absorption ignored; direct method (MULTAN78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and subsequent difference Fourier calculation; block-diagonal least squares (HBLS; Ohashi, 1975) with anisotropic thermal parameters for all non-H atoms; H atoms derived geometrically (C—H 1·08 Å) and refined; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + (0.03\,F)^2]^{-1}$; max. $(\varDelta/\sigma)$ 0·5; final $R = 0.044$; $\varDelta\rho$ excursions in final difference map 0·3 e Å^{-3}; atomic scattering factors from International Tables for X-ray Crystallography (1974); calculation carried out on HITAC M-280 computer at the Computer Center of the University of Tokyo.

Discussion. The final atomic parameters for non-H atoms are given in Table 1.* A projection of the structure along the c axis with the numbering scheme is given in Fig. 1 and a perspective drawing of the molecule is shown in Fig. 2. Bond distances and angles are listed in Table 2. The molecular structure is distorted to a large extent from a planar conformation due to steric repulsions between the intramolecular overcrowded hydrogen atoms; particularly H(C6) and H(C10). The repulsion is released mainly by the enlargement of the C(7)—C(8)—C(9) angle and distortion from the planar conformation. The degree of

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms, and positional and thermal parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43021 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

distortion was defined as the torsion angle around the pseudo-bond C(7)···C(9), C(6)—C(7)···C(9)—C(10) (Fujisawa, Oonishi, Aoki, Ohashi & Sasada, 1982). The values of C(7)—C(8)—C(9) and the torsion angle are 123·4 (5) and 38·2 (7)°, respectively. These are comparable to those of VEB: 124 (1) and 37 (1)°, but considerably different from those of the A molecule of 1,11-BisoVEB: 128·7 and 30·1°. The dihedral angles C(6)—C(7)—C(8)—C(9) and C(7)—C(8)—C(9)—C(10) are 18·2 and 25·4°, respectively. The deviations of the atoms from the mean molecular plane range from 1·07 (1) Å for C(11) to −0·74 (1) Å for C(5).

The molecules are stacked face-to-face to make a columnar structure along the c axis by van der Waals forces. The distance between the mean molecular planes is 3·60 (1) Å. There is no unusual short contact between the columns.

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8-Methylbenz[a]anthracene

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Abstract. $C_{19}H_{14}$, $M_r = 242.3$, monoclinic, $P2/a$, $a = 48.516$ (8), $b = 6.527$ (3), $c = 8.226$ (2) Å, $\beta = 92.15$ (2)°, $V = 2603.0$ Å^3, $Z = 8$, $D_m = 1.22$ (1) (NaI flotation), $D_x = 1.237$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 5.6$ cm^{-1}, $F(000) = 1024$, $T = 298$ K, final $R = 0.051$ for 2343 observed reflections. The two molecules in the asymmetric unit are almost planar with inclinations of 5·1 (3) and 2·4 (3)° between the outermost rings A/D. At the bay region the beach bond

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is 1·474 (4) Å; shortest C—C bonds are C(5)—C(6) = 1·345 (4) (K region), C(8)—C(9) = 1·351 (4), C(3)—C(4) = 1·362 (6) and C(10)—C(11) = 1·365 (4) Å.

Introduction. 8-Methylbenz[a]anthracene (8-MBA), originally designated 5-methylbenz-1:2-anthracene (Iball, 1938; Mason, 1956) is a moderately active member of an isomeric series of polycyclic hydrocarbons whose range of carcinogenic activity has been well documented (Jones & Matthews, 1974; Wislocki, Fiorentini, Fu, Yang & Lu, 1982). Of the related dimethylbenz[a]anthracenes (DMBA's), 7,8- and 8,12-DMBA are very active chemical carcinogens but 8,11-DMBA has low potency. The molecular structure of 8-MBA has been determined in view of the suspected influence of molecular shape and dimensions on the mechanism of carcinogenesis.

