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Structure of 10-[4-(Acetoxy)phenyl]-10-hydroxy-9(10*H*)-anthracenone*

By Zofia Skrzat and Aleksander Roszak

Institute of Chemistry, Nicolaus Copernicus University, Gagarina 7, 87–100 *Toruń, Poland*

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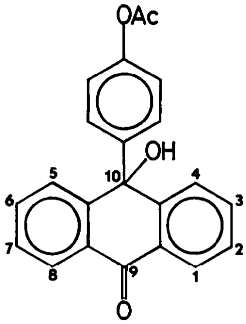
Abstract. $C_{22}H_{16}O_4$, M_r = 344·37, triclinic, $P\bar{1}$, a = 7·1485 (3), b = 9·3921 (5), c = 13·509 (1) Å, α = 108·186 (4), β = 99·371 (3), γ = 95·515 (3)°, V = 839·67 (9) Å^3, Z = 2, D_x = 1·362 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 0·72 mm^{-1}, F(000) = 360, room temperature, final R = 0·055 for 2722 unique observed reflections. The molecules, stacked head to tail, form dimers by hydrogen bonding between hydroxyl and carbonyl groups with distance O—H···O = 2·844 (2) Å.

Introduction. The structure determination of the title compound (I) forms part of an investigation of the relationship between structure and reactivity of a hydroxyl group attached to the central C(10) atom of various related compounds.



(I)

Experimental. Crystals pale yellow prisms (from ethanol), m.p. 554·1–555·1 K (Gronowska & Dzieleńdziak, 1982); D_m not determined; space group determined by film methods to be $P1$ or $P\bar{1}$; lattice parameters refined and intensity data obtained from a crystal approx. 0·7 × 0·3 × 0·2 mm sealed in a thin-walled glass capillary and mounted in random orientation on CAD-4 diffractometer, Ni-filtered Cu $K\alpha$ radiation, $\omega/2\theta$-scan mode, scan width (0·60 + 0·14tanθ)°, horizontal aperture (2·50 + 0·80tanθ) mm, vertical aperture 4 mm; max. sinθ/λ = 0·6285 Å^{-1}; hkl range: $h = -8 \rightarrow 8$, $k = -11 \rightarrow 11$, $l = 0 \rightarrow 16$. Three standard reflections monitored periodically showed no significant variation, 3490 reflections measured, 2724 with $I > 2\sigma(I)$ employed for structure determination. Data corrected for Lorentz and polarization factors, but not for absorption or extinction. Structure solved by direct methods with *SHELXS*84 (Sheldrick, 1984) in space group $P\bar{1}$, justified by calculated E statistics; successfully refined by full-matrix least squares (Sheldrick, 1976), using 2722 reflections in last cycle (two reflections suffering seriously from extinction omitted); $\sum w(|F_o| - |F_c|)^2$ minimized, $w^{-1} = \sigma^2(F)$. Seven H atoms located from Fourier difference map, rest determined from geometrical considerations. All H atoms (methyl group as rigid group with geometrical constraints: C—H 1·08 Å, H—C—H 109·5°) refined isotropically, non-H atoms anisotropically. Final R = 0·055, wR = 0·060, S = 3·76. Max. Δ/σ = 0·09 for positional and thermal parameters, and 0·7 for rotation parameters of the rigid methyl group, highest and lowest residual peaks in the final difference Fourier map +0·35 and −0·43 e Å^{-3}.

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* IUPAC name: 4-(9,10-dihydro-9-hydroxy-10-oxo-9-anthryl)-phenyl acetate.

0108-2701/86/091194-03$01.50

Scattering factors from *International Tables for X-ray Crystallography* (1974); other programs used: *CRYPOZ* system (Jaskólski, 1982), *PLUTO* (Motherwell & Clegg, 1978), *ORTEP*II (Johnson, 1976).

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond lengths and bond angles in Table 2. A view of the molecule with the atomic numbering is shown in Fig. 1.

