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Structure of 1,1-Diphenylphosphorinanium Bromide Monohydrate

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Abstract. $C_{17}H_{20}P^+.Br^-.H_2O$, $M_r = 353{\cdot}24$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}542\,(1)$, $b = 12{\cdot}202\,(2)$, $c = 15{\cdot}864\,(3)$ Å, $V = 1653{\cdot}5$ Å^3, $Z = 4$, $D_m = 1{\cdot}42$, $D_x = 1{\cdot}42$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 27{\cdot}2$ cm^{-1}, $F(000) = 728$, $T = 295$ K, $R = 0{\cdot}062$, $wR = 0{\cdot}067$, 1270 observed reflections. The phosphorus–carbon bond lengths are 1·794 (9), 1·806 (10), 1·796 (9) and 1·783 (10) Å. The phosphorus heterocyclic ring is in the chair form and the torsional angles indicate that it is highly symmetrical. The bond angles around the phosphorus atom are 103·7 (5), 111·7 (4), 110·5 (4), 109·8 (4), 111·0 (4) and 109·9 (4)°.

Experimental. Title compound prepared by previously described methods (Gray, Cremer & Marsi, 1976). D_m by flotation in ethanol and methyl iodide mixture. Colorless crystals suitable for X-ray analysis obtained by recrystallization from acetonitrile. Crystal size 0·2 × 0·25 × 0·43 mm, Nicolet *R*3*m* diffractometer, graphite monochromator, accurate unit-cell parameters (Campana, 1981) from least-squares refinement of 25 reflections (θ range 10–20°); three standard reflections (057, 434, 623) measured every 100 reflections, 4% variation. Range of *hkl*: $0 \leq h \leq 10$, $0 \leq k \leq 14$, $0 \leq l \leq 18$. Data taken as $\theta/2\theta$ scans, 1770 data measured ($3 \leq 2\theta \leq 55°$) with 1270 observed $I \geq 2\sigma(I)$, corrections for Lorentz and polarization effects (decay correction ignored). Absorption corrections by numerical Gaussian integration method from crystal dimensions and indexed faces. Max. and min. transmission coefficients 0·63 and 0·59, respectively. Trial structure from Patterson map provided Br^--ion position, refined by full-matrix least-squares procedures, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/[\sigma^2(F) + G(F)^2]$ and $G = 0{\cdot}001$; non-hydrogen atoms anisotropic, H atoms located in ΔF map, idealized coordinates calculated, not refined, isotropic thermal parameters

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assigned as 0·049 Å^2; $R = 0{\cdot}062$, $wR = 0{\cdot}067$, scattering factors for Br, P, O, C and H from *International Tables for X-ray Crystallography* (1962), $S = 1{\cdot}43$. Anomalous-dispersion corrections for P and Br applied toward end of refinement (Cromer, 1965). In final cycle, largest shift in any parameter was 0·08σ. Final difference map showed no peaks larger than 0·60 e Å^{-3} at 0·97 Å from Br. Calculations carried out with *SHELXTL* package on Nicolet *R*3*m* crystallographic system (Sheldrick, 1980).

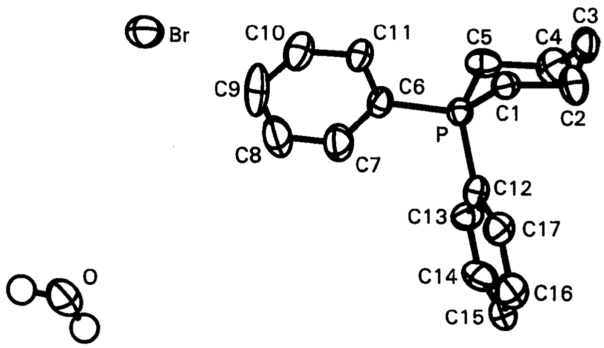

Fig. 1. The structure and numbering scheme for 1,1-diphenylphosphorinanium bromide monohydrate. Non-hydrogen atoms are represented as thermal ellipsoids scaled to enclose 50% probability. The hydrogen atoms are omitted for clarity.

