

Discussion. The structure of (3) with atom numbering is shown in Fig. 1. Table 1* lists the fractional atomic coordinates of the non-hydrogen atoms and Table 2 the bond lengths and angles.

The tetraester (3) possesses crystallographic *C_i* symmetry. All four ester groups are found to have the *exo* configuration in (3) as already observed (Kohnke *et al.*, 1988a) for the tetraester (1) derived by an analogous synthetic route. In contrast with (1), adjacent pairs of carbonyl functions are directed towards the *exo* face. However, they are not aligned in a parallel manner. The C(1)–C(2)–C(10)–O(10) and C(4)–C(3)–C(13)–O(13) torsion angles are 5.1 (2) and 40.2 (2)°, respectively. The dihedral angles between the C(1)–O(1)–C(4) and the C(1)–C(4)–C(4a)–C(9a) and C(1)–C(2)–C(3)–C(4) mean planes are 128 (1) and 121 (1)°, respectively, and that between the C(1)–C(2)–C(3)–C(4) and the C(1)–C(4)–C(4a)–C(9a) mean planes is 111 (1)°. The C(1)–O(1)–C(4) plane is canted further away from the mean plane of the aromatic ring. This observation is consistent with the increase of the C(2)–C(3) bond length [1.571 (2) Å] in (3) relative to the C(4a)–C(9a) bond [1.397 (2) Å] whereas, in tetraester (1), the situation was the converse. As in the case of the *anti* isomer (2) of 1,4:5,8-diepoxy-1,4,5,8-tetrahydroanthracene (Hart *et al.*, 1983; Kohnke *et al.*, 1988b), there is (Fig. 2) a slight but significant fold (4°) of the C(1)–C(4)–C(4a)–C(9a) plane out of that of the aromatic ring.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44646 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). C44, 742–745

***rel*-(1*R*,4*S*,5*R*,8*S*)-1,4:5,8-Diepoxy-1,4,5,8-tetrahydroanthracene**

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(Received 22 October 1987; accepted 4 January 1988)

Abstract. C₁₄H₁₀O₂, *M_r* = 210.2, monoclinic, *P*2₁/*n*, *a* = 8.046 (1), *b* = 14.573 (3), *c* = 8.718 (1) Å, β = 95.47 (1)°, *V* = 1018 Å³, *Z* = 4, *D_x* = 1.370 Mg m⁻³,

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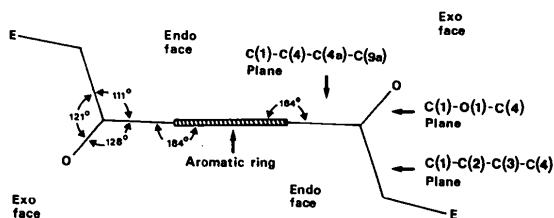


Fig. 2. Diagrammatic representation of the structure of (3) defining space around the molecule, mean planes in the molecule, and showing dihedral angles.

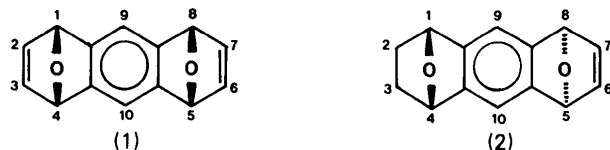
We thank the Science and Engineering Research Council and the University of Messina for financial support.

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Cu Kα radiation, λ = 1.54178 Å, μ = 0.696 mm⁻¹, *F*(000) = 440, room temperature, *R* = 0.040 for 1304 observed reflections with |*F_o*| > 3σ(|*F_o*|). Symmetry-related pairs of molecules of the title compound pack so that an olefinic hydrogen atom in one molecule interacts electrostatically with the π system on the *endo* face of another molecule.

Introduction. In view of the use (Kohnke, Slawin, Stoddart & Williams, 1987) of the *syn* isomer (1) of 1,4:5,8-diepoxy-1,4,5,8-tetrahydroanthracene (Hart, Raju, Meador & Ward, 1983) as a bisdienophile in the synthesis of some novel macropolycyclic systems based on Diels–Alder reactions, we decided to conduct a crystallographic investigation on (1). Here, we report the X-ray structure of (1) and draw comparisons of its molecular geometry and crystal packing with those (Kohnke, Stoddart, Slawin & Williams, 1988) of the *anti* isomer (2).



