cytosine ring is inclined 87° to the *ac* plane, namely almost perpendicular to the phosphate layers. Comparing the geometrical characteristics of the base rings in this structure (Table 2) and in the cytosine molecule (McClure & Craven, 1973), the effect of protonation of N(3) can be defined thus: an increase of the distances C(2)—N(3) and N(3)—C(4) by about 0·02 Å, an increase of the angle C(2)—N(3)—C(4) by about 4·5° and a decrease of the angles N(1)—C(2)—N(3) and N(3)—C(4)—C(5) by 4·5°. Similar deviations have been observed by Mandel (1977) in the structural analysis of cytosine hydrochloride.

Each cytosine cation is connected to two $H_2PO_4^-$ ions by three hydrogen bonds nearly situated in its plane and perpendicular to the anionic layers: N(1)—H(N1)···O(2), N(3)—H(N3)···O(1) and N(4) —H(2N4)···O(3).

The cytosine groups join themselves by weaker hydrogen bonds from the ammonium N(4) towards the carbonyl oxygen O, so as to form infinite chains running on both sides of the mirror *a*. Thus N(4) participates twice in the cohesion of the structure *via* its H atoms: H(1N4) links the cytosinium ions together along the [(**a**/2) + **c**] direction, while H(2N4) links the $C_4H_6N_3O^+$ cation to the $H_2PO_4^-$ anion along the **b** direction.

In the nucleic acids domain, it is known that, besides the hydrogen bonding, the base stacking also contributes to the stabilization of the structure. In this compound the cytosine rings stack in a direction which is inclined about 18° to the *a* axis. Adjacent base rings are related by the glide plane *a* with an interplanar spacing of 3·30 Å and a dihedral angle between them of 6·5°. Partial overlap of such adjacent rings occurs in each cytosine layer perpendicular to the *b* axis. This type of base packing is uncommon in the crystal structures of compounds containing protonated pyrimidines (Bugg, Thomas, Sundaralingam & Rao, 1971; Mandel, 1977).

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# Structure of 2-Acetoxy-10-hydroxy-3-methoxy-10-phenyl-9(10*H*)-anthracenone

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(*Received* 7 *March* 1989; *accepted* 21 *April* 1989)

**Abstract.** $C_{23}H_{18}O_5$, $M_r = 374{\cdot}40$, triclinic, $P\bar{1}$, $a = 12{\cdot}210\,(3)$, $b = 10{\cdot}557\,(3)$, $c = 8{\cdot}832\,(1)$ Å, $\alpha = 96{\cdot}39\,(2)$, $\beta = 121{\cdot}15\,(2)$, $\gamma = 103{\cdot}99\,(2)°$, $V = 906{\cdot}3\,(5)$ Å$^3$, $Z = 2$, $D_m = 1{\cdot}38$, $D_x = 1{\cdot}372$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}04$ cm$^{-1}$, $F(000) = 392$, $T = 293$ K, final $R = 0{\cdot}051$ for 3205 unique observed reflections. The anthracenone tricyclic system is nonplanar being bent about the C(9)···C(10) vector; the dihedral angle between the two halves is 11·1 (3)°. The acetoxy and methoxy groups are found in the expected 2- and 3-positions of the anthracenone system. The molecules form dimers by hydrogen bonding between the hydroxyl group and the 'ether' O atom of the acetoxy group; the O···O distance is 2·870 (2) Å and the O—H···O angle is 155 (2)°.

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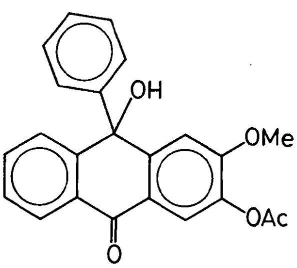
**Introduction.** The condensation reaction of 2-benzoylbenzoic acid with guaiacol (*o*-methoxyphenol) leads to the formation of 3-(4-hydroxy-3-methoxyphenyl)-3-phenylphthalide. This product can be reduced to 2-(4-hydroxy-3-methoxybenzhydryl)-benzoic acid which easily undergoes cyclization and acetylation to 2,9-diacetoxy-3-methoxy-10-phenylanthracene. This compound, in the presence of $HNO_3$/glacial AcOH, gives the anthracenone derivative (I: Aleksandrzak, 1988). An X-ray crystallographic study of (I) has been undertaken to confirm its chemical structure and, particularly, the positions of the acetoxy and methoxy groups. These results will also serve as an indirect confirmation of the reaction path described above.



