

it represents a stage in the synthesis of (+)-sarracenin. For the structure of the latter see Miles *et al.* (1976).

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Structure of Tetraphenylphosphonium Chloride

BY JOHN F. RICHARDSON,* JOANNE M. BALL AND P. MICHAEL BOORMAN

Department of Chemistry, University of Calgary, Calgary, Alberta, Canada T2N 1N4

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Abstract. $C_{24}H_{20}P^+.Cl^-$, $M_r = 374.85$, monoclinic, $P2_1/n$, $a = 9.3069$ (7), $b = 9.6235$ (5), $c = 22.225$ (2) Å, $\beta = 99.248$ (4)°, $V = 1964.7$ (2) Å³, $Z = 4$, $D_x = 1.27$ Mg m⁻³, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 2.50$ mm⁻¹, $F(000) = 784$, $T = 294$ K, $R = 0.038$ for 2865 unique reflections. The salt, isolated as an impurity in the preparation of $(Ph_4P)^+[S-(p\text{-tolyl})]^-$, is not found to be isostructural with the previously reported structure of $(Ph_4P)^+Br^-$ or powder studies of $(Ph_4P)^+I^-$ and $(Ph_4As)^+I^-$. The average P–C bond length is 1.788 (2) Å and dimensions of the phenyl rings are considered normal.

Experimental. Colourless block (cut), $0.3 \times 0.5 \times 0.5$ mm, Enraf–Nonius CAD-4F diffractometer, Ni pre-filtered Cu $K\alpha$ radiation; lattice parameters from least-squares refinement of 25 accurately centered reflections with $40 \leq \theta \leq 56^\circ$; space group uniquely determined from absences ($h0l$ $h + l = 2n + 1$, $0k0$ $k = 2n + 1$); 3337 unique reflections collected, 2865 considered observed at the $3\sigma(I)$ level [$\sigma(I)$ from counting statistics], $\theta_{max} = 65^\circ$, $\omega/2\theta$ scans, scan range of $1.5(0.66 + 0.142\tan\theta)^\circ$, variable scan speed $0.7^\circ - 3.4^\circ$ min⁻¹; three standard reflections ($1\bar{6}\bar{3}$, $1\bar{2}$, $1\bar{2}$, $3\bar{1}$, 10) measured every 1500 s of X-ray exposure time, max. variation +5.0%, data collected: $+h$, $-k$, $\pm l$ to max. indices of 10, 11, 26. Data corrected for background, Lp, drift in standards and absorption (Walker & Stuart, 1983); min., max. absorption corrections of 0.79 and 1.24. Structure solved by direct

methods, *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and difference Fourier syntheses, refined by full-matrix least squares based on F , minimizing the function $\sum w(|F_o| - |F_c|)^2$, w defined as $[\sigma^2(F_o) + 0.00001(F^2)]^{-1}$, *XRAY76* system of programs (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976); H atoms included in calculated positions with isotropic thermal parameters set to 1.1 times B_{eq} of the bonded atom, H parameters not refined, all non-H atoms refined with anisotropic thermal parameters. Model converged with: 2865 observations, 236 variables, $R = 0.038$, $wR = 0.038$, max. $\Delta/\sigma = 0.67$ for E but all others < 0.009 , $S = 1.51$, isotropic extinction parameter (E) = 9.9 (5) $\times 10^{-4}$, max. residual electron density = 0.28 e Å⁻³ associated with P. Scattering factors were those of Cromer & Mann (1968) for non-H atoms and Stewart, Davidson & Simpson (1965) for H atoms. Anomalous-dispersion corrections included for non-H atoms (*International Tables for X-ray Crystallography*, 1974). The closest interionic contact distance is 2.603 Å between H(33) and H(36) (at $1\frac{1}{2} - x$, $\frac{1}{2} + y$, $\frac{1}{2} - z$). An *ORTEP* plot of the unit cell is shown in Fig. 1, atomic coordinates are given in Table 1, and bond lengths and angles are listed in Table 2†

† Lists of structure factors, anisotropic thermal parameters for the non-H atoms, and positional and isotropic thermal parameters for the H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42968 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* Author to whom correspondence should be addressed.

Table 1. Coordinates ($\times 10^4$) and B_{eq} ($\times 10$) for the non-hydrogen atoms

	B_{eq} is one-third of the B_{ij} matrix.			
	x	y	z	$B_{eq}(\text{\AA}^2)$
Cl	890.3 (7)	4910.6 (7)	3690.3 (3)	48.1 (3)
P	6455.0 (6)	567.2 (6)	3715.8 (2)	30.0 (2)
C(1)	7474 (2)	-531 (2)	4273 (1)	32 (1)
C(2)	8343 (3)	-1567 (2)	4091 (1)	39 (1)
C(3)	8997 (3)	-2530 (3)	4505 (1)	49 (1)
C(4)	8809 (3)	-2456 (3)	5106 (1)	50 (1)
C(5)	7971 (3)	-1424 (3)	5295 (1)	53 (1)
C(6)	7300 (3)	-459 (3)	4887 (1)	46 (1)
C(11)	4735 (2)	-250 (2)	3458 (1)	33 (1)
C(12)	3874 (3)	132 (3)	2913 (1)	44 (1)
C(13)	2536 (3)	-490 (3)	2741 (1)	57 (1)
C(14)	2038 (3)	-1462 (3)	3102 (2)	62 (2)
C(15)	2870 (3)	-1853 (3)	3642 (1)	56 (1)
C(16)	4224 (3)	-1255 (3)	3822 (1)	42 (1)
C(21)	6174 (2)	2219 (2)	4044 (1)	32 (1)
C(22)	7288 (3)	2821 (3)	4461 (1)	39 (1)
C(23)	7151 (3)	4157 (3)	4664 (1)	45 (1)
C(24)	5915 (3)	4908 (3)	4460 (1)	47 (1)
C(25)	4784 (3)	4318 (3)	4064 (1)	44 (1)
C(26)	4910 (2)	2981 (2)	3856 (1)	36 (1)
C(31)	7464 (2)	825 (2)	3103 (1)	31 (1)
C(32)	8689 (3)	1679 (2)	3206 (1)	40 (1)
C(33)	9533 (3)	1866 (3)	2758 (1)	47 (1)
C(34)	9154 (3)	1225 (3)	2205 (1)	48 (1)
C(35)	7950 (3)	397 (3)	2096 (1)	53 (1)
C(36)	7107 (3)	177 (3)	2545 (1)	45 (1)

