Crystal structure of methyltriphenylphosphonium iodide, $C_{19}H_{18}IP$

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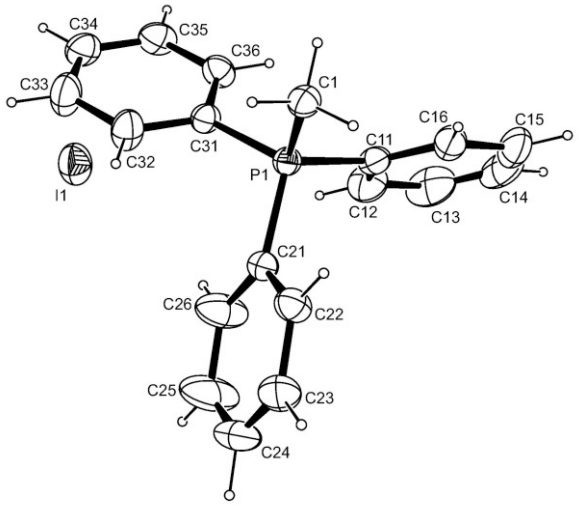

Abstract

$C_{19}H_{18}IP$, monoclinic, $P2_1/c$ (no. 14), a = 9.0703(3) Å, b = 19.0974(6) Å, c = 11.9927(3) Å, β = 120.079(2)°, V = 1797.7 Å^3, Z = 4, $R_{gt}(F)$ = 0.0197, $wR_{ref}(F^2)$ = 0.0464, T = 200 K.

Source of material

The compound was obtained commercially (KEK). Crystals suitable for the *X*-ray diffraction study were obtained upon recrystallization from water.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.243×0.487×0.594 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	18.62 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$2\theta_{max}$:	56.64°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15712, 4463
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4107
$N(param)_{refined}$:	191
Programs:	SHELXS-97 [6], ORTEP-3 [7], MERCURY [8], PLATON [9]

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2 U_{eq}(C). The H atoms of the methyl group were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density (HFIX 137 in the SHELX program suite [6], with U(H) set to 1.5 U_{eq}(C).

Discussion

The crystallization of ionic compounds is strongly influenced by the relative spatial size ratio of anion to cation, and the presence of different anions may influence on the conformation and as well as metric parameters of the cation in the case of bigger, organic cations. At the beginning of a comprehensive study of the influence of various anions on bond lengths and angles among a series of tetra-organo phosphonium compounds, we determined the molecular and crystal structure of the title compound. The molecular structure of the monohydrate as well as the dihydrate of tetraphenylphosphonium bromide are apparent in in the literature [1,2] as is information about the crystal structure of ethyltriphenylphosphonium bromide dihydrate [3]. The molecular geometry around the P atom is tetrahedral with the respective C–P–C angles covering a range of 108.56(8)–110.37(8)°, where the biggest angle is enclosed between two phenyl groups and the smallest angle is found between one of the phenyl groups and the methyl group. The least-squares planes defined by the carbon atoms of the aromatic moieties intersect at angles of 54.54(12)°, 68.66(12)° and 79.38(13)° (Fig. 1). In the crystal, C–H···I contacts whose range falls by more than 0.2 Å below the sum of van-der-Waals radii of the atoms participating are apparent. These are supported by one of the hydrogen atoms of the methyl group and connect the entities of the title compound to discrete ion pairs. In terms of graph-set analysis [4,5], the descriptor for this contact is *D* on the unitary level. The shortest intercentroid distance between two π-systems was measured at 3.8671(16) Å.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.0701	0.1531	–0.0292	0.042
H(1B)	4*e*	0.0396	0.1823	0.0827	0.042
H(1C)	4*e*	–0.0353	0.1075	0.0191	0.042
H(12)	4*e*	0.4691	–0.0207	0.2696	0.056
H(13)	4*e*	0.5350	–0.1164	0.1815	0.072
H(14)	4*e*	0.4042	–0.1293	–0.0401	0.071
H(15)	4*e*	0.2077	–0.0476	–0.1767	0.061
H(16)	4*e*	0.1386	0.0492	–0.0913	0.043
H(22)	4*e*	0.3200	0.2170	0.0486	0.040
H(23)	4*e*	0.5589	0.2872	0.1078	0.054
H(24)	4*e*	0.8030	0.2718	0.3037	0.066
H(25)	4*e*	0.8135	0.1872	0.4466	0.088
H(26)	4*e*	0.5772	0.1158	0.3902	0.068
H(32)	4*e*	0.2612	0.1763	0.3662	0.048
H(33)	4*e*	0.2171	0.1515	0.5378	0.056
H(34)	4*e*	0.1614	0.0376	0.5741	0.048
H(35)	4*e*	0.1465	–0.0524	0.4383	0.046
H(36)	4*e*	0.1894	–0.0290	0.2652	0.039

