Table 3. *Intermolecular distances shorter than* 3·6 Å

Roman numerals represent equivalent points, (i) x,y,z; (ii) $-x, \frac{1}{2}+y, -z$; and the other symbols represent cell translations.

O(10)···C(3, ii + a + c)	3·24 Å	O(11)···C(2, i + b − c)	3·41 Å
O(10)···C(3, ii + a)	3·32	O(11)···C(2, i + b)	3·51
O(10)···C(2, ii + a + c)	3·45	N(8)···C(6, ii − b + c)	3·46
O(11)···N(9, i − c)	3·32	N(8)···C(6, ii − b)	3·55
O(11)···O(10, i − c)	3·32	N(8)···C(5, i − b)	3·54
O(11)···C(4, i − c)	3·48	C(3)···C(4, i + c)	3·48
O(11)···C(7, i + b − c)	3·37	C(1)···C(6, i + c)	3·49
O(11)···N(8, i + b − c)	3·39		

related by screw axes at $x = 0$. In the belt-shaped chain, O(11) and the cyano C(7) of the successive molecules are arranged closely together, while in the molecular column along **c** O(11) and the nitro N(9) are in close contact.

From the viewpoint of intermolecular interactions, it is worth noting that there is no close contact suggesting dipole–dipole interactions between cyano groups or between nitro groups. But there is an anti-parallel close contact between the cyano and nitro groups of successive molecules within the belt-shaped chain, suggesting interactions between local dipoles.

The molecular arrangement described above seems better explained by Coulombic interactions, if we assume some residual charge on each atom: negative charges on O and N(cyano), positive charges on N(nitro), C and H. It may also be added that this assumption is supported qualitatively by a CNDO/2 calculation.

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Perfluorododecahydrotetracyclopentacyclooctene

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Abstract. $C_{20}F_{24}$, $M_r = 696{\cdot}2$, monoclinic, space group $C2/c$, $a = 15{\cdot}368$ (7), $b = 15{\cdot}326$ (7), $c = 10{\cdot}066$ (4) Å, $\beta = 105{\cdot}75$ (3)°, $V = 2281{\cdot}8$ Å^3, $Z = 4$, $D_c = 2{\cdot}026$, $D_o = 2{\cdot}01$ g cm^{-3}. The structure was solved by direct methods from Mo $K\alpha$ X-ray diffractometer intensities and refined by full-matrix least squares to a final R of 0·077 for 1200 observed reflexions (assuming space group $C2/c$). The molecule has crystallographic twofold symmetry and approximate $\bar{4}2m$ symmetry with the cyclooctatetraene ring in a boat conformation.

Introduction. Initial unit-cell dimensions were determined from photographs (Cu $K\alpha$ radiation) and absent reflexions hkl when $(h + k) = 2n + 1$ and $h0l$ when $l = 2n + 1$ indicated the space group to be either Cc or $C2/c$. Crystals sublimed at room temperature and were sealed in Lindemann-glass capillaries. Accurate cell dimensions were obtained by least-squares refinement of the setting angles of nine reflexions with 2θ values in the range 35–41°, centred on a Picker FACS-I four-circle diffractometer [λ(Mo $K\alpha_1$) = 0·70926 Å]. The crystal used for intensity measurements was a rectangular parallelepiped 0·18 × 0·30 × 0·25 mm and was mounted on the diffractometer with **b** off-set from the φ axis. Nb-filtered Mo radiation and a scintillation counter with pulse-height analysis were used. A θ–2θ scan was used with a scan speed of 2° min^{-1} and a scan width of 1·4° for reflexions with $2\theta < 35°$ and

1·5° for the remainder (increased for dispersion). Stationary background counts of 20 s were made at the limits of the scan. Reflexions with $2\theta \le 50°$ were measured, of which 1200 out of 2006 were accepted as observed $[I \ge 2{\cdot}3\sigma(I)]$. Two standard reflexions were measured every 60 reflexions. Lorentz and polarization but no absorption corrections were applied [μ(Mo $K\alpha$) = 2·83 cm^{-1}].

