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Di-*tert*-butylmethylphosphonium chloride

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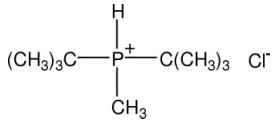
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.023; wR factor = 0.060; data-to-parameter ratio = 20.9.

Geometric parameters of the title compound, $C_9H_{22}P^+\cdot Cl^-$, are in the usual ranges. Cations and anions are connected by a P—H···Cl hydrogen bond.

Related literature

For related literature, see: Ruth *et al.* (2007).



Experimental

Crystal data

$C_9H_{22}P^+\cdot Cl^-$
M_r = 196.69
Orthorhombic, $P2_12_12_1$
a = 7.0601 (6) Å
b = 10.5406 (6) Å
c = 15.7074 (10) Å
V = 1168.91 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 173 (2) K
0.42 × 0.37 × 0.35 mm

Data collection

Stoe IPDSII two-circle diffractometer
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.846, T_{max} = 0.869
16039 measured reflections
2191 independent reflections
2138 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.060
S = 1.08
2191 reflections
105 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 905 Friedel pairs
Flack parameter: 0.00 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
P1—H1···Cl1	1.308 (19)	2.675 (19)	3.8400 (6)	147.2 (12)

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2409).

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Ruth, K., Müller, M., Bolte, M., Bats, J. W., Wagner, M. & Lerner, H.-W. (2007). *Z. Anorg. Allg. Chem.* **633**, 1485–1489.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2001). *X-AREA*. Stoe & Cie, Darmstadt, Germany.

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supplementary materials

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Di-*tert*-butylmethylphosphonium chloride

M. Müller, H.-W. Lerner and M. Bolte

Comment

Very recently, we have reported that treatment of Ph_2PCH_3 with nBuLi led to the formation of the lithium methanide $Li^+[CH_2PPh_2]^-$ (Ruth *et al.*, 2007). Thereby Ph_2PCH_3 reacts as a Brønsted acid. Here we describe a Lewis-base reaction of tBu_2PCH_3 with NH_4Cl which produces the phosphonium chloride $[tBu_2PHCH_3]^+ Cl^-$. X-ray quality crystals of the title compound have been obtained from a tetrahydrofuran solution at ambient temperature.

The title compound, $C_9H_{22}P^+\cdot Cl^-$, is composed of di(*t*-butyl)-methylphosphonium cations and chloride anions. Geometric parameters are in the usual ranges. Cations and anions are connected by a P—H···Cl hydrogen bond.

Experimental

At room temperature, 3.5 ml of a 1.0 *M* solution of NH_4Cl in water was added to a solution of 0.05 g (0.31 mmol) tBu_2PCH_3 in 3 ml pentane and 1 ml Et_2O. After removal of the solvent from the organic layer the residue was dissolved in 0.5 ml tetrahydrofuran. Colourless crystals of title compound were grown from this tetrahydrofuran solution at ambient temperature.

Refinement

H atoms were found in a difference map but those bonded to C were refined with fixed individual displacement parameters [U_{iso}(H) = 1.5 U_{eq}(C)] using a riding model with C—H = 0.98 Å. The H atom bonded to P was isotropically refined.

Figures

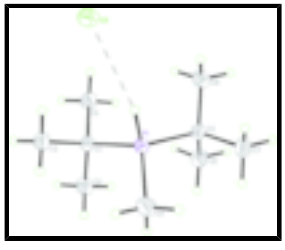

Fig. 1. Perspective view of the title compound with the atom numbering scheme. Displacement ellipsoids are at the 50% probability level. The H bond is drawn as a dashed line.

Fig. 2. The formation of the title compound.

