular hydrogen bond between hydroxyl and carbonyl groups, O(4)—H(17)···O(1), forms a chelate ring; H(17)···O(1) is 1·60 (4) Å and O(4)—H(17)···O(1) is 144 (3)°. The ester and the C(1)—C(6) phenyl groups are almost coplanar; the dihedral angle is 4°. Two

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38125 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

torsion angles of the bonds connecting the two phenyl groups, C(3)—C(4)—O(3)—C(11) and C(4)—O(3)—C(11)—C(16), are 16 and 55° respectively. Consequently the dihedral angle between the two rings is 64°. A pair of molecules related by a center of symmetry forms a dimer by an intermolecular hydrogen bond, O(5)—H(18)···O(4); O(5)···O(4) and H(18)···O(4) are 2·770 (3) and 1·88(4) Å respectively, and O(5)—H(18)···O(4) and H(18)···O(4)—C(2) are 171 (4) and 121 (1)° respectively. There is no abnormally short contact in this crystal.

Table 1. *Final atomic coordinates and isotropic thermal parameters (Å^2)*

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}/B
C(1)	−0·0032 (2)	0·2581 (3)	0·0574 (3)	2·9
C(2)	0·1236 (2)	0·2604 (4)	0·1415 (3)	3·1
C(3)	0·1591 (2)	0·2785 (4)	0·2831 (3)	3·1
C(4)	0·0663 (2)	0·2963 (4)	0·3434 (3)	3·2
C(5)	−0·0588 (2)	0·2992 (4)	0·2659 (3)	3·3
C(6)	−0·0958 (2)	0·2802 (4)	0·1244 (3)	3·1
C(7)	−0·0293 (2)	0·2304 (4)	−0·0924 (3)	3·3
C(8)	−0·1813 (3)	0·1815 (4)	−0·3219 (3)	4·2
C(9)	−0·3288 (3)	0·1301 (5)	−0·3908 (3)	5·5
C(10)	−0·2336 (3)	0·2852 (4)	0·0502 (3)	4·1
C(11)	0·2150 (2)	0·3478 (4)	0·5794 (3)	3·5
C(12)	0·2339 (2)	0·2213 (4)	0·6531 (3)	3·5
C(13)	0·3577 (3)	0·2626 (4)	0·7569 (3)	3·6
C(14)	0·4596 (2)	0·4295 (4)	0·7854 (3)	3·5
C(15)	0·4366 (2)	0·5548 (4)	0·7102 (3)	3·4
C(16)	0·3138 (3)	0·5159 (4)	0·6063 (3)	3·6
C(17)	0·3825 (3)	0·1250 (5)	0·8380 (3)	4·9
O(1)	0·0547 (2)	0·2179 (3)	−0·1453 (2)	4·4
O(2)	−0·1514 (2)	0·2151 (3)	−0·1709 (2)	3·8
O(3)	0·0865 (2)	0·3057 (3)	0·4807 (2)	4·2
O(4)	0·2174 (2)	0·2421 (3)	0·0848 (2)	3·9
O(5)	0·5320 (2)	0·7227 (3)	0·7357 (2)	4·7

