from the least-squares plane for this ring is 0·0137 (4) Å by the C(3′) atom. If the concept of pseudorotation is applied to the unsaturated pentafuranose ring, compound (2) exhibits a slight twist pucker ($\tau_m = 2·6°$, $P = -143·7°$). Conformations about the C(4′)—C(5′) bonds are *trans–gauche* and *gauche–trans* for (1) and (2) respectively.

Packing interactions are listed in Table 4 and illustrated in Fig. 2. The only close intermolecular contacts are those resulting from hydrogen bonding. In (1), a water of hydration sits on the twofold axis, accepting and donating H bonds to two symmetry-related pairs of molecules.

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Structure of Dimethyl Dibenzobarrelene-11,12-dicarboxylate

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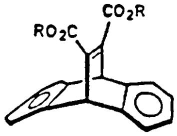
Abstract. Dimethyl 9,10-dihydro-9,10-ethenoanthracene-11,12-dicarboxylate, $C_{20}H_{16}O_4$, $M_r = 320·35$, triclinic, $P\bar{1}$, $a = 8·699\,(2)$, $b = 12·992\,(3)$, $c = 8·190\,(2)$ Å, $\alpha = 100·78\,(1)$, $\beta = 111·72\,(1)$, $\gamma = 97·01\,(1)°$, $V = 825·8\,(3)$ Å^3, $Z = 2$, $D_x = 1·289$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0·70930$ Å, $\mu = 0·84$ cm^{-1}, $F(000) = 336$, $T = 295$ K, $R = 0·043$ for 2599 unique observed reflections. The two ester groups have different orientations, C═C—C═O torsion angles 115·7 (2) and 164·2 (2)°, and hence different amounts of conjugation with the C═C double bond [cos^2(angle) = 0·19 (nearly non-conjugated) and 0·93 (conjugated)].

Introduction. As part of a crystal and photochemical study of the di-π-methane rearrangement of dibenzobarrelene derivatives (Garcia-Garibay, Scheffer, Trotter & Wireko, 1990), the structure of the symmetrical dimethyl ester has now been determined.



R = Me

Experimental. Crystal from methanol, approximate dimensions 0·30 × 0·35 × 0·25 mm. Nonius CAD-4F diffractometer, monochromatized Mo $K\alpha$ radiation, lattice parameters from 25 reflections with $\theta = 16–22°$. Intensities for $\theta \le 27·5°$, *hkl*: 0 to 11, −16 to 16, −10 to 9; ω–2θ scan, ω-scan width $(0·65 + 0·35\tan\theta)°$ at 1·1–10° min^{-1}, extended 25% on each side for background measurement; three standard reflections showed no significant variation. Lp but no absorption corrections. 3795 unique reflections, 2599

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(68·5%) with $I \geq 3\sigma(I)$ used in final refinement. Structure solved by direct methods, refined by full-matrix least-squares procedures. All ten H atoms of the dibenzobarrelene skeleton located on a difference map and refined with isotropic thermal parameters, methyl H atoms in calculated positions based on the difference peaks but not refined. Non-H atoms refined anisotropically. Refinement on F with $w = 1/\sigma^2(F)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04I)^2$, S = scan, B_1 and B_2 = background counts. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–102). Locally written or locally modified versions of standard computer programs [*ORFLS* and *ORFFE* (Busing, Martin & Levy, 1962, 1964), *FORDAP* (Zalkin, unpublished) and *ORTEP* (Johnson, 1976)]. Final $R = 0{\cdot}043$, $wR = 0{\cdot}057$, goodness of fit = 1·0 for 257 parameters. $R = 0{\cdot}072$ for all 3795 reflections, shift/e.s.d. in final cycle < 0·001 (mean), 0·003 (maximum), $\Delta\rho = -0{\cdot}26$ to $+0{\cdot}34$ e Å^{-3}.

Discussion. Final positional parameters are listed in Table 1 and bond lengths and angles in Table 2.* The dibenzobarrelene skeleton (Fig. 1) is similar to that in the parent hydrocarbon (Trotter & Wireko, 1990) and in related diester derivatives (Garcia-Garibay *et al.*, 1990); the aromatic rings show slight, but not chemically significant, deviations from exact planarity [maximum atom displacement 0·009 (2) Å] and the angles external to the aromatic rings average 126·7°. The two ester groups have different orientations and different amounts of conjugation with the ring-system double bond; the torsion angles are φ_1 = C12=C11—C13=O2 = 115·7 (2) and φ_2 = C11=C12—C15=O4 = 164·2 (2)°, *i.e.* anticlinal and antiperiplanar geometries, respectively, with $\cos^2\varphi_1 = 0{\cdot}19$ (nearly non-conjugated) and $\cos^2\varphi_2 = 0{\cdot}93$ (conjugated). However, the C11—C13 [1·484 (2)] and C12—C15 [1·483 (2) Å] bond lengths do not reflect this difference, both being about midway between those for non-conjugated [1·497 (4) Å] and conjugated [1·470 (3) Å] systems (Allen, 1981).

