Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

(Benzoylmethyl)triphenylphosphonium iodide: supramolecular zigzag chains *via* C—H···O hydrogen bonding stabilized by C—H···I interactions

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Received 21 January 2003
Accepted 24 January 2003
Online 31 January 2003

The title phosphonium salt, $C_{26}H_{22}OP^+\cdot I^-$, isomorphous with (benzoylmethyl)triphenylphosphonium nitrate, exhibits both intramolecular C—H···I and intermolecular C—H···O interactions. The latter interaction leads to supramolecular parallel zigzag chains along the *a* axis.

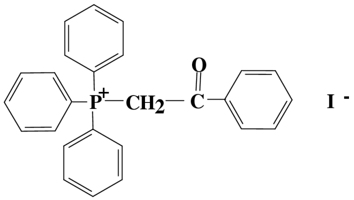
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.026
wR factor = 0.057
Data-to-parameter ratio = 16.2

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

Comment

Phosphonium keto ylides react with metal salts to form not only ylide complexes but also phosphonium salts (Antipin & Struchkov, 1984; Baby Mariyatra *et al.*, 2002) and phosphonium metalates (Albanese *et al.*, 1989; Bart *et al.*, 1982). The action of trimethyltin chloride on (benzoylmethylene)triphenylphosphorane led to the first O-coordinated ketoylide complex (Buckel *et al.*, 1972). The reaction of SnI_4 with the same ylide was used to prepare a six-coordinate complex, containing the ylide. However, the above reaction in benzene yielded the product of composition $[(C_6H_5)_3PCH_2CO\text{-}Ph]_2[SnI_6]$, as revealed by its elemental analysis (analysis found: C 37.98, H 2.78%; calculated: C 38.01, H 2.70%). The product was recrystallized from methanol. Its ^1H NMR in $CDCl_3$ indicates that it is a phosphonium salt, different from the initial phosphonium metalate. The investigation reported herein has been undertaken to assign the molecular and supramolecular structure of the title compound, (I).



(I)

Compound (I) is formed by the C protonation of the parent ylide, and its molecular structure is unexceptional. It is confirmed that compound (I) is an ion pair (Fig. 1), with a distance of 4.6231 (7) Å between the P^+ and I^- centres. The metrical parameters in (I) resemble those of the isomorphous nitrate salt (*Pbca* with $Z = 8$; Baby Mariyatra *et al.*, 2002), but differ from the parent ylide (Kalyanasundari *et al.*, 1994). The crystal structure can best be described as a zigzag chain formed by C—H···O hydrogen-bonding interactions (Table 2 and Fig. 2). The H1—C1—C2—O1 torsion angle [145.4 (14)°] indicates that atoms H1 and O1 are considerably distorted from an antiperiplanar arrangement, as expected for the formation of a zigzag chain. The I^- ions are embedded in the spaces between the chains and contribute to the three-dimensional arrangement *via* three C—H···I interactions (Table 2 and Fig. 3). The C···I distances [3.696 (2), 3.802 (2)

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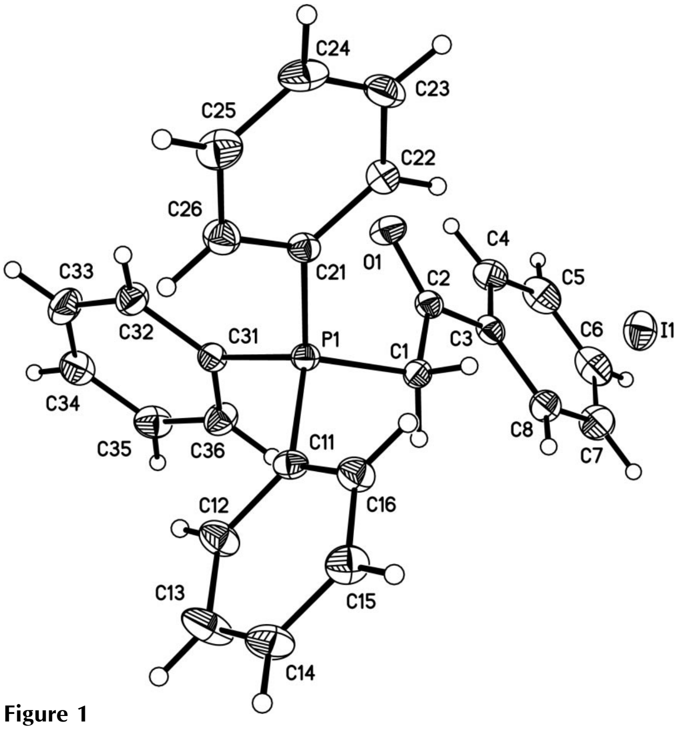

