

(3a*SR*,9a*SR*)-3a,4,9,9a-Tetrahydro-4-[(1*SR*)-1-methoxyethyl]-2-methyl-4,9-[1',2']benzeno-1*H*-benzo[*f*]isoindole-1,3-(2*H*)-dione**William Clegg* and Ross W. Harrington**School of Natural Sciences (Chemistry),
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Key indicators

Single-crystal X-ray study

 $T = 160\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$ R factor = 0.048 wR factor = 0.120

Data-to-parameter ratio = 12.8

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{22}\text{H}_{21}\text{NO}_3$, has a molecular structure with a rigid polycyclic nucleus, in which a carboxamide 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 carboxamide group.

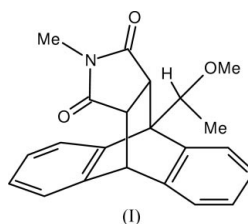
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Comment

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.



The molecular structure (Fig. 1) is essentially the same as that of the corresponding acid anhydride (Clegg & Harrington, 2002), in which an O atom takes the place of the NMe group in the title compound. This similarity extends to the orientation of the conformationally flexible 1-methoxyethyl substituent, which gives almost ideal staggering around the C9–C20 bond (Table 1), confirming that this is a molecular preference and is not affected by the crystal-packing arrangement, which is quite different in the two structures. There are only van der Waals interactions between molecules. The difference in size of the O and NMe groups in the anhydride and carboxamide, respectively, does not allow the two compounds to be isomorphous.

The Cambridge Structural Database (CSD, Version 5.23, April 2002, and 3 updates; Allen, 2002) contains as many as 16 structures with a carboxamide group bridging the 9,10-positions of an anthracene molecule, although these include different solvates or cocrystals of the same compound (Weber *et al.*, 1991, 1994, 1995; Kim, Hubig *et al.*, 2001; Kim, Lindeman & Kochi, 2001; Kishikawa *et al.*, 1997). The rigidity of the polycyclic core of these molecules is demonstrated by the small range of dihedral (hinge) angles found between pairs of mean planes defined as follows: plane 1 is the carboxamide

group; plane 2 is C9/C10/C15/C16 (using the atom numbering for the title compound); plane 3 is C9/C10/C13/C14; plane 4 is C9/C10/C11/C12. Dihedral angles for the previously reported carboxamide structures lie in the ranges: 117.4–120.7° for planes 1–2; 116.3–120.3° for planes 2–3 and 2–4; 122.3–125.7° for planes 3–4. The corresponding values for (I) are: 122.15 (8) (1–2), 119.08 (10) (2–3), 116.79 (9) (2–4) and 124.12 (8)° (3–4), each of them within or marginally outside the range for the other structures. In these and other aspects, the molecular structure of (I) closely resembles that of the corresponding anhydride (Clegg & Harrington, 2002). The benzene rings are essentially coplanar with planes 3 and 4; the respective dihedral angles in (I), demonstrating only very minor hinge folding at the ring fusions, are 177.45 (8) and 177.59 (15)°, with 180° corresponding to exact coplanarity.

Experimental

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

Crystal data

$C_{22}H_{21}NO_3$	$D_x = 1.327 \text{ Mg m}^{-3}$
$M_r = 347.40$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 11489 reflections
$a = 10.7625 (7) \text{ \AA}$	$\theta = 2.2\text{--}28.8^\circ$
$b = 9.6850 (6) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 16.7021 (11) \text{ \AA}$	$T = 160 (2) \text{ K}$
$\beta = 93.067 (2)^\circ$	Block, colourless
$V = 1738.45 (19) \text{ \AA}^3$	$0.70 \times 0.50 \times 0.50 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART 1K CCD diffractometer	2619 reflections with $I > 2\sigma(I)$
ω rotation with narrow frames	$R_{\text{int}} = 0.026$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
12142 measured reflections	$h = -12 \rightarrow 12$
3052 independent reflections	$k = -11 \rightarrow 11$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 1.7203P]$
$R[F^2 > 2\sigma(F^2)] = 0.048$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.120$	$(\Delta/\sigma)_{\text{max}} = 0.007$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
3052 reflections	$\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
238 parameters	
H-atom parameters constrained	

Table 1

Selected torsion angles ($^\circ$).

C11–C9–C20–O3	–62.8 (2)	C14–C9–C20–C21	–64.7 (2)
C11–C9–C20–C21	60.0 (2)	C16–C9–C20–O3	55.30 (19)
C14–C9–C20–O3	172.54 (14)	C16–C9–C20–C21	178.10 (16)

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.

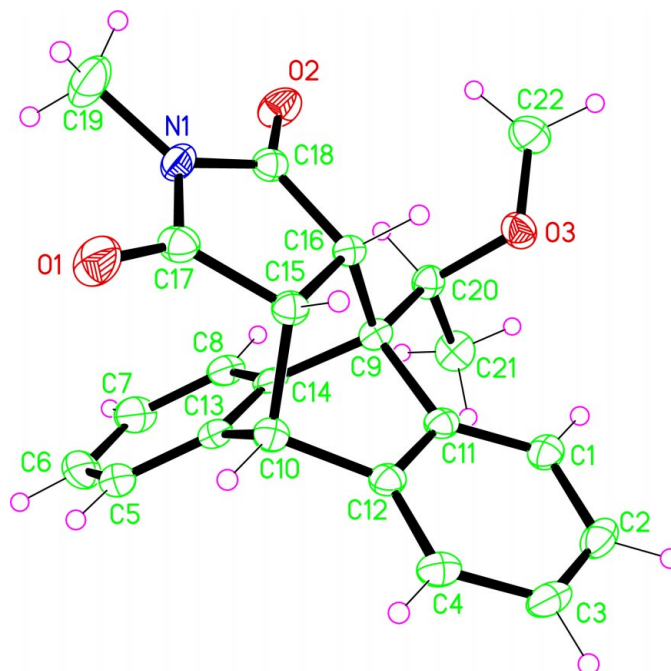


Figure 1

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

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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