The intermolecular environments of the two ester groups also differ,† but all contacts are of the normal van der Waals type. Since the two ester substituents are chemically identical, photolysis (solution or solid state) results in only one semibullvalene photoproduct (Garcia-Garibay *et al.*, 1990).

Table 1. *Positional* (*fractional* × 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3) *with e.s.d.'s in parentheses*

U_{eq} = 1/3(trace of diagonalized U).

	x	y	z	U_{eq}
C1	−3798 (3)	1732 (2)	2170 (2)	54
C2	−3323 (3)	2381 (3)	1182 (3)	72
C3	−2508 (4)	3435 (3)	1981 (3)	74
C4	−2143 (3)	3879 (2)	3796 (3)	58
C4a	−2588 (2)	3241 (1)	4791 (2)	42
C5	−4795 (2)	4055 (2)	7561 (2)	45
C6	−6378 (3)	3685 (2)	7522 (3)	55
C7	−7172 (3)	2621 (2)	6782 (3)	56
C8	−6404 (2)	1898 (2)	6038 (2)	46
C8a	−4844 (2)	2263 (1)	6055 (2)	36
C9	−3811 (2)	1605 (1)	5294 (2)	36
C9a	−3413 (2)	2175 (1)	3984 (2)	41
C10	−2285 (2)	3581 (1)	6795 (2)	37
C10a	−4028 (2)	3340 (1)	6838 (2)	36
C11	−2103 (2)	1759 (1)	6893 (2)	35
C12	−1335 (2)	2784 (1)	7686 (2)	35
C13	−1530 (2)	790 (1)	7401 (2)	39
C14	585 (3)	−233 (2)	7600 (3)	71
C15	288 (2)	3195 (1)	9304 (2)	38
C16	2349 (3)	2760 (2)	11754 (3)	82
O1	−176 (2)	652 (1)	7074 (2)	55
O2	−2277 (2)	180 (1)	7921 (2)	58
O3	737 (2)	2455 (1)	10206 (2)	58
O4	1095 (2)	4094 (1)	9744 (2)	56

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

C1—C2	1·399 (3)	C9—C9a	1·526 (2)
C1—C9a	1·383 (2)	C9—C11	1·534 (2)
C2—C3	1·374 (4)	C10—C10a	1·524 (2)
C3—C4	1·388 (3)	C10—C12	1·528 (2)
C4—C4a	1·381 (3)	C11—C12	1·333 (2)
C4a—C9a	1·391 (3)	C11—C13	1·484 (2)
C4a—C10	1·529 (2)	C12—C15	1·483 (2)
C5—C6	1·389 (3)	C13—O1	1·326 (2)
C5—C10a	1·379 (2)	C13—O2	1·198 (2)
C6—C7	1·377 (3)	C14—O1	1·451 (2)
C7—C8	1·393 (3)	C15—O3	1·330 (2)
C8—C8a	1·376 (2)	C15—O4	1·200 (2)
C8a—C9	1·527 (2)	C16—O3	1·446 (2)
C8a—C10a	1·398 (2)		
C2—C1—C9a	118·3 (2)	C4a—C10—C10a	105·49 (13)
C1—C2—C3	121·0 (2)	C4a—C10—C12	105·72 (13)
C2—C3—C4	120·5 (2)	C10a—C10—C12	105·38 (13)
C3—C4—C4a	119·0 (2)	C5—C10a—C8a	120·4 (2)
C4—C4a—C9a	120·6 (2)	C5—C10a—C10	126·82 (15)
C4—C4a—C10	126·7 (2)	C8a—C10a—C10	112·77 (14)
C9a—C4a—C10	112·71 (14)	C9—C11—C12	113·61 (14)
C6—C5—C10a	118·9 (2)	C9—C11—C13	118·15 (13)
C5—C6—C7	120·8 (2)	C12—C11—C13	128·21 (14)
C6—C7—C8	120·4 (2)	C10—C12—C11	114·26 (13)
C7—C8—C8a	119·0 (2)	C10—C12—C15	119·17 (14)
C8—C8a—C9	126·91 (15)	C11—C12—C15	126·57 (15)
C8—C8a—C10a	120·47 (15)	C11—C13—O1	111·30 (14)
C9—C8a—C10a	112·62 (14)	C11—C13—O2	123·5 (2)
C8a—C9—C9a	106·26 (13)	O1—C13—O2	125·0 (2)
C8a—C9—C11	105·42 (12)	C12—C15—O3	112·20 (14)
C9a—C9—C11	104·99 (13)	C12—C15—O4	123·63 (15)
C1—C9a—C4a	120·6 (2)	O3—C15—O4	124·18 (15)
C1—C9a—C9	126·5 (2)	C13—O1—C14	117·4 (2)
C4a—C9a—C9	112·93 (14)	C15—O3—C16	116·5 (2)

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* Lists of anisotropic thermal parameters, H-atom parameters, torsion angles, structure factors and a packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53671 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

† Packing diagram in deposited material.