Table 2. *Bond lengths (Å) and angles (°) for non-H atoms*

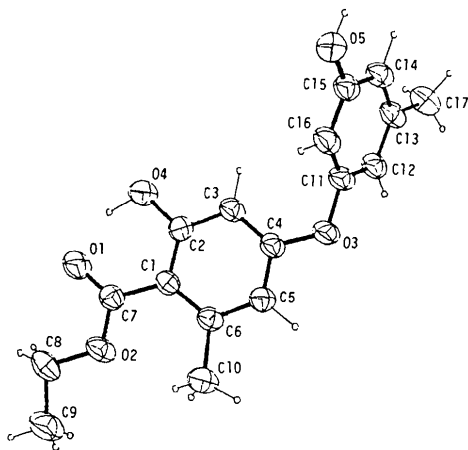|			
C(1)—C(2)	1·415 (4)	C(1)—C(6)	1·428 (4)
C(1)—C(7)	1·464 (4)	C(2)—C(3)	1·384 (4)
C(2)—O(4)	1·365 (3)	C(3)—C(4)	1·379 (4)
C(4)—C(5)	1·392 (4)	C(4)—O(3)	1·376 (4)
C(5)—C(6)	1·380 (4)	C(6)—C(10)	1·509 (4)
C(7)—O(1)	1·233 (4)	C(7)—O(2)	1·323 (4)
C(8)—C(9)	1·500 (5)	C(8)—O(2)	1·462 (4)
C(11)—C(12)	1·379 (4)	C(11)—C(16)	1·384 (4)
C(11)—O(3)	1·403 (5)	C(12)—C(13)	1·388 (4)
C(13)—C(14)	1·393 (4)	C(13) C(17)	1·512 (5)
C(14)—C(15)	1·388 (4)	C(15)—C(16)	1·384 (4)
C(15)—O(5)	1·367 (4)		
C(2)—C(1)—C(6)	117·7 (3)	C(2)—C(1)—C(7)	117·0 (2)
C(6)—C(1)—C(7)	125·3 (3)	C(1)—C(2)—C(3)	122·7 (3)
C(1)—C(2)—O(4)	120·8 (2)	C(3)—C(2)—O(4)	116·5 (3)
C(2)—C(3)—C(4)	117·9 (3)	C(3)—C(4)—C(5)	121·5 (3)
C(3)—C(4)—O(3)	123·8 (3)	C(5)—C(4)—O(3)	114·7 (3)
C(4)—C(5)—C(6)	121·2 (3)	C(1)—C(6)—C(5)	119·0 (3)
C(1)—C(6)—C(10)	124·1 (3)	C(5)—C(6)—C(10)	117·0 (3)
C(1)—C(7)—O(1)	123·3 (3)	C(1)—C(7)—O(2)	115·7 (2)
O(1)—C(7)—O(2)	120·9 (3)	C(9)—C(8)—O(2)	106·0 (3)
C(12)—C(11)—C(16)	122·7 (3)	C(12)—C(11)—O(3)	116·5 (3)
C(16)—C(11)—O(3)	120·6 (3)	C(11)—C(12)—C(13)	118·6 (3)
C(12)—C(13)—C(14)	120·1 (3)	C(12)—C(13)—C(17)	120·0 (3)
C(14)—C(13)—C(17)	120·0 (3)	C(13)—C(14)—C(15)	119·8 (3)
C(14)—C(15)—C(16)	121·0 (3)	C(14)—C(15)—O(5)	122·3 (3)
C(16)—C(15)—O(5)	116·7 (3)	C(11)—C(16)—C(15)	117·9 (3)
C(7)—O(2)—C(8)	116·9 (2)	C(4)—O(3)—C(11)	120·2 (2)

Fig. 1. A perspective view of funicin with the atomic-numbering scheme.

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Cyclohexylammonium μ-Disulfido-bis(trioxophosphate)(4–) Monohydrate, $4C_6H_{14}N_4^+.O_6P_2S_2^{4-}.H_2O$

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Abstract. $M_r = 640.82$, triclinic, space group $P\bar{1}$, $a = 10.646$ (3), $b = 14.469$ (4), $c = 11.565$ (3) Å, $\alpha = 102.62$ (2), $\beta = 96.34$ (1), $\gamma = 100.32$ (2)°, $V = 1689$ (1) Å³, $Z = 2$, $D_x = 1.260$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 2.63$ mm⁻¹, $T = 239$ K. Final $R = 5.7\%$ for 5118 unique X-ray diffractometer data. The anion displays P–O distances in the range 1.504 (3) to 1.522 (3) Å, the P–S and S–S bond lengths are 2.146 (2), 2.140 (1) and 2.072 (2) Å respectively, and the P–S–S–P torsion angle is 93 (1)°. The cyclohexyl-ammonium cations are hydrogen-bonded to phosphate and water O atoms, the latter being clathrated by three cyclohexyl groups in a basket-like arrangement.

Introduction. In the literature, the X-ray structures of several derivatives of dithiodiphosphonic acid have

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