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## Key indicators

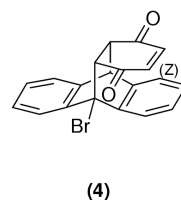
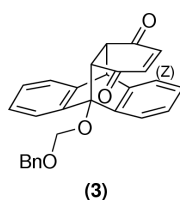
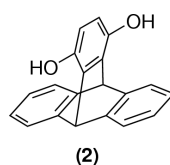
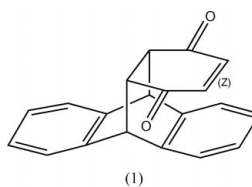
Single-crystal X-ray study  
 $T = 150\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
 $R$  factor = 0.047  
 $wR$  factor = 0.136  
Data-to-parameter ratio = 11.9For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.The Diels–Alder adduct of *p*-benzoquinone  
and anthracene: 9,10-tetrahydro-9,10[1',2']-  
benzenoanthracene-1',4'(2'*H*,3'*H*)-dione

The structure of the title compound,  $\text{C}_{20}\text{H}_{14}\text{O}_2$ , 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.

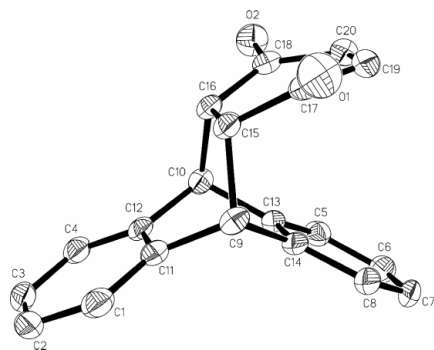
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## Comment

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.

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)

## 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

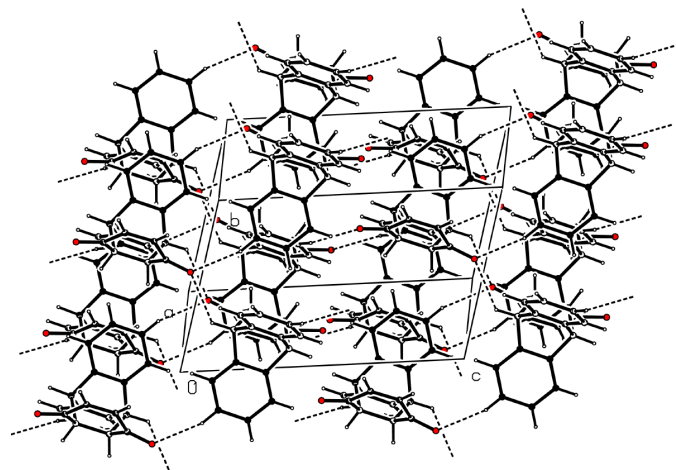
$C_{20}H_{14}O_2$	$Z = 2$
$M_r = 286.31$	$D_x = 1.375 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 6.870$ (3) Å	Cell parameters from 1866 reflections
$b = 8.333$ (4) Å	$\theta = 5.0\text{--}54.7^\circ$
$c = 12.707$ (5) Å	$\mu = 0.09 \text{ mm}^{-1}$
$\alpha = 78.567$ (7)°	$T =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

### Data collection

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

### Refinement

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



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

**Table 1**

Hydrogen-bonding geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
$C2\text{--}H2\cdots O1^i$	0.95	2.52	3.388 (3)	151
$C6\text{--}H6\cdots O2^{ii}$	0.95	2.46	3.385 (3)	164
$C10\text{--}H10\cdots O2^{iii}$	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  $U_{\text{iso}}(\text{H})$  values constrained to be 1.2 (1.5 for methyl groups) times  $U_{\text{eq}}$  of the carrier atom.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* (Bruker, 1997).

We thank the Department of Chemistry, University of Sheffield, for support (IO).

## References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Bharadwaj, P., Potenza, J. A., Ornaf, R. M., Rodrigues, K. E., Knapp, S. & Lalancette, R. A. (1985). *Acta Cryst.* **C41**, 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). *SHELXL97* and *SHELXS97*. 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.* **C44**, 381–383.

