Fig. 1 [drawn by *STRPL* (Cameron, 1973)] gives the packing of the molecules in the unit cell.

Discussion. The crystal is constructed from molecules with the formula I*a* (R = NHCHO), which are linked together to form chains parallel to **b** with a hydrogen bond between the phosphoryl oxygen atom O(1) and the amide nitrogen atom N(1). The chains stack side by side along **a** with the carbonyl group of one molecule fitting between the two methyl groups of a molecule in the adjacent stack. Along **c** the non-bonding contact between the stacks is made with the methyl and methylene groups of the phosphorinane ring (Fig. 1).

The phosphoryl O is equatorial, so the configuration at the P is apparently uninfluenced by the amido group. The P–O, P–N and N–C bond lengths of 1·461 (6), 1·702 (5) and 1·373 (9) Å can be compared with 1·476 (10), 1·649 (10) and 1·424 (9) Å in phenylaminophosphorinane (I*a*, R = NHPh) in which there is also a P=O···H–N hydrogen bond (Cameron & Karolak-Wojciechowska, 1976). The significant lengthening of only the P–N bond in the present compound is probably caused by the strong electron-withdrawing carbonyl group bonded to the N atom.

The configuration and dimensions of the dioxaphosphorinane ring are normal. The angle between the planes through P(1), O(2), O(3) and O(2), O(3), C(1) and C(3) is 32·1° and the angle between this second plane and that through C(1), C(2) and C(3) is 65·3°. The corresponding interplane angles in (I*a*) (R = NHPh) are 34·2 and 55·6°. The two P–O single bonds of 1·570 (6) Å [O(2)] and 1·544 (6) Å [O(3)] are not significantly different and are within the normal range (1·522–1·584 Å) for this type of compound (Gałdecki & Karolak-Wojciechowska, 1973). There is considerable spread in the C–C interatomic distances, particularly the C–Me, 1·488 (11) Å [C(4)] and 1·589 (10) Å [C(5)]. The methyl groups do not pack very closely (Fig. 1) and from the Fourier map it is clear that the methyl C atoms are strongly vibrating with ill defined positions.

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1,10-Phenanthrotricyclo[4.1.1.$0^{2,7}$]heptene

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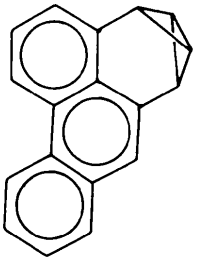
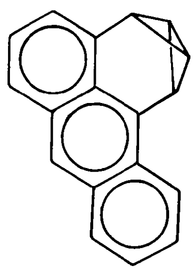
Abstract. $C_{18}H_{12}$. Orthorhombic, *Iba*2, a = 16·34 (1), b = 18·64 (1), c = 7·884 (5) Å, D_c = 1·26 g cm^{-3}. Z = 8. Full-matrix least-squares refinement gave R = 0·067 for 74 parameters and 409 significant reflexions [$I > 2\sigma(I)$]. The molecular geometry is normal, and the packing similar to that for 1,8-naphthotricyclo[4.1.1.$0^{2,7}$]heptene.

Introduction. The reaction of benz[*de*]anthracenyl anion with methylene chloride/*n*-butyllithium (Pagni, Burnett & Hazell, 1977) yielded 4,5-benzocyclohepta-[1,2,3-*de*]naphthalene and an isomeric compound. NMR showed the compound to possess a bicyclobutane structure and to be either 1,10-phenanthrotricyclo[4.1.1.$0^{2,7}$]heptene (I) or 1,9-anthrotricyclo-



(I)



(II)

[4.1.1.0^{2,7}]heptene (II). A crystal structure determination shows the compound to be (I), the structure favoured by NMR and mass spectra.

Intensities were collected on a computer-controlled Supper diffractometer with graphite-monochromated Mo $K\alpha$ radiation, a scintillation counter and a pulse-height analyser. 980 independent reflexions were measured by the background-peak-background method out to $\sin\theta = 0{\cdot}45$. The crystal, $0{\cdot}17 \times 0{\cdot}08 \times 0{\cdot}33$ mm, was mounted about **c**. No corrections were made for absorption.