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3)

	x	y	z	U_{eq}*
Br	−137 (1)	6591 (1)	4188 (1)	60 (1)
P	4080 (3)	1099 (2)	2509 (2)	31 (1)
O	2412 (10)	1554 (6)	9089 (5)	68 (3)
C(1)	4419 (11)	1659 (8)	1479 (5)	34 (3)
C(2)	5058 (13)	759 (7)	888 (6)	51 (3)
C(3)	3952 (13)	−216 (8)	819 (7)	49 (3)
C(4)	3698 (14)	−819 (8)	1644 (7)	55 (4)
C(5)	2884 (12)	−93 (8)	2308 (6)	43 (3)
C(6)	3060 (11)	2048 (7)	3177 (6)	33 (3)
C(7)	3308 (13)	2034 (10)	4034 (7)	54 (4)
C(8)	2559 (16)	2770 (10)	4550 (7)	68 (5)
C(9)	1566 (15)	3548 (8)	4215 (8)	66 (5)
C(10)	1371 (14)	3586 (9)	3351 (7)	54 (4)
C(11)	2058 (12)	2821 (8)	2835 (7)	43 (3)
C(12)	5897 (11)	722 (7)	2980 (6)	33 (3)
C(13)	5960 (12)	−105 (9)	3594 (6)	42 (3)
C(14)	7343 (14)	−405 (9)	3954 (7)	50 (4)
C(15)	8728 (13)	70 (10)	3723 (6)	49 (4)
C(16)	8698 (13)	929 (10)	3108 (7)	56 (4)
C(17)	7259 (12)	1211 (9)	2736 (6)	44 (3)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

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

P–C(1)	1·794 (9)	P–C(5)	1·806 (10)
P–C(6)	1·796 (9)	P–C(12)	1·783 (10)
C(1)–C(2)	1·544 (13)	C(2)–C(13)	1·524 (14)
C(3)–C(4)	1·516 (15)	C(4)–C(5)	1·542 (15)
C(6)–C(7)	1·376 (14)	C(6)–C(11)	1·384 (13)
C(7)–C(8)	1·373 (17)	C(8)–C(9)	1·379 (17)
C(9)–C(10)	1·382 (17)	C(10)–C(11)	1·372 (15)
C(12)–C(13)	1·405 (14)	C(12)–C(17)	1·363 (14)
C(13)–C(14)	1·362 (15)	C(14)–C(15)	1·367 (16)
C(15)–C(16)	1·432 (16)	C(16)–C(17)	1·407 (15)
C(1)–P–C(5)	103·7 (5)	C(1)–P–C(6)	111·7 (4)
C(5)–P–C(6)	110·5 (4)	C(1)–P–C(12)	109·8 (4)
C(5)–P–C(12)	111·0 (4)	C(6)–P–C(12)	109·9 (4)
P–C(1)–C(2)	109·8 (6)	C(1)–C(2)–C(3)	112·3 (9)
C(2)–C(3)–C(4)	114·0 (9)	C(3)–C(4)–C(5)	112·1 (8)
P–C(5)–C(4)	109·2 (7)	P–C(6)–C(7)	120·0 (8)
P–C(6)–C(11)	120·5 (7)	C(7)–C(6)–C(11)	119·4 (9)
C(6)–C(7)–C(8)	120·6 (11)	C(7)–C(8)–C(9)	120·4 (11)
C(8)–C(9)–C(10)	118·7 (11)	C(9)–C(10)–C(11)	121·1 (10)
C(6)–C(11)–C(10)	119·6 (10)	P–C(12)–C(13)	120·6 (7)
P–C(12)–C(17)	120·8 (7)	C(13)–C(12)–C(17)	118·6 (9)
C(12)–C(13)–C(14)	121·1 (10)	C(13)–C(14)–C(15)	121·7 (10)
C(14)–C(15)–C(16)	118·5 (10)	C(15)–C(16)–C(17)	118·7 (10)
C(12)–C(17)–C(16)	121·3 (10)		

The structure and numbering scheme of the title compound are shown in Fig. 1. The atomic fractional coordinates with standard deviations and U_{eq} values (Hamilton, 1959) are given in Table 1.* Table 2 gives a list of the interatomic distances and angles with their standard deviations.

Related literature. This structure is similar to two structures previously reported by Gallucci & Holmes (1980).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42544 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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