The space group was assumed to be $C2/c$ on the basis of the intensity statistics. With Z = 4, the molecule has either a centre of symmetry or twofold symmetry. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). Sets of phases for 222 reflexions with $E > 1{\cdot}5$ were generated and an E map using the phases from the set with the fifth highest combined figure of merit located 21 out of 22 atoms of the asymmetric unit of a recognizable molecule with twofold symmetry. However, the molecule was positioned incorrectly in the unit cell (being too close to a centre of symmetry) and packing considerations indicated a translation of ~1·5 Å parallel to **b** to give a reasonable model. The remaining F atom was generated in its expected position and full-matrix least-squares refinement initially with isotropic and finally anisotropic thermal parameters for all atoms gave a final R = 0·077. A difference map did not reveal any unusual features. The weighting scheme was $w = 1/\sigma(F)^2$ and an analysis as a function of $|F_o|$, $\sin\theta/\lambda$ and Miller indices showed no unusual variation of $w(|F_o| - |F_c|)^2$. On the last cycle the largest shift to estimated standard deviation ratio for any parameter was 0·04. The possibility that the space group is Cc could not be ruled out. Refinement in this space group was not considered worthwhile in view of the close approximation to $C2/c$ and the high correlation coefficients which would result.

Table 1. *Fractional atomic coordinates* ($\times 10^4$)

	x	y	z
C(1)	194 (3)	2004 (3)	1914 (5)
C(2)	67 (4)	1266 (3)	924 (6)
C(3)	376 (4)	1636 (4)	−292 (6)
C(4)	1029 (4)	2355 (4)	358 (6)
C(5)	726 (3)	2609 (3)	1610 (5)
C(6)	1013 (3)	3430 (3)	2373 (5)
C(7)	2007 (4)	3657 (4)	2933 (7)
C(8)	2007 (4)	4429 (4)	3884 (8)
C(9)	1041 (4)	4771 (4)	3445 (7)
C(10)	495 (3)	4047 (3)	2686 (5)
F(1)	−801 (2)	991 (2)	522 (3)
F(2)	561 (2)	567 (2)	1464 (3)
F(3)	758 (3)	1030 (2)	−913 (4)
F(4)	−331 (3)	1964 (3)	−1208 (4)
F(5)	1885 (2)	2054 (2)	743 (4)
F(6)	1014 (3)	3025 (2)	−496 (4)
F(7)	2501 (2)	2989 (2)	3551 (4)
F(8)	2350 (2)	3895 (3)	1880 (4)
F(9)	2628 (3)	5013 (3)	3852 (6)
F(10)	2181 (3)	4116 (3)	5175 (5)
F(11)	1000 (2)	5482 (2)	2649 (4)
F(12)	773 (2)	5042 (2)	4532 (4)

The programs used have been cited elsewhere (Einstein & Jones, 1972). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Final positional parameters are listed in Table 1* and interatomic distances and angles in Table 2. Fig. 1

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32566 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Interatomic distances and angles*

	Uncorrected	Corrected for libration
C(1)–C(1′)	1·459 (9) Å	1·464 Å
C(1)–C(5)	1·322 (6)	1·324
C(5)–C(6)	1·472 (7)	1·475
C(6)–C(10)	1·325 (7)	1·327
C(10)–C(10′)	1·464 (9)	1·469
C(1)–C(2)	1·480 (7)	1·483
C(2)–C(3)	1·535 (7)	1·538
C(3)–C(4)	1·510 (8)	1·512
C(4)–C(5)	1·508 (7)	1·508
C(6)–C(7)	1·518 (7)	1·524
C(7)–C(8)	1·516 (9)	1·520
C(8)–C(9)	1·521 (8)	1·526
C(9)–C(10)	1·468 (7)	1·472
C(2)–F(1)	1·350 (6)	1·379
C(2)–F(2)	1·334 (6)	1·349
C(3)–F(3)	1·337 (6)	1·359
C(3)–F(4)	1·317 (7)	1·351
C(4)–F(5)	1·348 (7)	1·376
C(4)–F(6)	1·331 (6)	1·367
C(7)–F(7)	1·321 (6)	1·355
C(7)–F(8)	1·354 (6)	1·388
C(8)–F(9)	1·310 (7)	1·366
C(8)–F(10)	1·341 (8)	1·383
C(9)–F(11)	1·339 (6)	1·356
C(9)–F(12)	1·335 (6)	1·362

