organic papers

The Diels–Alder adduct of *p*-benzoquinone and anthracene: 9,10-tetrahydro-9,10[1′,2′]-benzenoanthracene-1′,4′(2′*H*,3′*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.136
Data-to-parameter ratio = 11.9

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

The structure of the title compound, $C_{20}H_{14}O_2$, has a rigid bicyclic backbone, and the six-membered diketone ring is in a shallow boat conformation. Both carbonyl groups are orientated away from the underlying benzene rings. The structure is compared to other similar anthracene Diels–Alder adducts.

Comment

Diels–Alder adducts from the reaction of anthracene with dienophiles have been used in a variety of applications, including the synthesis of discrete molecular architectures such as molecular gears (Stevens & Richards, 1997). Although the crystal structures of a number of such derivatives have been disclosed, somewhat surprisingly the structure of the adduct (1) of *p*-benzoquinone and anthracene has not been previously reported. Tautomer (2) does appear in the Cambridge Structural Database (Version 5.25; Allen *et al.* 2002); however, its full structure has not been deposited (Hashimoto *et al.*, 1999).
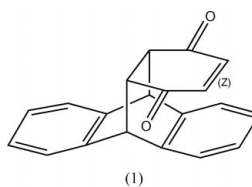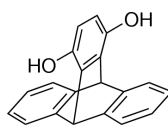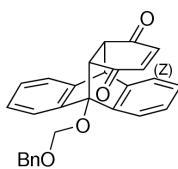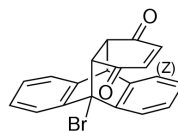

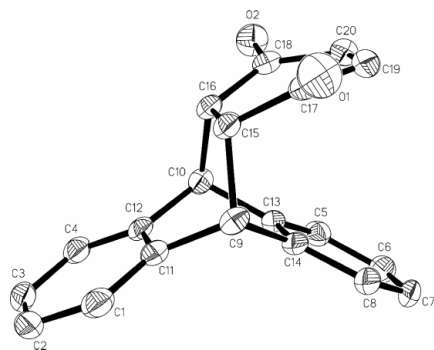The 2-ene-1,4-dione ring in (1) is in a shallow boat conformation in which the bonds C15—C16 and C19—C20 are parallel, and all the atoms of both these bonds are coplanar (r.m.s. deviation 0.007 Å). The two sets of atoms O2/C18/C16/C20 and O1/C17/C19/C15 (which contain the carbonyl groups) are essentially planar (r.m.s. deviations 0.009 and 0.007 Å, respectively) and these planar groups (O2/C18/C16/C20 and O1/C17/C19/C15) have dihedral angles with the previous plane (C15/C16/C19/C20) of 12.34 (13) and 18.99 (11)°,

Figure 1
View of (1) (50% probability displacement ellipsoids). H atoms are not shown.

Figure 2
Packing diagram (Spek, 2003) showing weak C—H···O interactions (dashed lines). O atoms are coloured red.

respectively. The difference in these angles is intriguing since the molecule itself is otherwise symmetrical. Unsymmetrical 9-substituted anthracene Diels–Alder adducts (3) and (4) also show similar deviations, although this is obviously more pronounced for the carbonyl group located proximal to the 9-substituent (Bharadwaj *et al.*, 1985; Watson & Nagl, 1988).

In the crystal structure, weak intermolecular C—H···O bonds (see Table 1) connect the molecules into a three-dimensional network (Fig. 2)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1[i]	0.95	2.52	3.388 (3)	151
C6—H6···O2[ii]	0.95	2.46	3.385 (3)	164
C10—H10···O2[iii]	1.00	2.57	3.302 (2)	130

Symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $2 - x, -y, -z$; (iii) $2 - x, 1 - y, -z$.

H atoms were positioned geometrically and refined with a riding model (including torsional freedom for methyl groups), with C—H = 0.95–0.98 Å, and with U_{iso}(H) values constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

Experimental

The title compound, (1), was prepared by the thermal Diels–Alder addition reaction of anthracene with *p*-benzoquinone (Wasielewski *et al.*, 1989). Suitable crystals for X-ray diffraction analysis were obtained by slow evaporation of a dichloromethane/petrol (60–80) solution, resulting in colourless crystals.
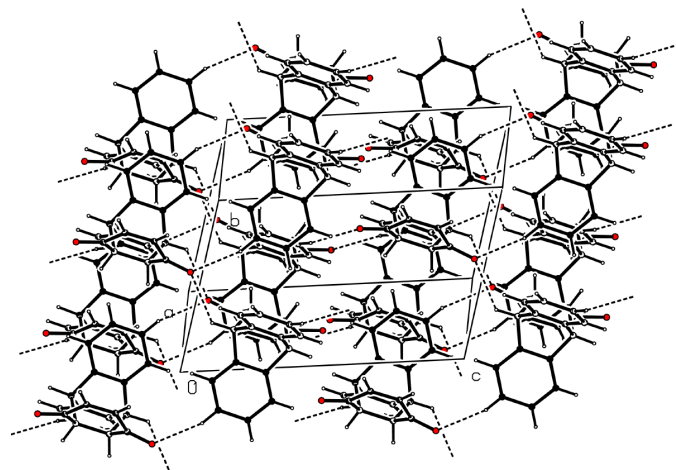
Crystal data

$C_{20}H_{14}O_2$	$Z = 2$
$M_r = 286.31$	$D_x = 1.375$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 6.870$ (3) Å	Cell parameters from 1866
$b = 8.333$ (4) Å	reflections
$c = 12.707$ (5) Å	$\theta = 5.0$–54.7°
$\alpha = 78.567$ (7)°	$\mu = 0.09$ mm^{-1}
$\beta = 78.991$ (7)°	$T = 150$ (2) K
$\gamma = 79.361$ (7)°	Block, colourless
$V = 691.7$ (5) Å^3	$0.46 \times 0.24 \times 0.16$ mm

Data collection

Bruker SMART 1000	2373 independent reflections
diffractometer	1741 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.025$
Absorption correction: multi-scan	$\theta_{max} = 25°$
(*SADABS*; Bruker, 1997)	$h = -8 \rightarrow 8$
$T_{min} = 0.961$, $T_{max} = 0.986$	$k = -9 \rightarrow 9$
4302 measured reflections	$l = -15 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0738P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.047$	$+ 0.2376P]$
$wR(F^2) = 0.136$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
2373 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
199 parameters	$\Delta\rho_{min} = -0.27$ e Å^{-3}
H-atom parameters constrained	

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bharadwaj, P., Potenza, J. A., Ornaf, R. M., Rodriques, K. E., Knapp, S. & Lalancette, R. A. (1985). *Acta Cryst.* C**41**, 1520–1522.
Bruker (1997). *SMART, SAINT, SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hashimoto, M., Takagi, H. & Yamamura, K. (1999). *Tetrahedron*, **40**, 6037–6040.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Spek, A. L. (2003). *PLATON*. University of Utrecht, The Netherlands.
Stevens, A. M. & Richards, C. J. (1997). *Tetrahedron Lett.* **38**, 7805–7808.
Wasielewski, M. R., Niemczyk, M. P., Johnson, D. G., Svec, W. A. & Minsek, D. W. (1989). *Tetrahedron*, **45**, 4785–4806.
Watson, H. W. & Nagl, A. (1988). *Acta Cryst.* C**44**, 381–383.