or less a similar probability from the same and opposite faces of (2), leading to the formation of (3) and (4), respectively, in approximately equal quantities. The absence of C_{2h} symmetry and the essentially orthogonal relationship of the C—Cl bonds in the vicinal C(2)—C(3) positions is probably a consequence of otherwise unfavourable steric interactions. The C(2)—C(3)—C(12)—Cl(2) and the C(3)—C(2)—C(11)—Cl(1) torsion angles are 172·0 (2) and 75·6 (2)°, respectively. The dihedral angles (Fig. 2) between C(1)—O(1)—C(4) and the C(1)—C(1*a*)—C(4*a*)—C(4) and the C(1)—C(2)—C(3)—C(4) mean planes are 127 (1) and 120 (1)°, respectively and that between the C(1)—C(2)—C(3)—C(4) and the C(1)—C(1*a*)—C(4*a*)—C(4) mean planes is 113 (1)°. The C(1)—O(1)—C(4) plane is canted further away from the mean plane of the aromatic ring by an extra 3°. These molecular deformations are virtually identical to those observed (Kohnke, Stoddart, Slawin & Williams, 1988) in methyl *rel*-(1*R*,2*R*,3*S*,4*S*,5*S*,6*S*,7*R*,8*R*)-1,4:5,8-diepoxy-1,2,3,4,5,6,7,8-octahydroanthracene-2,3,6,7-tetracarboxylate.

The packing is normal van der Waals with no short intermolecular contacts, the molecules interleaving with each other in the crystallographic **a** direction (Fig. 3).

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rel-(1*R*,4*S*,5*R*,8*S*)-1,4:5,8-Diepoxy-1,4,5,8-tetrahydro-2,3,6,7-tetramethyleneanthracene

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Abstract. $C_{18}H_{14}O_2$, $M_r = 262·3$, monoclinic, $P2_1/n$, $a = 10·567$ (2), $b = 8·149$ (2), $c = 16·128$ (3) Å, $\beta = 104·33$ (2)°, $V = 1346$ Å^3, $Z = 4$, $D_x = 1·29$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1·54178$ Å, $\mu = 0·63$ mm^{-1}, $F(000) = 552$, room temperature, $R = 0·053$ for 1632 observed reflections with $|F_o| > 3\sigma(|F_o|)$. Symmetry-related pairs of molecules of the title compound (5), prepared stereospecifically from the *syn* tetrachloride, pack so that an olefinic H atom in one molecule interacts electrostatically with the π system on the *endo* face of another molecule.

Introduction. The bisdiene (5) with the *syn* configuration is a key compound in the synthesis of polyacene and cyclacene derivatives using repetitive Diels–Alder reactions (Thomas & Miller, 1986; Chiba, Kenny & Miller, 1987). We have now obtained this compound as a pure diastereoisomer (5) stereospecifically from treatment of the *syn* tetrachloride (3) (Kohnke, Mathias, Stoddart, Slawin & Williams,

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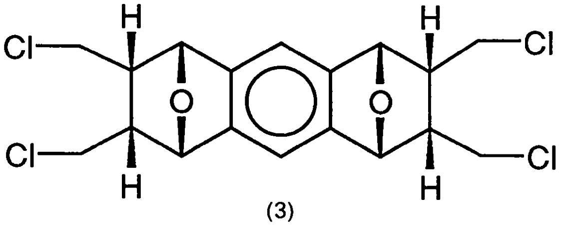
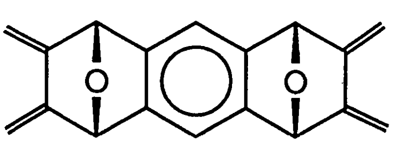
1990) with base, following an established procedure (Mahaim, Carrupt, Hagenbuch, Florey & Vogel, 1980). Here, we report a full structural analysis of (5), which not only confirms its *syn* stereochemistry, but also provides valuable information concerning the molecular geometry associated with its diene units.