	Uncorrected		Uncorrected
C(1′)–C(1)–C(5)	126·9 (4)°	C(5)–C(6)–C(10)	127·8 (5)°
C(1′)–C(1)–C(2)	122·4 (3)	C(5)–C(6)–C(7)	121·1 (5)
C(2)–C(1)–C(5)	110·6 (4)	C(7)–C(6)–C(10)	111·1 (5)
C(1)–C(2)–C(3)	104·1 (4)	C(6)–C(7)–C(8)	103·8 (5)
C(1)–C(2)–F(1)	112·1 (4)	C(6)–C(7)–F(7)	112·9 (5)
C(1)–C(2)–F(2)	112·1 (5)	C(6)–C(7)–F(8)	109·7 (5)
C(3)–C(2)–F(1)	111·6 (5)	C(8)–C(7)–F(7)	113·5 (6)
C(3)–C(2)–F(2)	110·7 (4)	C(8)–C(7)–F(8)	110·7 (5)
F(1)–C(2)–F(2)	106·4 (4)	F(7)–C(7)–F(8)	106·3 (5)
C(2)–C(3)–C(4)	103·6 (5)	C(7)–C(8)–C(9)	104·4 (5)
C(2)–C(3)–F(3)	112·9 (5)	C(7)–C(8)–F(9)	112·7 (6)
C(2)–C(3)–F(4)	108·8 (5)	C(7)–C(8)–F(10)	107·6 (6)
C(4)–C(3)–F(3)	112·3 (5)	C(9)–C(8)–F(9)	114·8 (6)
C(4)–C(3)–F(4)	110·7 (5)	C(9)–C(8)–F(10)	108·9 (5)
F(3)–C(3)–F(4)	108·5 (5)	F(9)–C(8)–F(10)	108·1 (6)
C(3)–C(4)–C(5)	103·2 (4)	C(8)–C(9)–C(10)	105·6 (5)
C(3)–C(4)–F(5)	111·1 (5)	C(8)–C(9)–F(11)	109·5 (5)
C(3)–C(4)–F(6)	112·6 (6)	C(8)–C(9)–F(12)	111·2 (5)
C(5)–C(4)–F(5)	110·3 (5)	C(10)–C(9)–F(11)	111·7 (5)
C(5)–C(4)–F(6)	112·9 (4)	C(10)–C(9)–F(12)	113·5 (4)
F(5)–C(4)–F(6)	106·9 (4)	F(11)–C(9)–F(12)	105·3 (4)
C(1)–C(5)–C(4)	111·4 (4)	C(6)–C(10)–C(9)	111·1 (4)
C(1)–C(5)–C(6)	126·1 (5)	C(6)–C(10)–C(10′)	126·1 (4)
C(4)–C(5)–C(6)	122·5 (4)	C(9)–C(10)–C(10′)	122·8 (4)

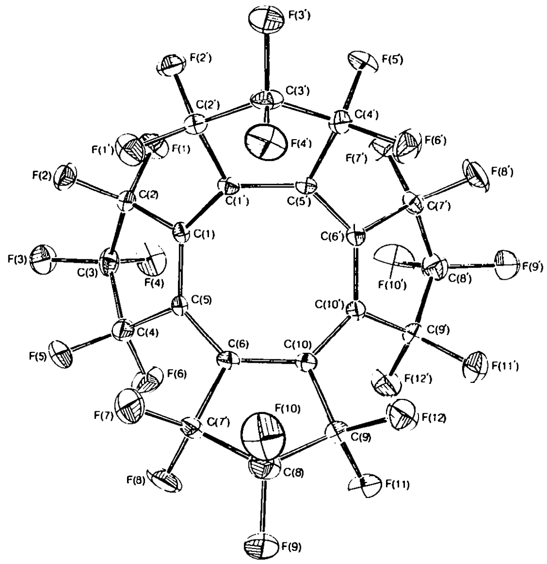


Fig. 1. Perspective view of the molecule drawn by *ORTEP* (Johnson, 1965). The thermal ellipsoids are shown with 20% probability.

shows the molecular conformation and the atom labelling.