## supporting information

*Acta Cryst.* (2004). E60, o690–o691 [https://doi.org/10.1107/S160053680400707X]

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

Harry Adams, Isaac Ojea-Jimenez and Simon Jones

(1)

### Crystal data

C<sub>20</sub>H<sub>14</sub>O<sub>2</sub>

*M<sub>r</sub>* = 286.31

Triclinic, *P* $\bar{1}$

Hall symbol: -P 1

*a* = 6.870 (3) Å

*b* = 8.333 (4) Å

*c* = 12.707 (5) Å

$\alpha$  = 78.567 (7)°

$\beta$  = 78.991 (7)°

$\gamma$  = 79.361 (7)°

*V* = 691.7 (5) Å<sup>3</sup>

*Z* = 2

*F*(000) = 300

*D<sub>x</sub>* = 1.375 Mg m<sup>-3</sup>

Mo *K* $\alpha$  radiation,  $\lambda$  = 0.71073 Å

Cell parameters from 1866 reflections

$\theta$  = 5.1–54.7°

$\mu$  = 0.09 mm<sup>-1</sup>

*T* = 150 K

Plate, colourless

0.46 × 0.24 × 0.16 mm

### Data collection

Bruker SMART 1000  
diffractometer

$\omega$  scans

Absorption correction: multi-scan  
(SADABS; Bruker, 1997)

*T<sub>min</sub>* = 0.961, *T<sub>max</sub>* = 0.986

4302 measured reflections

2373 independent reflections

1741 reflections with *I* > 2 $\sigma$ (*I*)

*R<sub>int</sub>* = 0.025

$\theta_{\max}$  = 25°,  $\theta_{\min}$  = 1.7°

*h* = -8→8

*k* = -9→9

*l* = -15→14

### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R* [*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.047

*wR*(*F*<sup>2</sup>) = 0.136

*S* = 1.03

2373 reflections

199 parameters

0 restraints

H-atom parameters constrained

*w* = 1/[ $\sigma^2(F_o^2) + (0.0738P)^2 + 0.2376P$ ]

where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

( $\Delta/\sigma$ )<sub>max</sub> < 0.001

$\Delta\rho_{\max}$  = 0.24 e Å<sup>-3</sup>

$\Delta\rho_{\min}$  = -0.27 e Å<sup>-3</sup>

### Special details

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O2	1.1857 (2)	0.32714 (19)	0.04934 (12)	0.0371 (4)
O1	1.0437 (3)	0.1850 (2)	0.47566 (13)	0.0435 (5)
C1	0.3560 (3)	0.4841 (3)	0.37728 (18)	0.0303 (5)
H1	0.3297	0.4512	0.454	0.036*
C2	0.2296 (3)	0.6139 (3)	0.3246 (2)	0.0344 (6)
H2	0.1158	0.6693	0.3657	0.041*
C3	0.2696 (3)	0.6620 (3)	0.2129 (2)	0.0329 (6)
H3	0.1821	0.7497	0.1781	0.039*
C4	0.4363 (3)	0.5838 (2)	0.15056 (18)	0.0283 (5)
H4	0.4637	0.6183	0.0741	0.034*
C5	0.7541 (3)	0.0568 (2)	0.11251 (18)	0.0267 (5)
H5	0.7846	0.09	0.036	0.032*
C6	0.7347 (3)	-0.1085 (3)	0.15683 (19)	0.0315 (5)
H6	0.7483	-0.1874	0.1102	0.038*
C7	0.6957 (3)	-0.1569 (2)	0.26878 (19)	0.0310 (5)
H7	0.6827	-0.2692	0.2982	0.037*
C8	0.6754 (3)	-0.0431 (2)	0.33860 (18)	0.0269 (5)
H8	0.6519	-0.0778	0.4152	0.032*
C9	0.6743 (3)	0.2622 (2)	0.35815 (16)	0.0231 (5)
H9	0.6397	0.2253	0.4384	0.028*
C10	0.7485 (3)	0.3545 (2)	0.14796 (16)	0.0227 (5)
H10	0.7706	0.389	0.0671	0.027*
C11	0.5212 (3)	0.4039 (2)	0.31528 (17)	0.0241 (5)
C12	0.5617 (3)	0.4541 (2)	0.20263 (16)	0.0230 (5)
C13	0.7283 (3)	0.1716 (2)	0.18166 (16)	0.0217 (5)
C14	0.6898 (3)	0.1220 (2)	0.29469 (16)	0.0219 (5)
C15	0.8831 (3)	0.3280 (2)	0.32630 (16)	0.0231 (5)
H15	0.8699	0.4303	0.3586	0.028*
C16	0.9310 (3)	0.3772 (2)	0.20054 (16)	0.0230 (5)
H16	0.9459	0.4972	0.1834	0.028*
C17	1.0366 (3)	0.1979 (3)	0.37928 (17)	0.0275 (5)
C18	1.1179 (3)	0.2805 (2)	0.14419 (17)	0.0253 (5)
C19	1.1718 (3)	0.0843 (3)	0.31257 (18)	0.0306 (5)
H19	1.2319	-0.0196	0.3475	0.037*
C20	1.2121 (3)	0.1233 (3)	0.20460 (18)	0.0301 (5)
H20	1.3041	0.0481	0.165	0.036*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O2	0.0384 (10)	0.0381 (9)	0.0304 (9)	-0.0078 (7)	0.0038 (7)	-0.0022 (7)
O1	0.0513 (11)	0.0500 (10)	0.0306 (10)	-0.0075 (8)	-0.0173 (8)	0.0000 (7)
C1	0.0308 (13)	0.0277 (11)	0.0351 (13)	-0.0117 (10)	-0.0005 (10)	-0.0095 (10)
C2	0.0226 (12)	0.0284 (12)	0.0553 (16)	-0.0053 (9)	-0.0009 (10)	-0.0185 (11)
C3	0.0289 (13)	0.0210 (10)	0.0518 (15)	-0.0033 (9)	-0.0124 (11)	-0.0080 (10)

