Table 2. *Bond distances* (Å), *angles* (°) *and torsion angles* (°)

Br—C(1)	1·909 (3)	C(3)—C(4)	1·380 (5)
O(1)—C(6)	1·425 (3)	C(4)—C(5)	1·379 (4)
O(1)—C(12)	1·405 (5)	C(5)—C(6)	1·517 (4)
O(2)—C(6)	1·422 (3)	C(6)—C(7)	1·522 (3)
O(2)—C(13)	1·394 (5)	C(7)—C(8)	1·378 (4)
N(1)—C(1)	1·314 (3)	C(8)—C(9)	1·394 (4)
N(1)—C(5)	1·343 (3)	C(9)—C(10)	1·368 (4)
N(2)—C(7)	1·336 (3)	C(10)—C(11)	1·390 (4)
N(2)—C(11)	1·352 (3)	C(11)—C(11′)	1·484 (4)
C(1)—C(2)	1·381 (4)	C(12)—C(13)	1·456 (7)
C(2)—C(3)	1·376 (5)		
C(6)—O(1)—C(12)	107·4 (3)	O(1)—C(6)—C(7)	107·9 (2)
C(6)—O(2)—C(13)	107·5 (3)	O(2)—C(6)—C(5)	109·8 (2)
C(1)—N(1)—C(5)	116·9 (2)	O(2)—C(6)—C(7)	108·9 (2)
C(7)—N(2)—C(11)	117·6 (2)	C(5)—C(6)—C(7)	113·3 (2)
Br—C(1)—N(1)	115·9 (2)	N(2)—C(7)—C(6)	115·3 (2)
Br—C(1)—C(2)	118·3 (2)	N(2)—C(7)—C(8)	123·4 (3)
N(1)—C(1)—C(2)	125·8 (3)	C(6)—C(7)—C(8)	121·3 (3)
C(1)—C(2)—C(3)	116·7 (3)	C(7)—C(8)—C(9)	118·5 (3)
C(2)—C(3)—C(4)	118·9 (3)	C(8)—C(9)—C(10)	118·9 (3)
C(3)—C(4)—C(5)	119·8 (3)	C(9)—C(10)—C(11)	119·3 (3)
N(1)—C(5)—C(4)	121·8 (3)	N(2)—C(11)—C(10)	122·2 (3)
N(1)—C(5)—C(6)	114·9 (2)	N(2)—C(11)—C(11′)	116·3 (3)
C(4)—C(5)—C(6)	123·3 (3)	C(10)—C(11)—C(11′)	121·5 (3)
O(1)—C(6)—O(2)	106·1 (2)	O(1)—C(12)—C(13)	107·4 (4)
O(1)—C(6)—C(5)	110·6 (2)	O(2)—C(13)—C(12)	106·1 (3)
N(1)—C(5)—C(6)—O(1)	170·9 (4)		
N(2)—C(7)—C(6)—O(2)	−159·5 (4)		
O(1)—C(12)—C(13)—O(2)	−9·3 (6)		
C(12)—C(13)—O(2)—C(6)	20·5 (5)		
C(13)—O(2)—C(6)—O(1)	−24·0 (5)		
O(2)—C(6)—O(1)—C(12)	17·9 (4)		
C(6)—O(1)—C(12)—C(13)	−5·5 (6)		

SDP (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974). Non-H atoms anisotropic; H atoms located by ΔF synthesis, placed in calculated positions with C—H 0·95 Å, $B = 5{\cdot}0$ Å^2. Final $R = 0{\cdot}037$ (0·053 for all data), $wR = 0{\cdot}049$, $S = 1{\cdot}563$ for 163 variables. Max. shift $0{\cdot}01\sigma$ in final cycle, largest residual density 0·62 e Å^{-3}, min. −0·45 e Å^{-3}. The molecule is depicted in Fig. 1, coordinates are given in Table 1, bond distances, angles and torsion angles are listed in Table 2.*

Related literature. Chisholm, Huffman, Rothwell, Bradley, Kress & Woodruff (1981); James & Williams (1973); Kvick (1976); Newkome, Taylor, Fronczek & Delord (1984).

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure factor amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42311 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Tetraphenylphosphonium Bromide

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Abstract. $C_{24}H_{20}P^+.Br^-$, $M_r = 419{\cdot}3$, tetragonal, $I\bar{4}$, $a = 11{\cdot}960\ (2)$, $c = 6{\cdot}967\ (2)$ Å, $V = 996{\cdot}6\ (3)$ Å^3, $Z = 2$, $D_m = 1{\cdot}45$, $D_x = 1{\cdot}40$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 21{\cdot}24$ cm^{-1}, $F(000) = 428$, ambient temperature. $R = 0{\cdot}066$ for 1006 observed reflections. The cation has $\bar{4}$ symmetry with P—C bond lengths of 1·800 (6) Å. The dimensions of the phenyl rings agree with standard values.

