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Acta Cryst. (1995). C**51**, 1641–1643

(±)-1-Acetoxy-8-hydroxy-1,4,4a,9a-tetrahydroanthraquinone

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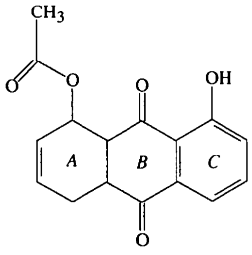
Abstract

The crystal structure of the title compound, methyl 8-hydroxy-9,10-dioxo-1,4,4a,9,9a,10-hexahydro-1-anthraceneacetate, $C_{16}H_{14}O_5$, is stabilized by intramolecular O—H···O and both intra- and intermolecular C—H···O hydrogen bonds. Ring *A*, carrying the acetoxy substituent, has a sofa conformation, the central ring *B* has an envelope conformation, while the aromatic ring *C* is planar.

Comment

The crystal and molecular structures of the title compound, (I), were investigated in order to determine the conformation and crystal packing, and also to confirm the stereochemistry.



(I)

A view of the molecule with the numbering scheme used is given in Fig. 1. The bond distances and angles are quite similar to those found in related compounds. From the torsion angles (see Table 2), it can be seen that rings *A* [puckering parameters φ_2 = 160.6 (3)°, θ_2 = 129.9 (2)°, Q = 0.434 (4) Å] and *B* [puckering parameters φ_2 = 172.4 (2)°, θ_2 = 120.2 (2)°, Q = 0.436 (4) Å] occur in sofa ($^6S_1$) and envelope (E_1) conformations, respectively (Boeyens, 1978). The aromatic ring *C* is planar to within 0.004 (2) Å.

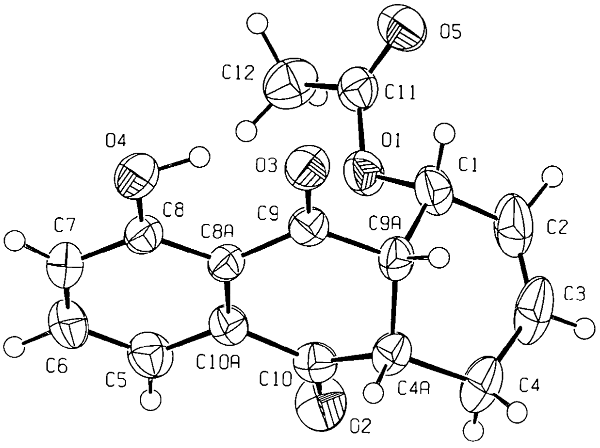


Fig. 1. The molecular structure of the title compound with the atom-labelling scheme and 50% probability displacement ellipsoids.

The hydroxy O4 atom forms an intramolecular hydrogen bond with the carbonyl O3 atom [O···O 2.561 (3) Å; see Table 3] (Allen, Kennard & Taylor, 1983), forming a six-membered ring composed of atoms C9, C8a, C8, O4, HO4 and O3, which is planar to within 0.02 (1) Å. This ring makes an interplanar angle of 4.8 (7)° with the aromatic ring *C* (C10a, C5–C8, C8a). The crystal structure (Fig. 2) is stabilized by O—H···O hydrogen bonds (Desiraju, 1991) and there are two intra- and three intermolecular C—H···O hydrogen bonds (Table 3).

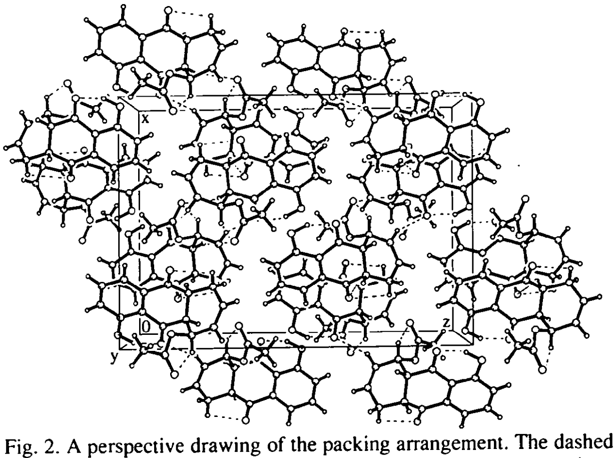


Fig. 2. A perspective drawing of the packing arrangement. The dashed lines indicate the hydrogen-bond contacts, details of which are given in Table 3.