(I)

**Experimental.** Light-brown parallelepipedal crystals grown from EtOH by slow evaporation, m.p. 472·2–473·8 K, density measured by flotation in aqueous KI solution; crystal specimen: 0·52 × 0·44 × 0·26 mm in sealed glass capillary on Enraf–Nonius CAD-4 diffractometer, $\omega$–$2\theta$ scan mode with graphite-monochromated Mo $K\alpha$ radiation; accurate cell parameters from least-squares refinement for 20 reflections with $16 < \theta < 24°$; three intensity standards ($\bar{3}\bar{1}5$, $3\bar{6}0$, $0\bar{5}3$) displayed no significant variation; $h$ −19/19, $k$ −17/17, $l$ 0/14; max.$(\sin\theta)/\lambda$ = 0·8060 $Å^{-1}$, 5370 unique reflections measured, 2165 unobserved reflections ($I < 2\sigma_I$); no absorption correction.

Solution by direct methods with *SHELXS*86 (Sheldrick, 1985); full-matrix least-squares refinement using *F* magnitudes with *SHELX*76 (Sheldrick, 1976); all H atoms located on difference Fourier maps and refined isotropically; anisotropic displacement parameters for non-H atoms; 326 paremeters refined.

Final $R = 0·051$, $wR = 0·037$ ($w^{-1} = \sigma_F^2$) and $S = 3·71$ for 3205 observed reflections; empirical isotropic extinction parameter $x$ (*SHELX*76, Sheldrick, 1976) converged at 0·0025 (2); max. $\Delta/\sigma = 0·09$ in last cycle; max. $\Delta\rho = 0·25$ and min. $\Delta\rho = -0·24$ e $Å^{-3}$ in final $\Delta F$ map. Scattering factors from *International Tables for X-ray Crystallography* (1974). Other computer programs used: *ORTEP*II (Johnson, 1976), *PLUTO* (Motherwell & Clegg, 1978) and *CRYPOZ* system (Jaskólski, 1982).

Table 1. *Atomic fractional coordinates and equivalent isotropic displacement parameters* ($Å^2$) *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| C(1) | 0·4395 (2) | −0·2624 (2) | 0·4447 (3) | 0·0490 (7) |
| C(2) | 0·4750 (2) | −0·1970 (2) | 0·3452 (3) | 0·0481 (7) |
| C(3) | 0·4079 (2) | −0·1127 (2) | 0·2526 (3) | 0·0459 (7) |
| C(4) | 0·3016 (2) | −0·0986 (2) | 0·2622 (3) | 0·0413 (6) |
| C(5) | 0·0436 (2) | −0·1686 (2) | 0·5519 (3) | 0·0466 (7) |
| C(6) | 0·0239 (2) | −0·2182 (2) | 0·6767 (3) | 0·0565 (9) |
| C(7) | 0·0909 (3) | −0.3109 (3) | 0·7647 (4) | 0·0596 (9) |
| C(8) | 0·1788 (2) | −0·3347 (2) | 0·7291 (3) | 0·0562 (9) |
| C(9) | 0·2986 (2) | −0·3187 (2) | 0·5701 (3) | 0·0547 (8) |
| C(10) | 0·1449 (2) | −0·1481 (2) | 0·3678 (3) | 0·0342 (5) |
| C(11) | 0·1326 (2) | −0·1999 (2) | 0·5142 (3) | 0·0373 (6) |
| C(12) | 0·2009 (2) | −0·2842 (2) | 0·6036 (3) | 0·0445 (7) |
| C(13) | 0·3329 (2) | −0·2475 (2) | 0·4572 (3) | 0·0414 (6) |
| C(14) | 0·2644 (2) | −0·1648 (2) | 0·3653 (3) | 0·0364 (5) |
| C(15) | 0·0125 (2) | −0·2245 (2) | 0·1777 (3) | 0·0337 (5) |
| C(16) | −0·0591 (2) | −0·1579 (2) | 0·0571 (3) | 0·0440 (7) |
| C(17) | −0·1753 (2) | −0·2297 (2) | −0·1166 (3) | 0·0528 (8) |
| C(18) | −0·2226 (2) | −0·3695 (2) | −0·1714 (3) | 0·0546 (8) |
| C(19) | −0·1532 (2) | −0·4372 (2) | −0·0507 (3) | 0·0577 (7) |
| C(20) | −0·0364 (2) | −0·3657 (2) | 0·1215 (3) | 0·0489 (7) |
| C(21) | 0·5525 (2) | −0·3112 (2) | 0·1964 (3) | 0·0564 (8) |
| C(22) | 0·6753 (3) | −0·3109 (4) | 0·1999 (6) | 0·0802 (12) |
| C(23) | 0·3879 (3) | 0·0365 (3) | 0·0627 (5) | 0·0689 (12) |
| O(1) | 0·3505 (2) | −0·4011 (2) | 0·6344 (3) | 0·0931 (9) |
| O(2) | 0·1578 (1) | −0·0068 (1) | 0·3955 (2) | 0·0422 (4) |
| O(3) | 0·5851 (1) | −0·2071 (1) | 0·3386 (2) | 0·0619 (5) |
| O(4) | 0·4389 (2) | −0·3862 (2) | 0·0924 (3) | 0·1008 (7) |
| O(5) | 0·4531 (2) | −0·0513 (2) | 0·1586 (2) | 0·0628 (6) |