C4	0.0296 (13)	0.0228 (11)	0.0356 (13)	-0.0083 (9)	-0.0104 (10)	-0.0032 (9)
C5	0.0240 (12)	0.0277 (11)	0.0303 (12)	-0.0038 (9)	-0.0059 (9)	-0.0079 (9)
C6	0.0267 (13)	0.0238 (11)	0.0486 (15)	-0.0035 (9)	-0.0096 (10)	-0.0140 (10)
C7	0.0271 (13)	0.0153 (10)	0.0511 (15)	-0.0050 (8)	-0.0111 (10)	-0.0005 (9)
C8	0.0231 (12)	0.0227 (10)	0.0341 (12)	-0.0047 (9)	-0.0083 (9)	0.0020 (9)
C9	0.0267 (12)	0.0228 (10)	0.0205 (10)	-0.0091 (9)	-0.0021 (8)	-0.0025 (8)
C10	0.0258 (12)	0.0203 (10)	0.0228 (11)	-0.0054 (8)	-0.0052 (9)	-0.0021 (8)
C11	0.0233 (12)	0.0215 (10)	0.0304 (12)	-0.0083 (9)	-0.0046 (9)	-0.0065 (8)
C12	0.0242 (12)	0.0177 (10)	0.0311 (12)	-0.0084 (8)	-0.0082 (9)	-0.0047 (8)
C13	0.0179 (11)	0.0195 (10)	0.0295 (11)	-0.0042 (8)	-0.0067 (8)	-0.0041 (8)
C14	0.0183 (11)	0.0207 (10)	0.0284 (11)	-0.0062 (8)	-0.0065 (8)	-0.0023 (8)
C15	0.0248 (12)	0.0195 (10)	0.0267 (11)	-0.0069 (8)	-0.0050 (9)	-0.0043 (8)
C16	0.0242 (12)	0.0185 (10)	0.0276 (11)	-0.0077 (8)	-0.0047 (9)	-0.0024 (8)
C17	0.0286 (12)	0.0281 (11)	0.0280 (12)	-0.0133 (9)	-0.0074 (9)	0.0013 (9)
C18	0.0249 (12)	0.0259 (11)	0.0273 (12)	-0.0111 (9)	-0.0037 (9)	-0.0036 (9)
C19	0.0242 (12)	0.0260 (11)	0.0399 (14)	-0.0046 (9)	-0.0101 (10)	0.0037 (9)
C20	0.0231 (12)	0.0268 (11)	0.0397 (14)	-0.0034 (9)	-0.0030 (10)	-0.0060 (10)