8-MBA
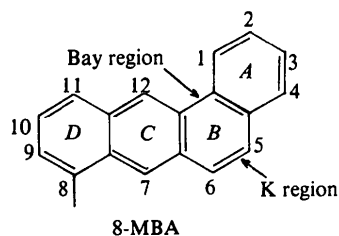
Experimental. Powder sample recrystallized from warm acetone in sealed tube to yield colourless prisms. In addition to systematic absences $h0l$ with h odd, X-ray photography showed a very weak 050 as the only odd $0k0$ reflection, so that all three space groups, $P2_1/a$, $P2/a$, and Pa (despite centrosymmetric intensity distribution), were considered before $P2/a$ was confirmed (standard $P2/c$, No. 13). Crystal dimensions 0·7 × 0·3 × 0·05 mm. Nonius CAD-4 diffractometer, Ni-filtered Cu $K\alpha$; 25 reflections $20 \leq \theta \leq 30°$ for lattice parameters; intensity data measured using $\omega/2\theta$ scan over θ range 2–60°, index ranges h 0/54, k 0/7, l −9/9, no significant intensity change for two standard reflections (22,1,0, 3$\bar{2}$4) checked every hour; 3823 unique data of which 2343 had $I \geq 1·5\sigma(I)$.

Structure solution by direct methods with SHELXS84 (Sheldrick, 1984) in $P2/a$ ($Z = 8$) yielded two 8-MBA molecules but R did not refine below 0·37. Solution by direct methods in noncentrosymmetric Pa gave two pairs of nearly complete molecules (one molecule of one pair was complete and the other three contained 18 of the 19 C atoms); corresponding atoms in each pair, with common y, were related by a twofold axis at $x = 0·168$, $z = 0·575$. Shifting coordinates of one molecule in each pair by (0·082, 0, −0·075) enabled least-squares refinement to proceed in $P2/a$ (twofold axis in standard position, 0·25, y, 0·50) isotropically to $R = 0·213$ and, with all C atoms included (remainder located from difference Fourier

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å^2) (*e.s.d.'s in parentheses*)

	x	y	z	U^*_{eq}
C(1)	3070 (1)	6583 (7)	2196 (4)	0·081 (3)
C(2)	2783 (1)	6497 (9)	2129 (5)	0·106 (3)
C(3)	2652 (1)	4800 (9)	2786 (5)	0·110 (4)
C(4)	2796 (1)	3206 (8)	3462 (4)	0·084 (3)
C(5)	3239 (1)	1586 (7)	4244 (3)	0·074 (3)
C(6)	3516 (1)	1628 (6)	4375 (3)	0·069 (3)
C(7)	3956 (1)	3435 (6)	4063 (3)	0·057 (2)
C(8)	4406 (1)	5228 (6)	3816 (3)	0·058 (2)
C(9)	4540 (1)	6867 (6)	3224 (3)	0·065 (2)
C(10)	4400 (1)	8475 (6)	2396 (3)	0·070 (2)
C(11)	4121 (1)	8431 (6)	2180 (3)	0·067 (2)
C(12)	3680 (1)	6651 (6)	2521 (3)	0·057 (2)
C(13)	3224 (1)	4972 (6)	2923 (3)	0·063 (2)
C(14)	3086 (1)	3276 (6)	3540 (3)	0·071 (2)
C(15)	3668 (1)	3359 (5)	3821 (3)	0·052 (2)
C(16)	4111 (1)	5095 (5)	3559 (3)	0·050 (2)
C(17)	3970 (1)	6735 (5)	2745 (3)	0·055 (2)
C(18)	3528 (1)	5038 (5)	3066 (3)	0·054 (2)
C(19)	4558 (1)	3570 (8)	4719 (5)	0·080 (3)
C(1')	1706 (1)	2017 (7)	773 (4)	0·072 (2)
C(2')	1993 (1)	1976 (8)	758 (4)	0·088 (3)
C(3')	2135 (1)	340 (8)	1413 (4)	0·092 (3)
C(4')	1998 (1)	−1274 (8)	2055 (4)	0·085 (3)
C(5')	1561 (1)	−2980 (6)	2775 (4)	0·072 (2)
C(6')	1285 (1)	−2963 (6)	2854 (3)	0·067 (2)
C(7')	838 (1)	−1240 (6)	2355 (3)	0·058 (2)
C(8')	379 (1)	342 (6)	1774 (3)	0·057 (2)
C(9')	237 (1)	1899 (6)	1064 (3)	0·062 (2)
C(10')	367 (1)	3560 (6)	333 (3)	0·067 (2)
C(11')	649 (1)	3652 (6)	325 (3)	0·060 (2)
C(12')	1099 (1)	2040 (5)	943 (3)	0·054 (2)
C(13')	1560 (1)	402 (5)	1440 (3)	0·056 (2)
C(14')	1706 (1)	−1277 (6)	2080 (3)	0·066 (2)
C(15')	1124 (1)	−1255 (5)	2241 (3)	0·052 (2)
C(16')	675 (1)	347 (5)	1734 (2)	0·050 (2)
C(17')	807 (1)	2041 (5)	1009 (3)	0·051 (2)
C(18')	1258 (1)	430 (5)	1525 (3)	0·052 (2)
C(19')	232 (1)	−1370 (8)	2602 (4)	0·076 (2)