**Discussion.** The final atomic parameters for non-H atoms are given in Table 1.* A perspective view of the molecule with the atom-numbering scheme is shown in Fig. 1. Bond lengths and bond angles are listed in Table 2.

The anthracenone tricyclic system is nonplanar, being bent about the C(9)···C(10) vector: the dihedral angle between the two halves of the system is 11·1° (e.s.d.'s for dihedral angles are 0·2–0·3°) and between the two outer, planar rings is 10·5°; the central ring adopts a boat-like conformation. A similar conformation was observed for 10,10-divanillyl-9(10*H*)-anthracenone (Brown & Fullerton, 1980), whilst in 10-(4-acetoxyphenyl)-10-hydroxy-9(10*H*)-anthracenone (APHA hereafter; Skrzat & Roszak, 1986) a chair-like conformation was found. In APHA the whole tricyclic system was almost planar [the dihedral angle between the outer rings was only 2·8 (3)°].

The phenyl ring and the hydroxy group bonded to atom C(10) are almost perpendicular to the least-

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* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52167 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

| | | | |
|---|---|---|---|
| C(1)—C(13) | 1·406 (3) | C(10)—C(14) | 1·522 (2) |
| C(1)—C(2) | 1·350 (3) | C(10)—C(15) | 1·531 (2) |
| C(2)—C(3) | 1·399 (3) | C(10)—O(2) | 1·441 (2) |
| C(2)—O(3) | 1·404 (2) | C(11)—C(12) | 1·394 (2) |
| C(3)—C(4) | 1·384 (3) | C(13)—C(14) | 1·389 (2) |
| C(3)—O(5) | 1·355 (2) | C(15)—C(16) | 1·376 (2) |
| C(4)—C(14) | 1·393 (3) | C(15)—C(20) | 1·387 (2) |
| C(5)—C(11) | 1·388 (3) | C(16)—C(17) | 1·382 (3) |
| C(5)—C(6) | 1·375 (3) | C(17)—C(18) | 1·373 (3) |
| C(6)—C(7) | 1·378 (3) | C(18)—C(19) | 1·376 (3) |
| C(7)—C(8) | 1·370 (3) | C(19)—C(20) | 1·377 (3) |
| C(8)—C(12) | 1·399 (3) | C(21)—C(22) | 1·482 (3) |
| C(9)—C(12) | 1·481 (3) | C(21)—O(3) | 1·381 (2) |
| C(9)—C(13) | 1·477 (3) | C(21)—O(4) | 1·177 (2) |
| C(9)—O(1) | 1·218 (2) | C(23)—O(5) | 1·429 (3) |
| C(10)—C(11) | 1·521 (2) | O(2)—H(9) | 0·89 (2) |