Discussion. The molecule has crystallographic twofold symmetry, the axis passing through two bonds of the cyclooctatetraene ring. Bond lengths were corrected for thermal motion. Analysis of the thermal parameters in terms of rigid-body motion of the whole molecule (Schomaker & Trueblood, 1968) indicated independent motion of the F atoms (r.m.s. ΔU_{ij} = 0·0146 Å^2 compared with an average σU_{ij} of 0·0029 Å^2). Treatment of only the C-atom skeleton as a rigid-body gave reasonable agreement between observed U_{ij} values and those calculated from the **T**, **L** and **S** tensors (r.m.s. ΔU_{ij} = 0·0047 Å^2). Bond distances were corrected for libration by assuming riding motion for the F atoms and rigid-body motion for the C-atom skeleton. The corrected interatomic distances are shown in Table 2.

The cyclooctatetraene ring is in a boat conformation with approximate $\bar{4}2m$ symmetry and is very similar to cyclooctatetraene (C_8H_8) in the vapour phase (Trætteberg, 1966).

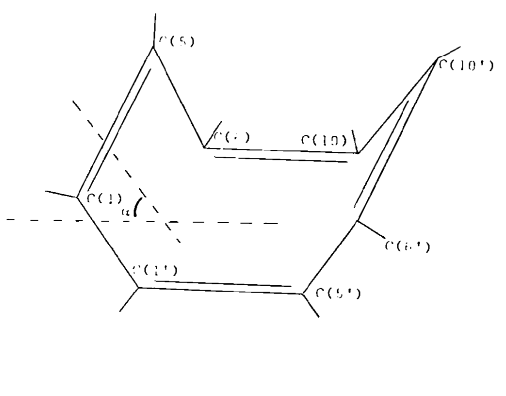


Table 3. *Deviations of atoms* (Å) *from least-squares planes*

The equations of the planes are referred to orthogonal axes *a*, *b′* and *c**.

(*a*) Plane through C(1′), C(5′), C(6) and C(10)

$$-0.3166x + 0.0006y - 0.9485z + 2.4696 = 0$$

C(1′)	−0·003	C(1)	0·784	
C(5′)	0·004	C(5)	0·778	$\chi^2 = 2.01$
C(6)	0·004	C(6′)	0·779	
C(10)	−0·003	C(10′)	0·788	

(*b*) Plane through C(1′), C(1), C(5) and C(6)

$$-0.6122x + 0.4691y - 0.5608z - 0.5375 = 0$$

C(1′)	−0·004	C(2)	−0·032	
C(1)	0·008	C(4)	−0·061	$\chi^2 = 7.47$
C(5)	−0·009			
C(6)	0·004			

(*c*) Plane through C(5′), C(6′), C(10′) and C(10)

$$0.2145x - 0.4700y - 0.8562z + 5.1192 = 0$$

C(5′)	0·001	C(7′)	0·002	
C(6′)	−0·004	C(9′)	−0·022	$\chi^2 = 1.06$
C(10′)	0·003			
C(10)	−0·001			

(*d*) Plane through C(1), C(2), C(4), and C(5)

$$-0.6642x + 0.4596y - 0.5896z - 0.4587 = 0$$

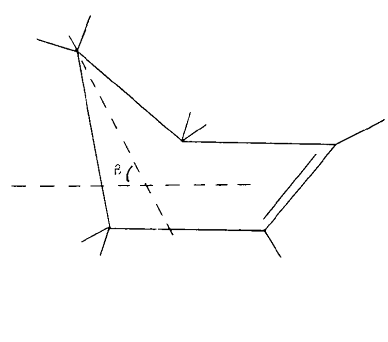
C(1)	0·001	C(3)	0·417	
C(2)	−0·001	C(1′)	−0·060	$\chi^2 = 0.055$
C(4)	0·001	C(6)	−0·017	
C(5)	−0·001			

