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Tetraphenylphosphonium chloride dihydrate

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The structure of the title compound, $C_{24}H_{20}P^+\cdot Cl^-\cdot 2H_2O$, comprises hydrogen-bonded chains of water molecules and chloride anions separated by hydrophobic regions of tetraphenylphosphonium cations. Within the hydrogen-bonded chains the chloride anions are bridged by pairs of water molecules. Each water molecule donates two hydrogen bonds, while each chloride accepts four.

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.098
Data-to-parameter ratio = 20.0

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

Comment

The tetraphenylphosphonium cation is widely used as a counter-cation for the isolation of organic and inorganic anions. Two modifications are known for tetraphenylphosphonium chloride, a monoclinic α form (Richardson *et al.*, 1986) and a triclinic β form (Gruber & Müller, 1998), and a monohydrate has also been reported (Schweizer *et al.*, 1989). Herein we report the structure of a dihydrate, (I), formed from a reaction mixture which included tetraphenylphosphonium bromide and $K_2[PdCl_4]$.

$$[PPh_4]Cl\cdot 2H_2O$$

(I)

The structure of (I) comprises hydrogen-bonded chains of water molecules and chloride anions running along the [010] direction, separated by hydrophobic regions containing the tetraphenylphosphonium cations. Within each chain, the chloride anions are bridged by pairs of water molecules, each water molecule contributing two hydrogen bonds, with each chloride accepting four hydrogen bonds.

Experimental

The title compound was isolated from a reaction mixture containing 4-(pyridin-2-yl)-[1,3]dithiol-2-one, $K_2[PdCl_4]$ and NaOH (2/1/10) in EtOH/H_2O, which was treated with $[PPh_4]Br$. Crystals were grown by dissolving the compound in CH_2Cl_2 and slowly diffusing hexane into the solution.

Crystal data

$C_{24}H_{20}P^+\cdot Cl^-\cdot 2H_2O$
M_r = 410.85
Triclinic, $P\bar{1}$
a = 9.4849 (11) Å
b = 11.0024 (13) Å
c = 11.4028 (13) Å
α = 75.090 (2)°
β = 73.748 (2)°
γ = 71.050 (2)°
V = 1062.2 (4) Å^3

Z = 2
D_x = 1.285 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4088 reflections
θ = 2.3–29.6°
μ = 0.27 mm^{-1}
T = 150 (2) K
Tablet, pale orange
0.44 × 0.28 × 0.14 mm

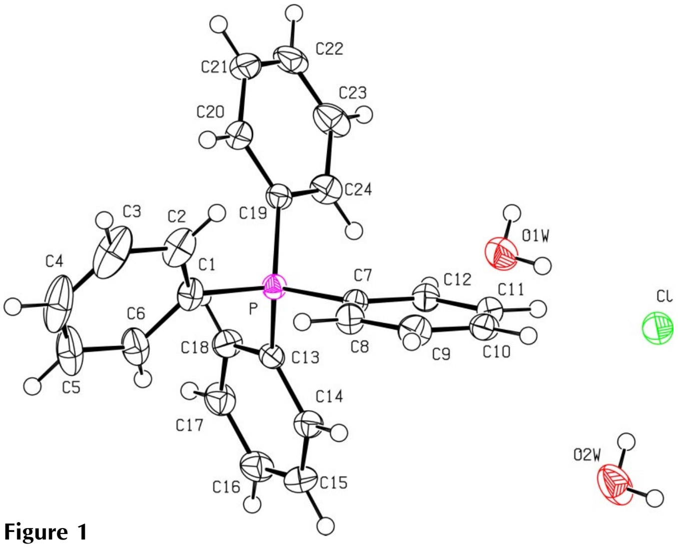

Figure 1
A view of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

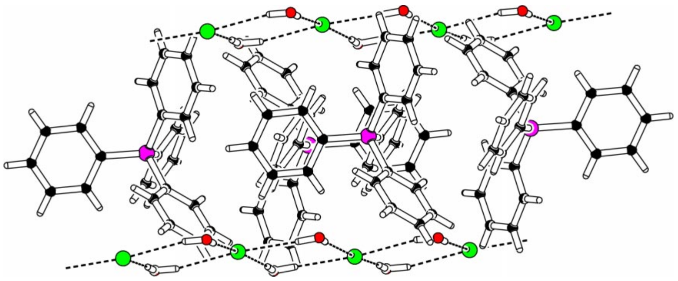

Figure 2
A view of the structure, showing hydrogen-bonded chains of water molecules and chloride anions separated by a hydrophobic region of tetraphenylphosphonium cations. Atom types are identified as black for C, red for O, purple for P and green for Cl.

Data collection

Bruker SMART1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SHEXLTL*; Bruker, 2001)
$T_{\min} = 0.876$, $T_{\max} = 0.961$
9902 measured reflections
5302 independent reflections
4045 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 29.6°$
$h = -12 \rightarrow 13$
$k = -15 \rightarrow 15$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.098$
$S = 1.04$
5295 reflections
265 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.051P)^2P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.41$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O1*W*—H1*W*1···Cl	0.85 (1)	2.60 (1)	3.4322 (13)	169 (2)
O1*W*—H1*W*2···Cl$^{\rm i}$	0.85 (2)	2.38 (2)	3.2253 (12)	175 (2)
O2*W*—H2*W*1···Cl$^{\rm ii}$	0.85 (1)	2.39 (2)	3.2405 (13)	175 (2)
O2*W*—H2*W*2···Cl	0.84 (3)	2.51 (3)	3.3405 (13)	167 (2)

Symmetry codes: (i) $2-x, 1-y, 1-z$; (ii) $2-x, -y, 1-z$.

Water H atoms were located from difference Fourier syntheses and refined with restraints to the O—H distances and the H—O—H angles. Phenyl H atoms were included at geometrically calculated positions and constrained to ride at a distance of 0.95 Å from their parent C atoms, with $U_{\rm iso}(\text{H}) = 1.2U_{\rm eq}(\text{C})$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* and *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002); software used to prepare material for publication: *SHELXL*97 and *PLATON*.

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