zero Δ ($= b - a$) value (where a and b are the *ipso–ortho* and *ortho–meta* C—C bond lengths respectively). In the crystal structure there are close non-bonded C—H···O intermolecular contacts, C(2)···O(1) ($x, -\frac{1}{2} - y, -\frac{1}{2} + z$) 3·050 (6) Å and C(7)···O(4) ($x, \frac{1}{2} - y, -\frac{1}{2} + z$) 3·254 (6) Å, somewhat longer than those reported for phenacyl phenyl sulfone (Krawiec, Krygowski & Zakrzewski, 1989).

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Structure of the Tetrabutylammonium Salt of 2,4,4,6,6-Pentachloro-1,3,5,2λ^5,4λ^5,6λ^5-triazatriphosphorinine 2-Oxide

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Abstract. [N(C$_4$H$_9$)$_4$][N$_3$P$_3$Cl$_5$O], $M_r = 570·67$, monoclinic, $P2_1/c$, $a = 10·184$ (2), $b = 18·072$ (3), $c = 16·448$ (2) Å, $\beta = 106·88$ (2)°, $V = 2896·8$ (9) Å^3, $Z = 4$, $D_x = 1·308$ g cm^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71073 Å, $\mu = 6·8$ cm^{-1}, $F(000) = 1192$, $T = 298$ K, $R_F = 0·082$, $wR = 0·069$ for 2089 unique observed reflections with $I \geq 1·5\sigma(I)$ and 263 parameters. The inorganic ring skeleton is nearly flat, tending somewhat to a crown conformation. The P—N bond lengths (mean values) are: N—P(OCl) 1·606 (5) Å and N—P(Cl$_2$) 1·540 (5), 1·58 (1) Å.

Experimental. Suitable crystals were obtained by recrystallization from a heptane–chloroform mixture. A block-shaped, transparent, colourless crystal (0·07 × 0·15 × 0·20 mm) was glued on top of a glass fibre and transferred to an Enraf-Nonius CAD-4F diffractometer interfaced to a MicroVAX 2000 computer. Graphite-monochromated Mo $K\bar{\alpha}$ radiation used to obtain 4957 reflections (h −12→0, k 0→22, l −12→19); $1·13 < \theta < 26·0°$; $[(\sin\theta)]_{\max}/\lambda = 0·6168$ Å^{-1}; $\omega/2\theta$ scan, $\Delta\omega = (1·0 + 0·35 \operatorname{tg}\theta)°$. Unit-cell parameters and their e.s.d.'s were derived from setting angles of 22 reflections ($13·84 < \theta < 16·87°$) in four alternate settings (de Boer & Duisenberg, 1984). Reduced cell calculations did not indicate any higher lattice symmetry (Spek, 1988). Three reference reflections measured every 2 h (012: r.m.s.d. 2·6%, $\bar{2}00$: r.m.s.d. 2·5%, $\bar{1}04$: r.m.s.d. 2·9%) showed a linear decay of 11·8% during the 89·5 h of X-ray exposure time. A 360° ψ scan for a reflection close to axial ($\bar{4}2\bar{1}$) showed a variation in intensity of up to 10% about the mean value. The intensity data were corrected for this decay, for Lorentz and polarization effects, but not for absorption. Standard deviations in the intensities based on counting statistics were increased according to an analysis of the excess variance (McCandlish, Stout &

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Table 1. *Final fractional atomic coordinates and equivalent isotropic thermal parameters for non-*H *atoms, with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
Residue (1): $[N_3P_3OCl_5]^-$				
Cl(1)	0·4501 (3)	0·0821 (2)	0·2685 (2)	0·106 (1)
Cl(2)	0·1386 (3)	0·3442 (2)	0·1854 (2)	0·123 (2)
Cl(3)	0·0748 (4)	0·1987 (2)	0·2583 (2)	0·137 (2)
Cl(4)	0·1945 (3)	0·1905 (1)	−0·0616 (1)	0·081 (1)
Cl(5)	0·1369 (3)	0·0569 (1)	0·0360 (2)	0·092 (1)
P(1)	0·4448 (3)	0·1776 (1)	0·2003 (2)	0·0633 (9)
P(2)	0·1895 (3)	0·2382 (1)	0·1880 (2)	0·071 (1)
P(3)	0·2222 (2)	0·1575 (1)	0·0577 (1)	0·0540 (8)
O(1)	0·5849 (5)	0·2048 (3)	0·2189 (3)	0·079 (2)
N(1)	0·3440 (7)	0·2305 (4)	0·2338 (4)	0·072 (3)
N(2)	0·1255 (6)	0·2062 (4)	0·0968 (4)	0·065 (3)
N(3)	0·3768 (6)	0·1525 (4)	0·1039 (4)	0·056 (3)
Residue (2): $[NBu_4]^+$				
N(4)	0·7055 (6)	0·2692 (3)	0·4748 (4)	0·058 (3)
C(1)	0·5710 (8)	0·2317 (4)	0·4728 (5)	0·072 (4)
C(2)	0·5760 (9)	0·1780 (5)	0·5432 (6)	0·090 (4)
C(3)	0·439 (1)	0·1418 (6)	0·5291 (9)	0·160 (8)
C(4)	0·420 (1)	0·0868 (7)	0·572 (1)	0·27 (1)
C(5)	0·8187 (8)	0·2130 (4)	0·4839 (5)	0·068 (3)
C(6)	0·7967 (9)	0·1538 (5)	0·4162 (5)	0·083 (4)
C(7)	0·922 (1)	0·1055 (5)	0·4363 (6)	0·115 (5)
C(8)	0·914 (1)	0·0483 (5)	0·3751 (6)	0·143 (6)
C(9)	0·7494 (8)	0·3227 (4)	0·5495 (5)	0·063 (3)
C(10)	0·6683 (9)	0·3918 (4)	0·5451 (5)	0·068 (4)
C(11)	0·726 (1)	0·4376 (4)	0·6244 (5)	0·088 (4)
C(12)	0·645 (1)	0·5077 (4)	0·6235 (5)	0·114 (6)
C(13)	0·6761 (8)	0·3094 (4)	0·3906 (5)	0·065 (3)
C(14)	0·7947 (8)	0·3509 (5)	0·3756 (5)	0·068 (3)
C(15)	0·748 (1)	0·3919 (6)	0·2941 (6)	0·108 (5)
C(16)	0·862 (1)	0·4301 (6)	0·2690 (6)	0·138 (7)