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Experimental

The title compound was recrystallized from a benzene solution at room temperature.

Crystal data

$C_{16}H_{14}O_5$
$M_r = 286.27$
Orthorhombic
Pbca
$a = 14.383\,(4)$ Å
$b = 9.579\,(2)$ Å
$c = 20.122\,(5)$ Å
$V = 2772.3\,(12)$ Å^3
$Z = 8$
$D_x = 1.372$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 15\text{–}45°$
$\mu = 0.856$ mm^{-1}
$T = 293\,(2)$ K
Needle
0.30 × 0.20 × 0.20 mm
Pale yellow

Data collection

*P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1845 measured reflections
1844 independent reflections
1599 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.1303$

$\theta_{\rm max} = 56.73°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 10$
$l = -21 \rightarrow 1$
3 standard reflections monitored every 100 reflections intensity decay: 3%

Refinement

Refinement on F^2
$R(F) = 0.0478$
$wR(F^2) = 0.1427$
$S = 1.081$
1844 reflections
246 parameters
All H atoms were refined isotropically

$w = 1/[\sigma^2(F_o^2) + (0.0810P)^2 + 1.5334P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.016$
$\Delta\rho_{\rm max} = 0.225$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.164$ e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
C1	0.5587 (2)	0.1146 (3)	0.29169 (14)	0.0481 (7)
C2	0.5930 (3)	0.1761 (4)	0.2283 (2)	0.0702 (11)
C3	0.6744 (3)	0.1486 (4)	0.2035 (2)	0.0699 (11)
C4	0.7440 (3)	0.0540 (4)	0.23402 (15)	0.0571 (9)
C4a	0.7187 (2)	0.0070 (3)	0.30400 (13)	0.0384 (7)
C5	0.7785 (2)	0.1041 (3)	0.48043 (14)	0.0471 (7)
C6	0.7536 (2)	0.0632 (4)	0.54421 (15)	0.0572 (9)
C7	0.6773 (2)	−0.0189 (4)	0.55472 (15)	0.0525 (8)
C8	0.6236 (2)	−0.0621 (3)	0.50128 (12)	0.0391 (7)
C8a	0.6469 (2)	−0.0228 (3)	0.43682 (12)	0.0339 (6)
C9	0.5872 (2)	−0.0597 (3)	0.38089 (12)	0.0362 (6)
C9a	0.6141 (2)	−0.0145 (3)	0.31199 (12)	0.0383 (6)
C10	0.7571 (2)	0.0973 (3)	0.35832 (13)	0.0390 (6)
C10a	0.7266 (2)	0.0611 (3)	0.42686 (12)	0.0352 (6)
C11	0.4984 (2)	0.3030 (3)	0.35818 (13)	0.0421 (7)
C12	0.5236 (3)	0.4114 (4)	0.4077 (2)	0.0607 (9)
O1	0.57082 (12)	0.2200 (2)	0.34428 (9)	0.0454 (5)
O2	0.81466 (14)	0.1883 (2)	0.34869 (10)	0.0578 (6)
O3	0.51368 (12)	−0.1239 (2)	0.38955 (9)	0.0483 (6)
O4	0.54838 (14)	−0.1425 (2)	0.51409 (10)	0.0504 (6)
O5	0.42382 (15)	0.2906 (2)	0.33282 (10)	0.0619 (6)

Table 2. *Selected geometric parameters* (Å, °)

