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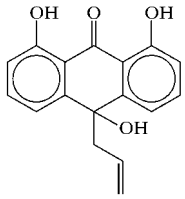
The central ring of the anthrone system in the title compound, $C_{17}H_{14}O_4$, has a shallow envelope conformation, and each of the two outer rings is inclined at an angle of 17.41 (3)°. In the solid state, the molecules exist as centrosymmetrically related O—H···O hydrogen-bonded dimers. Two intramolecular O—H···O hydrogen bonds, involving the central carbonyl O atom and having a graph-set motif of $S(6)$, are observed. These intramolecular interactions lead co-operatively to an O—H···O···H—O pattern that has a binary graph-set motif of $R_2^1(10)$.

Comment

Anthracenones substituted at atom C10 have attained paramount significance because of their wide range of biological activities, including antipsoriatic activity and leukotriene biosynthesis inhibition (Hayden *et al.*, 1994; Muller & Prinz, 1997; Earl *et al.*, 1998). The 5-LO (LO is lipoxygenase) pathway has been the major focus of study because of the pronounced pro-inflammatory role of leukotrienes and the approval of 5-LO inhibitors for the treatment of asthma (Young, 1999). Although less well characterized, the 12-LO pathway may also play an important role in the progression of human diseases such as cancer (Honn *et al.*, 1994) and psoriasis (Ikai, 1999). In this paper, we report the crystal structure of the title compound, (I), and the interesting hydrogen-bond patterns observed in the solid state.

Fig. 1 shows a perspective view of (I), with the atom-numbering scheme. Most of the bond lengths and angles are unexceptional and comparable to those reported for related structures (Brown & Fullerton, 1980; Skrzat & Roszak, 1986; Roszak & Engelen, 1990). The anthrone carbonyl C9═O9 distance [1.2603 (13) Å] is significantly longer than that usually observed for carbonyl bonds, probably because atom O9 is involved in two intramolecular hydrogen bonds. The tricyclic anthracenone ring system is non-planar; the dihedral angle between the two halves of the system is 16.30 (3)° and that between the two outer planar rings is 17.41 (3)°. The central ring adopts a shallow envelope conformation [Cremer & Pople (1975) puckering parameters are $Q = 0.206$ (1) Å, $q_2 = 0.189$ (1) Å, $q_3 = -0.083$ (1) Å, $\theta = 113.8$ (4)° and $\varphi_2 = 357.4$ (4)° for the C9—C12—C11—C10—C14—C13 atom sequence (Table 1)]. Atom C10 lies 0.291 (2) Å from the plane defined by the other five ring atoms. However, in related compounds, shallow boat-like (Brown & Fullerton, 1980; Roszak & Engelen, 1990) and chair-like conformations (Skrzat & Roszak, 1986) have been reported.



(I)

The propene and hydroxy substituents at atom C10 are nearly perpendicular to the least-squares plane of the anthracenone system, with dihedral angles of 86.47 (6) and 86.93 (1)°, respectively (the plane of the OH group is defined by atoms C10, O15 and H15). The hydroxy group is oriented roughly perpendicular to the central ring of the anthrone system in related compounds (Skrzat & Roszak, 1986; Roszak & Engelen, 1990).

There are two intramolecular hydrogen bonds between the 1,8-hydroxy groups and the carbonyl O atom of the central ring of (I) (Table 2), each generating an $S(6)$ graph-set motif (Bernstein *et al.*, 1995). Thus, the carbonyl O atom is an acceptor of two hydrogen bonds, and these intramolecular interactions lead co-operatively to an O—H···O···H—O pattern, which has a binary graph-set motif of $R_2^1(10)$. The

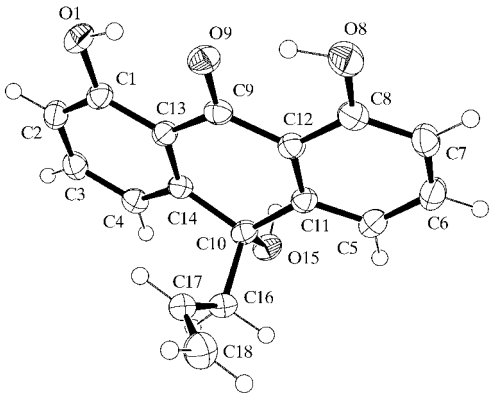


Figure 1
A view of the asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids have been drawn at the 50% probability level and H atoms are represented by circles of arbitrary radii.

