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2-Bromobenz[*a*]anthracene-5,6,7,12-tetraone-5-dimethylketal

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Key indicators

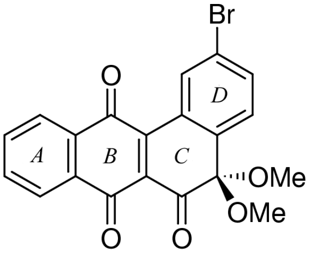
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.066
wR factor = 0.165
Data-to-parameter ratio = 16.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, 2-bromo-5,5-dimethoxybenzo[*a*]-anthracene-6,7,12(5*H*)trione, $C_{20}H_{13}BrO_5$, the two aromatic rings are essentially planar and make a dihedral angle of 15.1 (2)° with one another, while the dimethoxy-substituted cyclohexane ring is non-planar, as expected. An intramolecular C—H···O interaction is present, with C···O = 2.814 (5) Å and C—H···O = 125°.

Comment

The analogues of angucyclines have been intensively investigated due to their biological properties (Krohn & Rohr, 1997). In order to generate a new analogue, we have prepared the title compound, (I). We have undertaken an X-ray structural determination of compound (I) to define the conformation of this system, especially the conformation of the bromophenyl with respect to the substituted anthracene moiety.



(I)

In (I), the bond lengths and angles have normal values. In the aromatic rings *A* and *D*, the average C—C bond distances are 1.387 and 1.395 Å, respectively. In comparison, the C—C bond distances within the cyclohexanedione (ring *B*) and that of C6*A*—C12*A* are slightly shorter than in the related structure, benz[*a*]anthracene-7,12-dione, (II), previously studied (Kuroda *et al.*, 1982), while the observed intramolecular interaction between C1*A*—H1*A* and O5 in (I) is slightly longer than in (II) (2.058 Å).

The aromatic rings *A* and *D* are essentially planar, and atom Br1 deviates slightly (by 0.035 Å) from the least-squares plane through ring *D*. The angle between these two aromatic rings is 15.1 (2)° and is much larger than in (II) benz[*a*]anthracene-7,12-dione (Kuroda *et al.*, 1982), because in (I), ring *C*, bridging between rings *A* and *D*, is non-planar. The close interaction between O5 and H1*A*, and the non-bonded lone-pair–lone-pair repulsions of the O atoms [the non-bonded distance between O1 and O2 is 2.880 (5) Å] are two reasons for the distortions from planarity. Atom O1 lies on, while atom O5

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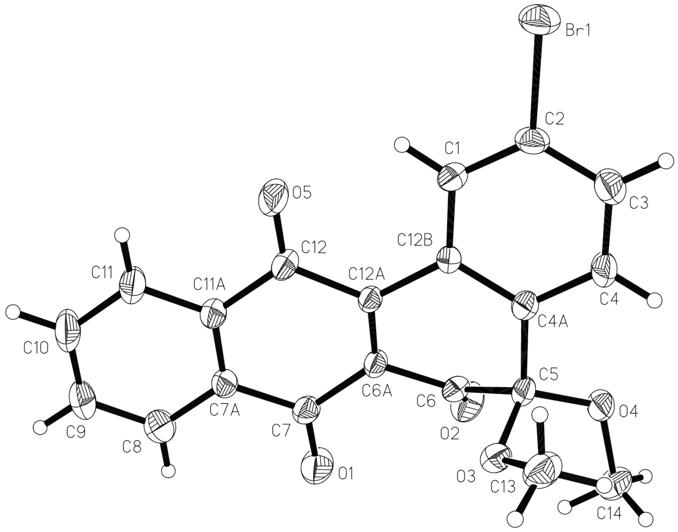

Figure 1
The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

deviates slightly by −0.121 (4) Å from the least-squares plane through ring *B* having a maximum deviation of 0.052 (4) Å (C12*A*). Atom O2 attached to ring *C* at C6 significantly deviates by −1.087 (4) Å, while atom C6 deviates by −0.317 (4) Å from the least-squares plane through ring *C*. The puckering parameters of ring *C* (Cremer & Pople, 1975), which adopts a half-chair conformation, are Q_T = 0.478 (5) Å, θ = 115.5 (6)° and φ_2 = 275.5 (6)°.

Experimental

To a solution of 2-bromo-6-hydroxy-5-methoxybenz[a]anthracene in methanol was added an equimolar amount of phenyliodonium acetate. The mixture was stirred for 3 h at room temperature. After stirring, the solvent was removed *in vacuo*, and the residue was purified by column chromatography. The residue afforded a brown–red solid and was recrystallized from a mixture of ethyl acetate and petroleum ether (25/75).

Crystal data

$C_{20}H_{13}BrO_5$
M_r = 413.21
Triclinic, $P\bar{1}$
a = 7.7414 (1) Å
b = 10.3084 (1) Å
c = 10.9583 (2) Å
α = 74.415 (1)°
β = 78.835 (1)°
γ = 81.192 (1)°
V = 821.65 (2) Å^3
Z = 2
D_x = 1.670 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3602 reflections
θ = 2.0–28.3°
μ = 2.53 mm^{-1}
T = 293 (2) K
Slab, brown–red
0.36 × 0.26 × 0.10 mm

Data collection

Siemens SMART CCD diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.463, $T_{\max}$ = 0.786
5992 measured reflections
3910 independent reflections
2584 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.055
$\theta_{\max}$ = 28.3°
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 13$
$l = -14 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.165
S = 0.94
3910 reflections
237 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0817P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.97 e Å^{-3}
$\Delta\rho_{\min}$ = −1.79 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.896 (4)	C4—C4*A*	1.402 (6)
O1—C7	1.216 (5)	C1—C12*B*	1.400 (6)
O2—C6	1.200 (5)	C4*A*—C12*B*	1.416 (5)
O3—C5	1.430 (5)	C4*A*—C5	1.525 (5)
O4—C5	1.388 (5)	C5—C6	1.534 (6)
O5—C12	1.208 (5)	C6—C6*A*	1.499 (5)
C1—C2	1.386 (6)	C6*A*—C12*A*	1.353 (6)
C2—C3	1.385 (6)	C7*A*—C11*A*	1.388 (6)
C3—C4	1.379 (6)	C12*A*—C12*B*	1.498 (5)
C5—O3—C13	116.3 (3)	O3—C5—O4	112.9 (3)
C5—O4—C14	117.3 (3)	O2—C6—C5	123.4 (4)
C1—C2—C3	121.7 (4)	O2—C6—C6*A*	124.1 (4)
C1—C2—Br1	120.4 (3)	C5—C6—C6*A*	112.5 (3)
C3—C2—Br1	118.0 (3)		
C12*B*—C4*A*—C5—C6	33.5 (5)	C11—C11*A*—C12—O5	−4.4 (7)
O4—C5—C6—O2	3.7 (6)	C11*A*—C12—C12*A*—C12*B*	−173.0 (4)
O2—C6—C6*A*—C7	47.0 (6)	C12—C12*A*—C12*B*—C1	−13.3 (6)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O5	0.93	2.18	2.814 (5)	125

After checking their presence in the difference map, all H atoms were geometrically fixed and allowed to ride on their attached atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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