C1—C9a	1.526 (4)	C8—C8a	1.392 (4)
C1—O1	1.473 (3)	C8a—C10a	1.414 (3)
C1—C2	1.489 (5)	C8a—C9	1.459 (4)
C2—C3	1.300 (6)	C9—O3	1.235 (3)
C3—C4	1.483 (5)	C9a—C9	1.503 (4)
C4—C4a	1.523 (4)	C9a—C4a	1.527 (4)
C4a—C10	1.499 (4)	C10—O2	1.218 (3)
C5—C10a	1.374 (4)	C10—C10a	1.488 (4)
C5—C6	1.389 (4)	C11—O5	1.194 (3)
C6—C7	1.367 (5)	C11—O1	1.340 (3)
C7—C8	1.388 (4)	C11—C12	1.484 (5)
C8—O4	1.352 (3)		
O1—C1—C2	107.8 (3)	C10a—C8a—C9	120.4 (2)
O1—C1—C9a	107.5 (2)	O3—C9—C8a	121.0 (2)
C2—C1—C9a	112.2 (3)	O3—C9—C9a	119.6 (2)
C3—C2—C1	123.1 (3)	C8a—C9—C9a	119.3 (2)
C2—C3—C4	125.0 (3)	C9—C9a—C1	110.2 (2)
C3—C4—C4a	113.7 (3)	C9—C9a—C4a	112.9 (2)
C10—C4a—C4	114.5 (2)	C1—C9a—C4a	112.1 (2)
C10—C4a—C9a	111.3 (2)	O2—C10—C10a	121.0 (2)
C4—C4a—C9a	111.9 (2)	O2—C10—C4a	123.2 (2)
C10a—C5—C6	120.0 (3)	C10a—C10—C4a	115.6 (2)
C7—C6—C5	120.8 (3)	C5—C10a—C8a	120.0 (2)
C6—C7—C8	120.0 (3)	C5—C10a—C10	119.8 (2)
O4—C8—C7	117.9 (2)	C8a—C10a—C10	120.2 (2)
O4—C8—C8a	121.6 (2)	O5—C11—O1	123.4 (3)
C7—C8—C8a	120.5 (3)	O5—C11—C12	125.2 (3)
C8—C8a—C10a	118.8 (2)	O1—C11—C12	111.4 (3)
C8—C8a—C9	120.8 (2)	C11—O1—C1	117.7 (2)
C4a—C9a—C1—C2	44.5 (3)	C1—C9a—C4a—C4	−54.2 (3)
C9a—C1—C2—C3	−18.0 (5)	C10a—C8a—C9—C9a	3.4 (4)
C1—C2—C3—C4	−0.1 (6)	C4a—C9a—C9—C8a	25.9 (3)
C2—C3—C4—C4a	−9.1 (6)	C9a—C4a—C10—C10a	46.7 (3)
C3—C4—C4a—C9a	35.8 (4)	C9—C8a—C10a—C10	−7.8 (4)
C9—C9a—C4a—C10	−49.9 (3)	C4a—C10—C10a—C8a	−18.3 (3)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—HO4···O3	1.00 (4)	1.64 (4)	2.561 (3)	153 (2)
C1—H1···O5	0.97 (3)	2.29 (4)	2.700 (3)	104 (3)
C2—H2···O3$^{\rm i}$	1.01 (3)	2.48 (4)	3.412 (3)	154 (3)
C4—H41···O2	0.91 (4)	2.48 (5)	2.831 (3)	103 (4)
C4a—H4*A*···O2$^{\rm ii}$	0.96 (3)	2.49 (3)	3.219 (3)	133 (2)
C9a—H9*A*···O5$^{\rm iii}$	0.98 (3)	2.59 (3)	3.504 (3)	156 (2)

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $\frac{3}{2} - x, y - \frac{1}{2}, z$; (iii) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

Data collection: *P*4 diffractometer software. Cell refinement: *XSCANS* (Siemens, 1991). Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1057). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two 2,3-Naphthalenic Schiff Bases

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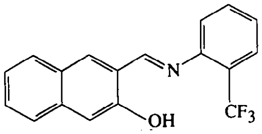
Abstract