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P(1)	4*e*	0.25242(5)	0.09924(2)	0.16355(4)	0.0230(2)	0.0249(2)	0.0215(2)	–0.0007(1)	0.0111(2)	0.0008(1)
C(1)	4*e*	0.0601(2)	0.14014(9)	0.0458(2)	0.0239(8)	0.0336(8)	0.0260(8)	0.0031(6)	0.0116(6)	0.0024(6)
C(11)	4*e*	0.2950(2)	0.02292(9)	0.0974(2)	0.0273(8)	0.0270(8)	0.0374(9)	–0.0011(6)	0.0201(7)	–0.0016(6)
C(12)	4*e*	0.4148(3)	–0.0262(1)	0.1787(2)	0.037(1)	0.049(1)	0.053(1)	0.0130(9)	0.022(1)	0.0042(9)
C(13)	4*e*	0.4542(3)	–0.0827(1)	0.1265(3)	0.049(1)	0.045(1)	0.097(2)	0.019(1)	0.045(1)	0.007(1)
C(14)	4*e*	0.3768(3)	–0.0902(1)	–0.0049(3)	0.067(2)	0.040(1)	0.103(2)	–0.003(1)	0.068(2)	–0.017(1)
C(15)	4*e*	0.2601(3)	–0.0419(1)	–0.0860(2)	0.070(2)	0.043(1)	0.063(1)	–0.014(1)	0.051(1)	–0.018(1)
C(16)	4*e*	0.2186(3)	0.01543(9)	–0.0354(2)	0.045(1)	0.0303(9)	0.040(1)	–0.0063(7)	0.0276(9)	–0.0049(7)
C(21)	4*e*	0.4264(2)	0.15909(9)	0.2136(2)	0.0253(8)	0.0301(8)	0.0258(8)	–0.0035(6)	0.0125(7)	–0.0017(6)
C(22)	4*e*	0.4204(2)	0.21042(9)	0.1296(2)	0.0343(9)	0.0318(9)	0.0319(9)	–0.0055(7)	0.0143(8)	0.0001(7)
C(23)	4*e*	0.5624(3)	0.2521(1)	0.1652(2)	0.052(1)	0.040(1)	0.050(1)	–0.0162(9)	0.030(1)	–0.0034(8)
C(24)	4*e*	0.7063(3)	0.2432(1)	0.2810(2)	0.041(1)	0.064(2)	0.059(1)	–0.027(1)	0.024(1)	–0.012(1)
C(25)	4*e*	0.7129(3)	0.1929(2)	0.3654(3)	0.038(1)	0.101(2)	0.050(1)	–0.028(1)	–0.001(1)	0.011(1)
C(26)	4*e*	0.5728(3)	0.1506(1)	0.3320(2)	0.038(1)	0.073(2)	0.037(1)	–0.017(1)	0.0028(9)	0.018(1)
C(31)	4*e*	0.2326(2)	0.07592(8)	0.3004(2)	0.0273(8)	0.0295(8)	0.0234(7)	–0.0014(6)	0.0134(6)	0.0027(6)
C(32)	4*e*	0.2388(3)	0.1297(1)	0.3809(2)	0.061(1)	0.0295(9)	0.040(1)	–0.0050(8)	0.034(1)	–0.0007(7)
C(33)	4*e*	0.2122(3)	0.1149(1)	0.4825(2)	0.068(1)	0.043(1)	0.044(1)	–0.007(1)	0.039(1)	–0.0060(9)
C(34)	4*e*	0.1787(3)	0.0474(1)	0.5038(2)	0.040(1)	0.054(1)	0.0319(9)	–0.0079(9)	0.0219(8)	0.0040(8)
C(35)	4*e*	0.1703(2)	–0.0060(1)	0.4235(2)	0.041(1)	0.037(1)	0.037(1)	–0.0078(8)	0.0192(8)	0.0084(7)
C(36)	4*e*	0.1963(2)	0.00776(9)	0.3210(2)	0.0362(9)	0.0292(8)	0.0297(8)	–0.0051(7)	0.0147(7)	0.0005(6)
I(1)	4*e*	0.09171(2)	0.318820(6)	0.23763(1)	0.04470(7)	0.03178(6)	0.02828(6)	–0.00329(5)	0.02108(5)	–0.00247(4)

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