* Calculated as: $\frac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^* \mathbf{a}_i.\mathbf{a}_j$, i.e. the orthogonalized U_{ij} tensor.

map), to 0·137. After anisotropic least-squares refinement for all C atoms, all 28 H atoms located in difference Fourier map, and unconstrained final full-matrix least-squares refinement on F (C atoms anisotropic, H atoms isotropic), $w = [\sigma^2(F) + 0·00015 |F_o|^2]^{-1}$, for 2343 reflections and 455 variables converged to $R = 0·051$ (and $wR = 0·052$), $(\Delta/\sigma)_{max} = 0·05$, residual electron density in final difference map −0·16 to 0·11 e Å^{-3}. Scattering factors from Stewart, Davidson & Simpson (1965) for H and Cromer & Mann (1968) for C. No correction for absorption or secondary extinction.*

Discussion. Final fractional coordinates of C atoms are in Table 1 and bond lengths and angles in Table 2. Fig. 1 shows the atom numbering. The shapes of the two crystallographically independent molecules are similar but not identical and correspond to the type-I (Briant,

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* Lists of structure factors, anisotropic thermal parameters, parameters of H atoms, bond lengths and angles involving H atoms and some additional experimental details have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 43019 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Jones & Shaw, 1985) or nearly planar methylbenz-[a]anthracenes (MBA's). For individual aromatic rings, none of the C atoms deviates by more than 0·02 Å from its own ring plane; typically, deviations are about 0·01 Å, or as low as 0·005 Å for rings A (molecule 1) and A' and B' (molecule 2). The overall molecular curvatures are intermediate between the rather pronounced bowing of 3,9-DMBA (Briant et al., 1985) and the almost planar 2-MBA (Briant & Jones, 1983) and 11-MBA (Briant & Jones, 1984). Thus the inclination A/D between extreme rings is 5·1 (3)° in molecule 1 of 8-MBA and 2·4 (3)° in molecule 2. With respect to the best plane through atoms C(1,2,3,4,14,13) of ring A in molecule 1, C(8) and C(9) of ring D lie about 0·43 Å above and the ring methyl C(19) is 0·56 Å above, whereas in the less bowed molecule 2 the largest C

deviants from the corresponding A' plane are the methyl C(19') at 0·25 Å and C(7') and C(8') at 0·15 Å.*