Di-*tert*-butylmethylphosphonium chloride

Crystal data

$C_9H_{22}P^+\cdot Cl^-$ $F_{000} = 432$

M_r = 196.69	D_x = 1.118 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 14606 reflections
a = 7.0601 (6) Å	θ = 3.5–25.8º
b = 10.5406 (6) Å	μ = 0.41 mm^{-1}
c = 15.7074 (10) Å	T = 173 (2) K
V = 1168.91 (14) Å^3	Block, colourless
Z = 4	0.42 × 0.37 × 0.35 mm

Data collection

Stoe IPDSII two-circle diffractometer	2191 independent reflections
Radiation source: fine-focus sealed tube	2138 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.065
T = 173(2) K	θ_{max} = 25.6º
ω scans	θ_{min} = 3.5º
Absorption correction: multi-scan (MULABS; Spek, 2003; Blessing, 1995)	h = −8→8
T_{min} = 0.846, T_{max} = 0.869	k = −12→12
16039 measured reflections	l = −19→18

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0316P)^2 + 0.2388P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.023	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.060	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
S = 1.08	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
2191 reflections	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
105 parameters	Extinction coefficient: 0.021 (2)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 905 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.00 (7)
Hydrogen site location: inferred from neighbouring sites	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.22208 (5)	0.43238 (4)	0.79852 (3)	0.02524 (12)
P1	0.64522 (5)	0.49139 (4)	0.65011 (2)	0.01714 (11)
H1	0.512 (3)	0.5180 (18)	0.7049 (13)	0.029 (5)*
C1	0.7006 (2)	0.32277 (14)	0.67016 (10)	0.0190 (3)
C2	0.8916 (2)	0.28544 (16)	0.62979 (11)	0.0255 (4)
H2A	0.8829	0.2931	0.5677	0.038*
H2B	0.9221	0.1976	0.6449	0.038*
H2C	0.9913	0.3419	0.6510	0.038*
C3	0.7165 (3)	0.30947 (16)	0.76817 (10)	0.0253 (3)
H3A	0.5953	0.3324	0.7945	0.038*
H3B	0.8160	0.3661	0.7893	0.038*
H3C	0.7481	0.2216	0.7827	0.038*
C4	0.5391 (2)	0.23663 (16)	0.63818 (12)	0.0271 (4)
H4A	0.4198	0.2622	0.6650	0.041*
H4B	0.5671	0.1483	0.6529	0.041*
H4C	0.5277	0.2447	0.5762	0.041*
C5	0.5531 (2)	0.53377 (15)	0.54320 (10)	0.0206 (3)
C6	0.6720 (2)	0.47265 (17)	0.47158 (10)	0.0284 (4)
H6A	0.8045	0.4990	0.4773	0.043*
H6B	0.6233	0.5001	0.4161	0.043*
H6C	0.6637	0.3801	0.4758	0.043*
C7	0.5622 (3)	0.67927 (16)	0.53430 (12)	0.0311 (4)
H7A	0.4854	0.7186	0.5791	0.047*
H7B	0.5130	0.7042	0.4784	0.047*
H7C	0.6939	0.7075	0.5397	0.047*
C8	0.3434 (2)	0.49260 (18)	0.53649 (11)	0.0291 (4)
H8A	0.2706	0.5320	0.5826	0.044*
H8B	0.3348	0.4001	0.5412	0.044*
H8C	0.2919	0.5197	0.4815	0.044*
C9	0.8495 (3)	0.58718 (16)	0.67419 (12)	0.0303 (4)
H9A	0.8202	0.6766	0.6633	0.045*
H9B	0.9558	0.5609	0.6382	0.045*
H9C	0.8838	0.5762	0.7342	0.045*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02263 (18)	0.02150 (19)	0.0316 (2)	−0.00030 (15)	0.00044 (16)	−0.00198 (15)
P1	0.01824 (18)	0.01498 (19)	0.01820 (19)	0.00116 (15)	0.00001 (14)	−0.00105 (14)

