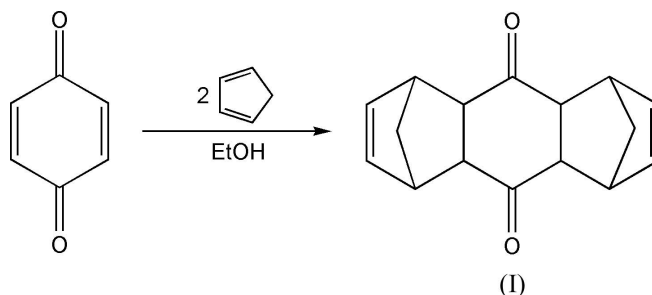


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Key indicators

Single-crystal X-ray study
 $T = 120\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
 R factor = 0.042
 wR factor = 0.084
Data-to-parameter ratio = 12.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Redetermination of the Diels–Alder diadduct of
1,4-benzoquinone and cyclopentadiene: 1,4:5,8-di-
methano-1,1a,4,4a,5,5a,8,8a-octahydroanthracene-
9,10-dioneThe title diadduct, $\text{C}_{16}\text{H}_{16}\text{O}_2$, has been redetermined by X-ray
diffraction methods at 120 K. In this work, we report more
exact values of bond and angles.Received 3 March 2005
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Comment

The Diels–Alder reaction between 1,4-benzoquinone and two
equivalents of cyclopentadiene gives the title diadduct 1,4:5,8-
dimethano-1,1a,4,4a,5,5a,8,8a-octahydroanthracene-9,10-di-
one, (I) (Fig. 1 and Table 1).

The structure of (I) was first reported in 1974 but with a space-group ambiguity (Brown *et al.*, 1974). The corrected room-temperature structure was reported in 1999 (Gunes *et al.*, 1999). To define more precisely the molecular structure of (I), we have studied the structure at low temperature, *viz.* 120 K. The analysis confirms that the stereochemistry is *endo,cis,anti,cis,endo*.

Despite of the slight change in the deviations of the bond lengths and angles (0.001 Å for bond lengths and 0.01° for angles), the bond lengths and angles themselves have changed dramatically. For example, in our work, the C=O distances are in the range 1.2211 (16)–1.2189 (16) Å and the C–C distances are in the range 1.5115 (19)–1.5798 (19) Å, while in Gunes *et al.* (1999), the C=O is 1.215 (2) Å and the C–C distances are in the range 1.499 (3)–1.579 (3) Å. The increase in distances is connected with a significant reduction in the atomic anisotropic displacement parameters and the measurement of the present data at 120 K. It results in a reduction of the amplitudes of thermal vibration of atoms and their localization. Accordingly, the atomic coordinates have much smaller deviations than in the previous work. Thus, in our work, more exact values of bonds lengths and angles are presented.

Experimental

The title compound was prepared according to the known procedure of Brown *et al.* (1974) (see scheme). Suitable single crystals were

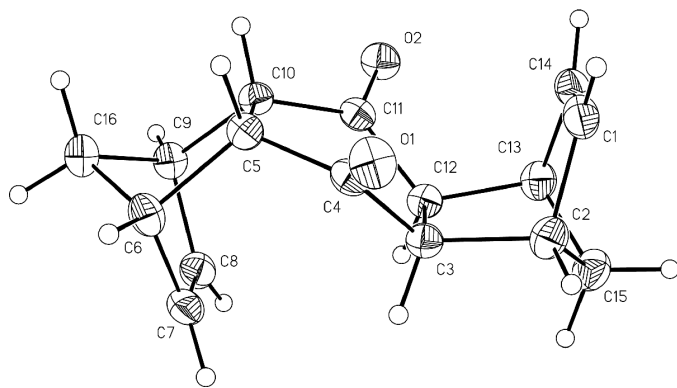


Figure 1
A view of (I), showing the atom labeling and displacement ellipsoids at the 50% probability level.

obtained by isothermic evaporation of its solution in ethanol at room temperature.

Crystal data

$C_{16}H_{16}O_2$
 $M_r = 240.29$
Monoclinic, $P2_1/n$
 $a = 11.909$ (4) Å
 $b = 6.166$ (2) Å
 $c = 16.937$ (6) Å
 $\beta = 110.278$ (7)°
 $V = 1166.6$ (7) Å³
 $Z = 4$

$D_x = 1.368$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 944 reflections
 $\theta = 2.8$ – 29.9 °
 $\mu = 0.09$ mm⁻¹
 $T = 120$ (2) K
Prism, colorless
 $0.4 \times 0.3 \times 0.2$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1998)
 $T_{\min} = 0.964$, $T_{\max} = 0.981$
7972 measured reflections

2922 independent reflections
2435 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 28.6$ °
 $h = -13 \rightarrow 15$
 $k = -8 \rightarrow 8$
 $l = -22 \rightarrow 22$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.084$
 $S = 1.00$
2922 reflections
227 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.1078P)^2 + 0.097P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1
Selected geometric parameters (Å, °).

O2—C11	1.2211 (16)	C10—C11	1.5183 (18)
O1—C4	1.2189 (16)	C11—C12	1.5115 (19)
C5—C4	1.5135 (19)	C12—C13	1.5610 (19)
C5—C10	1.5586 (18)	C1—C14	1.338 (2)
C5—C6	1.5798 (19)	C1—C2	1.520 (2)
C3—C4	1.5133 (18)	C8—C7	1.336 (2)
C3—C2	1.5621 (19)	C2—C15	1.545 (2)
C3—C12	1.5650 (18)	C14—C13	1.522 (2)
C9—C8	1.515 (2)	C6—C7	1.513 (2)
C9—C16	1.5449 (19)	C6—C16	1.542 (2)
C9—C10	1.5794 (19)	C13—C15	1.540 (2)
C4—C5—C10	116.42 (10)	C14—C1—C2	107.54 (13)
C4—C5—C6	114.05 (11)	C7—C8—C9	107.23 (12)
C10—C5—C6	102.59 (10)	O1—C4—C3	121.25 (12)
C4—C3—C2	113.46 (11)	O1—C4—C5	119.97 (12)
C4—C3—C12	116.03 (11)	C3—C4—C5	118.77 (11)
C2—C3—C12	102.91 (10)	C1—C2—C15	100.15 (12)
C8—C9—C16	100.49 (11)	C1—C2—C3	106.04 (11)
C8—C9—C10	107.75 (11)	C15—C2—C3	100.15 (11)
C16—C9—C10	99.22 (10)	C1—C14—C13	107.53 (13)
C11—C10—C5	116.39 (11)	C7—C6—C16	100.29 (11)
C11—C10—C9	113.66 (11)	C7—C6—C5	107.92 (11)
C5—C10—C9	102.70 (10)	C16—C6—C5	99.15 (11)
O2—C11—C12	121.01 (12)	C8—C7—C6	107.91 (13)
O2—C11—C10	119.99 (12)	C6—C16—C9	93.60 (10)
C12—C11—C10	118.97 (11)	C14—C13—C15	100.33 (12)
C11—C12—C13	113.96 (11)	C14—C13—C12	106.15 (11)
C11—C12—C3	116.64 (10)	C15—C13—C12	100.26 (11)
C13—C12—C3	102.62 (11)	C13—C15—C2	93.94 (11)

The H atoms were refined with individual isotropic displacement parameters; the range of C—H was 0.964 (17)–1.023 (17) Å.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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