Pairs of corresponding bond lengths are the same within 2σ except for C(2)—C(3), while most pairs of corresponding C—C—C angles agree within 1σ; pairs of angles involving C(3) differ by about 1° and C(3) has the largest thermal coefficient [$U_{22} = 0·138$ (5) Å²]. C—H apparent bond lengths are in the range 0·93 (3)–1·09 (4) Å [0·94 (3)–1·00 (3) Å for methyl C—H]. In the 'bay' (Bartle & Jones, 1967) region, the beach (Briant & Jones, 1983) bond, C(13)—C(18) = 1·474 (3) Å, is, as usual (Briant et al., 1985), the longest; the beach bond angles are also enlarged to C(1)—C(13)—C(18) = 121·9 (3) and C(12)—C(18)—C(13) = 122·9 (3)° (both typically 122·9° in type-I structures). Of the short C—C bonds, C(5)—C(6) at the K region is the shortest at 1·345 (4) Å but barely shorter than C(8)—C(9) = 1·351 (4) Å adjoining the substituent, while C(3)—C(4) = 1·362 (6) Å and C(10)—C(11) = 1·365 (4) Å are also short, as in other reliable type-I BA structures. Methyl substitution in BA's often causes the adjacent C—C bond(s) to lengthen [C(8)—C(16) at 1·441 (3) Å in 8-MBA is significantly longer than the type-I mean of

* E.s.d.'s of atom-to-plane distances are ca 0·005 Å.

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

C(1)—C(2)	1·389 (5)	C(1')—C(2')	1·393 (5)
C(1)—C(13)	1·410 (5)	C(1')—C(13')	1·394 (5)
C(2)—C(3)	1·397 (7)	C(2')—C(3')	1·369 (6)
C(3)—C(4)	1·361 (7)	C(3')—C(4')	1·362 (6)
C(4)—C(14)	1·405 (5)	C(4')—C(14')	1·414 (5)
C(5)—C(6)	1·345 (5)	C(5')—C(6')	1·345 (5)
C(5)—C(14)	1·439 (5)	C(5')—C(14')	1·445 (5)
C(6)—C(15)	1·434 (5)	C(6')—C(15')	1·440 (5)
C(7)—C(15)	1·402 (4)	C(7')—C(15')	1·393 (4)
C(7)—C(16)	1·390 (5)	C(7')—C(16')	1·390 (5)
C(8)—C(9)	1·353 (5)	C(8')—C(9')	1·348 (5)
C(8)—C(16)	1·443 (4)	C(8')—C(16')	1·439 (4)
C(8)—C(19)	1·493 (6)	C(8')—C(19')	1·500 (7)
C(9)—C(10)	1·410 (5)	C(9')—C(10')	1·402 (5)
C(10)—C(11)	1·361 (5)	C(10')—C(11')	1·368 (5)
C(11)—C(17)	1·415 (5)	C(11')—C(17')	1·407 (5)
C(12)—C(17)	1·411 (4)	C(12')—C(17')	1·419 (4)
C(12)—C(18)	1·372 (5)	C(12')—C(18')	1·379 (5)
C(13)—C(14)	1·399 (5)	C(13')—C(14')	1·397 (5)
C(13)—C(18)	1·476 (4)	C(13')—C(18')	1·471 (4)
C(15)—C(18)	1·421 (5)	C(15')—C(18')	1·417 (5)
C(16)—C(17)	1·423 (5)	C(16')—C(17')	1·422 (5)