The tricyclic anthrone system in the present compound is almost planar (r.m.s. deviation 0·035 Å), the dihedral angle between the planes of the outer rings (which are planar with r.m.s. deviations of 0·006 and 0·007 Å respectively) being only 2·8° (e.s.d.'s for the dihedral angles are 0·2–0·3°), similar to 10,10-divanillyl-9(10*H*)-anthracenone (Brown & Fullerton, 1980). The atoms of the central ring of the tricyclic system lie alternately above and below its least-squares plane [maximum deviations ±0·021 (2) Å] in a 'crown'-like conformation, whereas in the structure mentioned above the central ring adopts a boat-like conformation.

The phenyl ring to which the acetoxy group is attached shows slight but significant deviation from planarity (r.m.s. deviation 0·010 Å) probably due to the effect of packing. The mean plane of the phenyl ring forms a dihedral angle of 87·7° with the mean plane of the anthrone moiety. The almost planar acetoxy group (r.m.s. deviation 0·008 Å) makes dihedral angles of 66·5 and 63·7° with the mean plane of the phenyl ring and of the tricyclic ring system, respectively.

Most of the lengths and angles agree with expected values within experimental error. The lengths of the C=O bonds differ slightly from the value of 1·215 (5) Å proposed by Sutton (1965). The anthrone carbonyl bond C(9)=O(1) distance of 1·235 (2) Å is larger than this, probably as the result of the involve-

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}(Å^2)
C(1)	0·3669 (3)	1·2486 (3)	0·6263 (2)	0·0580 (7)
C(2)	0·4772 (4)	1·3469 (3)	0·7203 (2)	0·0664 (8)
C(3)	0·5727 (4)	1·2911 (3)	0·7941 (2)	0·0644 (8)
C(4)	0·5564 (3)	1·1372 (3)	0·7748 (2)	0·0553 (6)
C(5)	0·2991 (3)	0·6078 (2)	0·5304 (2)	0·0543 (7)
C(6)	0·1926 (4)	0·5119 (3)	0·4337 (2)	0·0647 (8)
C(7)	0·0958 (4)	0·5684 (3)	0·3611 (2)	0·0624 (7)
C(8)	0·1088 (3)	0·7229 (3)	0·3846 (2)	0·0551 (7)
C(9)	0·2357 (3)	0·9876 (2)	0·5020 (1)	0·0457 (6)
C(10)	0·4270 (3)	0·8666 (2)	0·6649 (1)	0·0416 (5)
C(11)	0·3110 (3)	0·7646 (2)	0·5562 (1)	0·0424 (5)
C(12)	0·2168 (3)	0·8232 (2)	0·4822 (1)	0·0432 (5)
C(13)	0·3495 (3)	1·0909 (2)	0·6050 (2)	0·0449 (6)
C(14)	0·4431 (3)	1·0346 (2)	0·6803 (1)	0·0434 (5)
C(15)	0·3318 (3)	0·8378 (2)	0·7525 (1)	0·0418 (5)
C(16)	0·1441 (3)	0·8642 (3)	0·7552 (2)	0·0490 (6)
C(17)	0·0582 (3)	0·8496 (3)	0·8369 (2)	0·0535 (6)
C(18)	0·1600 (3)	0·8066 (3)	0·9152 (1)	0·0539 (6)
C(19)	0·3428 (3)	0·7753 (3)	0·9130 (2)	0·0690 (8)
C(20)	0·4292 (3)	0·7911 (3)	0·8307 (2)	0·0598 (7)
C(21)	−0·0649 (4)	0·6999 (3)	0·9942 (2)	0·0680 (8)
C(22)	−0·1244 (5)	0·7163 (4)	1·0971 (2)	0·0937 (12)
O(1)	0·1638 (2)	1·0377 (2)	0·4328 (1)	0·0609 (5)
O(2)	0·6157 (2)	0·8258 (2)	0·6804 (1)	0·0511 (4)
O(3)	0·0847 (2)	0·8040 (2)	1·0050 (1)	0·0714 (5)
O(4)	−0·1391 (5)	0·6119 (3)	0·9109 (2)	0·1593 (12)