Figure 1
The molecular structure of the title compound, (I), showing 50% probability displacement ellipsoids.

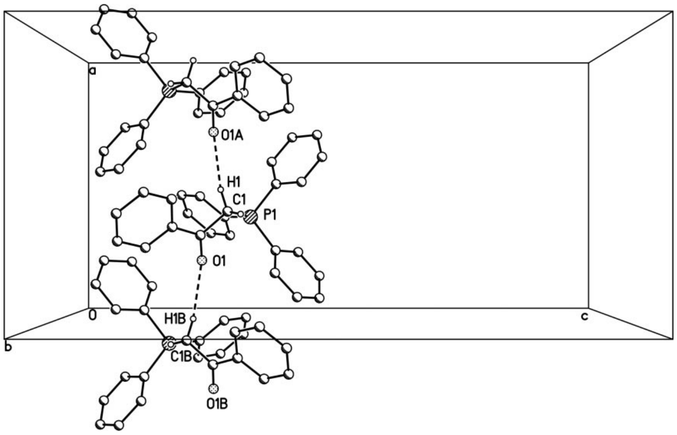

Figure 2
View of the chain along the *a* axis, due to C—H$\cdots$O interactions (dashed lines).

and 3.983 (2) Å] are comparable to those observed in the fulleride salt $[(C_6H_5)_4P]_2(C_{60})I_{0.35}$ (C$\cdots$I$^-$ = 3.67 Å; Penicaud *et al.*, 1993) and those observed in (α-ferrocenylphenacyl)triphenylphosphonium iodide [C$\cdots$I = 3.836 (7), 3.932 (7) and 3.995 (7) Å; Ahmed *et al.*, 1996].

The present results indicate that the phosphonium salt is present in the C-protonated form both in solution and in the solid state. The ylidic C atom is involved in hydrogen-bonding interactions, as observed in many similar structures (Yufit *et al.*, 2000; Baby Mariyatra *et al.*, 2002). We attribute the predominance of the C-protonated form to the considerable carbanionic character of the ylidic C atom in spite of resonance stabilization.

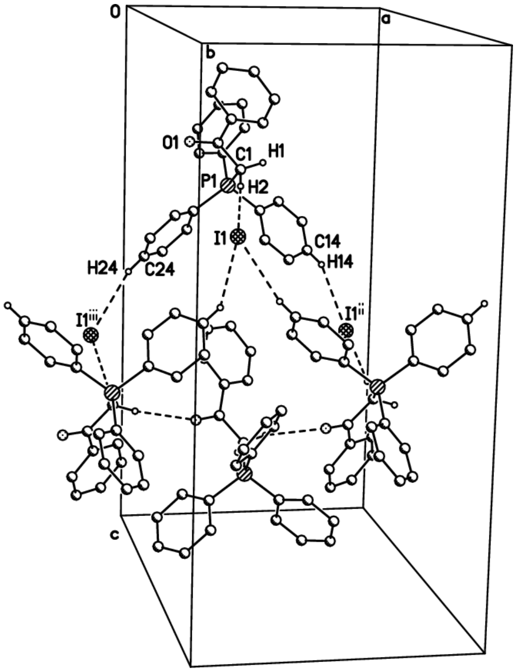

Figure 3
Diagram showing both the C—H$\cdots$I and C—H$\cdots$O interactions (dashed lines). The symmetry codes are as in Table 2.

Experimental

The title phosphonium salt, (I), was obtained when the product of the reaction between stannic iodide and the phosphonium ylide, (benzoylmethylene)triphenylphosphorane, was crystallized from dry methanol.