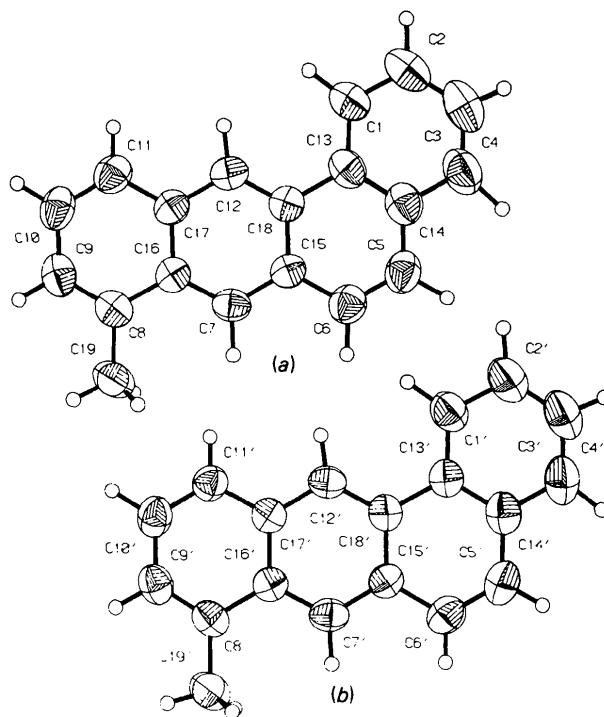| C(1)—C(2)—C(3)	119·1 (6)	C(1')—C(2')—C(3')	119·8 (6)
C(1)—C(13)—C(14)	119·4 (4)	C(1')—C(13')—C(14')	118·9 (4)
C(1)—C(13)—C(18)	121·7 (4)	C(1')—C(13')—C(18')	122·1 (4)
C(2)—C(3)—C(4)	121·9 (5)	C(2')—C(3')—C(4')	120·8 (5)
C(3)—C(4)—C(14)	119·5 (5)	C(3')—C(4')—C(14')	120·6 (5)
C(4)—C(14)—C(5)	119·6 (5)	C(4')—C(14')—C(5')	120·6 (5)
C(4)—C(14)—C(13)	120·0 (5)	C(4')—C(14')—C(13')	119·1 (5)
C(5)—C(6)—C(15)	121·5 (5)	C(5')—C(6')—C(15')	121·5 (5)
C(5)—C(14)—C(13)	120·4 (4)	C(5')—C(14')—C(13')	120·3 (4)
C(6)—C(15)—C(7)	120·3 (4)	C(6')—C(15')—C(7')	120·6 (4)
C(6)—C(15)—C(18)	120·1 (4)	C(6')—C(15')—C(18')	119·7 (4)
C(7)—C(15)—C(18)	119·5 (4)	C(7')—C(15')—C(18')	119·7 (4)
C(7)—C(16)—C(8)	123·2 (4)	C(7')—C(16')—C(8')	123·1 (4)
C(7)—C(16)—C(17)	118·1 (3)	C(7')—C(16')—C(17')	118·2 (3)
C(8)—C(9)—C(10)	122·3 (4)	C(8')—C(9')—C(10')	122·6 (4)
C(8)—C(16)—C(17)	118·7 (4)	C(8')—C(16')—C(17')	118·7 (4)
C(9)—C(10)—C(11)	120·4 (5)	C(9')—C(10')—C(11')	120·2 (4)
C(10)—C(11)—C(17)	119·9 (5)	C(10')—C(11')—C(17')	119·7 (4)
C(11)—C(17)—C(12)	120·8 (4)	C(11')—C(17')—C(12')	121·0 (4)
C(11)—C(17)—C(16)	119·9 (4)	C(11')—C(17')—C(16')	120·0 (3)
C(12)—C(17)—C(16)	119·3 (4)	C(12')—C(17')—C(16')	118·9 (4)
C(12)—C(18)—C(13)	123·1 (4)	C(12')—C(18')—C(13')	122·6 (4)
C(12)—C(18)—C(15)	118·6 (3)	C(12')—C(18')—C(15')	118·5 (4)
C(13)—C(1)—C(2)	120·0 (5)	C(13')—C(1')—C(2')	120·8 (4)
C(13)—C(18)—C(15)	118·3 (4)	C(13')—C(18')—C(15')	118·9 (4)
C(14)—C(5)—C(6)	121·0 (5)	C(14')—C(5')—C(6')	121·0 (4)
C(14)—C(13)—C(18)	119·0 (4)	C(14')—C(13')—C(18')	118·9 (4)
C(15)—C(7)—C(16)	122·0 (4)	C(15')—C(17')—C(16')	122·4 (4)
C(16)—C(8)—C(9)	118·9 (4)	C(16')—C(8')—C(9')	118·8 (4)
C(16)—C(8)—C(19)	120·0 (4)	C(16')—C(8')—C(19')	120·2 (4)
C(17)—C(12)—C(18)	122·2 (4)	C(17')—C(12')—C(18')	122·2 (4)
C(19)—C(8)—C(9)	121·1 (4)	C(19')—C(8')—C(9')	121·4 (4)