Experimental. Title compound recrystallized from MeOH/MeCN to yield large colourless block-like crystals. Density measured by flotation. Data collected with a Syntex $P2_1$ four-circle diffractometer. Maximum 2θ 60°, scan range ±1° (2θ) around $K\alpha_1$–$K\alpha_2$ angles; scan speed 2–29° min^{-1} depending on intensity of 2 s prescan; backgrounds measured at each end of the scan for 0·25 of scan time. Three standard reflections

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monitored every 200 reflections, slight changes during data collection; data rescaled to correct for this. $h = 0$ to 16, $k = 0$ to 16, $l = -7$ to 7. Unit-cell dimensions and standard deviations obtained by least-squares fit to 15 reflections ($28 < \theta < 30°$), 1629 reflections measured, 1006 observed $[I/\sigma(I) > 3{\cdot}0]$ used in refinement, and corrected for Lorentz, polarization and absorption effects, the last with *ABSCOR* (Alcock, 1970); maximum and minimum transmission factors 0·89 and 0·67; crystal dimensions 0·17 × 0·25 × 0·10 mm.

Heavy atoms located by the Patterson-interpretation section of *SHELXTL* (Sheldrick, 1983) and the light atoms then found on successive Fourier syntheses. Anisotropic temperature factors for all non-H atoms. H atoms inserted at calculated positions and not refined; P and Br atoms in special positions, symmetry $\bar{4}$. Final refinement on F by cascaded least-squares methods. Largest positive and negative peaks on a final difference Fourier synthesis of height +0·7 and −1·1 e Å^{-3}.

Weighting scheme of the form $w = 1/[\sigma^2(F) + gF^2]$ with $g = 0{\cdot}0035$ used and shown to be satisfactory by a weight analysis. Final $R = 0{\cdot}066$, $wR = 0{\cdot}072$. Maximum Δ/σ in final cycle 0·01. Computing with *SHELXTL* (Sheldrick, 1983) on a Data General DG30, apart from absorption correction on a Burroughs B6800. Scattering factors in the analytical form and anomalous-dispersion factors taken from *International Tables for X-ray Crystallography* (1974). Final atomic coordinates are given in Table 1,* and bond lengths and angles in Table 2. Fig. 1 shows the packing.

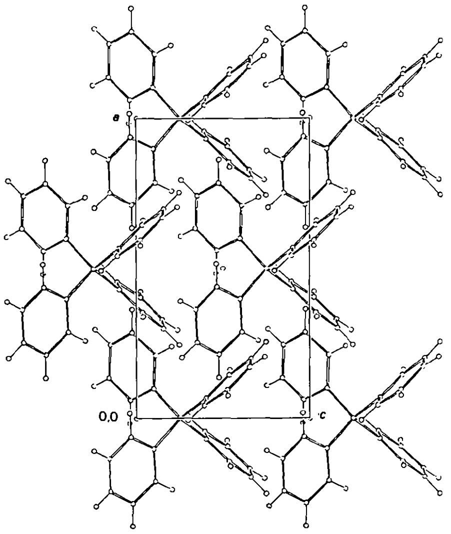

Fig. 1. Packing diagram for $[Ph_4P]^+Br^-$, viewed down **b**.

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 ×10^3) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Br	0	0	0	72 (1)
P(1)	0	5000	7500	52 (1)
C(1)	−686 (5)	6005 (5)	5977 (8)	53 (1)
C(2)	−437 (5)	7118 (5)	6081 (11)	65 (2)
C(3)	−952 (6)	7861 (5)	4749 (16)	75 (2)
C(4)	−1704 (7)	7455 (7)	3482 (11)	76 (2)
C(5)	−1993 (8)	6348 (8)	3405 (12)	80 (3)
C(6)	−1448 (6)	5616 (6)	4658 (9)	69 (2)

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

P(1)–C(1)	1·800 (6)	P(1)–C(1^{i})	1·800 (6)
P(1)–C(1^{ii})	1·800 (6)	P(1)–C(1^{iii})	1·800 (6)
C(1)–C(2)	1·367 (9)	C(2)–C(3)	1·424 (11)
C(3)–C(4)	1·351 (12)	C(4)–C(5)	1·369 (13)
C(5)–C(6)	1·398 (11)	C(6)–C(1)	1·375 (9)
C(1)–P(1)–C(1^{i})	107·8 (4)	C(1)–P(1)–C(1^{ii})	110·3 (2)
C(1)–P(1)–C(1^{iii})	110·3 (2)	P(1)–C(1)–C(2)	121·4 (5)
P(1)–C(1)–C(6)	118·0 (5)	C(2)–C(1)–C(6)	120·6 (6)
C(1)–C(2)–C(3)	118·6 (6)	C(2)–C(3)–C(4)	119·3 (6)
C(3)–C(4)–C(5)	122·8 (8)	C(4)–C(5)–C(6)	117·6 (8)
C(1)–C(6)–C(5)	121·0 (7)		

Symmetry code: (i) $-x, 1-y, z$; (ii) $-\frac{1}{2}+y, \frac{1}{2}-x, 1\frac{1}{2}-z$; (iii) $\frac{1}{2}-y, \frac{1}{2}+x, 1\frac{1}{2}-z$.

Related literature. The structures of the tetraphenylphosphonium salts of: pentabromocarbonate (Lindner & Kitschke-von Gross, 1976), dibromoiodide (Müller, 1979), tribromide (Bogaard & Rae, 1982) and octachlorodiarsenate (Mohammed & Müller, 1985) have been reported.

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