Experimental. Single crystals of (1), platy rhombs, (110) prominent, suitable for X-ray crystallography were grown at room temperature from chloroform and had m.p. 464–466 K. Crystal size 0.11 × 0.28 × 0.46 mm. Refined unit-cell parameters obtained from setting angles of 18 reflections with $12 \leq \theta \leq 32^\circ$. Nicolet R3m diffractometer. 1371 independent reflections ($\theta \leq 58^\circ$) measured, Cu K α radiation (graphite monochromator), ω scan. 1304 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range $h -8/8, k 0/15, l 0/9$; two check reflections measured every 50 reflections, net count constant; Lorentz and polarization corrections, numerical absorption correction (face indexed crystal) with minimum and maximum transmission factors of 0.738 and 0.932. Structure solved by direct methods; non-hydrogen atoms refined anisotropically; positions of all H atoms calculated (C–H = 0.96 Å), assigned isotropic thermal parameters, $U(H) = 1.2U_{eq}(C)$, and allowed to ride on parent C atoms. An empirical extinction correction was applied [$g = 0.016(3)$]. Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0.040$; $wR = 0.054$ [$w^{-1} = \sigma^2(F) + 0.00020F^2$]. $(\Delta/\sigma)_{max} = 0.03$; residual electron density in difference map within -0.16 and

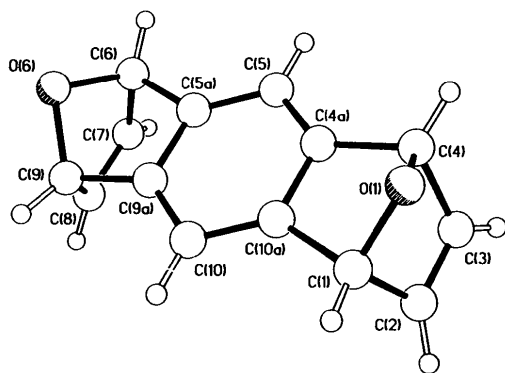


Fig. 1. Ball-and-stick representation of the structure of (1) showing the atomic numbering scheme.

Table 1. Atom coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	6965 (2)	7077 (1)	748 (1)	52 (1)
C(1)	7799 (2)	7878 (1)	1431 (2)	48 (1)
C(2)	8481 (3)	7498 (2)	2987 (3)	57 (1)
C(3)	7385 (3)	6892 (1)	3362 (2)	58 (1)
C(4)	5994 (3)	6902 (1)	2030 (2)	50 (1)
C(4a)	5108 (2)	7828 (1)	2128 (2)	40 (1)
C(5)	3559 (2)	8087 (1)	2531 (2)	44 (1)
C(5a)	3240 (2)	9019 (1)	2501 (2)	43 (1)
C(6)	1743 (3)	9607 (1)	2870 (2)	54 (1)
O(6)	1842 (2)	10334 (1)	1752 (2)	59 (1)
C(7)	2293 (3)	10119 (1)	4360 (2)	56 (1)
C(8)	3395 (3)	10726 (1)	3975 (2)	55 (1)
C(9)	3543 (2)	10585 (1)	2261 (2)	51 (1)
C(9a)	4398 (2)	9660 (1)	2097 (2)	43 (1)
C(10)	5965 (2)	9398 (1)	1706 (2)	43 (1)
C(10a)	6281 (2)	8465 (1)	1733 (2)	40 (1)

Table 2. Bond lengths (Å) and bond angles ($^\circ$)