Crystal data

$C_{26}H_{22}OP^+\cdot I^-$
M_r = 508.31
Orthorhombic, *Pbca*
a = 10.870 (2) Å
b = 18.932 (2) Å
c = 22.135 (3) Å
V = 4555.2 (11) Å^3
Z = 8
D_x = 1.482 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 805 reflections
θ = 10.2–23.6°
μ = 1.49 mm^{-1}
T = 120 (2) K
Block, light yellow
0.46 × 0.32 × 0.28 mm

Data collection

Bruker SMART 1K CCD diffractometer
ω scans
Absorption correction: by integration (*XPREP* in *SHELXTL*; Bruker, 1998)
T_{min} = 0.555, T_{max} = 0.684
29262 measured reflections
5663 independent reflections
4873 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 28.3°
$h = -13 \rightarrow 14$
$k = -25 \rightarrow 24$
$l = -29 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.057
S = 1.05
5663 reflections
350 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0193P)^2 + 4.7P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.62 e Å^{-3}
$\Delta\rho_{min}$ = −0.74 e Å^{-3}

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Table 1
Selected geometric parameters (Å, °).

P1—C1	1.807 (2)	C1—C2	1.518 (3)
O1—C2	1.225 (2)		
C31—P1—C1	108.33 (9)	O1—C2—C1	119.90 (18)
C2—C1—P1	113.28 (14)	C3—C2—C1	118.17 (17)
O1—C2—C3	121.92 (18)		
P1—C1—C2—O1	22.6 (2)	P1—C1—C2—C3	−158.66 (14)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C1—H1$\cdots$O1^{i}	0.96 (2)	2.50 (2)	3.445 (3)	165.2 (18)
C1—H2$\cdots$I1	0.92 (3)	2.78 (3)	3.696 (2)	177 (2)
C14—H14$\cdots$I1^{ii}	0.93 (3)	3.03 (3)	3.802 (2)	141 (2)
C24—H24$\cdots$I1^{iii}	0.92 (3)	3.09 (3)	3.983 (2)	164 (2)

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

All the H atoms were located from difference Fourier maps and their positional and $U_{\rm iso}$ parameters were refined. The C—H bond distances range from 0.91 (3) to 0.98 (2) Å.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SMART-NT*; data reduction: *SAINT-NT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *PLATON*97 (Spek, 1997); software used to prepare material for publication: *SHELXL*97.

References

Ahmed, S. Z., Glidewell, C. & Ferguson, G. (1996). *Acta Cryst.* C**52**, 1634–1636.

Albanese, J. A., Staley, D. L., Rheingold, A. L. & Burmeister, J. L. (1989). *Acta Cryst.* C**45**, 1128–1131.

Antipin, M. Yu. & Struchkov, Yu. T. (1984). *Zh. Strukt. Khim.* **25**, 122–131.

Baby Mariyatra, M., Panchanatheswaran, K. & Goeta, A. E. (2002). *Acta Cryst.* E**58**, o807–o809.

Bart, J. C. J., Bassi, I. W. & Calcaterra, M. (1982). *Inorg. Chim. Acta*, **57**, 261–268.

Bruker (1998). *SMART-NT* (Version 5.0), *SAINT-NT* (Version 5.0) and *SHELXTL-NT* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.

Buckel, J., Harrison, P. G., King, T. J. & Richards J. A. (1972). *Chem. Commun.* pp. 1104–1105.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Kalyanasundari, M., Panchanatheswaran, K., Parthasarathi, V., Robinson, W. T. & Huo, W. (1994). *Acta Cryst.* C**50**, 1738–1741.

Penicaud, A., Perez-Benitez, A., Gleason, V. R., Munoz, P. E. & Escudro, R. (1993). *J. Am. Chem. Soc.* **113**, 10392–10393.

Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.

Spek, A. L. (1997). *PLATON*97. University of Utrecht, The Netherlands.

Yufit, D. S., Howard, J. A. K. & Davidson, M. G. (2000). *J. Chem. Soc. Perkin Trans.* pp. 249–253.

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