| | | | |
|---|---|---|---|
| C(13)—C(1)—C(2) | 120·5 (2) | C(8)—C(12)—C(9) | 119·6 (2) |
| C(1)—C(2)—C(3) | 121·4 (2) | C(8)—C(12)—C(11) | 119·6 (2) |
| C(1)—C(2)—O(3) | 120·6 (2) | C(9)—C(12)—C(11) | 120·8 (2) |
| C(3)—C(2)—O(3) | 118·0 (2) | C(12)—C(13)—C(9) | 118·9 (2) |
| C(2)—C(3)—C(4) | 118·7 (2) | C(1)—C(13)—C(14) | 119·0 (2) |
| C(2)—C(3)—O(5) | 116·1 (2) | C(9)—C(13)—C(14) | 122·0 (2) |
| C(4)—C(3)—O(5) | 125·2 (2) | C(4)—C(14)—C(10) | 118·4 (2) |
| C(3)—C(4)—C(14) | 120·6 (2) | C(4)—C(14)—C(13) | 119·9 (2) |
| C(6)—C(5)—C(11) | 120·8 (2) | C(10)—C(14)—C(13) | 121·7 (2) |
| C(5)—C(6)—C(7) | 120·4 (2) | C(10)—C(15)—C(16) | 122·2 (2) |
| C(6)—C(7)—C(8) | 119·8 (2) | C(10)—C(15)—C(20) | 119·5 (2) |
| C(7)—C(8)—C(12) | 120·5 (2) | C(16)—C(15)—C(20) | 118·2 (2) |
| C(12)—C(9)—C(13) | 117·3 (2) | C(15)—C(16)—C(17) | 120·9 (2) |
| C(12)—C(9)—O(1) | 121·8 (2) | C(16)—C(17)—C(18) | 120·5 (2) |
| C(13)—C(9)—O(1) | 120·8 (2) | C(17)—C(18)—C(19) | 119·1 (2) |
| C(11)—C(10)—C(14) | 113·3 (2) | C(18)—C(19)—C(20) | 120·5 (2) |
| C(11)—C(10)—C(15) | 109·2 (1) | C(15)—C(20)—C(19) | 120·8 (2) |
| C(11)—C(10)—O(2) | 109·6 (1) | O(3)—C(21)—C(22) | 111·3 (2) |
| C(14)—C(10)—C(15) | 108·8 (1) | O(4)—C(21)—C(22) | 128·5 (3) |
| C(14)—C(10)—O(2) | 109·6 (1) | O(3)—C(21)—O(4) | 120·2 (2) |
| C(15)—C(10)—O(2) | 106·1 (1) | C(2)—O(3)—C(21) | 115·7 (2) |
| C(5)—C(11)—C(10) | 118·5 (2) | C(3)—O(5)—C(23) | 118·1 (2) |
| C(5)—C(11)—C(12) | 118·8 (2) | C(10)—O(2)—H(9) | 108 (1) |
| C(10)—C(11)—C(12) | 122·6 (2) | | |

squares plane of the anthracenone system – the appropriate dihedral angles are 87·5 and 83·4° [the plane of the OH group was defined by atoms C(10), O(2) and H(O2)]. These angles in APHA were 87·7 (3) and 85·6 (3)°, respectively.

The acetoxy and methoxy groups were found in the expected 2- and 3-positions. The acetoxy group is planar; it is oriented perpendicular to the bonded ring with a dihedral angle of 89·3°, and on the same side of the anthracenone system as the 10-phenyl group. The methoxy group is directed away from the acetoxy group and is coplanar with the ring; the dihedral angle between the plane defined by atoms C(3), O(5) and C(23), and the ring plane is 0·7°. Both groups adopt the most typical orientations relative to the aromatic ring plane (Hummel, Huml & Bürgi, 1988; Hummel, Roszak & Bürgi, 1988).

The molecules of the title compound form dimers around the centre of symmetry ($\frac{1}{2}$,0,$\frac{1}{2}$) *via* hydrogen bonds between the hydroxyl group and the 'ether' oxygen atom, O(3), of the acetoxy group (Fig. 2). Hydrogen-bond geometry: $O_D\cdots O_A$ = 2·870 (2), $O_D$—H = 0·89 (2), $O_A\cdots$H = 2·04 (2) Å, angle $O_D$—H$\cdots O_A$ = 155 (2)°. The molecules of APHA were also linked as dimers but through a hydrogen bond between the hydroxyl group and the carbonyl O atom of the anthracenone; the length of this carbonyl bond was 1·235 (5) Å. In the present structure the corresponding carbonyl bond, C(9)—O(1), is not involved in hydrogen bonding and its length is 1·218 (2) Å. These two different values agree well with values of 1·242 (3) and 1·218 (3) Å for the two C═O bonds in 4,5-dihydroxy-1,8-dinitroanthraquinone (Brown & Colclough, 1983), only the first of which was involved in hydrogen bonding.

The other carbonyl bond in the title structure, C(21)═O(4) in the acetoxy group, is relatively short [1·177 (2) Å]. Similar values are frequently observed (Hecht & Luger, 1974; Low & Wilson, 1984; Roszak, 1986; Hummel, Roszak & Bürgi, 1988), and in APHA the length of the corresponding bond was 1·171 (4) Å. Thermal motion can explain only a part of this significant contraction: corrections for librational motions in 13 OAc groups bonded to aromatic rings led to a weighted average of 1·201 (1) Å (Roszak, 1986).