O(1)–C(1)	1.447 (2)	O(1)–C(4)	1.446 (2)
C(1)–C(2)	1.519 (3)	C(1)–C(10a)	1.534 (3)
C(2)–C(3)	1.311 (3)	C(3)–C(4)	1.534 (3)
C(4)–C(4a)	1.533 (3)	C(4a)–C(5)	1.380 (3)
C(4a)–C(10a)	1.389 (3)	C(5)–C(5a)	1.382 (3)
C(5a)–C(6)	1.536 (3)	C(5a)–C(9a)	1.388 (3)
C(6)–O(6)	1.446 (2)	C(6)–C(7)	1.527 (3)
O(6)–C(9)	1.445 (2)	C(7)–C(8)	1.318 (3)
C(8)–C(9)	1.524 (3)	C(9)–C(9a)	1.527 (3)
C(9a)–C(10)	1.390 (3)	C(10)–C(10a)	1.384 (3)
C(1)–O(1)–C(4)	95.0 (1)	O(1)–C(1)–C(2)	100.8 (1)
O(1)–C(1)–C(10a)	100.1 (1)	C(2)–C(1)–C(10a)	106.0 (2)
C(1)–C(2)–C(3)	105.9 (2)	C(2)–C(3)–C(4)	105.4 (2)
O(1)–C(4)–C(3)	100.3 (2)	O(1)–C(4)–C(4a)	100.1 (1)
C(3)–C(4)–C(4a)	106.0 (1)	C(4)–C(4a)–C(5)	133.7 (2)
C(4)–C(4a)–C(10a)	104.1 (2)	C(5)–C(4a)–C(10a)	122.1 (2)
C(4a)–C(5)–C(5a)	115.8 (2)	C(5)–C(5a)–C(6)	133.8 (2)
C(5)–C(5a)–C(9a)	122.5 (2)	C(6)–C(5a)–C(9a)	103.7 (2)
C(5a)–C(6)–O(6)	100.0 (2)	C(5a)–C(6)–C(7)	106.3 (2)
O(6)–C(6)–C(7)	100.7 (2)	C(6)–O(6)–C(9)	95.2 (1)
C(6)–C(7)–C(8)	105.0 (2)	C(7)–C(8)–C(9)	106.1 (2)
O(6)–C(9)–C(8)	100.1 (2)	O(6)–C(9)–C(9a)	99.8 (1)
C(8)–C(9)–C(9a)	106.8 (2)	C(5a)–C(9a)–C(9)	104.5 (2)
C(5a)–C(9a)–C(10)	121.7 (2)	C(9)–C(9a)–C(10)	133.8 (2)
C(9a)–C(10)–C(10a)	115.7 (2)	C(1)–C(10a)–C(4a)	104.0 (2)
C(1)–C(10a)–C(10)	133.8 (2)	C(4a)–C(10a)–C(10)	122.2 (2)

0.15 e \AA^{-3} ; atomic scattering factors and dispersion corrections from *International Tables for X-ray Crystallography* (1974). Computations carried out on an Eclipse S140 computer using the *SHELXTL* program system (Sheldrick, 1983).

Discussion. The structure of (1) with atomic numbering scheme is shown in Fig. 1. Table 1* lists the fractional atomic coordinates of the nonhydrogen atoms and Table 2 the bond lengths and angles.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44647 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

In contrast with the *anti* isomer (2) which possesses (Kohnke *et al.*, 1988) crystallographic C_i symmetry, the *syn* isomer (1) is not subject to any symmetry constraints. However, it does have pseudo C_{2v} molecular symmetry. Furthermore, all the equivalent bond

lengths are within statistical significance the same as those (Kohnke *et al.*, 1988) for the *anti* isomer (2). The dihedral angles (Fig. 2) between the C(1)—O(1)—C(4) and the C(1)—C(4)—C(4a)—C(10a) and C(1)—C(2)—C(3)—C(4) mean planes are 123 (1) and 126 (1)°, respectively, and that between the C(1)—C(2)—C(3)—C(4) and the C(1)—C(4)—C(4a)—C(10a) mean planes is 111 (1)°. The corresponding values (Fig. 2) for the other oxanorbornadiene unit are 122 (1), 126 (1), and 112 (1)°, respectively. These dihedral angles do not differ significantly from those observed (Kohnke *et al.*, 1988) for the *anti* isomer (2). The fold angles of the aromatic ring plane are 181°, representing in each case (Fig. 2) a slight reduction when compared (Kohnke *et al.*, 1988) with the values of 182° for the *anti* isomer (2).