(3)



(5)

Experimental. Treatment of the *syn* tetrachloride (3) at room temperature with 8 mol equivs of potassium *tert*-butoxide in tetrahydrofuran afforded the *syn* tetraene (5) in quantitative yield. Single crystals (found: C, 82·6; H, 5·22%; $C_{18}H_{14}O_2$ requires C, 82·4; H, 5·38%) of (5), suitable for X-ray crystallography, were grown by vapour diffusion of pentane into a dichloromethane solution and had m.p. > 501 K with decomposition. Crystal size 0·50 × 0·23 × 0·27 mm. Refined unit-cell parameters obtained from setting angles of 18 reflections with $9 \le \theta \le 29°$. Nicolet *R3m* diffractometer. 1817 independent reflections ($\theta \le 58°$) measured, Cu $K\alpha$ radiation (graphite monochromator), ω scan. 1632 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range h −11/11, k 0/8, l 0/17; two check reflections measured every 50 reflections, no significant change in net counts; Lorentz and polarization corrections, no absorption correction. Structure solved by direct methods; non-H atoms refined anisotropically; positions of H atoms on C(1), C(4), C(5), and C(8) located from ΔF map and refined isotropically subject to a C—H distance constraint. Positions of remaining H atoms calculated (C—H = 0·96 Å), assigned isotropic thermal parameters, $U(\text{H}) = 1{\cdot}2U_{\text{eq}}(\text{C})$, and allowed to ride on parent C atoms. The exocyclic methylene groups refined as rigid bodies. An empirical extinction correction was applied [$g = 0{\cdot}011\,(2)$]. Refinement using F magnitudes by block-cascade full-matrix least squares; $R = 0{\cdot}053$; $wR = 0{\cdot}061$ [$w^{-1} = \sigma^2(F) + 0{\cdot}00091F^2$]; $S = 1{\cdot}86$. $(\Delta/\sigma)_{\max} = 0{\cdot}23$; residual electron density in difference map within −0·20 and 0·21 e Å^{-3}; atomic scattering factors and dispersion corrections from *International Tables for X-ray Crystallography* (1974). Computations carried out on an Eclipse S140 computer using *SHELXTL* program system (Sheldrick, 1983).

Discussion. The structure of (5) with the atomic numbering scheme is shown in Fig. 1. Table 1* lists the fractional atomic coordinates of the non-H atoms and Table 2 the bond lengths and angles.

The molecule has the *syn* configuration (5) with non-crystallographic C_{2v} symmetry. As expected, there is a significant shortening of the C(2)—C(3) and C(6)—C(7) bonds to 1·483 (3) and 1·473 (3) Å, respectively, *cf.* 1·568 (4) Å for C(2)—C(3) in the *anti* tetrachloride (4) (Kohnke *et al.*, 1990). The lengths of the exocyclic double bonds range from 1·314 (3) Å for C(3)—C(12) to 1·319 (3) Å for C(2)—C(11). The dihedral angles (Fig. 2) between the C(1)—O(1)—C(4) and the C(1)—C(4)—C(4*a*)—C(9*a*) and the C(1)—C(2)—C(3)—C(4) mean planes are 125 (1) and 123 (1)° respectively and that between the C(1)—C(2)—C(3)—C(4) and C(1)—C(4)—C(4*a*)—C(9*a*) mean planes is 112 (1)°. The equivalent dihedral angles with respect to the planes subtending the C(5)—O(5)—C(8) plane are 124 (1), 123 (1) and 113 (1)°, respectively. These dihedral angles are similar to those observed (Kohnke, Stoddart, Slawin & Williams, 1988*a*,*b*) in other 1,4:5,8-diepoxy-1,4,5,8-tetrahydroanthracenes. There are small (1·3 and 2·5°), but probably not significant, tilts of the planes incorporating each diene unit in an *endo* direction. A similar small distortion has also been reported (Pinkerton, Schwarzenbach, Stibbard, Carrupt & Vogel, 1983) in a 5,6-bis(methylene)-7-oxonorborn-2-ene derivative.

