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The 'absolute structure' of dimethyldiphenylphosphonium iodide

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The structure of the title compound, $C_{14}H_{16}P^{+}\cdot I^{-}$, has been reported previously by Staples, Carlson, Wang & Fackler [*Acta Cryst*. (1995), C**51**, 498–500] in the space group $P6_522$ without any information about the determination of the absolute structure. We present here a redetermination of this structure from new intensity data in the space group $P6_122$ with the direction of the polar axis unequivocally determined for this particular crystal. Both the cation and anion are located on twofold rotation axes.

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Key indicators

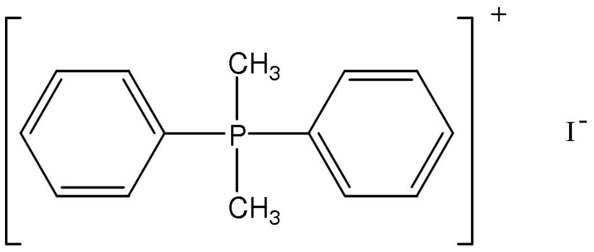
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.020
wR factor = 0.047
Data-to-parameter ratio = 19.9

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

Comment

Recently, we reported that, in the thermolysis of $Me_3PH^{+}\cdot Cl^{-}$, the phosphonium salts $Me_4P^{+}\cdot Cl^{-}$ and $Me_2PH_2^{+}\cdot Cl^{-}$ were formed in CD_2Cl_2 at 323 K (Margraf *et al.*, 2002). Interestingly, we have now observed a reaction of Ph_2PH with CH_3I at room temperature in tetrahydrofuran which produces the phosphonium salts $Ph_2PMe_2^{+}\cdot I^{-}$ and $Ph_2PH_2^{+}\cdot I^{-}$ in quantitative yield (see scheme). X-ray quality crystals of the title compound, (I), were obtained from the reaction solution.

$$2Ph_2PH + 2CH_3I \xrightarrow[\text{tetrahydrofuran}]{\text{r. t.}} Ph_2PMe_2^{+}I^{-} + Ph_2PH_2^{+}I^{-}$$



(I)

A perspective view of (I) is shown in Fig. 1. The structure is composed of discrete $[C_{14}H_{16}P]^{+}$ cations and I^{-} anions. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.7 plus one update; *MOGUL* Version 1.0; Allen, 2002). The cation and anion are each located on twofold rotation axes. Compound (I) has previously been reported in the space group $P6_522$ without any information on the absolute structure (Staples *et al.*, 1995). We present here a redetermination of this structure from new intensity data in the space group $P6_122$ with the direction of the polar axis unequivocally determined for this particular crystal. Apart from the different space groups (corresponding to different absolute structures), there are no significant differences between the two structure determinations.

Experimental

CH_3I (0.201 g) was added to a solution of Ph_2PH (0.210 g) in tetrahydrofuran (3 ml) at room temperature. Colourless crystals of (I) were grown from an acetonitrile solution at ambient temperature.

Crystal data

$C_{14}H_{16}P^+ \cdot I^-$
$M_r = 342.14$
Hexagonal, $P6_122$
$a = 11.7463\ (6)$ Å
$c = 18.2841\ (10)$ Å
$V = 2184.8\ (2)$ Å^3
$Z = 6$
$D_x = 1.560$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 15 434 reflections
$\theta = 3.6$–$26.7°$
$\mu = 2.28$ mm^{-1}
$T = 173\ (2)$ K
Block, colourless
0.22 × 0.20 × 0.17 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{min} = 0.634$, $T_{max} = 0.698$
15 434 measured reflections

1475 independent reflections
1445 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 26.3°$
$h = -14 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -22 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.047$
$S = 1.20$
1475 reflections
74 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2 + 0.9694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.68$ e Å^{-3}
Absolute structure: Flack (1983) 546 Friedel pairs
Flack parameter = −0.02 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7C···I1^{ii}	0.98	3.01	3.983 (2)	174

Symmetry codes: (ii) $1 + x - y, x, \frac{1}{6} + z$.

H atoms were refined with fixed individual displacement parameters [$U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5\ U_{eq}(C_{methyl})$] using a riding model, with C—H = 0.95 and 0.98 Å for aromatic and methyl H atoms, respectively.

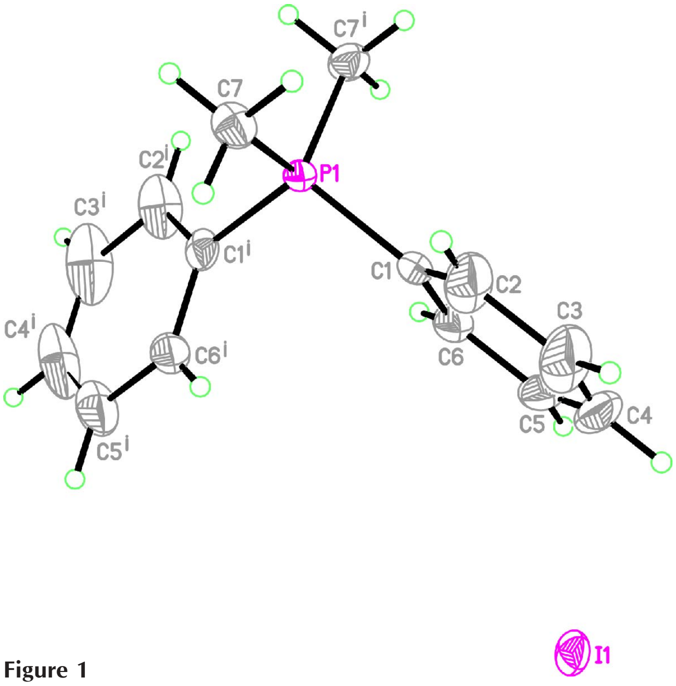


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i) $1 - y, 1 - x, \frac{5}{6} - z$.]

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* and *PLATON* (Spek, 2003).

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