*Geometric parameters (Å, °)*

O2—C18	1.220 (2)	C9—C11	1.517 (3)
O1—C17	1.218 (3)	C9—C14	1.524 (3)
C1—C11	1.393 (3)	C9—C15	1.582 (3)
C1—C2	1.401 (3)	C9—H9	1
C1—H1	0.95	C10—C12	1.521 (3)
C2—C3	1.385 (3)	C10—C13	1.524 (3)
C2—H2	0.95	C10—C16	1.585 (3)
C3—C4	1.395 (3)	C10—H10	1
C3—H3	0.95	C11—C12	1.398 (3)
C4—C12	1.392 (3)	C13—C14	1.401 (3)
C4—H4	0.95	C15—C17	1.516 (3)
C5—C13	1.389 (3)	C15—C16	1.556 (3)
C5—C6	1.401 (3)	C15—H15	1
C5—H5	0.95	C16—C18	1.518 (3)
C6—C7	1.387 (3)	C16—H16	1
C6—H6	0.95	C17—C19	1.473 (3)
C7—C8	1.394 (3)	C18—C20	1.484 (3)
C7—H7	0.95	C19—C20	1.333 (3)
C8—C14	1.392 (3)	C19—H19	0.95
C8—H8	0.95	C20—H20	0.95
C11—C1—C2	118.9 (2)	C1—C11—C12	120.52 (19)
C11—C1—H1	120.6	C1—C11—C9	126.16 (19)
C2—C1—H1	120.6	C12—C11—C9	113.30 (17)
C3—C2—C1	120.3 (2)	C4—C12—C11	120.49 (19)
C3—C2—H2	119.9	C4—C12—C10	125.93 (19)
C1—C2—H2	119.9	C11—C12—C10	113.58 (17)
C2—C3—C4	121.1 (2)	C5—C13—C14	120.47 (18)

C2—C3—H3	119.4	C5—C13—C10	126.32 (19)
C4—C3—H3	119.4	C14—C13—C10	113.12 (17)
C12—C4—C3	118.7 (2)	C8—C14—C13	120.11 (18)
C12—C4—H4	120.6	C8—C14—C9	126.38 (19)
C3—C4—H4	120.6	C13—C14—C9	113.48 (17)
C13—C5—C6	119.3 (2)	C17—C15—C16	116.20 (17)
C13—C5—H5	120.4	C17—C15—C9	107.70 (16)
C6—C5—H5	120.4	C16—C15—C9	109.37 (16)
C7—C6—C5	120.0 (2)	C17—C15—H15	107.8
C7—C6—H6	120	C16—C15—H15	107.8
C5—C6—H6	120	C9—C15—H15	107.8
C6—C7—C8	120.90 (19)	C18—C16—C15	116.24 (16)
C6—C7—H7	119.6	C18—C16—C10	106.80 (16)
C8—C7—H7	119.6	C15—C16—C10	109.35 (16)
C14—C8—C7	119.2 (2)	C18—C16—H16	108.1
C14—C8—H8	120.4	C15—C16—H16	108.1
C7—C8—H8	120.4	C10—C16—H16	108.1
C11—C9—C14	107.99 (16)	O1—C17—C19	120.9 (2)
C11—C9—C15	105.89 (16)	O1—C17—C15	120.5 (2)
C14—C9—C15	106.44 (16)	C19—C17—C15	118.58 (18)
C11—C9—H9	112	O2—C18—C20	120.10 (19)
C14—C9—H9	112	O2—C18—C16	120.82 (19)
C15—C9—H9	112	C20—C18—C16	119.02 (18)
C12—C10—C13	107.89 (16)	C20—C19—C17	121.79 (19)
C12—C10—C16	106.94 (15)	C20—C19—H19	119.1
C13—C10—C16	105.24 (16)	C17—C19—H19	119.1
C12—C10—H10	112.1	C19—C20—C18	122.1 (2)
C13—C10—H10	112.1	C19—C20—H20	118.9
C16—C10—H10	112.1	C18—C20—H20	118.9

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C2—H2 $\cdots$ O1 <sup>i</sup>	0.95	2.52	3.388 (3)	151
C6—H6 $\cdots$ O2 <sup>ii</sup>	0.95	2.46	3.385 (3)	164
C10—H10 $\cdots$ O2 <sup>iii</sup>	1.00	2.57	3.302 (2)	130

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