

(11*SR*,15*SR*)-9,10,11,15-Tetrahydro-9-[(1*SR*)-1-methoxyethyl]-9,10-[3',4']-furanoanthracene-12,14-dione**William Clegg* and Ross W. Harrington**School of Natural Sciences (Chemistry),
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Key indicatorsSingle-crystal X-ray study
 $T = 160\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.051
 wR factor = 0.112
Data-to-parameter ratio = 12.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{21}\text{H}_{18}\text{O}_4$, has a molecular structure with a rigid polycyclic nucleus, in which an acid anhydride ring bridges the two CH positions of the central ring of anthracene. The 1-methoxyethyl substituent, by contrast, has conformational freedom and adopts an orientation minimizing steric and electronic repulsions with the anhydride group.

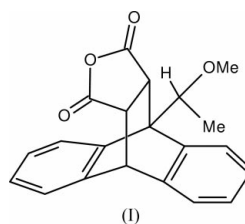
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Comment

The title compound, (I), was prepared as part of a study of diastereoselective Diels–Alder addition reactions of chiral anthracene derivatives substituted at one of the central ring C atoms (Atherton & Jones, 2002). The primary purpose of the structure determination was to confirm the relative stereochemistry, as evidence for the proposed mechanism of the reaction. Both the starting material and the product, (I), of the Diels–Alder reaction are racemic; (I) crystallizes in a centrosymmetric space group.



Detailed examination of the molecular structure of (I) (Fig. 1) shows further points of interest in addition to the qualitative identification required for the mechanistic study. Most of the molecule is relatively rigid, but there is conformational freedom for the 1-methoxyethyl group. Torsion angles for this group are given in Table 1. The antiperiplanar arrangement of the methyl group relative to the ring system and the orientation of the anhydride ring away from the methoxy group are in accord with expected steric and electronic interactions in the starting material and during the course of the reaction, and almost ideal staggering of the substituents around the C10–C19 bond is retained in the solid state, apparently with no significant crystal-packing effects. There are only van der Waals interactions between molecules.

A search of the Cambridge Structural Database (CSD, Version 5.23 of April 2002, and 3 updates; Allen, 2002) reveals 232 structures in which a bridge of two C atoms is added across the opposite CH groups of the central ring of anthracene. Of these, almost one-half are triptycene derivatives, in which the two-atom bridge is part of a third benzene ring. In six structures, the bridge is the C–C bond of an acid anhydride, as in (I) (Bulgarovskaya *et al.*, 1979; Schwartz *et al.*, 1992; Plummer *et al.*, 1997; Weber *et al.*, 1993; Díaz de Dalgado *et al.*, 2002).

The rigidity of the polycyclic core of these molecules is demonstrated by the small range of dihedral angles found between pairs of mean planes defined as follows: plane 1 is the anhydride group; plane 2 is C9/C10/C15/C16 (using the atom numbering for the title compound; see Fig. 1); plane 3 is C9/C10/C13/C14; plane 4 is C9/C10/C11/C12. Dihedral angles for the previously reported anhydride structures (defined as hinge angles, such that 180° corresponds to exact coplanarity, as in anthracene itself) lie in the ranges: $118.8\text{--}122.3^\circ$ for planes 1–2; $117.4\text{--}119.8^\circ$ for planes 2–3 and 2–4; $121.7\text{--}124.6^\circ$ for planes 3–4. The corresponding values for (I) are: 120.12 (9) (1–2), 117.69 (11) (2–3), 116.57 (12) (2–4) and 125.74 (10) (3–4), each of them within or marginally outside the range for the other structures. There is no systematic difference between the dihedral angles 2–3 and 2–4, the C9/C15/C16/C10 bridge lying essentially symmetrically above the anthracene nucleus, despite the orientation of the anhydride group to one side. The dihedral angle 3–4 is increased, and both 2–3 and 2–4 are decreased from the 120° of a symmetrical triptycene structure. The differences are small, but systematic, and are probably a consequence of the longer bonds from C9 and C10 to the anhydride group than to the benzene rings, leading to a slightly lower steric demand by the anhydride group.

