Crystal structure of 1,4:5,8-dimethano-1,1a,4,4a,5,5a,8,8a-octahydro-anthracene-9,10-dione, $C_{16}H_{16}O_2$

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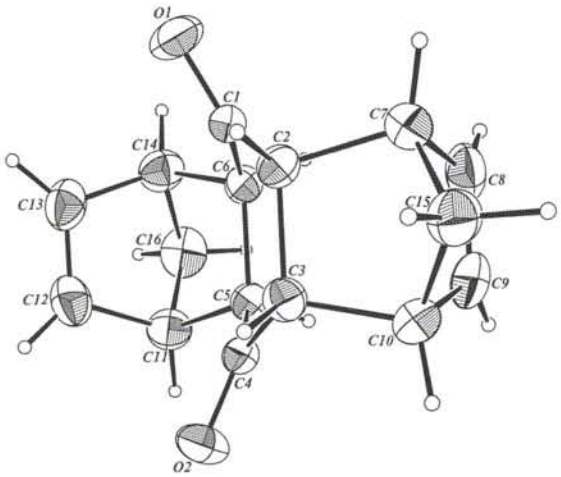

Abstract

$C_{16}H_{16}O_2$, monoclinic, $P12_1/n1$ (No. 14), a = 12.0005(7) Å, b = 6.2388(9) Å, c = 16.973(2) Å, β = 110.609(6)°, V = 1189.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.047, $R_w(F^2)$ = 0.108, T = 293 K.

Source of material

The title compound is a Diels-Alder adduct of a [4+2] cycloaddition of 1,3-cyclopentadiene with *p*-benzoquinone. Cyclopentadiene freshly distilled (40.32 g, 0.6 mol) in methanol (20 ml) was added to a solution of *p*-benzoquinone (32.43 g, 0.3 mol) in methanol (100 ml) at 203 K. The resultant colorless solid was recrystallized from ethanol.

Discussion

The *p*-benzoquinone (PBQ) molecule used to obtain the title compound was found to be planar at room temperature [1], then, at 113 K, the significant deviations from planarity were observed, and the PBQ molecule was in the chair conformation [2]. In contrast to these results, it has been found that the 1,4-cyclohexadione, which is a chemically changed form of PBQ molecule, is not planar and has a boat conformation. The near zero torsion angles of this boat are –0.6(3)° and 0.7(3)°. The dihedral angles between 1,4-cyclohexadione ring and the planes formed by norbornenyl atoms of C2,C3,C7,C10 and C5,C6,C11,C14 are 70.68(9)° and 39.08(7)°, respectively. In the 1,4-cyclohexadione ring, the bond length of O=C is 1.215(2) Å, the C–C distance in the range 1.499(3) Å – 1.562(3) Å. O=C–C, C(=O)–C–C and C–C(=O)–C angles in the range 120.0(2)° – 120.9(2)°, 115.8(2)° –116.6(2)° and 119.1(2)°, respectively. For the norbornenyl atoms, the C–C and C=C distances in the range 1.501(3) Å – 1.579(3) Å and 1.317(3) Å – 1.318(3) Å, respectively. The bond lengths and angles are comparable with corresponding values observed in related molecules [3, 4]. The molecules are linked together by weak C–H···O hydrogen bonds. The bond lengths and angles are comparable with corresponding values observed in related molecules [3, 4].

Table 1. Data collection and handling.