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

C(1)–C(2)	1·374 (4)	C(10)–C(15)	1·537 (2)
C(1)–C(13)	1·407 (3)	C(10)–O(2)	1·438 (3)
C(2)–C(3)	1·381 (4)	C(11)–C(12)	1·397 (2)
C(3)–C(4)	1·376 (4)	C(13)–C(14)	1·394 (3)
C(4)–C(14)	1·400 (3)	C(15)–C(16)	1·392 (3)
C(5)–C(6)	1·381 (4)	C(15)–C(20)	1·379 (3)
C(5)–C(11)	1·394 (3)	C(16)–C(17)	1·383 (4)
C(6)–C(7)	1·377 (4)	C(17)–C(18)	1·373 (3)
C(7)–C(8)	1·375 (4)	C(18)–C(19)	1·370 (3)
C(8)–C(12)	1·405 (3)	C(18)–O(3)	1·411 (2)
C(9)–C(12)	1·471 (3)	C(19)–C(20)	1·396 (4)
C(9)–C(13)	1·475 (3)	C(21)–C(22)	1·486 (4)
C(9)–O(1)	1·235 (2)	C(21)–O(3)	1·334 (3)
C(10)–C(11)	1·528 (3)	C(21)–O(4)	1·171 (4)
C(10)–C(14)	1·516 (3)	O(2)–H(9)	0·91 (3)

C(13)–C(1)–C(2)	120·3 (2)	C(9)–C(12)–C(11)	121·4 (1)
C(1)–C(2)–C(3)	120·1 (2)	C(1)–C(13)–C(9)	119·3 (2)
C(2)–C(3)–C(4)	120·4 (2)	C(1)–C(13)–C(14)	119·7 (2)
C(3)–C(4)–C(14)	120·7 (2)	C(9)–C(13)–C(14)	121·0 (2)
C(6)–C(5)–C(11)	120·3 (2)	C(4)–C(14)–C(10)	118·2 (2)
C(5)–C(6)–C(7)	121·0 (2)	C(4)–C(14)–C(13)	118·8 (2)
C(6)–C(7)–C(8)	119·3 (2)	C(10)–C(14)–C(13)	122·9 (1)
C(7)–C(8)–C(12)	120·8 (2)	C(10)–C(15)–C(16)	119·2 (2)
C(12)–C(9)–C(13)	118·3 (1)	C(10)–C(15)–C(20)	121·8 (2)
C(12)–C(9)–O(1)	120·8 (1)	C(16)–C(15)–C(20)	118·9 (2)
C(13)–C(9)–O(1)	120·8 (2)	C(15)–C(16)–C(17)	120·9 (2)
C(11)–C(10)–C(14)	113·8 (1)	C(16)–C(17)–C(18)	119·0 (2)
C(11)–C(10)–C(15)	109·2 (1)	C(17)–C(18)–C(19)	121·5 (2)
C(14)–C(10)–C(15)	107·9 (1)	C(17)–C(18)–O(3)	120·8 (2)
C(11)–C(10)–O(2)	109·3 (1)	C(19)–C(18)–O(3)	117·6 (2)
C(14)–C(10)–O(2)	109·7 (1)	C(18)–C(19)–C(20)	119·2 (2)
C(15)–C(10)–O(2)	106·7 (1)	C(19)–C(20)–C(15)	120·4 (2)
C(5)–C(11)–C(10)	118·7 (1)	O(3)–C(21)–C(22)	111·9 (2)
C(5)–C(11)–C(12)	119·1 (2)	O(3)–C(21)–O(4)	121·4 (2)
C(10)–C(11)–C(12)	122·2 (1)	O(4)–C(21)–C(22)	126·6 (3)
C(8)–C(12)–C(9)	119·2 (2)	C(18)–O(3)–C(21)	119·8 (2)
C(8)–C(12)–C(11)	119·3 (2)	C(10)–O(2)–H(9)	105 (2)

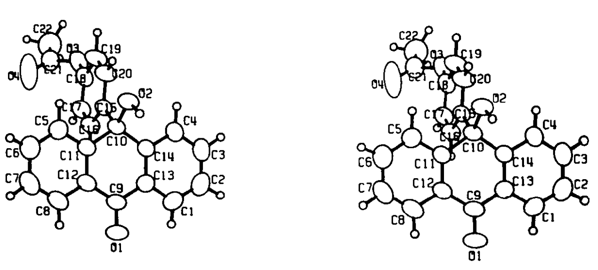

Fig. 1. Stereoview of the molecule with 50% probability thermal ellipsoids. H atoms shown as small spheres of arbitrary size.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, least-squares planes data and some shorter intermolecular distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42864 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