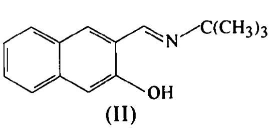
The crystal structures of 2-[2-(trifluoromethyl)phenyliminomethyl]-3-naphthol, (I), $C_{18}H_{12}F_3NO$, and 2-(*tert*-butyliminomethyl)-3-naphthol, (II), $C_{15}H_{17}NO$, have been determined. Both naphthalenic Schiff base molecules crystallize in the enol–imine tautomeric form. It is claimed that in the crystal of compound (I) there are both intra- and intermolecular hydrogen bonds, while in compound (II) there are only intramolecular hydrogen bonds, although a C—H···O interaction is found that may be considered as a weak intermolecular hydrogen bond.

Comment

Despite the considerable number of X-ray studies on the structures of Schiff bases derived from salicylaldehyde (Moustakali-Mavridis, Hadjoudis & Mavridis, 1980; Hadjoudis, Vitorakis & Moustakali-Mavridis, 1986), only a small number of 1,2-naphthalenic Schiff base analogues have been structurally characterized by X-ray crystallography (Acevedo-Arauz, Fernández-G., Rosales-Hoz & Toscano, 1992) and, to our knowledge, there are no reports of single-crystal X-ray diffraction studies on any 2,3-naphthalenic Schiff base analogues. We report here the structures of 2-[2-(trifluoromethyl)phenyliminomethyl]-3-naphthol, (I), and 2-(*tert*-butyliminomethyl)-3-naphthol, (II).



(I)



(II)

In compound (I), the mean C—F bond length is 1.331 (3) Å. Substitution of the trifluoromethyl group at atom C(13) does not cause any perturbation in the geometry and conformation of the benzene ring. The crystals of both compounds, the molecules are in the enol–imine tautomeric form. Examination of the crystal packing reveals that in both structures, the conformation of the side chain at atom C(2) orients the imino N atom in such a way that an intramolecular hydrogen bond is formed from the O(1) hydroxyl group at C(3) to atom N(1). Considering the interactions quoted in Table 3, it appears that in both compounds there is an intramolecular hydrogen bond of the O—H···N type and also intermolecular interactions of the C—H···O type that can be considered as weak hydrogen bonds (Taylor & Kennard, 1982; Berkovitch-Yellin & Leiserowitz, 1984). In addition, in compound (I) there are intra- and intermolecular attractive interactions of the O—H···F and C—H···F types; the intramolecular ones determine the orientation of the trifluoromethyl group with respect to the phenyl ring, while the intermolecular ones contribute to the packing in the crystal.

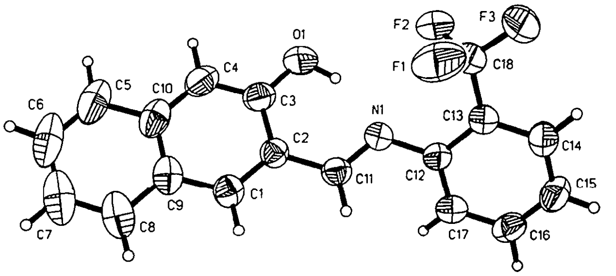


Fig. 1. The molecular structure of compound (I) with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

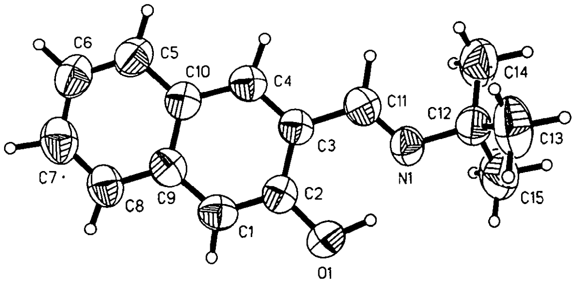


Fig. 2. The molecular structure of compound (II) with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

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† Contribution No. 1302 of the Instituto de Química, UNAM.