C1	0.0221 (8)	0.0142 (7)	0.0206 (7)	0.0002 (6)	−0.0004 (6)	0.0001 (5)
C2	0.0263 (9)	0.0228 (8)	0.0274 (9)	0.0063 (7)	0.0021 (7)	−0.0004 (7)
C3	0.0281 (8)	0.0262 (8)	0.0216 (8)	0.0025 (7)	0.0002 (7)	0.0035 (6)
C4	0.0302 (9)	0.0197 (8)	0.0313 (9)	−0.0054 (7)	−0.0058 (8)	0.0021 (7)
C5	0.0223 (7)	0.0205 (8)	0.0191 (8)	0.0006 (6)	−0.0002 (6)	0.0030 (6)
C6	0.0317 (9)	0.0345 (10)	0.0191 (7)	0.0036 (7)	0.0038 (6)	0.0013 (6)
C7	0.0363 (9)	0.0221 (9)	0.0348 (10)	0.0022 (7)	−0.0027 (8)	0.0062 (7)
C8	0.0229 (7)	0.0353 (9)	0.0290 (8)	−0.0007 (8)	−0.0048 (6)	0.0020 (8)
C9	0.0297 (8)	0.0198 (8)	0.0413 (10)	−0.0039 (7)	−0.0114 (8)	0.0002 (7)

Geometric parameters (Å, °)

P1—C9	1.8009 (17)	C5—C7	1.541 (2)
P1—C1	1.8470 (15)	C5—C6	1.544 (2)
P1—C5	1.8554 (16)	C5—C8	1.546 (2)
P1—H1	1.308 (19)	C6—H6A	0.9800
C1—C2	1.541 (2)	C6—H6B	0.9800
C1—C4	1.542 (2)	C6—H6C	0.9800
C1—C3	1.550 (2)	C7—H7A	0.9800
C2—H2A	0.9800	C7—H7B	0.9800
C2—H2B	0.9800	C7—H7C	0.9800
C2—H2C	0.9800	C8—H8A	0.9800
C3—H3A	0.9800	C8—H8B	0.9800
C3—H3B	0.9800	C8—H8C	0.9800
C3—H3C	0.9800	C9—H9A	0.9800
C4—H4A	0.9800	C9—H9B	0.9800
C4—H4B	0.9800	C9—H9C	0.9800
C4—H4C	0.9800		
C9—P1—C1	109.51 (8)	C7—C5—C6	109.05 (14)
C9—P1—C5	109.63 (8)	C7—C5—C8	108.23 (14)
C1—P1—C5	117.40 (7)	C6—C5—C8	110.71 (14)
C9—P1—H1	108.6 (8)	C7—C5—P1	107.88 (11)
C1—P1—H1	104.3 (9)	C6—C5—P1	111.61 (11)
C5—P1—H1	106.9 (8)	C8—C5—P1	109.25 (11)
C2—C1—C4	111.26 (13)	C5—C6—H6A	109.5
C2—C1—C3	108.81 (14)	C5—C6—H6B	109.5
C4—C1—C3	108.89 (13)	H6A—C6—H6B	109.5
C2—C1—P1	111.15 (11)	C5—C6—H6C	109.5
C4—C1—P1	110.76 (11)	H6A—C6—H6C	109.5
C3—C1—P1	105.77 (10)	H6B—C6—H6C	109.5
C1—C2—H2A	109.5	C5—C7—H7A	109.5
C1—C2—H2B	109.5	C5—C7—H7B	109.5
H2A—C2—H2B	109.5	H7A—C7—H7B	109.5
C1—C2—H2C	109.5	C5—C7—H7C	109.5
H2A—C2—H2C	109.5	H7A—C7—H7C	109.5
H2B—C2—H2C	109.5	H7B—C7—H7C	109.5
C1—C3—H3A	109.5	C5—C8—H8A	109.5
C1—C3—H3B	109.5	C5—C8—H8B	109.5
H3A—C3—H3B	109.5	H8A—C8—H8B	109.5