Table 2. *Selected data on the geometry*

Residue (1): $[N_3P_3OCl_5]^-$

Bond distances (Å)

Cl(1)—P(1)	2·052 (4)	P(1)—N(1)	1·612 (8)
Cl(2)—P(2)	1·983 (4)	P(1)—N(3)	1·601 (7)
Cl(3)—P(2)	1·998 (5)	P(2)—N(1)	1·541 (8)
Cl(4)—P(3)	1·991 (3)	P(2)—N(2)	1·562 (7)
Cl(5)—P(3)	2·002 (3)	P(3)—N(2)	1·589 (7)
P(1)—O(1)	1·455 (6)	P(3)—N(3)	1·539 (7)

Bond angles (°)

Cl(1)—P(1)—O(1)	107·5 (3)	Cl(4)—P(3)—Cl(5)	99·6 (1)
N(1)—P(1)—N(3)	111·8 (4)	P(1)—N(1)—P(2)	123·2 (4)
N(1)—P(2)—N(2)	121·2 (4)	P(2)—N(2)—P(3)	117·3 (4)
Cl(2)—P(2)—Cl(3)	99·5 (2)	P(1)—N(3)—P(3)	124·0 (4)
N(2)—P(3)—N(3)	119·8 (4)		

Residue (2): $[NBu_4]^+$

Bond distances (Å)

N(4)—C(1)	1·52 (1)	C(6)—C(7)	1·50 (1)
N(4)—C(5)	1·51 (1)	C(7)—C(8)	1·43 (1)
N(4)—C(9)	1·53 (1)	C(9)—C(10)	1·49 (1)
N(4)—C(13)	1·52 (1)	C(10)—C(11)	1·51 (1)
C(1)—C(2)	1·50 (1)	C(11)—C(12)	1·51 (1)
C(2)—C(3)	1·49 (2)	C(13)—C(14)	1·50 (1)
C(3)—C(4)	1·26 (2)	C(14)—C(15)	1·48 (1)
C(5)—C(6)	1·51 (1)	C(15)—C(16)	1·51 (2)

Bond angles (°)

C(1)—N(4)—C(5)	111·1 (5)	C(5)—N(4)—C(9)	108·0 (6)
C(1)—N(4)—C(9)	110·6 (6)	C(5)—N(4)—C(13)	110·8 (6)
C(1)—N(4)—C(13)	104·9 (6)	C(9)—N(4)—C(13)	111·5 (5)

Andrews, 1975) of the three reference reflections: $\sigma^2(I) = \sigma^2_{cs}(I) + (0{\cdot}065I)^2$, where $\sigma^2_{cs}(I)$ represents the contribution from counting statistics. Equivalent reflections were averaged, resulting in 2089 reflections satisfying the $I \geq 1{\cdot}5\sigma(I)$ criterion of observability (due to weak scattering power of the crystal). Structure was solved by Patterson methods and subsequent partial structure expansion (*SHELXS*86; Sheldrick, 1986) and refined on F by block-diagonal least-squares techniques with anisotropic thermal parameters for the non-H atoms. The H atoms were included with their positions calculated by using sp^3 hybridization at the corresponding C atom and a fixed C—H distance of 1·0 Å and refined in the riding mode with one common isotropic temperature factor. Convergence with 263 parameters was reached at $R_F = 0{\cdot}082$; $wR = 0{\cdot}069$, $w = 1/\sigma^2(F)$; $S = 1{\cdot}265$; average $\Delta/\sigma = 0{\cdot}017$, max. $\Delta/\sigma = 0{\cdot}308$. Relatively high values of R can be ascribed to crystal instability. Min. and max. residual densities in final Fourier map $-0{\cdot}53$ and $0{\cdot}42$ e Å^{-3}. High thermal