Table 1. *Atomic coordinates* (Å) *relative to molecular axes*

The molecular origin is at $X_o = 2{\cdot}147$ (11), $Y_o = 3{\cdot}511$ (11), $Z_o = 0{\cdot}000$; the orientation is given by the Euler angles (Goldstein, 1959) $\psi = 0{\cdot}455$ (11), $\theta = 2{\cdot}114$ (10), $\varphi = 1{\cdot}313$ (7) rad.

	x	y	z
C(1)	0·00	0·00	0·00
C(2)	0·00	−1·39 (2)	0·00
C(3)	1·24 (2)	−2·05 (3)	0·02 (3)
C(4)	2·43 (2)	−1·35 (4)	0·01 (3)
C(5)	2·39 (2)	0·07 (3)	0·00
C(6)	1·20 (2)	0·74 (2)	0·00 (2)
C(7)	1·11 (4)	2·22 (2)	−0·05 (3)
C(8)	0·03 (4)	2·86 (2)	0·72 (4)
C(9)	−0·04 (4)	2·84 (2)	−0·75 (4)
C(10)	−1·14 (4)	2·23 (2)	0·07 (4)
C(11)	−1·25 (2)	0·74 (2)	0·02 (2)
C(12)	−2·43 (2)	0·09 (3)	0·06 (3)
C(13)	−2·46 (2)	−1·33 (3)	0·04 (2)
C(14)	−1·28 (2)	−2·10 (2)	0·03 (2)
C(15)	−1·40 (4)	−3·48 (2)	−0·02 (3)
C(16)	−2·58 (4)	−4·13 (4)	0·03 (4)
C(17)	−3·77 (3)	−3·37 (4)	0·05 (4)
C(18)	−3·71 (2)	−2·01 (4)	0·02 (3)

The structure was determined by trial and error with the structure of 1,8-naphthotricyclo[4.1.1.0^{2,7}]heptene (Hazell, 1977) as a starting point. The geometry of the molecule was assumed and the Euler angles (Goldstein, 1959) and molecular origin were refined by full-matrix least squares (*KONSLS*, Pawley, 1971). The four possibilities gave $R(A) = 0{\cdot}51$, $R(B) = 0{\cdot}48$, $R(C) = 0{\cdot}29$ and $R(D) = 0{\cdot}11$ for the 21 lowest-order reflexions. Refinement of model D gave $R = 0{\cdot}067$ for 74 parameters and 409 significant reflexions* [$I > 2\sigma(I)$]. The positions of the H atoms were not refined; thermal parameters were described by the tensors **T**, **L**, and **S**. Unit weights were employed. The scattering

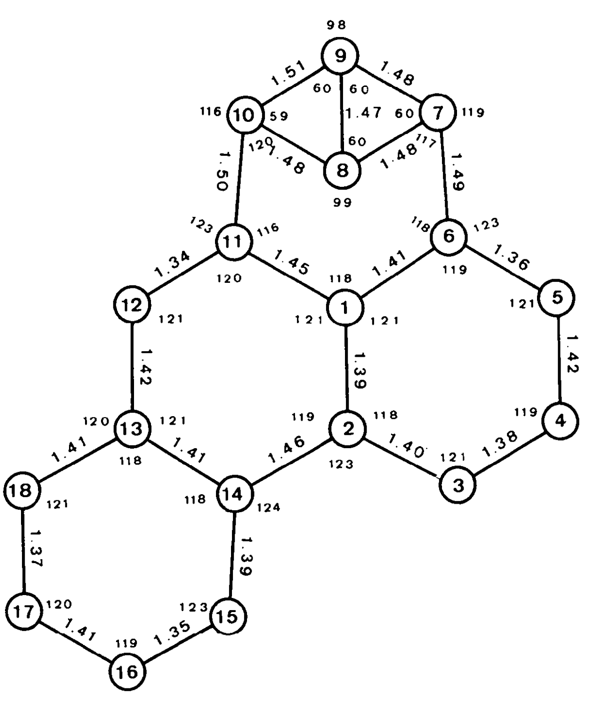


Fig. 1. Bond distances (Å) and angles (°). The average standard deviations are: σ(C–C) $\simeq$ 0·04 Å, σ(C–C–C) 2 to 3°.