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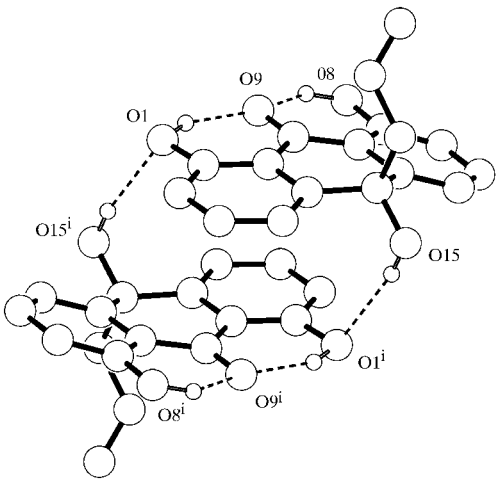

Figure 2
The connection of molecules of (I) into dimers [symmetry code: (i) $1-x$, $-y$, $-z$]. H atoms bonded to C atoms have been omitted for clarity.

hydroxy substituent on atom C10 forms an intermolecular hydrogen bond with the hydroxy O atom on atom C1 of a neighbouring centrosymmetrically related molecule. This interaction links the molecules into O—H···O hydrogen-bonded dimers that have a graph-set motif of $R_2^2(14)$ (Fig. 2).

Experimental

1,8-Dihydroxyanthraquinone (750 mg, 3.1 mmol), allyl bromide (654 mg, 5.4 mmol) and indium metal (413 mg, 3.6 mmol) were added to a mixture of tetrahydrofuran (10 ml), CH_3OH (10 ml) and water (5 ml), and the mixture was stirred at 303–305 K for 4–6 h. The reaction mixture was then quenched with saturated brine solution and dilute HCl until the mixture became clear. The product was extracted ($CHCl_3$), the extract was dried (Na_2SO_4) and the solvent was removed. Column chromatography (silica gel, 60–120 mesh) was used to isolate a pure yellow solid from the residue. The resulting yellow solid was recrystallized from a mixture of ethyl acetate and methanol to afford crystals of (I) (yield 90%, m.p. 394–396 K). MS (*m*/*z*, mass/relative intensity): 282 [M^+]; ^{1}H NMR ($CDCl_3$): δ 2.56 (1H, *s*, OH), 2.59–2.62 (2H, *m*, CH_2), 4.58–4.90 (2H, *m*, $=CH_2$), 5.10–5.24 (1H, *m*, $=CH$), 6.83 (2H, *d*, J_o = 8.2 Hz, H-2,7), 7.32 (2H, *d*, J_o = 8.2 Hz, H-4,5), 7.51 (2H, *t*, J_o = 8.2 Hz, H-3,6), 12.04 (2H, *s*, 2 × OH); ^{13}C NMR (normal/DEPT-135; $CDCl_3$): 54.23 (−ve, CH_2), 72.96 (ab, C), 114.32 (ab, C), 116.87 (+ve, CH), 119.87 (−ve, CH_2), 130.84 (+ve, CH), 136.58 (+ve, CH), 148.58 (ab, C), 161.77 (ab, C), 192.13 (ab, C); UV_{max} (EtOH, nm): 374 (8×10^2), 299 (7.4×10^2), 267 (6.2×10^2). Analysis found: C 72.6, H 4.7%; $C_{17}H_{14}O_4$ requires: C 72.34, H 4.96%.

Crystal data

$C_{17}H_{14}O_4$
M_r = 282.28
Monoclinic, $P2_1/c$
a = 9.0267 (1) Å
b = 20.0965 (3) Å
c = 8.0592 (1) Å
β = 116.0327 (9)°
V = 1313.65 (3) Å^3
Z = 4
D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3936 reflections
θ = 2.0–30.0°
μ = 0.10 mm^{-1}
T = 160 (2) K
Tablet, yellow
0.30 × 0.25 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans with κ offsets
34 734 measured reflections
3832 independent reflections
2963 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 30.0°
$h = -12 \rightarrow 11$
$k = -28 \rightarrow 0$
$l = 0 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.135
S = 1.05
3832 reflections
202 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0739P)^2 + 0.264P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Selected torsion angles (°).

C14—C10—C11—C12	22.51 (15)	C12—C9—C13—C14	5.78 (16)
C10—C11—C12—C9	−10.05 (17)	C9—C13—C14—C10	8.45 (16)
C13—C9—C12—C11	−4.99 (16)	C11—C10—C14—C13	−21.75 (15)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O9	0.925 (19)	1.74 (2)	2.5753 (13)	148.0 (17)
O8—H8···O9	1.00 (2)	1.64 (2)	2.5555 (14)	149 (2)
O15—H15···O1^i	0.87 (2)	2.15 (2)	2.9985 (13)	166.3 (17)

Symmetry code: (i) $1-x$, $-y$, $-z$.

Hydroxy H atoms were located from difference Fourier maps and their positions and individual isotropic displacement parameters were refined freely. The remaining H atoms were placed in idealized positions (C—H = 0.95–0.99 Å) and were constrained to ride on their parent atoms, with U_{iso}(H) values equal to $1.5U_{eq}$(C). Reflection 020 was partially obscured by the beam stop and hence was omitted.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1634). Services for accessing these data are described at the back of the journal.

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