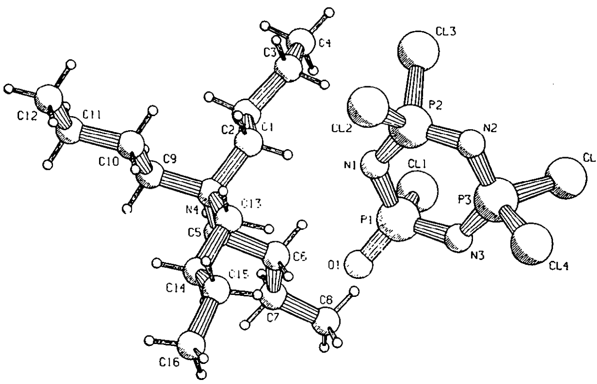

Fig. 1. *PLUTO* drawing of the molecule illustrating the puckering and the adopted numbering scheme.

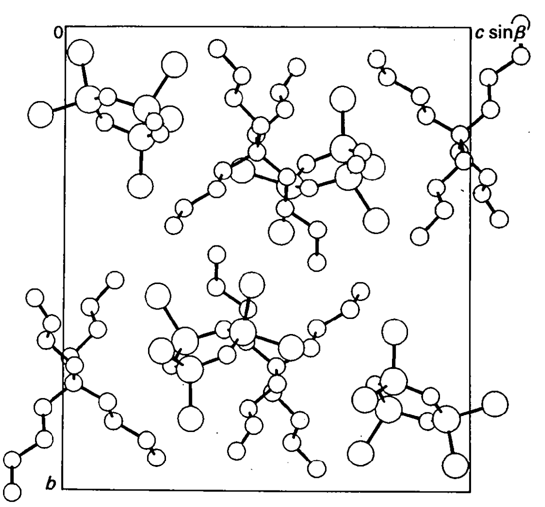

Fig. 2. Projection of the crystal structure down [100].

motion but no resolvable disorder was found at the C(4)-atom position, resulting in an observed short bond distance of C(3)—C(4). Scattering factors were taken from Cromer & Mann (1968). Anomalous-dispersion factors are those given by Cromer & Liberman (1970). All calculations carried out on the CDC-Cyber 170/760 computer of the University of Groningen with the program packages *XTAL* (Hall & Stewart, 1987), *EUCLID* (Spek, 1982; calculations of geometric data) and a locally modified version of the program *PLUTO* (Meetsma, 1986; Motherwell & Clegg, 1978; preparation of illustrations). Final fractional atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms are presented in Table 1.* Intramolecular bond distances and angles are summarized in Table 2. Fig. 1 shows the molecular struture with the adopted numbering scheme; Fig. 2 shows the arrangement of the compound in the unit cell viewed along **a**.

The observed conformation of the inorganic PNP ring approaches that of a crown with torsion angles ranging from $-17{\cdot}3$ (7) to $15{\cdot}5$ (7)° and lowest asymmetry parameter value ΔC_2 [N(1)—P(2), P(1)—N(3)] = 2·2 (7)° (Duax, Weeks & Rohrer, 1976). Puckering parameters are $Q = 0{\cdot}138$ (6) Å, $\theta = 151$ (2)°, $\varphi = 164$ (5)° [Cremer & Pople (1975); e.s.d.'s following Norrestam (1981)].

Related literature. The structural parameters of the inorganic ring skeleton can be compared with those found in related structures: $[(NSOCl)_2NPClO]^-$ (van Bolhuis, de Ruiter & van de Grampel, 1981), $[(NSOClNHPO_2NPCl_2]^-$ (Winter, 1986) and $[NPHBEt_3(NPF)_2CpFeCp]^-$ (Manners, Coggio, Mang, Parvez & Allcock, 1989).

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* Lists of structure factors, anisotropic thermal parameters of the non-H atoms, H-atom parameters, bond lengths and angles excluding H and an *ORTEP* plot (Johnson, 1965) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52518 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Room-Temperature Structure of Cordiaquinone B

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Abstract. $C_{21}H_{24}O_3$, $M_r = 324{\cdot}42$, monoclinic, $P2_1$, $a = 7{\cdot}729$ (1), $b = 7{\cdot}180$ (1), $c = 16{\cdot}206$ (3) Å, $\beta = 100{\cdot}24$ (1)°, $V = 885{\cdot}1$ (5) Å^3, $Z = 2$, $D_x = 1{\cdot}21$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}05$ mm^{-1}, $F(000) = 346$, $T = 293$ K, $R = 0{\cdot}041$ for 1155 unique observed reflections. The naphtho-

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