The striking feature of the structure is the packing of symmetry-related pairs of molecules (Fig. 3) whereby one [C(8)—H(9)] of the C—H bonds in one molecule is directed into the centre of the aromatic ring on the *endo* face of another. The distance of H(8) from the aromatic ring centroid is 2.92 Å and the C(8)—centroid vector is inclined by 88° to the aromatic-ring mean plane. The edge-to-face stacking (Gould, Gray, Taylor & Walkinshaw, 1985; Burley & Petsko, 1985, 1986) involving an attractive electrostatic interaction between a $\delta(+)$ hydrogen atom and a $\delta(-)$ π -electron cloud has been observed recently both intramolecularly (Slawin, Spencer, Stoddart & Williams, 1987; Ashton, Chrystal, Mathias, Parry, Slawin, Spencer, Stoddart & Williams, 1988) and intermolecularly (Alston, Slawin, Stoddart, Williams & Zarzycki, 1987; Moody, Owusu, Slawin, Spencer, Stoddart, Thomas & Williams, 1987; Anelli, Slawin, Stoddart & Williams, 1988) in the context of different supramolecular phenomena. Despite the efficient packing interaction in the case of the *syn* isomer (1), the overall resulting packing density is noticeably lower than that observed for either polymorph (Hart *et al.*, 1983; Kohnke *et al.*, 1988) of the *anti* isomer.

We thank the Science and Engineering Research Council and the University of Messina for financial support.

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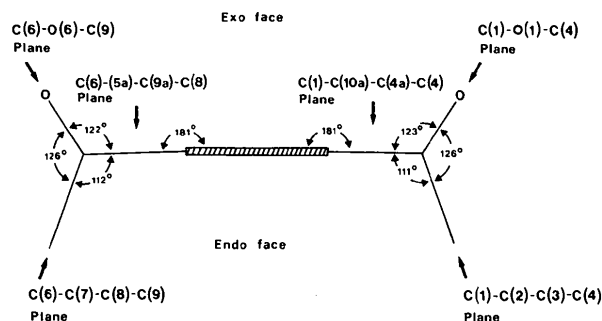


Fig. 2. Diagrammatic representation of the structure of (1) defining space around the molecule, mean planes in the molecule, and showing dihedral angles.

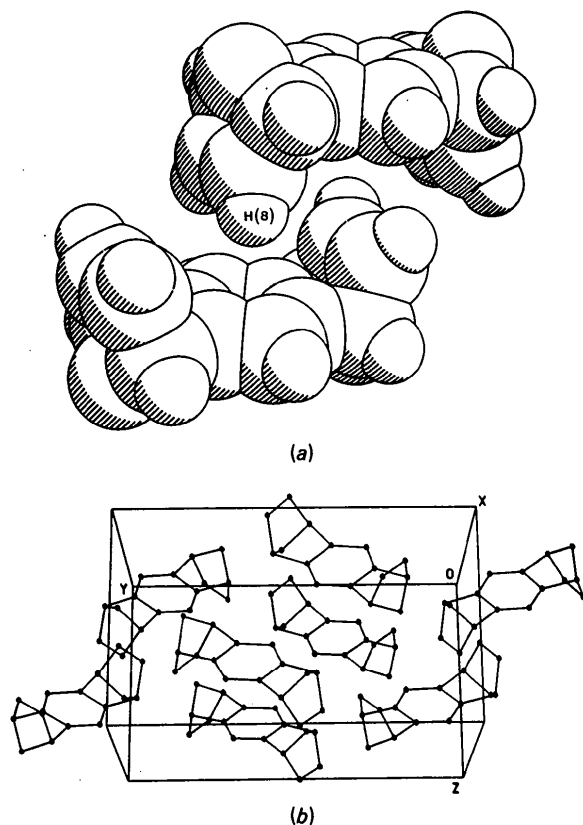


Fig. 3. (a) Space-filling representation of the packing of a centrosymmetrically related pair of molecules of (1). (b) Packing diagram of the title compound (1).

