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The 4:2 Diels–Alder Adduct of 1,3-Cyclopentadiene with 1,4-Naphthoquinone

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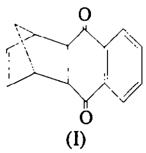
Abstract

The title compound, 1,4-methano-1,4,4a,9a-tetrahydroanthracene-9,10-dione, $C_{15}H_{12}O_2$, is a Diels–Alder adduct resulting from a [4+2] cycloaddition of 1,3-cyclopentadiene with *p*-naphthoquinone.

Comment

The structure analysis of the title compound is part of an ongoing program of obtaining cage compounds by photochemical [2+2] cycloaddition (Maruyama, Terada & Yamamoto, 1981; Kaftory & Weisz, 1984). One aim is to obtain the norbornediene derivatives from the title compound (Diels & Alder, 1929). The present structural analysis has been carried out in order to establish more details of the molecular geometry of the title compound, (I), thus providing a basis of comparison for our further studies.



(I)

The unit cell contains two independent molecules, related by a pseudo-*c* glide perpendicular to the *a* axis, in the asymmetric unit, the average deviation from *c* glide symmetry being 0.02 (2) Å between the two molecules. The C and O atoms of each dihydronaphthoquinone ring are coplanar (Pizzotti, Cenini, Ugo & Demartin, 1991). The r.m.s. deviations of fitted atoms for these planes are 0.025 and 0.057 Å, the maximum deviations from these planes are 0.04 (3) Å for C6 and 0.164 (3) Å for C1*A*. The two five-membered rings adopt envelope conformations with C15 and C15*A* at the flaps. The dihedral angles between the envelope planes through C11–C14 and C11*A*–C14*A*, respectively, and the dihydronaphthoquinone moieties are 55.5 (1) and 49.7 (2)° for each independent molecule. The dihedral angle is 29.8 (2)° between the two envelope planes and 57.7 (1)° between the two dihydronaphthoquinone moieties in the asymmetric unit.

The bond lengths and angles are quite normal and comparable with corresponding values observed in related molecules (Kerr, 1987; Beddoes, Gorman & McNeeney, 1993).

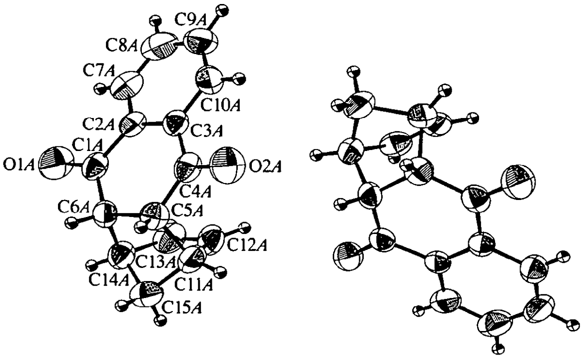


Fig. 1. A perspective view of the molecular structure of the title compound with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

Experimental

p-Naphthoquinone (31.63 g, 0.20 mol) was suspended in benzene (100 ml). A cyclopentadiene (13.88 g, 0.21 mol) solution in cooled benzene (20 ml) was added to the above suspension. After keeping the mixture overnight, the resulting colourless solid was separated and recrystallized from ethanol.

Crystal data

$C_{15}H_{12}O_2$
$M_r = 224.25$
Triclinic
$P\bar{1}$
$a = 9.4077 (12)$ Å
$b = 10.1752 (10)$ Å
$c = 12.5542 (13)$ Å
$\alpha = 71.698 (9)°$
$\beta = 86.980 (9)°$
$\gamma = 77.019 (10)°$
$V = 1111.6 (2)$ Å^3
$Z = 4$
$D_x = 1.340$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–18°$
$\mu = 0.088$ mm^{-1}
$T = 293 (2)$ K
Prismatic
0.64 × 0.44 × 0.24 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scan (Fair, 1990)
$T_{\min} = 0.924$, $T_{\max} = 0.999$
4502 measured reflections
4502 independent reflections
2917 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 26.29°$
$h = 0 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity decay: 2.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.129$
$S = 1.481$
4502 reflections
404 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0237P)^2 + 0.5769P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.182$ e Å^{-3}
$\Delta\rho_{\min} = -0.172$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.008 (1)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

	Molecule 1	Molecule 2†
O1—C1	1.217 (3)	1.218 (3)
O2—C4	1.221 (3)	1.223 (3)
C12—C13	1.319 (4)	1.327 (5)
O1—C1—C2	119.6 (3)	120.3 (3)
O1—C1—C6	120.2 (3)	119.5 (3)
O2—C4—C3	121.0 (3)	119.6 (3)
O2—C4—C5	118.7 (3)	120.0 (3)
C3—C4—C5	120.3 (2)	120.4 (2)
C6—C1—C2	120.2 (2)	120.3 (2)
C14—C15—C11	93.6 (2)	93.1 (3)
C11—C12—C13—C14	0.3 (3)	−0.1 (4)
C13—C12—C11—C15	32.5 (3)	−33.3 (3)
C15—C14—C13—C12	−33.3 (3)	33.6 (3)

† The atomic labels are each appended by *A*.

H atoms were located from difference Fourier maps and refined isotropically. The needle-shaped fragile crystals were difficult to cut so, eventually, a specimen of length 0.64 mm had to be used.

Data collection: *CAD*-4-*PC* (Enraf–Nonius, 1993). Cell refinement: *CAD*-4-*PC*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1411). Services for accessing these data are described at the back of the journal.

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(±)-*tert*-Butyl 3-Hydroxy-4-phenyl-2-(*p*-toluenesulfonylamino)pentanoate–Dichloromethane (1/1): a Pseudo Centre of Symmetry in an Enantiomeric Pair

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Abstract

The title compound, $C_{22}H_{29}NO_5S.CH_2Cl_2$, was investigated in order to study the stereochemistry of the β-hydroxy-α-amino acid derivatives formed by the aldol reaction of an ester enolate with an aldehyde. The racemate crystallizes with two independent formula units in the non-centrosymmetric space group *Pn* as hydrogen-bonded dimers in which the two enantiomers are related by a pseudo centre of symmetry.

Comment

Among non-proteinogenic unnatural α-amino acids, β-hydroxy-α-amino acids are of special interest, especially in view of their activity as enzyme inhibitors (Rando, 1975; Walsh, Metzler, Powell & Jacobsen, 1980; Abeles,