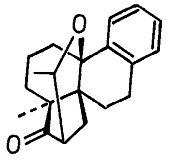
(122 and 21$\bar{2}$) monitored periodically showed no significant variation in intensity. No absorption correction. Lorentz–polarization correction. Structure determined by direct phasing using *MITHRIL* (Gilmore, 1984). H atoms located in a difference Fourier synthesis. Full-matrix least-squares calculations on F with *SHELX76* (Sheldrick, 1976); anisotropic thermal parameters for C and O atoms and isotropic for H. Convergence at $R = 0{\cdot}036$, $wR = 0{\cdot}038$, $\Delta/\sigma < 0{\cdot}1$, with $w = 1/\sigma^2(|F_o|)$. Final $\Delta\rho$ max. 0·15, min. −0·18 e Å^{-3}. Scattering factors for C, H and O atoms were those incorporated in *SHELX76*.



(I)

Atomic coordinates are listed in Table 1 and molecular dimensions in Table 2.* Fig. 1, drawn with *ORTEP* (Johnson, 1965), illustrates the molecular structure.

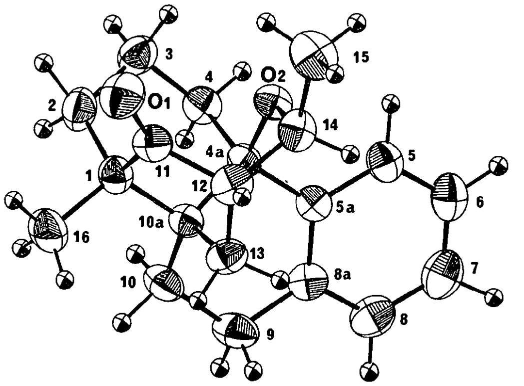


Fig. 1. Molecular structure and atomic numbering (Ghatak *et al.*, 1980). The thermal ellipsoids of the C and O atoms are drawn at the 50% probability level and the H atoms are represented by spheres of radius 0·1 Å.

Related literature. For the preparation of the compound see Ghatak, Sanyal, Ghosh, Sarkar, Raju & Wenkert (1980).

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Structure of 3,3,5,5,7,7-Hexaphenyl-1,2,4,6,3,5,7-tetraazatriphosphepine Hydrochloride Monohydrate

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Abstract. $C_{36}H_{32}N_4P_3^+.Cl^-.H_2O$, $NHNHPPh_2NPPh_2NPPh_2^+.Cl^-.H_2O$, $M_r = 667{\cdot}1$, monoclinic, $P2_1/n$, $a = 8{\cdot}646\,(4)$, $b = 20{\cdot}969\,(4)$, $c = 19{\cdot}276\,(3)$ Å, $\beta = 96{\cdot}14\,(3)°$, $V = 3475\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}27$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 2{\cdot}77$ cm^{-1}, $F(000) = 1392$, $T = 293$ K, $R = 0{\cdot}047$ for 3805 unique observed reflections. The highly puckered P_3N_4 ring has P—N distances of 1·568 (2) to 1·659 (2) Å, N—N of

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1·430 (3) Å, P angles of 112·0 (1) to 118·7 (1)°, PNP angles of 132·3 (3)°, and PNN angles of 112·2 (7)°. Two water molecules and two chloride ions form a hydrogen-bonded trapezoid around a crystallographic center; the cation is hydrogen-bonded to both the water and the chloride ion *via* the N—H hydrogen atoms.