Fig. 1. Projections of the two independent (but nearly screw-axis-related) 8-MBA molecules, (a) molecule 1, (b) molecule 2, showing crystallographic atom numbering and 50% probability thermal vibration ellipsoids for the C atoms.

1·423 (3) Å] and invariably affects the bond angles: C(16)—C(8)—C(9), typically 121° in other MBA's, is reduced in 8-MBA to 118·9 (3)° and C(7)—C(16)—C(8) and C(8)—C(9)—C(10), typically 122·4 and 121·4°, are increased to 123·2 (3) and 122·5 (3)°, respectively. Although the *D* ring carries the substituent in 8-MBA, most of the deviations from the most reliable mean bond angles for type-I BA's (Briant *et al.*, 1985) are in or near ring *A*, namely enlargement by about 1° of angles C(2)—C(3)—C(4) and C(1)—C(13)—C(14), and reduction by about 1° of angles C(13)—C(1)—C(2), C(1)—C(2)—C(3), C(3)—C(4)—C(14), C(14)—C(5)—C(6), C(18)—C(13)—C(1), and C(10)—C(11)—C(17). ¹H NMR chemical shifts for 8-MBA in CS₂ solution are all within about 0·1 p.p.m. [or less for H(1—6) and H(12)] of the corresponding shifts in unsubstituted BA's (Jones & Mokoena, 1982; Evans, Fu & Cairns, 1981).

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The Structure of a Tricyclic Peroxide*

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Abstract. 10,11,14,15-Tetraoxa-1,8-diazatricyclo[6.4.4.0²,⁷]hexadeca-2(7),3,5-triene, BTDD, $C_{10}H_{12}N_2O_4$, $M_r = 224.22$, orthorhombic, *Pbca*, $a = 7.635$ (1), $b = 11.041$ (1), $c = 24.249$ (2) Å, $V = 2044.1$ (6) Å³, $Z = 8$, $D_m = 1.46$ (3), $D_x = 1.46$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.23$ cm⁻¹, $F(000) = 944$, $T = 294$ K, $R = 0.0707$ for 1785 reflections. The bridgehead N atoms adopt a nearly planar configuration [0·16 (1) and 0·10 (1) Å out of the planes of the C atoms to which they are bonded]. N—C and peroxide bonds are slightly short [N—C(—O) average 1·430 (3), N—C(—C) average 1·424 (4), O—O average 1·465 (10) Å], as expected from similar compounds. The four O atoms are disordered.

Introduction. Schaefer, Fourkas & Tiemann (1985) reported that the bridgehead N atoms of HMTD, hexamethylene triperoxide diamine, N(CH₂OOCH₂)₃N (3,4,8,9,12,13-hexaoxa-1,6-diazabicyclo[4.4.4]tetradecane), are coplanar with the C atoms to which they are bonded. We have synthesized several similar compounds to study this unusual nitrogen geometry. BTDD (benzene tetramethylene diperoxide diamine) is one such compound.

Experimental. 45 g H₂O₂ and 60 ml formaldehyde solution (37%) are mixed and cooled to 273 K; 1 g *o*-phenylenediamine added, stirred for one hour; crystals collected, washed with ethanol; recrystallization from pentyl acetate; elemental analysis satisfactory for N, C, H; irregular crystal 0·33 × 0·33 × 0·44 mm; D_m by flotation; Nonius CAD-4 diffrac-