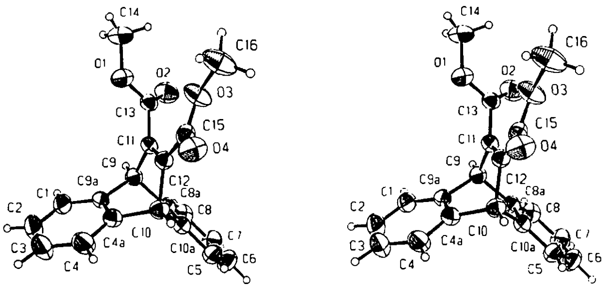

Fig. 1. Stereoview of the molecule.

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Structures of Two Polymorphic Forms of Diethyl Dibenzobarrelene-11,12-dicarboxylate

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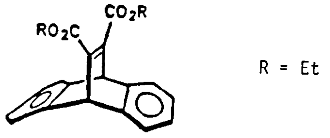
Abstract. Diethyl 9,10-dihydro-9,10-ethenoanthracene-11,12-dicarboxylate, $C_{22}H_{20}O_4$, M_r = 348·40, T = 295 K; form a, monoclinic, $P2_1/c$, a = 15·037 (2), b = 29·438 (4), c = 8·186 (1) Å, β = 91·06 (1)°, V = 3623·0 (7) Å^3, Z = 8 (two molecules per asymmetric unit), D_x = 1·277 g cm^{-3}, Mo $K\alpha_1$, λ = 0·70930 Å, μ = 0·81 cm^{-1}, $F(000)$ = 1472, R = 0·041 for 2680 unique observed reflections; form b, orthorhombic, $P2_12_12_1$, a = 9·0157 (5), b = 13·5253 (9), c = 14·9749 (7) Å, V = 1826·0 (2) Å^3, Z = 4, D_x = 1·267 g cm^{-3}, Cu $K\alpha_1$, λ = 1·54056 Å, μ = 6·7 cm^{-1}, $F(000)$ = 736, R = 0·035 for 1602 unique observed reflections. The two ester groups have different orientations, with one group in each molecule not conjugated to the C=C double bond and the other fully conjugated.

Introduction. As part of a crystal and photochemical study of the di-π-methane rearrangement of dibenzobarrelene derivatives (Garcia-Garibay, Scheffer, Trotter & Wireko, 1990*a*,*b*; Trotter & Wireko, 1991*a*) the structure of the symmetrical diethyl ester has now been determined. The material crystallizes in two crystal forms, one of which has two molecules per asymmetric unit.



Experimental. Crystals grown from ethanol exhibit two different morphologies, plate-like (Et/Et-*a*) and prisms (Et/Et-*b*). Approximate dimensions of cut crystals 0·35 × 0·3 × 0·3 mm for form *a* [0·30 × 0·33 × 0·29 mm for form *b*]. Nonius CAD-4F diffractometer, monochromatized Mo $K\alpha$ [Cu $K\alpha$] radiation, lattice parameters from 25 reflections with θ = 10–19 [30–56]°. Intensities for $\theta \leq 25$ [75]°; *hkl*: −17 to 17 [0 to 11], 0 to 34 [0 to 16], −9 to 0 [0 to 18]; ω–2θ scan, ω-scan width $(0{\cdot}65 + k\tan\theta)°$, k = 0·35 [0·14] at 1·6–10 [1·1–10]° min^{-1}, extended 25% on each side for background measurement. Three standard reflections showed no significant variation. Lp but no absorption corrections. 6367 [2152] unique reflections, 2680 (42·1%) [1602 (74·4%)] with $I \geq 3\sigma(I)$ used in final refinement. Structure solved by direct methods, refined by full-matrix least-squares procedures. H atoms fixed [refined with isotropic thermal parametes], non-H atoms refined anisotropically. Refinement on F with $w = 1/\sigma^2(F)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0{\cdot}04I)^2$, S = scan, B_1 and B_2 = background counts. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99–102). Locally written, or locally modified versions of standard computer programs [*ORFLS* and *ORFFE* (Busing, Martin & Levy, 1962, 1964), *FORDAP* (Zalkin, unpublished) and *ORTEP*II (Johnson, 1976)]. Final R = 0·041 [0·035], wR = 0·041 [0·039], goodness of fit = 1·32 [1·47] for 469 [316] parameters, R = 0·146 [0·056] for all 6367