Table 1. *Fractional atomic coordinates, equivalent isotropic thermal parameters, and their e.s.d.'s*

	x	y	z	B_{eq}(Å^2)
Cl	0·4512 (1)	0·09605 (6)	0·41822 (6)	4·70 (2)
P(1)	0·5844 (1)	0·12497 (4)	0·69748 (5)	2·45 (2)
P(2)	0·5370 (1)	0·10518 (5)	0·84199 (5)	2·70 (2)
P(3)	0·4285 (1)	−0·01294 (4)	0·77534 (5)	2·42 (2)
O	0·3272 (3)	0·0227 (2)	0·5445 (1)	4·89 (7)
N(1)	0·6204 (4)	0·1328 (2)	0·7785 (2)	3·11 (7)
N(2)	0·4309 (3)	0·0429 (1)	0·8296 (2)	2·79 (6)
N(3)	0·5229 (3)	0·0058 (1)	0·7076 (1)	2·54 (6)
N(4)	0·4676 (3)	0·0644 (1)	0·6761 (1)	2·59 (6)
C(1)	0·4784 (4)	0·1904 (2)	0·6560 (2)	2·93 (8)
C(2)	0·4390 (5)	0·1908 (2)	0·5837 (2)	4·4 (1)
C(3)	0·3494 (7)	0·2380 (3)	0·5527 (3)	6·7 (1)
C(4)	0·2961 (7)	0·2860 (3)	0·5931 (3)	8·0 (2)
C(5)	0·3371 (7)	0·2879 (2)	0·6639 (3)	7·4 (2)
C(6)	0·4289 (5)	0·2393 (2)	0·6971 (3)	4·7 (1)
C(7)	0·7624 (4)	0·1163 (2)	0·6595 (2)	2·82 (7)
C(8)	0·8990 (5)	0·1412 (2)	0·6923 (2)	4·6 (1)
C(9)	1·0364 (5)	0·1356 (3)	0·6591 (3)	6·2 (1)
C(10)	1·0363 (5)	0·1054 (3)	0·5976 (3)	5·9 (1)
C(11)	0·9021 (5)	0·0797 (3)	0·5658 (3)	6·0 (1)
C(12)	0·7651 (5)	0·0852 (2)	0·5971 (2)	4·5 (1)
C(13)	0·6879 (4)	0·0909 (2)	0·9122 (2)	3·16 (8)
C(14)	0·6491 (5)	0·0647 (2)	0·9740 (2)	4·1 (1)
C(15)	0·7623 (5)	0·0525 (2)	1·0288 (2)	5·0 (1)
C(16)	0·9140 (6)	0·0679 (3)	1·0223 (2)	6·0 (1)
C(17)	0·9557 (6)	0·0935 (3)	0·9615 (3)	6·7 (1)
C(18)	0·8416 (5)	0·1053 (2)	0·9066 (2)	5·1 (1)
C(19)	0·4161 (5)	0·1658 (2)	0·8748 (2)	3·27 (8)
C(20)	0·2602 (5)	0·1700 (2)	0·8525 (3)	5·3 (1)
C(21)	0·1703 (6)	0·2189 (3)	0·8732 (3)	7·2 (2)
C(22)	0·2339 (7)	0·2647 (2)	0·9169 (3)	7·2 (1)
C(23)	0·3879 (8)	0·2614 (2)	0·9410 (3)	7·7 (2)
C(24)	0·4790 (6)	0·2121 (2)	0·9199 (3)	5·8 (1)
C(25)	0·2298 (4)	−0·0328 (2)	0·7469 (2)	2·91 (8)
C(26)	0·1095 (4)	−0·0116 (2)	0·7836 (2)	3·86 (9)
C(27)	−0·0422 (5)	−0·0280 (3)	0·7613 (3)	5·4 (1)
C(28)	−0·0750 (5)	−0·0639 (3)	0·7027 (3)	6·1 (1)
C(29)	0·0418 (6)	−0·0844 (3)	0·6658 (3)	6·6 (1)
C(30)	0·1954 (5)	−0·0691 (2)	0·6880 (2)	5·1 (1)
C(31)	0·5293 (4)	−0·0830 (2)	0·8095 (2)	2·98 (8)
C(32)	0·5868 (5)	−0·0854 (2)	0·8784 (2)	4·6 (1)
C(33)	0·6653 (6)	−0·1386 (3)	0·9064 (3)	6·1 (1)
C(34)	0·6892 (6)	−0·1888 (2)	0·8655 (3)	6·8 (1)
C(35)	0·6382 (8)	−0·1869 (3)	0·7967 (3)	8·7 (2)
C(36)	0·5561 (7)	−0·1345 (2)	0·7677 (3)	6·5 (1)
H(N3)	0·542 (4)	−0·025 (2)	0·680 (2)	
H(N4)	0·419 (4)	0·060 (2)	0·637 (2)	
H(O1)	0·343 (4)	0·040 (2)	0·514 (2)	
H(O2)	0·385 (5)	−0·016 (2)	0·549 (2)	

Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as $B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table 2. *Selected bond distances* (Å) *and bond angles* (°) *and their e.s.d.'s*

P(1)	N(1)	1·568 (2)	P(1)	C(1)	1·790 (2)
P(1)	N(4)	1·647 (2)	P(1)	C(7)	1·783 (2)
P(2)	N(1)	1·595 (2)	P(2)	C(13)	1·802 (2)
P(2)	N(2)	1·598 (2)	P(2)	C(19)	1·802 (2)
P(3)	N(2)	1·568 (2)	P(3)	C(25)	1·795 (2)
P(3)	N(3)	1·659 (2)	P(3)	C(31)	1·798 (2)
O	H(O1)	0·72 (4)	Cl	O	3·163 (2)
O	H(O2)	0·95 (4)	Cl′	O	3·177 (2)
N(3)	N(4)	1·430 (3)	Cl	N(3)	3·257 (2)
N(3)	H(N3)	0·86 (4)	O	N(4)	2·831 (3)
N(4)	H(N4)	0·83 (4)			

N(1)	P(1)	N(4)	112·4 (1)	P(1)	N(4)	H(N4)	123 (3)
N(1)	P(2)	N(2)	118·7 (1)	N(3)	N(4)	H(N4)	114 (3)
N(2)	P(3)	N(3)	112·0 (1)	C(1)	P(1)	C(7)	108·5 (1)
H(O1)	O	H(O2)	110 (4)	C(13)	P(2)	C(19)	104·7 (1)
P(1)	N(1)	P(2)	132·0 (2)	C(25)	P(3)	C(31)	109·4 (1)
P(2)	N(2)	P(3)	132·6 (2)	Cl	H(N3)	N(3)	165 (4)
P(3)	N(3)	N(4)	111·5 (2)	O	H(N4)	N(4)	163 (4)
P(3)	N(3)	H(N3)	117 (3)	Cl	H(O1)	O	169 (4)
N(4)	N(3)	H(N3)	117 (3)	Cl	H(O2)	O	167 (4)
P(1)	N(4)	N(3)	112·9 (2)				

Experimental. Compound prepared by the literature reaction of hydrazine monohydrochloride with chlorodiphenylphosphine (Schmidpeter & Stoll, 1971). $P_3(C_6H_5)_6N_4H_2^+.Cl^-.H_2O$ crystals obtained from acetonitrile solutions. Colorless data crystal 0·40 × 0·40 × 0·50 mm mounted on glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4 to 16° min^{-1} in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $17 < 2\theta < 21°$. Analytical absorption correction based on crystal shape varied from 0·94 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·60 Å^{-1}, $-11 \leq h \leq 11$, $0 \leq k \leq 25$, $-23 \leq l \leq 0$. Four standard reflections ($31\bar{6}$, $\bar{1}8\bar{4}$, 0,10,0, $\bar{4}51$) varied ±0·3% over 53·7 h of data collection. 6278 reflections measured, 6090 unique ($R_{int} = 0{\cdot}02$), 2285 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$. The water and N-bonded H atoms were refined with fixed isotropic thermal parameters of 1·2 × B of bonded O or N. Phenyl H atoms constrained to idealized (C—H = 0·95 Å) positions with isotropic $B = 1{\cdot}2 \times B$ of bonded C atom. All non-H atoms refined anisotropically for a total of 418 variables. $R = 0{\cdot}047$, $wR = 0{\cdot}062$, $S = 1{\cdot}67$, where non-Poisson $w^{-1} = [\sigma^2(I) + 0{\cdot}0025I^2]/4F^2$. Final $(\Delta/\sigma)_{max} < 0{\cdot}01$,