In (I), as in the other anhydride structures, and also generally in bridged anthracene derivatives, the two benzene rings are essentially coplanar with planes 3 and 4; the respective dihedral (hinge) angles in (I) are 175.26 (19) and 177.42 (18) $^\circ$.

Experimental

The synthesis has been described by Atherton & Jones (2002).

Crystal data

$C_{21}H_{18}O_4$	$Z = 2$
$M_r = 334.35$	$D_x = 1.340 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.5923$ (8) \AA	Cell parameters from 4879 reflections
$b = 10.1129$ (8) \AA	$\theta = 2.4\text{--}28.3^\circ$
$c = 10.9661$ (9) \AA	$\mu = 0.09 \text{ mm}^{-1}$
$\alpha = 112.886$ (2) $^\circ$	$T = 160$ (2) K
$\beta = 94.278$ (2) $^\circ$	Plate, colourless
$\gamma = 117.285$ (2) $^\circ$	$0.70 \times 0.35 \times 0.08 \text{ mm}$
$V = 828.41$ (12) \AA^3	

Data collection

Bruker SMART 1K CCD diffractometer	2332 reflections with $I > 2\sigma(I)$
ω rotation with narrow frames	$R_{\text{int}} = 0.023$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
5885 measured reflections	$h = -11 \rightarrow 11$
2885 independent reflections	$k = -12 \rightarrow 11$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 0.5015P]$
$R[F^2 > 2\sigma(F^2)] = 0.051$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.112$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.13$	$\Delta\rho_{\text{max}} = 0.28 \text{ e \AA}^{-3}$
2885 reflections	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
229 parameters	Extinction correction: <i>SHELXTL</i>
H-atom parameters constrained	Extinction coefficient: 0.013 (3)

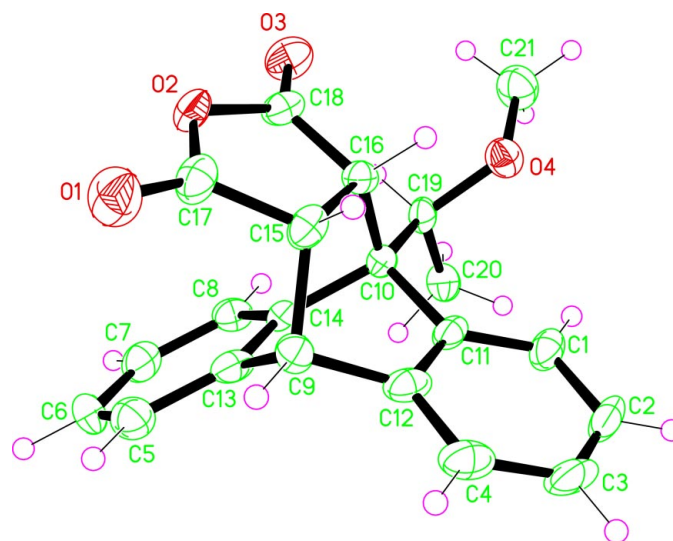


Figure 1
The molecular structure of (I), with atom labels and 50% probability ellipsoids for non-H atoms.

Table 1

Selected torsion angles ($^\circ$).

C11–C10–C19–C20	61.5 (2)	C14–C10–C19–O4	171.45 (16)
C11–C10–C19–O4	–62.2 (2)	C16–C10–C19–C20	178.81 (17)
C14–C10–C19–C20	–64.8 (2)	C16–C10–C19–O4	55.0 (2)

H atoms were placed geometrically and refined with a riding model (including free rotation about C–C bonds), and with U_{iso} constrained to be 1.2 (1.5 for methyl groups) times U_{eq} of the carrier atom.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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