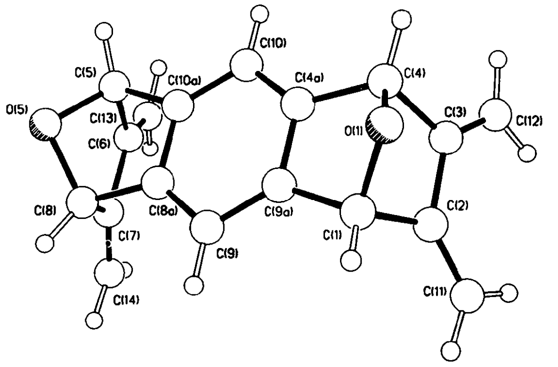


Fig. 1. Ball-and-stick representation of the structure of (5) showing the atomic numbering scheme.

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* 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 52552 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* (× 10^4) *and temperature factors* ($Å^2 \times 10^3$) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	3529 (2)	7545 (3)	8565 (1)	44 (1)
O(1)	4321 (1)	8979 (2)	8879 (1)	50 (1)
C(2)	2226 (2)	8366 (3)	8142 (1)	41 (1)
C(3)	2189 (2)	9781 (3)	8715 (1)	41 (1)
C(4)	3481 (2)	9627 (3)	9392 (1)	45 (1)
C(4*a*)	3344 (2)	8147 (3)	9929 (1)	41 (1)
C(5)	2854 (2)	5634 (3)	11839 (1)	46 (1)
O(5)	3590 (1)	4113 (2)	11872 (1)	55 (1)
C(6)	1459 (2)	4957 (3)	11649 (1)	48 (1)
C(7)	1507 (2)	3541 (3)	11091 (1)	49 (1)
C(8)	2917 (2)	3539 (3)	11016 (1)	48 (1)
C(8*a*)	3087 (2)	5020 (3)	10484 (1)	42 (1)
C(9)	3232 (2)	5204 (3)	9654 (1)	41 (1)
C(9*a*)	3371 (2)	6797 (3)	9399 (1)	40 (1)
C(10)	3155 (2)	7960 (3)	10748 (1)	43 (1)
C(10*a*)	3052 (2)	6368 (3)	11015 (1)	41 (1)
C(11)	1368 (2)	7926 (3)	7435 (1)	56 (1)
C(12)	1281 (2)	10902 (3)	8669 (1)	53 (1)
C(13)	505 (3)	5491 (3)	11970 (2)	64 (1)
C(14)	577 (3)	2500 (4)	10738 (2)	76 (1)

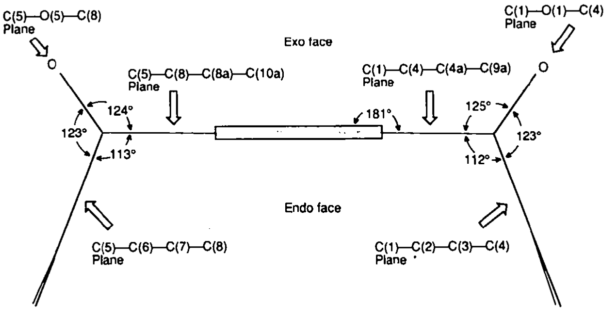

Fig. 2. Diagrammatic representation of the structure of (5) defining space around the molecule, mean planes in the molecule, and showing dihedral angles.

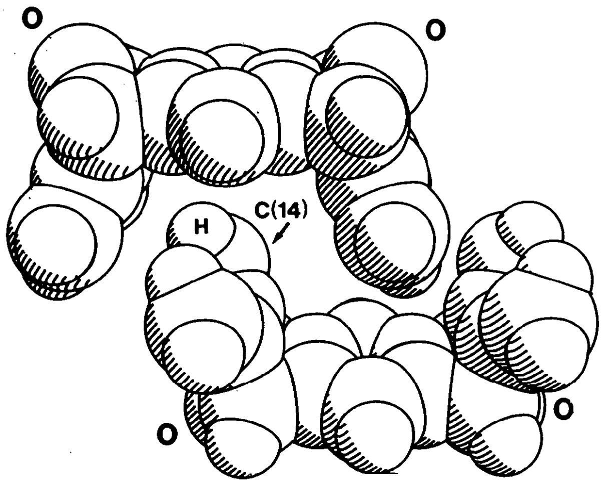

Fig. 3. Space-filling representation of the packing of a centro-symmetrically related pair of molecules (5).