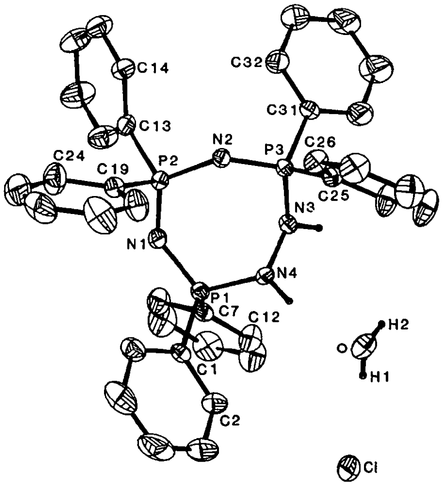

Fig. 1. *ORTEP* diagram (Johnson, 1976) and atom-numberng scheme. Non-H ellipsoids at 30% probability level, H atoms given arbitrary radii.

$\Delta\rho_{\max} = 1{\cdot}03\ (3)$ and $\Delta\rho_{\min} = -0{\cdot}28\ (3)\ \mathrm{e\ Å^{-3}}$ on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP*.* Table 1 gives the atom coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the molecule with the numbering scheme.

Related literature. The title compound has been considered to be both acyclic (Moran & Reider, 1969) and cyclic (Schmidpeter & Stoll, 1971). The present study confirms the latter conformation.

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Structure of 3,5-Diphenyl-4*H*-1,2,4,6-thiatriazine 1-Oxide

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Abstract. $C_{14}H_{11}N_3OS$, $M_r = 269{\cdot}3$, orthorhombic, *Pbca*, $a = 15{\cdot}682\ (4)$, $b = 8{\cdot}872\ (2)$, $c = 18{\cdot}062\ (5)$ Å, $V = 2513\ (2)\ \mathrm{Å^3}$, $Z = 8$, $D_x = 1{\cdot}42\ \mathrm{g\ cm^{-3}}$, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 2{\cdot}40\ \mathrm{cm^{-1}}$, $F(000) = 1120$, $T = 293$ K, $R = 0{\cdot}044$ for 838 unique observed reflections. The N_3C_2 portion of the ring is planar within $\pm 0{\cdot}06$ Å; the S atom is 0·549 (2) Å out of this plane. The S—N bonds average 1·677 (5) Å and the NSN angle is 103·2 (2)°.

Experimental. Compound prepared by the hydrolysis of 3,5-diphenyl-1,2,4,6-thiatriazine 1-chloride in wet acetonitrile. $S(O)N_3C_2(C_6H_5)_2$ crystals obtained from acetonitrile solutions. Colorless needle data crystal $0{\cdot}14 \times 0{\cdot}12 \times 0{\cdot}50$ mm mounted on glass fiber. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4 to 16° $\mathrm{min^{-1}}$ in θ. Unit cell determined from least-squares analysis of angle data for 25 reflections with $16 < 2\theta < 20°$. Analytical absorption correction based on crystal shape varied from 0·98 to 1·00. Data collected to $(\sin\theta)/\lambda$ of $0{\cdot}62\ \mathrm{Å^{-1}}$, $-10 \le h \le 0$, $0 \le k \le 19$, $0 \le l \le 22$. Three standard reflections ($\bar{3}23$, $\bar{2}35$, $\bar{1}\bar{6}0$) varied $\pm 3{\cdot}0\%$ over 19·8 h of data collection; the contribution of each standard to the decay correction was weighted according to the relative distance between reciprocal-lattice points. 2459 reflections measured, 1621 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$. H atoms constrained to idealized (C—H = 0·95; N—H = 0·93 Å) positions with isotropic $B = 1{\cdot}2 \times B$ of bonded C or N atom. All non-H atoms refined anisotropically for a total of 172 variables. $R = 0{\cdot}044$, $wR = 0{\cdot}050$, $S = 1{\cdot}18$, where non-Poisson

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