Crystal:	colorless, prismatic, size 0.20 x 0.36 x 0.64 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.87 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω/2θ
$2\theta_{max}$:	52.58°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2424, 2293
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1774
$N(param)_{refined}$:	228
Programs:	MolEN [5], SHELXS-86 [6], SHELXL-93 [7], ORTEP [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	4*e*	1.125(1)	0.596(4)	0.448(1)	0.043(6)
H(3)	4*e*	1.223(1)	0.611(4)	0.362(1)	0.044(6)
H(5)	4*e*	0.987(1)	0.280(3)	0.203(1)	0.036(5)
H(6)	4*e*	0.887(1)	0.267(3)	0.291(1)	0.033(5)
H(7)	4*e*	1.203(2)	0.346(4)	0.559(1)	0.051(6)
H(8)	4*e*	1.193(2)	–0.014(5)	0.493(2)	0.069(8)
H(9)	4*e*	1.307(2)	0.001(5)	0.393(2)	0.075(8)
H(10)	4*e*	1.388(2)	0.377(4)	0.401(1)	0.049(6)
H(11)	4*e*	0.986(2)	0.614(4)	0.144(1)	0.050(6)
H(12)	4*e*	1.075(2)	0.868(4)	0.263(1)	0.056(7)
H(13)	4*e*	0.963(2)	0.860(4)	0.360(1)	0.060(7)
H(14)	4*e*	0.800(2)	0.590(3)	0.304(1)	0.045(6)
H(15A)	4*e*	1.411(2)	0.359(4)	0.553(2)	0.066(8)
H(15B)	4*e*	1.347(2)	0.587(5)	0.516(1)	0.064(8)
H(16A)	4*e*	0.791(2)	0.728(4)	0.159(1)	0.063(7)
H(16B)	4*e*	0.793(2)	0.468(4)	0.157(1)	0.061(7)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*e*	0.9709(1)	0.2632(3)	0.4431(1)	0.063(1)	0.056(1)	0.0544(9)	–0.0062(9)	0.0353(8)	0.0083(8)
O(2)	4*e*	1.2023(1)	0.2869(3)	0.24604(9)	0.060(1)	0.063(1)	0.0520(9)	0.0151(9)	0.0329(8)	–0.0043(8)
C(1)	4*e*	1.0134(2)	0.3560(3)	0.3974(1)	0.046(1)	0.027(1)	0.043(1)	0.0023(9)	0.025(1)	–0.0012(9)
C(2)	4*e*	1.1358(2)	0.4531(3)	0.4320(1)	0.048(1)	0.031(1)	0.037(1)	–0.0003(9)	0.0203(9)	–0.0042(9)
C(3)	4*e*	1.2075(2)	0.4633(3)	0.3712(1)	0.039(1)	0.031(1)	0.044(1)	–0.0013(9)	0.0198(9)	0.0004(9)
C(4)	4*e*	1.1457(2)	0.3720(3)	0.2843(1)	0.048(1)	0.029(1)	0.039(1)	0.0044(9)	0.0245(9)	0.0034(9)
C(5)	4*e*	1.0130(2)	0.3913(3)	0.2448(1)	0.047(1)	0.032(1)	0.034(1)	0.0002(9)	0.0177(9)	–0.0059(9)
C(6)	4*e*	0.9418(2)	0.3813(3)	0.3053(1)	0.039(1)	0.031(1)	0.043(1)	–0.0075(9)	0.0181(9)	–0.0046(9)
C(7)	4*e*	1.2233(2)	0.3314(4)	0.5092(1)	0.060(1)	0.053(1)	0.033(1)	0.002(1)	0.016(1)	–0.002(1)
C(8)	4*e*	1.2339(2)	0.1066(4)	0.4797(1)	0.063(2)	0.043(1)	0.047(1)	0.005(1)	0.011(1)	0.009(1)
C(9)	4*e*	1.2946(2)	0.1166(4)	0.4286(2)	0.060(1)	0.047(1)	0.049(1)	0.014(1)	0.013(1)	0.002(1)
C(10)	4*e*	1.3258(2)	0.3484(4)	0.4222(1)	0.041(1)	0.056(1)	0.052(1)	0.002(1)	0.018(1)	0.000(1)
C(11)	4*e*	0.9690(2)	0.6087(4)	0.1963(1)	0.052(1)	0.048(1)	0.044(1)	0.009(1)	0.024(1)	0.009(1)
C(12)	4*e*	1.0114(2)	0.7886(4)	0.2591(2)	0.050(1)	0.030(1)	0.076(2)	0.004(1)	0.021(1)	0.012(1)
C(13)	4*e*	0.9504(2)	0.7805(4)	0.3102(2)	0.055(1)	0.030(1)	0.060(1)	0.010(1)	0.018(1)	–0.002(1)
C(14)	4*e*	0.8661(2)	0.5943(4)	0.2832(1)	0.039(1)	0.044(1)	0.049(1)	0.006(1)	0.021(1)	0.002(1)
C(15)	4*e*	1.3421(2)	0.4256(5)	0.5113(2)	0.053(1)	0.062(2)	0.051(1)	–0.004(1)	0.005(1)	–0.009(1)
C(16)	4*e*	0.8377(2)	0.5996(5)	0.1879(1)	0.046(1)	0.055(2)	0.046(1)	0.007(1)	0.014(1)	0.004(1)

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