(*e*) Plane through C(6), C(7), C(9) and C(10)

$$0.2130x + 0.4624y - 0.8607z - 0.6234 = 0$$

C(6)	0·007	C(8)	−0·310	
C(7)	−0·006	C(5)	0·016	$\chi^2 = 5.33$
C(9)	0·006	C(10′)	0·002	
C(10)	−0·007			

C(1′), C(5′), C(6) and C(10) form the base of the boat and are planar, and the dihedral angle, α, of 42° is close to that of 43·1 (5)° for C_8H_8 (Beagley, 1973). Bond lengths and angles of the ring are similar in both compounds; average corrected values are C–C = 1·471 (5), C=C = 1·326 (5) Å, ∠C=C–C = 126·7 (2)° in $C_{20}F_{24}$ compared with 1·478 (1), 1·342 (1) Å and 126·1 (5)° for C_8H_8.



The cyclopentene rings are non-planar and have a partly opened envelope conformation. Calculations for relevant planes are given in Table 3. The flap angles, β, for the two crystallographically non-equivalent cyclopentene rings are 26 and 19° respectively for rings defined by C(1) to C(5) and C(6) to C(10). These values are comparable with the angle of 21·9 (2)° in perfluorocyclopentene (Chang & Bauer, 1971) but smaller than that of 29 (1)° in cyclopentene (Davis & Muecke, 1970). Average corrected bond lengths for the cyclopentene rings are similar to those of perfluorocyclopentene (shown in square brackets); –C=C– = 1·326 (5) [1·342], =C–C– = 1·497 (4) [1·510 (9)], –C–C– = 1·524 (4) [1·539 (13) Å]. The C–F lengths are comparable with those in perfluoro-1,2-3,4-5,6-triethanobenzene (Cobbledick & Einstein, 1976). There are no particularly short intermolecular contacts.

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2-Formylamino-2-oxo-5,5-dimethyl-1,3,2-dioxaphosphorinane

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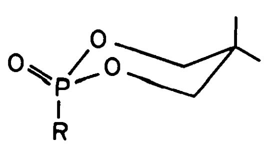
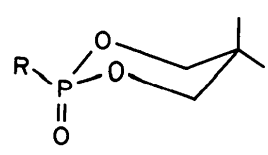
Abstract. $C_6H_{12}NO_4P$, M_r = 193·1, orthorhombic, *Pbca*, a = 10·17 (1), b = 10·01 (1), c = 18·36 (2) Å, Z = 8, D_c = 1·372, D_m = 1·360 g cm^{-3}, μ(Cu $K\alpha$) = 24·7 cm^{-1}, λ = 1·5418 Å. The structure was solved by the heavy-atom method and refined to R = 0·08. The molecule has the chair configuration with the phosphoryl O atom equatorial. This atom forms a hydrogen bond to the amide group of an adjacent molecule.

Introduction. Most of the information on the preferred configuration of the P substituents in the chair form of 2-R-2-oxo-1,3,2-dioxaphosphorinanes suggests that the molecule with the O atom equatorial (I*a*) rather than axial (I*b*) is thermodynamically more stable (White, McEwen, Bertrand & Verkade, 1971). However, when R = NMe_2 this O is axial (Wadsworth, 1977), although it is equatorial when R = NHPh (Cameron & Karolak-Wojciechowska, 1976). The present structure, where R = NHCHO, has been examined to see if a strong electronegative group at the N atom has any influence on the P configuration.



(I*a*)



(I*b*)

The compound was prepared by A. Zwierzak (Technical University, Łodz). The crystal system and cell dimensions were determined from Weissenberg photographs; systematic absences 0*kl*, $k = 2n + 1$; *h*0*l*, $l = 2n + 1$; *hk*0, $h = 2n + 1$ uniquely determined the space group *Pbca*. 1030 independent reflexions were visually estimated from equi-inclination Weissenberg

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