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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

By Franz H. Kohnke,* John P. Mathias and J. Fraser Stoddart

Department of Chemistry, The University, Sheffield S3 7HF, England

and Alexandra M. Z. Slawin, David J. Watts and David J. Williams

Department of Chemistry, Imperial College, London SW7 2AY, England

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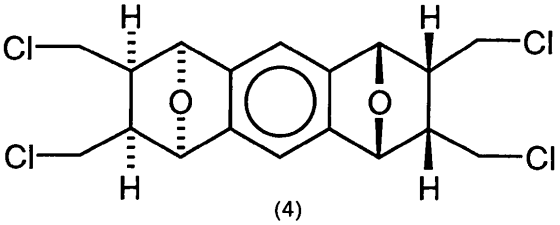
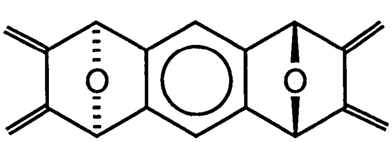
Abstract. $C_{18}H_{14}O_2$, $M_r = 262{\cdot}3$, monoclinic, $P2_1/n$, $a = 10{\cdot}442\,(5)$, $b = 5{\cdot}691\,(2)$, $c = 11{\cdot}467\,(4)$ Å, $\beta = 96{\cdot}69\,(3)^\circ$, $V = 677$ Å^3, $Z = 2$ (the molecule possesses a centre of symmetry), $D_x = 1{\cdot}29$ Mg m^{-3}, Cu $K\alpha$ radiation, $\lambda = 1{\cdot}54178$ 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.


(4)


(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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* Permanent address: Dipartimento di Chimica Organica e Biologica dell' Università di Messina, Contrada Papardo, Salita Sperone, 98100 Messina, Italy.

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parameters obtained from setting angles of 23 reflections with $11 \leq \theta \leq 29°$. Nicolet *R*3*m* diffractometer. 906 independent reflections ($\theta \leq 58°$) measured, Cu *Kα* radiation (graphite monochromator), *ω* scan, 857 [$|F_o| > 3\sigma(|F_o|)$] considered observed, index range *h* −11/11, *k* 0/6, *l* 0/12; 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) and C(4) 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, $U(\mathrm{H}) = 1{\cdot}2U_{\mathrm{eq}}(\mathrm{C})$, allowed to ride on parent C atoms. The exocyclic methylene groups refined as rigid bodies. An empirical extinction correction was applied [$g = 0{\cdot}024\,(7)$]. Refinement using *F* magnitudes by block-cascade full-matrix least squares; $R = 0{\cdot}046$; $wR = 0{\cdot}056$ [$w^{-1} = \sigma^2(F) + 0{\cdot}00120F^2$]; $S = 1{\cdot}91$; $(\Delta/\sigma)_{\max} = 0{\cdot}049$; residual electron density in difference map within −0·17 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 (6) 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.

Table 1. *Atomic coordinates* (× 10^4) *and temperature factors* (Å^2 × 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)	5199 (2)	3275 (3)	1904 (2)	41 (1)
O(1)	3857 (1)	3069 (2)	2113 (1)	52 (1)
C(2)	5850 (2)	1868 (3)	2937 (1)	42 (1)
C(3)	4914 (2)	−56 (3)	3059 (1)	45 (1)
C(4)	3822 (1)	504 (3)	2109 (1)	46 (1)
C(4*a*)	4317 (1)	−14 (3)	944 (1)	38 (1)
C(9)	5907 (2)	1835 (3)	−145 (1)	40 (1)
C(9*a*)	5202 (1)	1771 (2)	806 (1)	37 (1)
C(11)	4985 (2)	−1845 (3)	3797 (2)	64 (1)
C(12)	6953 (2)	2373 (4)	3571 (2)	58 (1)