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—O(1)	1·454 (3)	C(1)—C(2)	1·530 (3)
C(1)—C(9*a*)	1·522 (3)	O(1)—C(4)	1·454 (3)
C(2)—C(3)	1·483 (3)	C(2)—C(11)	1·319 (3)
C(3)—C(4)	1·527 (3)	C(3)—C(12)	1·314 (3)
C(4)—C(4*a*)	1·513 (3)	C(4*a*)—C(9*a*)	1·398 (3)
C(4*a*)—C(10)	1·392 (3)	C(5)—O(5)	1·457 (3)
C(5)—C(6)	1·532 (3)	C(5)—C(10*a*)	1·519 (3)
O(5)—C(8)	1·464 (2)	C(6)—C(7)	1·473 (3)
C(6)—C(13)	1·315 (4)	C(7)—C(8)	1·525 (3)
C(7)—C(14)	1·316 (4)	C(8)—C(8*a*)	1·517 (3)
C(8*a*)—C(9)	1·392 (3)	C(8*a*)—C(10*a*)	1·399 (3)
C(9)—C(9*a*)	1·381 (3)	C(10)—C(10*a*)	1·380 (3)

O(1)—C(1)—C(2)	100·4 (2)	O(1)—C(1)—C(9*a*)	101·1 (2)
C(2)—C(1)—C(9*a*)	106·6 (2)	C(1)—O(1)—C(4)	96·3 (1)
C(1)—C(2)—C(3)	102·8 (1)	C(1)—C(2)—C(11)	128·0 (2)
C(3)—C(2)—C(11)	129·1 (2)	C(2)—C(3)—C(4)	103·0 (2)
C(2)—C(3)—C(12)	129·5 (2)	C(4)—C(3)—C(12)	127·5 (2)
O(1)—C(4)—C(3)	100·8 (2)	O(1)—C(4)—C(4*a*)	101·2 (2)
C(3)—C(4)—C(4*a*)	106·2 (2)	C(4)—C(4*a*)—C(9*a*)	104·9 (2)
C(4)—C(4*a*)—C(10)	133·4 (2)	C(9*a*)—C(4*a*)—C(10)	121·6 (2)
O(5)—C(5)—C(6)	100·4 (2)	O(5)—C(5)—C(10*a*)	100·3 (2)
C(6)—C(5)—C(10*a*)	107·5 (2)	C(5)—O(5)—C(8)	96·1 (1)
C(5)—C(6)—C(7)	103·1 (2)	C(5)—C(6)—C(13)	126·5 (2)
C(7)—C(6)—C(13)	130·2 (2)	C(6)—C(7)—C(8)	103·3 (2)
C(6)—C(7)—C(14)	129·4 (2)	C(8)—C(7)—C(14)	127·3 (2)
O(5)—C(8)—C(7)	100·5 (2)	O(5)—C(8)—C(8*a*)	100·5 (2)
C(7)—C(8)—C(8*a*)	107·4 (2)	C(8)—C(8*a*)—C(9)	133·3 (2)
C(8)—C(8*a*)—C(10*a*)	104·7 (2)	C(9)—C(8*a*)—C(10*a*)	122·0 (2)
C(8*a*)—C(9)—C(9*a*)	115·7 (2)	C(1)—C(9*a*)—C(4*a*)	104·4 (2)
C(1)—C(9*a*)—C(9)	133·1 (2)	C(4*a*)—C(9*a*)—C(9)	122·5 (2)
C(4*a*)—C(10)—C(10*a*)	116·1 (2)	C(5)—C(10*a*)—C(8*a*)	104·9 (2)
C(5)—C(10*a*)—C(10)	133·0 (2)	C(8*a*)—C(10*a*)—C(10)	122·1 (2)

The striking feature of the structure is the packing of pairs of symmetry-related molecules (Fig. 3) whereby one of the C(14)—H bonds is directed into the centre of the aromatic ring on the *endo* face of another. The distance of this olefinic H atom from the aromatic ring centre is 3·20 Å and the C(14)–centroid vector is inclined by 84° to the aromatic ring mean plane.