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Acta Cryst. (1988). **C44**, 745–746

Structure of 1-(2-Chloro-4,5-dimethoxyphenyl)-2-(isopropylamino)ethanol

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(Received 28 September 1987; accepted 26 November 1987)

Abstract. $C_{13}H_{20}ClNO_3$, $M_r = 273.8$, monoclinic, $P2_1/a$, $a = 8.389$ (3), $b = 18.39$ (1), $c = 9.395$ (2) Å, $\beta = 94.46$ (3)°, $V = 1445.4$ Å³, $Z = 4$, $D_x = 1.26$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 2.2$ cm⁻¹, $F(000) = 584$, $T = 298$ K, $R = 0.0318$ for 2018 unique reflections. The ethylamine side chain is oriented perpendicular to the plane of the phenyl ring; the isopropyl group is *trans* to the chiral centre of the molecule. The molecules form a one-dimensional chain linked by O—H...N hydrogen bonds.

Introduction. The title compound is one of a series of structurally related β -adrenoceptor blocking drugs, studied by molecular-mechanics calculations in this laboratory (Squier, Van der Schyf, Venter, Oliver, Van Rooyen & Dillen, 1986). Synthesis and pharmacological results are reported in the literature (Squier, Van der Schyf, Oliver & Venter, 1986).

Experimental. Colourless regular shaped crystal, ca $0.3 \times 0.3 \times 0.4$ mm used for data collection. Intensities measured at 298 K; Enraf–Nonius CAD-4 diffractometer; graphite-monochromated Mo $K\alpha$ radiation; ω scan; variable scan speed with a maximum of $5.49^\circ \text{ min}^{-1}$, and a minimum corresponding to a measuring time of 50 s per reflection; ω -scan angle changed as $(0.63 + 0.34 \tan\theta)^\circ$, horizontal aperture as $(1.17 + 0.77 \tan\theta)$ mm, limited to 1.3–5.0 mm; vertical slit fixed to 4 mm. Unit-cell parameters determined with 25 reflections in the θ range 7–19°; total exposure time 18.3 h; intensity control every hour using three standard reflections ($2\bar{5}1$, $1\bar{5}2$, $0\bar{5}3$), no significant loss in intensity; orientation control every 200 reflections, allowed deviation 0.1°. Monoclinic data set collected up to $(\sin\theta)/\lambda = 0.595$ Å⁻¹ ($h = 0 \rightarrow 9$, $k =$

$0 \rightarrow 21$, $l = -11 \rightarrow 11$) resulting in 2650 reflections of which 311 were unobserved [$I < \sigma(I)$]. Data corrected for Lorentz and polarization effects; ψ -scan empirical absorption correction (transmission 94.29 to 99.27%).

The structure was solved by direct methods (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), all H atoms were located from subsequent Fourier maps. Non-H atoms were refined anisotropically, H atoms with a common isotropic temperature factor $U = 0.079$ (2) Å²; *SHELX76* (Sheldrick, 1976); full-matrix method with $1/\sigma^2(F)$ weights; 2018 unique reflections used for 224 refined parameters; $R_{\text{int}} = 0.0195$, $(\Delta/\sigma)_{\text{max}} = 0.59$; $(\Delta\rho)_{\text{max}} = 0.36$, $(\Delta\rho)_{\text{min}} = -0.33$ e Å⁻³; $R = 0.0318$, $wR = 0.0478$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Table 1* lists final refined positional parameters with equivalent isotropic temperature factors. The coordinates refer to the (*R*)-enantiomer of the compound, hereafter referred to as (1). Selected bond lengths and torsion angles are given in Table 2. A view of the molecule is given in Fig. 1.

The phenyl ring is planar; the r.m.s. deviation of the C atoms is 0.002 Å. The methoxy substituents are coplanar with the ring. The ethylamine side chain adopts a perpendicular-*trans* conformation. This is the conformation which occurs most frequently in crystalline phenylethanolamine derivatives (Murray-Rust,

* Lists of structure factors, anisotropic thermal parameters, bond lengths and valence angles, and H-atom data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44576 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.