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

C(1)—O(1)	1·453 (2)	C(1)—C(2)	1·523 (2)
C(1)—C(9*a*)	1·523 (2)	O(1)—C(4)	1·460 (2)
C(2)—C(3)	1·485 (2)	C(2)—C(12)	1·320 (2)
C(3)—C(4)	1·516 (2)	C(3)—C(11)	1·320 (2)
C(4)—C(4*a*)	1·517 (2)	C(4*a*)—C(9*a*)	1·395 (2)
C(4*a*)—C(9*a*)	1·385 (2)	C(9)—C(9*a*)	1·385 (2)
C(9)—C(4*a*′)*	1·385 (2)		

O(1)—C(1)—C(2)	100·5 (1)	O(1)—C(1)—C(9*a*)	100·7 (1)
C(2)—C(1)—C(9*a*)	107·7 (1)	C(1)—O(1)—C(4)	96·0 (1)
C(1)—C(2)—C(3)	102·8 (1)	C(1)—C(2)—C(12)	126·5 (2)
C(3)—C(2)—C(12)	130·6 (2)	C(2)—C(3)—C(4)	103·0 (1)
C(2)—C(3)—C(11)	129·8 (2)	C(4)—C(3)—C(11)	127·2 (2)
O(1)—C(4)—C(3)	101·0 (1)	O(1)—C(4)—C(4*a*)	100·7 (1)
C(3)—C(4)—C(4*a*)	106·7 (1)	C(4)—C(4*a*)—C(9*a*)	104·9 (1)
C(4)—C(4*a*)—C(9*a*)	132·8 (1)	C(9*a*)—C(4*a*)—C(9*a*)	122·2 (1)
C(9*a*)—C(9)—C(4*a*′)*	115·9 (1)	C(1)—C(9*a*)—C(4*a*)	104·5 (1)
C(1)—C(9*a*)—C(9)	133·5 (1)	C(4*a*)—C(9*a*)—C(9)	121·9 (1)

* Indicates the operation 1 − *x*, −*y*, −*z*.

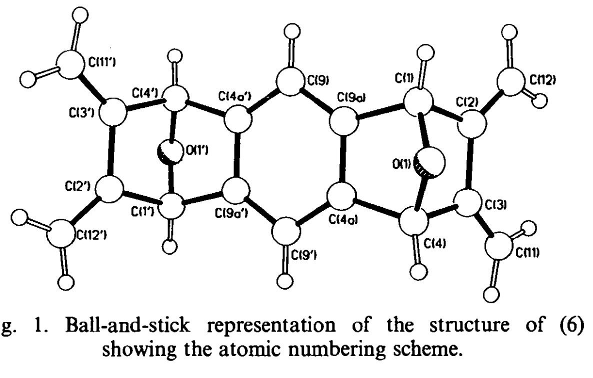

Fig. 1. Ball-and-stick representation of the structure of (6) showing the atomic numbering scheme.

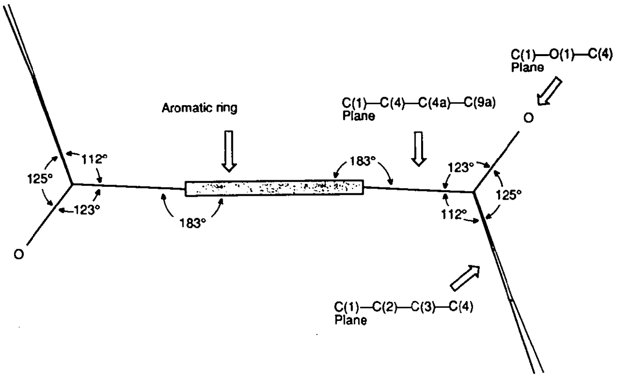

Fig. 2. Diagrammatic representation of the structure of (6) defining space around the molecule, mean planes in the molecule, and showing dihedral angles.