Edge-to-face stacking (Gould, Gray, Taylor & Walkinshaw, 1985; Burley & Petsko, 1985, 1986), involving an attractive electrostatic interaction between a δ(+) H atom and a δ(−) π-electron cloud, has also been observed (Kohnke *et al.*, 1988*b*) in the *syn* isomer of 1,4:5,8-diepoxy-1,4,5,8-tetrahydroanthracene.

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rel-(1*R*,4*S*,5*S*,8*R*)-1,4:5,8-Diepoxy-1,4,5,8-tetrahydro-2,3,6,7-tetramethyleneanthracene

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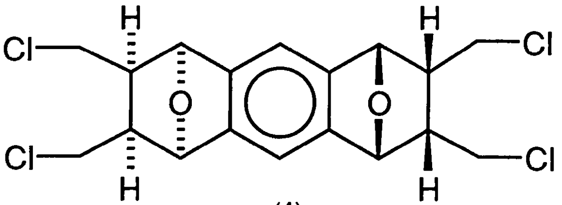
Abstract. $C_{18}H_{14}O_2$, $M_r = 262{\cdot}3$, monoclinic, $P2_1/n$, $a = 10{\cdot}442\,(5)$, $b = 5{\cdot}691\,(2)$, $c = 11{\cdot}467\,(4)$ Å, $\beta = 96{\cdot}69\,(3)^\circ$, $V = 677$ Å^3, $Z = 2$ (the molecule possesses a centre of symmetry), $D_x = 1{\cdot}29$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}54178$ Å, $\mu = 0{\cdot}62$ mm^{-1}, $F(000) = 276$, room temperature, $R = 0{\cdot}046$ for 857 observed reflections with $|F_o| > 3\sigma(|F_o|)$. Lattice-translated molecules of the title compound (6), prepared stereospecifically from the *anti* tetrachloride, are involved in weak diene–diene interactions linking molecules in a chain along the crystallographic **c** direction.

Introduction. The bisdiene (6) with the *anti* configuration is a key compound in the synthesis of polyacene derivatives with ladder-like structures using repetitive Diels–Alder reactions (Thomas & Miller, 1986; Chiba, Kenny & Miller, 1987). We have now obtained this compound as a pure diastereoisomer (6) stereospecifically from treatment of the *anti* tetrachloride (4) (Kohnke, Mathias, Stoddart, Slawin & Williams, 1990), following an established procedure (Mahaim, Carrupt, Hagenbuch, Florey & Vogel, 1980) which we had previously employed successfully in the *syn* series (Kohnke, Mathias, Stoddart, Slawin, Watts & Williams, 1990). Here, we report a full structural analysis of (6), which not only confirms its *anti* stereochemistry, but also provides valuable information concerning the molecular geometry associated with its diene units.



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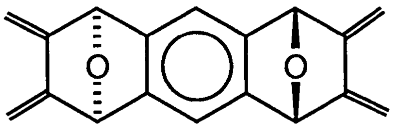

(6)

Experimental. Treatment of the *anti* tetrachloride (4) at room temperature with 8 mol equivs of potassium *tert* butoxide in tetrahydrofuran afforded the *anti* tetraene (6) in quantitative yield. Single crystals (found: C, 82·4; H, 5·58%; $C_{18}H_{14}O_2$ requires C, 82·4; H, 5·38%) of (6), suitable for X-ray crystallography, were grown by vapour diffusion of pentane into a dichloromethane solution and had m.p. 513 K. Crystal size 0·47 × 0·33 × 0·27 mm. Refined unit-cell

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