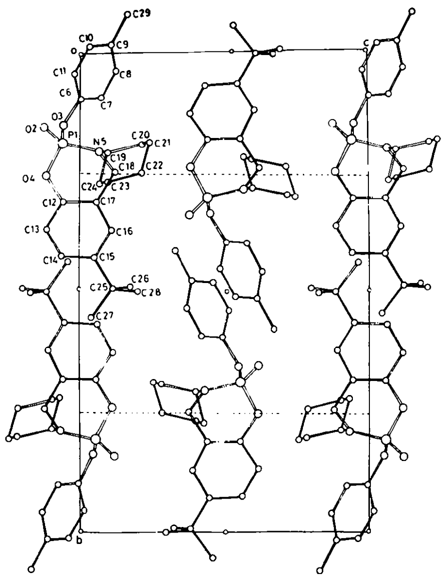

Fig. 2. A perspective view of the molecules in the unit cell.

& Bentrude, 1980) because, firstly, the substituent at N(5) is rotated away by 104·7° and, secondly, the distance O(2)—C(19) is 3·02 (2) Å. The phosphoryl O(2) atom occupies an axial position. The least-squares plane of the 4-methylphenyl ring is inclined at an angle of 126 (1)° to the least-squares plane of the phosphorine ring. The *tert*-butyl and phenyl groups are *trans* to each other and they occupy equatorial positions on the phosphorine ring. The dihedral angle between the two phenyl rings is 47·9°. The cyclohexyl ring adopts a chair conformation with the N(5) atom occupying an equatorial position. Fig. 2 shows a perspective view of the molecules in the unit cell. Molecular packing only involves van der Waals contacts.

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Structure of 9,10-Dihydro-1,9:4,10-diethano-9,10-ethenoanthracene

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Abstract. $C_{20}H_{16}$, $M_r = 256{\cdot}34$, orthorhombic, $Pnm2_1$, $a = 8{\cdot}750\,(1)$, $b = 9{\cdot}419\,(1)$, $c = 8{\cdot}042\,(1)$ Å, $V = 662{\cdot}7\,(1)$ Å^3, $Z = 2$, $D_m = 1{\cdot}30$, $D_x = 1{\cdot}30$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 0·0790 mm^{-1}, $F(000) = 272$, $T = 293$ K, final $R = 0{\cdot}054$ for 691 observed reflections. The molecule has mirror symmetry in the crystal. All the bond lengths are normal, but the unusual bond angles around the bridgehead suggest that there is much strain in this molecule.

Introduction. The title hydrocarbon, possessing a dibenzobarrelene structure, has recently been synthesized by hydrolysis of *anti*-4-amino-[2.2](1,4)-

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal factors with their e.s.d.'s for non*-H *atoms*

$$B_{eq} = \tfrac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
C(1)	1·2076 (3)	0·0751 (3)	0·1252 (5)	4·2 (1)
C(2)	1·0694 (3)	0·1517 (3)	0·1120 (5)	4·0 (2)
C(3)	0·9417 (3)	0·0725 (3)	0·1003 (5)	3·7 (2)
C(4)	0·7886 (3)	0·1402 (3)	0·0881 (5)	4·0 (2)
C(5)	0·6987 (3)	0·0770 (4)	0·2340 (4)	4·1 (1)
C(6)	0·6234 (4)	0·1482 (4)	0·3573 (5)	5·9 (2)
C(7)	0·5484 (4)	0·0716 (4)	0·4834 (4)	7·3 (2)
C(8)	0·7133 (3)	0·0691 (4)	−0·0661 (4)	4·1 (1)
C(9)	1·0173 (4)	0·3068 (3)	0·1062 (6)	5·0 (2)
C(10)	0·8383 (3)	0·3007 (3)	0·0870 (6)	4·5 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1·412 (5)	C(1)—C(1′)	1·415 (7)
C(2)—C(3)	1·347 (6)	C(2)—C(9)	1·531 (6)
C(3)—C(4)	1·487 (5)	C(3)—C(3′)	1·366 (8)
C(4)—C(5)	1·533 (5)	C(4)—C(8)	1·556 (5)
C(4)—C(10)	1·573 (6)	C(5)—C(6)	1·366 (5)
C(5)—C(5′)	1·451 (7)	C(6)—C(7)	1·407 (6)
C(7)—C(7′)	1·349 (8)	C(8)—C(8′)	1·302 (7)
C(9)—C(10)	1·575 (7)		