ment of O(1) in an intermolecular hydrogen bond. The length of the C=O bond of the acetoxy group is shorter, being only 1·171 (4) Å. This is probably due to the high thermal vibration of O(4). However, as Low & Wilson (1984) noted, this bond is frequently shorter than expected for a conjugated C=O bond. This conclusion is confirmed by the data retrieved (Roszak, 1986) from the Cambridge Structural Database (CSD) (Allen, Bellard, Brice, Cartwright, Doubleday, Higgs, Hummelink, Hummelink-Peters, Kennard, Motherwell, Rodgers & Watson, 1979) for 102 acetoxy groups substituted on aromatic six-membered carbon rings, and even better for 20 of these in which there is no *ortho* substituent in the ring. The mean values of the C=O bond lengths in these two samples are 1·188 (2) and 1·181 (3) Å with the standard deviation of the sample distribution (σ_s), 0·016 and 0·015 Å, respectively.

Similarly, the rather large value of 119·8 (2)° for the C(18)—O(3)—C(21) angle and the rather small value of 1·334 (3) Å for the O(3)—C(21) bond length are within $2\sigma_s$ from the mean values of 118·1 (2)° and 1·357 (2) Å (with $\sigma_s = 1{\cdot}6°$ and 0·018 Å, respectively) for the CSD data in the first sample mentioned above and from the mean values of 118·5 (3)° and 1·346 (4) Å (with $\sigma_s = 1{\cdot}2°$ and 0·020 Å, respectively) for the second sample.

The substituted phenyl ring is slightly deformed as reported in the literature (Domenicano, Vaciago & Coulson, 1975). The internal angles at C(15) and C(18) are 118·9 (2) and 121·5 (2)°, respectively, due to the σ-electron-releasing and σ-electron-withdrawing character, respectively, of the substituents. Very similar values, 118·7 (1) and 121·3 (1)°, occur in the structure of 10-(4-acetoxyphenyl)-9-anthryl acetate (Roszak & Duax, 1986), which suggests that the influence of these large ring systems on the phenyl ring is much the same, independent of the hybridization state of the atom connecting the ring system with the aromatic ring. The value of 118·9 (2)° at C(15) in the title compound also agrees well with the value of 118·7 (5)° observed by Albinati, Meille, Arnoldi & Galli (1985) for the corresponding angle in 1-(4-fluorophenyl)-1-(3-pyridyl)but-3-yn-1-ol; however in this compound the fluoro substituent *para* with respect to this angle has stronger σ-electron-withdrawing character.

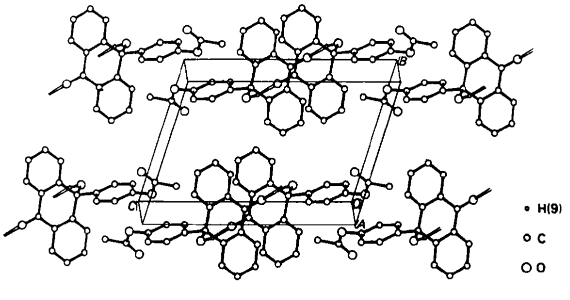

Fig. 2. Molecular packing in the unit cell showing hydrogen bonds as double lines.

The molecules, stacked head to tail, form dimers around the centre of symmetry ($\frac{1}{2},1,\frac{1}{2}$) by hydrogen bonding through the hydroxyl, O(2)—H(9), and carbonyl, C(9)=O(1), groups. Hydrogen-bond geometry: $O_D\cdots O_A = 2{\cdot}844\ (2)$, $O_D\text{—}H = 0{\cdot}91\ (3)$, $O_A\cdots H = 1{\cdot}95\ (3)$ Å, angle $O_D\text{—}H\cdots O_A = 165\ (2)°$. The value 165 (2)° is in very good agreement with the value 165·8 (12)° determined by Ceccarelli, Jeffrey & Taylor (1981) for the group of hydrogen bonds with distance $O_A\cdots H > 1{\cdot}812$ Å. The packing of the dimers, illustrated in Fig. 2, is governed by van der Waals contacts.

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