In addition to possessing crystallographic C_i symmetry, the compound (6) also exhibits C_{2h} molecular symmetry, consistent with it having the *anti* configuration. As in the case of the *syn*-1,4:5,8-diepoxy-1,4,5,8-tetrahydro-2,3,6,7-tetramethyleneanthracene (Kohnke, Mathias, Stoddart, Slawin, Watts & Williams, 1990), there is a significant shortening of the C(2)—C(3) bond to 1·485 (2) Å. The lengths of the conjugated exocyclic double bonds are 1·320 (2) Å for both C(2)—C(12) and C(3)—C(11). The dihedral angles (Fig. 2) between the C(1)—O(1)—C(4) and the C(1)—C(2)—C(3)—C(4) and C(1)—C(4)—C(4*a*)—C(9*a*)

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* List 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 52553 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

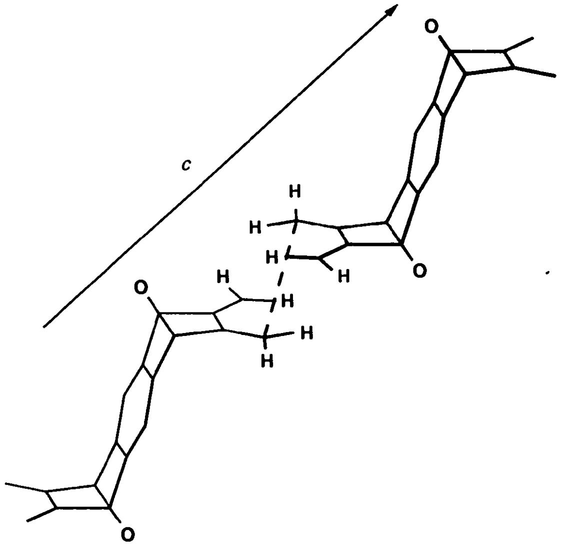

Fig. 3. Line drawing of a pair of lattice-translated molecules (6).

mean planes are 125 and 123° 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°. The C(1)—O(1)—C(4) plane is canted further away from the mean plane of the aromatic ring by a further 3°. These dihedral angles are similar to those observed in the *syn* isomer (Kohnke, Mathias, Stoddart, Slawin, Watts & Williams, 1990) and in other 1,4:-5,8-diepoxy-1,4,5,8-tetrahydroanthracenes (Kohnke, Stoddart, Slawin & Williams, 1988*a*,*b*). The tilt of the diene units towards the *endo* faces is only a modest 2° and is probably not significant (Pinkerton, Schwarzenbach, Stibbard, Carrupt & Vogel, 1983).

The only notable intermolecular interaction (Fig. 3) is a chain of weak diene–diene interactions between lattice-translated molecules in the crystallographic **c** direction. The interplanar separation is 3·00 Å with the shortest carbon–carbon distance being 3·48 Å between C(11) and C(11′).

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Structure of Ethyl 1-Cyanomethyl-5-methoxy-3-phenylindole-2-carboxylate

By T. N. Chandrakantha, K. A. Nirmala and Puttaraja

Department of Physics, Bangalore University, Bangalore-560 056, *India*

and M. Nethaji*

Department of Crystallography and Biophysics, Madras University, Madras-600 025, *India*

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Abstract. $C_{20}H_{18}N_2O_3$, $M_r = 334{\cdot}38$, orthorhombic, $Pbca$, $a = 7{\cdot}179\,(3)$, $b = 33{\cdot}014\,(3)$, $c = 14{\cdot}847\,(1)$ Å, $V = 3518{\cdot}85$ Å^3, $Z = 8$, $D_x = 1{\cdot}262$, $D_m = 1{\cdot}263$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 0{\cdot}62$ mm^{-1}, $F(000) = 1408$, $T = 300$ K. Final $R = 0{\cdot}041$ for 1713 observed reflections. The indole ring system is planar with the methoxy group coplanar with the rings. The phenyl ring connected to the indole moiety is rotated about the C(3)—C(10) bond

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* Present address: Department of Inorganic and Physical Chemistry, Indian Institute of Science, Bangalore-560 012, India.

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