C(1′)—C(1)—C(2)	120·7 (3)	C(1)—C(2)—C(3)	115·6 (4)
C(1)—C(2)—C(9)	138·1 (4)	C(3)—C(2)—C(9)	106·2 (4)
C(2)—C(3)—C(4)	121·0 (4)	C(2)—C(3)—C(3′)	123·6 (4)
C(3′)—C(3)—C(4)	115·4 (4)	C(3)—C(4)—C(5)	104·2 (3)
C(3)—C(4)—C(8)	104·5 (3)	C(3)—C(4)—C(10)	99·4 (3)
C(5)—C(4)—C(8)	103·0 (3)	C(5)—C(4)—C(10)	121·3 (3)
C(8)—C(4)—C(10)	121·8 (3)	C(4)—C(5)—C(6)	127·8 (3)
C(4)—C(5)—C(5′)	112·9 (3)	C(5′)—C(5)—C(6)	119·4 (3)
C(5)—C(6)—C(7)	119·8 (4)	C(6)—C(7)—C(7′)	120·9 (4)
C(4)—C(8)—C(8′)	115·5 (3)	C(2)—C(9)—C(10)	105·3 (4)
C(4)—C(10)—C(9)	108·0 (4)		

naphthalenoparacyclophane (Mori, Takemura & Tsuchiya, 1988). The X-ray crystallographic study of this compound has been undertaken to confirm its structure.

Experimental. A colourless platelike crystal obtained from *n*-hexane solution, 0·4 × 0·4 × 0·2 mm, mounted on a Rigaku AFC-5 four-circle diffractometer with graphite-monochromatized Mo *Kα* radiation (λ = 0·71073 Å). Cell parameters were refined from least-squares procedure for setting angles of 25 reflections with $25{\cdot}58 < 2\theta < 31{\cdot}23°$. Intensities were measured by θ–2θ scan method with scan width (θ) $(1{\cdot}0 + 0{\cdot}5\tan\theta)°$, scan speed ($\theta$) 4° min^{-1}, backgrounds for 1·0 s at both ends of every scan. Three standard reflections (015, 00$\bar{6}$ and 0$\bar{1}$5) monitored every 100 measurements, changed −1·3 to 1·6% of F's throughout the data collection. 2167 reflections measured up to $2\theta < 60°$, index range $0 \leq h \leq 12$, $0 \leq k \leq 13$, $-11 \leq l \leq 11$, $R_{\rm int} = 0{\cdot}011$. All reflections $0kl$, $k + l$ odd, and $00l$, l odd, were too weak to be observed as required by space group $Pnm2_1$. Of the 1087 independent reflections, 691 with $|F_o| \geq 3\sigma(F)$ were used for further calculations. Lorentz and polarization corrections were made, absorption was neglected. Density was determined by flotation with aqueous K_2HgI_4 solution. Structure was solved by *MULTAN*84 (Main, Germain & Woolfson, 1984). All the non-H atoms were found on the Fourier map, and after several cycles of the block-diagonal least-squares refinement, the locations of H atoms were calculated. Final refinement with anisotropic thermal factors for non-H atoms and fixed isotropic thermal factors for H atoms, $B_{\rm iso} = 4{\cdot}0$ Å^2. Final $R = 0{\cdot}054$ and $wR = 0{\cdot}029$, $S = 0{\cdot}65$. Function minimized $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma^2(F)$, $(\Delta/\sigma)_{\rm max} = 0{\cdot}74$, $(\Delta\rho)_{\rm max} = 0{\cdot}09$, $(\Delta\rho)_{\rm min} = -0{\cdot}1$ e Å^{-3} in the final difference Fourier map. Scattering factors from *International Tables for X-ray Crystallography* (1974). The calculations were performed on a PANAFACOM U-1200 II computer with the Rigaku *RASA*-5P program package and the HITAC M-680H at the Computer Centre of the University of Tokyo with the *UNICS* (Sakurai, 1967) program system.