Table 2. Bond lengths (\AA) and angles ($^\circ$)

P-C(1)	1.781 (2)	P-C(21)	1.786 (2)
P-C(11)	1.793 (2)	P-C(31)	1.791 (2)
C(1)-C(2)	1.385 (3)	C(21)-C(22)	1.400 (3)
C(1)-C(6)	1.402 (3)	C(21)-C(26)	1.392 (3)
C(2)-C(3)	1.377 (3)	C(22)-C(23)	1.375 (4)
C(3)-C(4)	1.377 (4)	C(23)-C(24)	1.372 (4)
C(4)-C(5)	1.370 (4)	C(24)-C(25)	1.381 (3)
C(5)-C(6)	1.376 (4)	C(25)-C(26)	1.379 (3)
C(11)-C(12)	1.390 (3)	C(31)-C(32)	1.394 (3)
C(11)-C(16)	1.393 (3)	C(31)-C(36)	1.380 (3)
C(12)-C(13)	1.379 (4)	C(32)-C(33)	1.376 (4)
C(13)-C(14)	1.361 (5)	C(33)-C(34)	1.370 (4)
C(14)-C(15)	1.372 (4)	C(34)-C(35)	1.365 (4)
C(15)-C(16)	1.385 (4)	C(35)-C(36)	1.381 (4)
C(1)-P-C(11)	108.1 (1)	C(11)-P-C(21)	109.8 (1)
C(1)-P-C(21)	109.9 (1)	C(11)-P-C(31)	111.6 (1)
C(1)-P-C(31)	109.0 (1)	C(21)-P-C(31)	108.4 (1)
P-C(1)-C(2)	119.7 (2)	P-C(21)-C(22)	119.8 (2)
P-C(1)-C(6)	121.0 (2)	P-C(21)-C(26)	121.2 (2)
C(2)-C(1)-C(6)	119.0 (2)	C(22)-C(21)-C(26)	118.7 (2)
C(1)-C(2)-C(3)	120.4 (2)	C(21)-C(22)-C(23)	120.4 (2)
C(2)-C(3)-C(4)	120.1 (2)	C(22)-C(23)-C(24)	120.1 (2)
C(3)-C(4)-C(5)	120.2 (2)	C(23)-C(24)-C(25)	120.4 (2)
C(4)-C(5)-C(6)	120.5 (2)	C(24)-C(25)-C(26)	120.0 (2)
C(1)-C(6)-C(5)	119.8 (2)	C(21)-C(26)-C(25)	120.3 (2)
P-C(11)-C(12)	121.5 (2)	P-C(31)-C(32)	118.1 (2)
P-C(11)-C(16)	119.3 (1)	P-C(31)-C(36)	122.7 (2)
C(12)-C(11)-C(16)	119.1 (2)	C(32)-C(31)-C(36)	119.1 (2)
C(11)-C(12)-C(13)	119.7 (2)	C(31)-C(32)-C(33)	120.4 (2)
C(12)-C(13)-C(14)	120.8 (2)	C(32)-C(33)-C(34)	119.7 (2)
C(13)-C(14)-C(15)	120.4 (3)	C(33)-C(34)-C(35)	120.5 (2)
C(14)-C(15)-C(16)	119.9 (3)	C(34)-C(35)-C(36)	120.5 (2)
C(11)-C(16)-C(15)	120.0 (2)	C(31)-C(36)-C(35)	119.8 (2)

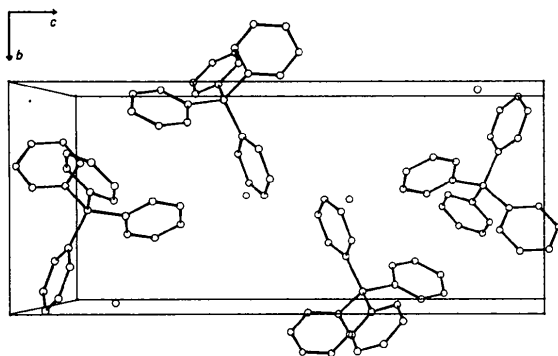


Fig. 1. ORTEP plot (Johnson, 1965) showing the unit-cell contents. Atoms are drawn as spheres of radius 0.1 Å and H atoms are omitted for clarity.

Related literature. A number of related simple salts have been reported: $(\text{Ph}_4\text{As})^+\text{I}^-$ (Mooney, 1940), $(\text{Ph}_4\text{As})^+\text{Br}_3^-$ (Ollis, James, Ollis & Bogaard, 1976; Bogaard, Peterson & Rae, 1979), $(\text{Ph}_4\text{As})^+\text{Cl}_3^-$ (Bogaard, Peterson & Rae, 1981), $(\text{Ph}_4\text{P})^+\text{Br}_3^-$ (Bogaard & Rae, 1982), $(\text{Ph}_4\text{P})^+\text{Br}_2\text{I}^-$ (Müller, 1979), and most recently $(\text{Ph}_4\text{P})^+\text{Br}^-$ (Alcock, Pennington & Willey, 1985).

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