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Triphenylphosphonium perchlorate

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Key indicators

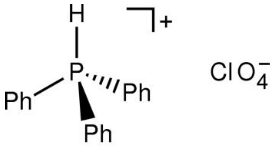
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.006 Å
R factor = 0.053
wR factor = 0.149
Data-to-parameter ratio = 14.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound consists of $C_{18}H_{15}PH^{+}\cdot ClO_4^{-}$ ion pairs, each ion lying on a crystallographic mirror plane. The P atom has a slightly distorted tetrahedral geometry. The P—H distance is 1.25 (5) Å. C—H···O hydrogen bonds are observed between the cation and anion.

Comment

The title compound, (I), was obtained from an acidic reaction of triphenylphosphine treated with an equivalent amount of perchloric acid.



(I)

The structure contains $[C_{18}H_{15}PH]^{+}$ and $[ClO_4]^{-}$ ion pairs. Atoms H1, P1, C7, and C10 lie on a crystallographic mirror plane; Cl1, O1 and O3 lie on a second mirror plane (Fig. 1). The P atom has a slightly distorted tetrahedral geometry with average C—P—C and C—P—H angles of 111.61 (13) and 107.2 (12)°, respectively. In the perchlorate anion O—Cl—O angles lie in the range 106.03 (18)–113.0 (4)°. The P—H distance of 1.25 (5) Å in (I) is comparable to that in $[Ph_3PH]_2[SnCl_6]$ [1.285 (3) Å; Yatsenko *et al.*, 1986], $[(2\text{-}MePh)_3PH][BCl_4]$ [1.289 (19) Å; Burke *et al.*, 2000] and $[Ph_3PH][(FSO_2)_2N]$ [1.28 (2) Å; Hiemisch *et al.*, 1996], but shorter than that in $[Ph_3PH]Br$ [1.61 (9) Å] where the proton is directed towards the bromide anion (Bricklebank *et al.*, 1993).

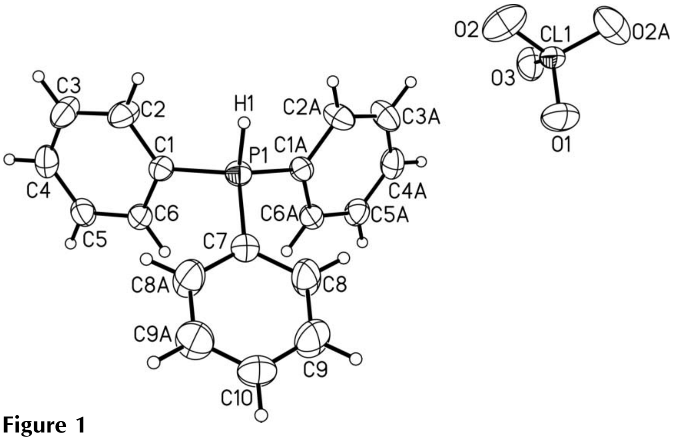


Figure 1
The molecular structure of (I), showing the atom-numbering scheme, and with displacement ellipsoids at the 50% probability level for non-H atoms. Atoms labelled with the suffix A are at the symmetry position (x, $\frac{1}{2} - y$, z) in the $[Ph_3PH]^{+}$ cation and (x, $\frac{3}{2} - y$, z) in the $[ClO_4]^{-}$ anion.

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The H atom of the phosphonium cation is not involved in hydrogen bonding. However, C—H···O-type hydrogen-bonding interactions (Table 1) exist between the cation and anion.

Experimental

A tetrahydrofuran solution (20 ml) of triphenylphosphine (2.62 g, 10 mmol) was treated with an equivalent amount of perchloric acid (1.56 g in 65% *w*/*w* aqueous solution, 10 mmol). Diffusion of this solution into hexane over a period of three days afforded colourless crystals of (I).

Crystal data

$C_{18}H_{16}P^+\cdot ClO_4^-$
M_r = 362.73
Orthorhombic, *Pnma*
a = 10.6695 (18) Å
b = 13.113 (2) Å
c = 12.488 (2) Å
V = 1747.2 (5) Å^3
Z = 4
D_x = 1.379 Mg m^{-3}
Mo *Kα* radiation
μ = 0.33 mm^{-1}
T = 294 (2) K
Block, colourless
0.12 × 0.10 × 0.06 mm

Data collection

Bruker APEX area-detector diffractometer
φ and *ω* scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.958, T_{max} = 0.981
10334 measured reflections
1725 independent reflections
1270 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 25.7°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.149
S = 1.03
1725 reflections
121 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0909P)^2 + 0.6474P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O2^{i}	0.93	2.39	3.320 (5)	174
C6—H6···O3^{ii}	0.93	2.58	3.507 (5)	176

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

The H atom bonded to the P atom was located in a difference map and refined freely. The remaining H atoms were placed in calculated positions (C—H = 0.93 Å) and allowed to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-PLus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL97*.

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