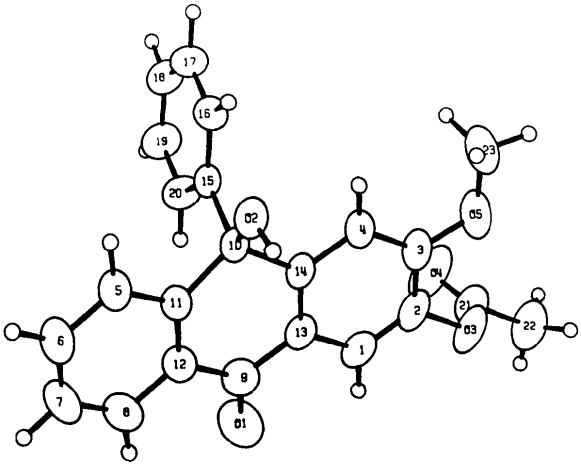

Fig. 1. Perspective view of the molecule with the atom-numbering scheme. Thermal ellipsoids are shown at the 50% probability level; H atoms as small spheres of arbitrary size.

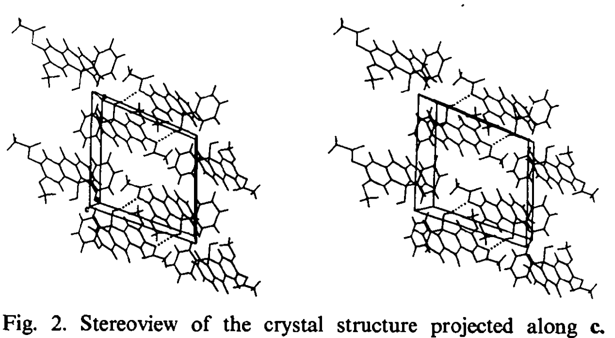

Fig. 2. Stereoview of the crystal structure projected along **c**. Hydrogen bonds are indicated by dotted lines.

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# Structures of Two Model Peptides: *N*-Acetyl-D,L-valine and *N*-Acetyl-L-valyl-L-leucine

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**Abstract.** $C_7H_{13}NO_3$, $M_r = 159\cdot19$, monoclinic, $P2_1/c$, $a = 6\cdot626\,(2)$, $b = 13\cdot002\,(5)$, $c = 10\cdot031\,(2)$ Å, $\beta = 105\cdot37\,(3)°$, $V = 833\cdot2$ Å$^3$, $Z = 4$, $D_x = 1\cdot269$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71073 Å, $\mu = 0\cdot93$ cm$^{-1}$, $F(000) = 344$, $T = 297$ K, final $R = 0\cdot058$ for 1429 unique observed reflections. $C_{13}H_{24}N_2O_4$, $M_r = 272\cdot35$, monoclinic, $P2_1$, $a = 9\cdot458\,(1)$, $b = 9\cdot523\,(1)$, $c = 9\cdot409\,(1)$ Å, $\beta = 114\cdot55\,(1)°$, $V = 770\cdot9$ Å$^3$, $Z = 2$, $D_x = 1\cdot173$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71073 Å, $\mu = 0\cdot81$ cm$^{-1}$, $F(000) = 296$, $T = 297$ K, final $R = 0\cdot034$ for 1333 unique observed reflections. *N*-Acetyl-D,L-valine and *N*-acetyl-L-valyl-L-leucine have been used as model peptides in solid-state NMR spectroscopy. The X-ray crystal structure determinations were undertaken to provide opportunities for direct comparisons between solid-state NMR spectroscopy and X-ray diffraction.

**Introduction.** *N*-Acetyl-D,L-valine is a popular model peptide for solid-state NMR spectroscopy (Stark, Haberkorn & Griffin, 1978; Stark, Jelinski, Ruben, Torchia & Griffin, 1983; Tycko, Stewart & Opella, 1986; Ramanathan & Opella, 1988) because it has one peptide linkage per molecule and only two magnetically unique molecules per unit cell. *N*-Acetyl-L-valyl-L-leucine has two peptide linkages per molecule and two unique molecules in the unit cell and can serve as a model system with two adjoining peptide planes for solid-state NMR spectroscopy (Stewart, Tycko & Opella, 1988).

**Experimental.** *N*-Acetyl-D,L-valine and L-valyl-L-leucine were obtained from Sigma Chemical Company. *N*-Acetyl-L-valyl-L-leucine was prepared by acetylation of L-valyl-L-leucine. Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Table 1 lists data collection parameters for the title compounds. Lattice parameters were refined from 25 reflections in the range $6\cdot5 < \theta < 15\cdot7°$. Intensities of standard reflections measured every 3000 s of X-ray exposure showed no significant decay. Data corrected for Lorentz and polarization effects and for secondary extinction but not for absorption. Structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H atoms found

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