Table 2. *Thermal parameters* (**T** *in* Å$^2 \times 10^{-3}$, **L** *in* deg^2, **S** *in* deg Å $\times 10^{-3}$)

L and **S** are referred to an origin $X = 2{\cdot}145$, $Y = 4{\cdot}097$, $Z = 0{\cdot}329$ Å.

$$\mathbf{T} = \begin{pmatrix} 29\,(3) & -5\,(2) & 3\,(2) \\ & 54\,(2) & 3\,(2) \\ & & 46\,(2) \end{pmatrix} \qquad \mathbf{L} = \begin{pmatrix} 8{\cdot}0\,(1{\cdot}5) & -0{\cdot}5\,(1{\cdot}9) & -1{\cdot}6\,(1{\cdot}3) \\ & 27{\cdot}2\,(3{\cdot}3) & 1{\cdot}3\,(1{\cdot}5) \\ & & 9{\cdot}2\,(1{\cdot}6) \end{pmatrix}$$

$$\mathbf{S} = \begin{pmatrix} * & 86\,(47) & 58\,(32) \\ 57\,(60) & * & -113\,(78) \\ 37\,(37) & -88\,(56) & * \end{pmatrix} \qquad \begin{aligned} S_{33} - S_{22} &= 183\,(94) \\ S_{11} - S_{33} &= 67\,(65) \end{aligned}$$

* It is not possible to determine all three diagonal elements from diffraction data; the differences between the diagonal terms are given.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32618 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

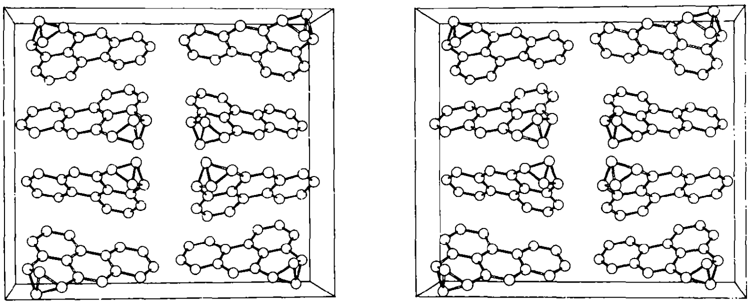

Fig. 2. A stereoscopic view of the cell contents as seen down [001]; **b** is across, **a** up the page. The molecules at $z = \frac{1}{2}$ are shown with filled bonds.

Table 3. *Comparison of* 1,10-*phenanthrotricycloheptene* (I) *with* 1,8-*naphthotricycloheptene* (II)

	a(Å)	b(Å)	c(Å)	X_o(Å)	Y_o(Å)	Z_o(Å)	ψ(rad)	θ(rad)	φ(rad)
(I)	16·34	18·64	7·88	2·147 (11)	3·511 (11)	0·000	0·455 (11)	2·114 (10)	1·313 (7)
(II)	17·42	13·95	8·00	2·232 (8)	3·389 (8)	0·000	0·327 (9)	2·162 (8)	1·310 (6)

factors were those of Cromer & Mann (1968) for C and of Stewart, Davidson & Simpson (1965) for H.

Atomic coordinates are given in Table 1, thermal parameters in Table 2. Bond lengths and angles are shown in Fig. 1.

Discussion. Bond lengths and angles are similar to those in phenanthrene (Kay, Okaya & Cox, 1971), bicyclobutane (Cox, Harmony, Nelson & Wiberg, 1969) and 1,8-naphthotricyclo[4.1.1.0^{2,7}]heptene.

The packing, Fig. 2, is very similar to that for 1,8-naphthotricycloheptene; the cell dimensions, molecular origin and Euler angles for the two compounds are compared in Table 3.

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