Discussion. The final atomic parameters are listed in Table 1, and the bond lengths and angles are shown in Table 2. A perspective view of the molecule with the atom numbering is shown in Fig. 1.*

The basic structure of the present molecule consists of a barrelene (bicyclo[2.2.2]octatriene) skeleton with two benzene rings condensed on both sides, only one of which is bridged to the central barrelene ring by two ethano groups. The molecule possesses crystallographic mirror symmetry about a plane bisecting the central etheno group, C(8)—C(8′). All the bond lengths are within a range of standard values, and they are approximately the same as those of 2,3-(tetrafluorobenzo)bicyclo[2.2.2]octatriene

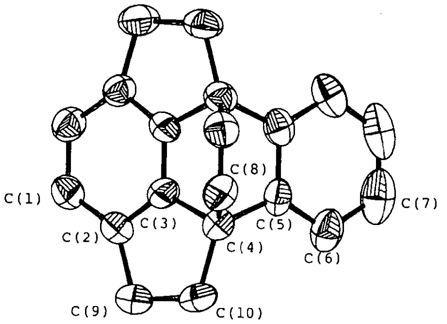

Fig. 1. *ORTEP* (Johnson, 1976) view of the molecule with the atom numbering. H atoms have been omitted for clarity. Thermal ellipsoids are drawn at the 50% probability level.

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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 51939 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

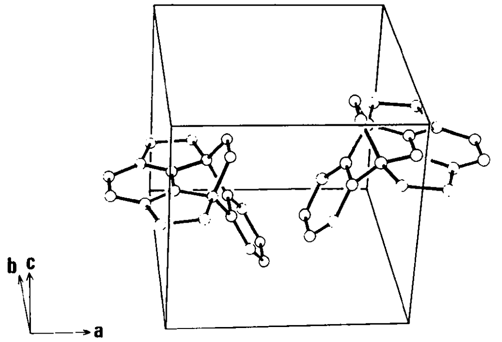

Fig. 2. The molecular packing arrangement.

(Hamor & Hamor, 1979) and pyracene (Simmons & Lingafelter, 1961). However, the two ethano bridges make the geometry of each side of the molecule unsymmetrical: the lengths of C(3)—C(3′) and C(3)—C(4) on the bridged side [1·366 (8), 1·487 (6) Å, respectively] are significantly shorter than the corresponding bonds on the non-bridged side, C(5)—C(5′) and C(4)—C(5) [1·451 (7), 1·533 (5) Å, respectively]. This ethano-bridge effect is more distinct about the bond angles: for example, C(3)—C(4)—C(10) of 99·4 (3)° on the bridged side is much smaller than the corresponding C(5)—C(4)—C(10) of 121·3 (3)° on the non-bridged side. The values of both angles deviate significantly from the ideal sp^3 angle of 109·5°, suggesting the existence of a large distortion at the bridgehead C(4) atom. Similar unusual bond angles have also been observed at the bridgehead of 1,10:6,7-bis(ethano)-9-methoxytriptycene (Fukazawa, Kikuchi, Kajita & Ito, 1984). In contrast, the bond angles at the bridgehead of non-bridged bicyclo[2.2.2] compounds do not deviate as much from the tetrahedral angle (Sawyer, Gomes & Yates, 1986).

Two benzene rings are planar within their e.s.d.'s, forming a dihedral angle of 118·3 (1)°, which is almost equal to that observed in 1,10:6,7-bis(ethano)-9-methoxytriptycene (Fukazawa, Kikuchi, Kajita & Ito, 1984). The five-membered ring C(2)—C(3)—C(4)—C(10)—C(9) is also planar within ±0·02 Å. Molecular packing in a unit cell is illustrated in Fig. 2. There are no intermolecular atomic contacts less than the sum of the van der Waals radii.

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Structure of *N*,*N*-Dimethyl-3-{[1-(phenylmethyl)cycloheptyl]oxy}-1-propanamine Fumarate (Bencyclane Hydrogen Fumarate)

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Abstract. $C_{19}H_{32}NO^+.C_4H_3O_4^-$, vasodilator, spasmolytic, Ca^{2+} antagonist, $M_r = 405·54$, triclinic, $P\bar{1}$, $a = 6·461\ (1)$, $b = 9·063\ (1)$, $c = 19·607\ (5)$ Å, $\alpha = 80·82\ (2)$, $\beta = 87·48\ (2)$, $\gamma = 88·78\ (1)°$, $V = 1132$ Å^3, $Z = 2$, $D_x = 1·19$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 6·78$ cm^{-1}, $F(000) = 440$, room temperature, $R = 0·079$ for 2143 observed reflections. The aliphatic chain is almost completely extended and the cycloheptane ring is in a twist-chair conformation. The molecules are held together by N—H···O hydrogen bonds and van der Waals contacts.

Introduction. First prepared nearly 25 years ago (Pallos, Zolyomi, Budai, Komlos & Petocz, 1965),

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