C1—C3—H3C	109.5	C5—C8—H8C	109.5
H3A—C3—H3C	109.5	H8A—C8—H8C	109.5
H3B—C3—H3C	109.5	H8B—C8—H8C	109.5
C1—C4—H4A	109.5	P1—C9—H9A	109.5
C1—C4—H4B	109.5	P1—C9—H9B	109.5
H4A—C4—H4B	109.5	H9A—C9—H9B	109.5
C1—C4—H4C	109.5	P1—C9—H9C	109.5
H4A—C4—H4C	109.5	H9A—C9—H9C	109.5
H4B—C4—H4C	109.5	H9B—C9—H9C	109.5
C9—P1—C1—C2	−47.69 (14)	C9—P1—C5—C7	−41.13 (14)
C5—P1—C1—C2	78.12 (13)	C1—P1—C5—C7	−166.88 (11)
C9—P1—C1—C4	−171.92 (12)	C9—P1—C5—C6	78.65 (13)
C5—P1—C1—C4	−46.11 (14)	C1—P1—C5—C6	−47.10 (14)
C9—P1—C1—C3	70.25 (13)	C9—P1—C5—C8	−158.57 (12)
C5—P1—C1—C3	−163.93 (10)	C1—P1—C5—C8	75.67 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
P1—H1···Cl1	1.308 (19)	2.675 (19)	3.8400 (6)	147.2 (12)

Fig. 1

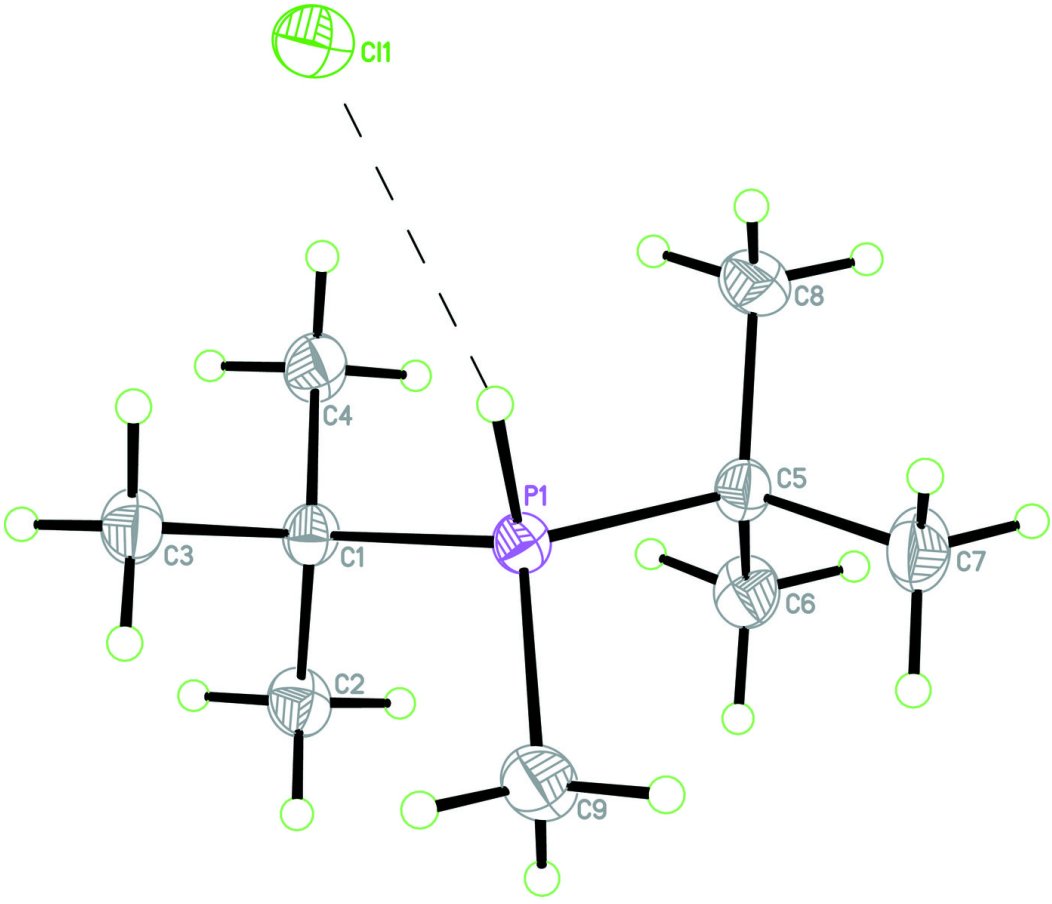

Fig. 2