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The Crystal and Molecular Structure of 3,3-Dimethyl-1,1-diphenylphosphetanium Iodide

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Abstract

$C_{17}H_{20}P^+ \cdot I^-$ is monoclinic, space group $P2_1/n$, $a = 11.264$ (1), $b = 12.276$ (3), $c = 12.193$ (3) Å, $\beta = 94.60$ (15)°, $Z = 4$. The structure was refined to $R = 0.029$ for 2529 counter reflections. The phosphetane ring is puckered with a dihedral angle of 18.6°.

Introduction

During the past decade the chemistry of the four-membered heterocycle, phosphetane, has been studied

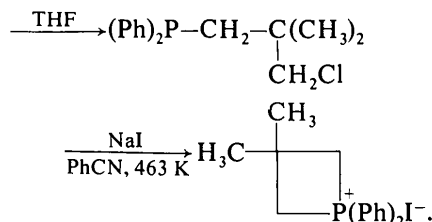
extensively (Trippett, 1970–1976). The ring constraints in this system provide a structural asset for the analysis of P stereochemistry, particularly where pseudo-rotational processes are involved (Westheimer, 1968). A ring methyl substituent *cis* or *trans* to a functional group provides a convenient probe to enable the following stereochemical changes (retention and inversion) about the P atom in chemical reactions (Trippett, 1970–1976; Cremer & Trivedi, 1969).

X-ray studies have provided confirmation of stereochemical assignments and important data on a number of phosphetane derivatives (Swank & Caughlan, 1968; Moret & Trefonas, 1969; Mazhar-ul-Haque, 1970,

1971; Mazhar-ul-Haque, Rashid & Cremer, 1978; Fitzgerald, Campbell, Smith, Caughlan & Cremer, 1978). The average puckering angle for the five symmetrically substituted compounds (Swank & Caughlan, 1968; Moret & Trefonas, 1969; Mazhar-ul-Haque, 1970, 1971) is 22.9°. The two unsymmetrically substituted compounds (Fitzgerald *et al.*, 1978) have puckering angles of 16.7 and 29.8°, which differ from this mean value. It is expected that an unsubstituted phosphetane ring system would exhibit less puckering. The present compound is unsubstituted at C(2) and C(4).

Experimental

The compound was synthesized by a method described by Gray, Cremer & Marsi (1976):



Crystal data

$\text{C}_{17}\text{H}_{20}\text{P}^+\text{I}^-$, $M_r = 382.22$, monoclinic, $a = 11.264$ (1), $b = 12.276$ (3), $c = 12.193$ (3) Å, $\beta = 94.60$ (15)°, $U = 1680.6$ Å³, $D_m = 1.50$, $Z = 4$, $D_c = 1.509$ Mg m⁻³, $F(000) = 760$. Space group $P2_1/n$ uniquely from systematic absences, Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu(\text{Mo } K\alpha) = 2.009$ mm⁻¹.

Preliminary crystal data were obtained with a Nonius-Weissenberg camera. A crystal 0.5 × 0.8 × 0.8 mm was used for data collection. The Enraf-Nonius program *SEARCH* was used to obtain 13 centered reflections which were used in the program *INDEX* to obtain approximate cell dimensions and the orientation matrix for data collection. Accurate cell dimensions were obtained by least squares from 22 accurately centered reflections.

The intensities of 2963 independent reflections [$\theta(\text{Mo } K\alpha) \leq 25^\circ$] were measured on an Enraf-Nonius CAD-4 diffractometer controlled by a PDP8/A computer, with Mo $K\alpha$ radiation from a square-graphite-crystal monochromator at a take-off angle of 5°. Data were collected by the $\omega-(\frac{1}{2})2\theta$ scan technique with the ZIGZAG routine; background counts were taken on each side of the peak. The intensities of two standard reflections were measured after every 90 reflections and showed no significant change. 2529 reflections were considered observed with $I > 2.5\sigma(I)$. Lorentz and

polarization corrections were applied. Form factors for non-hydrogen atoms were those of Cromer & Waber (1965), those for H were of Stewart, Davidson & Simpson (1965). Anomalous terms for I and P were included.

All calculations were carried out on the University of Petroleum and Minerals IBM 370/158 computer with the programs of Ahmed, Hall, Pippy & Huber (1966).

The coordinates of the I and P atoms were determined from a Patterson synthesis, and the complete structure from a Fourier map. Isotropic block-diagonal least-squares refinement reduced R to 0.098. Anisotropic refinement reduced R to 0.043. A difference map at this stage revealed all 20 H atoms. Refinement with anisotropic temperature factors for non-hydrogen atoms and isotropic for H yielded an R of 0.029.* A final difference map showed no significant peaks. A Hughes (1941) weighting scheme of the form $w = 1$ for $|F_o| \leq 20$ and $w^{1/2} = 20/|F_o|$ for $|F_o| > 20$ was used during the final stages.

Results and discussion of the structure

Fig. 1 is an ORTEP drawing (Johnson, 1976) illustrating 30% probability ellipsoids for the non-hydrogen atoms; Fig. 2 shows the packing of the molecules. Atomic coordinates are given in Tables 1 and 2, bond lengths and angles in Table 3, and intermolecular contacts < 4.0 Å in Table 4.

The internal angle at the P atom is 82.2 (1)° with the adjacent angles 88.6 (2) and 88.4 (3)°. The internal angle opposite the P atom is 97.9 (2)°. The phosphetane ring is puckered; P(1) and C(3) are 0.08 and 0.11 Å below and C(2) and C(4) 0.09 Å above the best least-squares plane. The angle between the planes C(2)-P(1)-C(4) and C(2)-C(3)-C(4) is 18.6°.

* Lists of structure factors and all thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34627 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

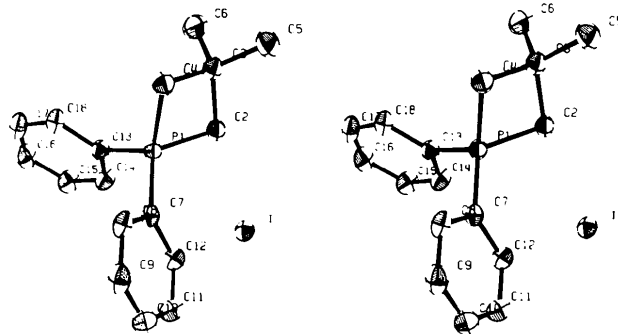


Fig. 1. Stereopair for 3,3-dimethyl-1,1-diphenylphosphetanium iodide, showing the atom numbering.

Table 1. Fractional coordinates (for I and P $\times 10^5$, Table 3. Bond lengths (\AA) and angles ($^\circ$) with e.s.d.'s for C $\times 10^4$), with e.s.d.'s in parentheses

	x	y	z
I	-7097 (2)	46597 (2)	68198 (2)
P(1)	22655 (6)	21074 (6)	79300 (6)
C(2)	2035 (3)	3460 (3)	8452 (3)
C(3)	2968 (3)	3198 (3)	9438 (3)
C(4)	3514 (3)	2138 (3)	8951 (3)
C(5)	3882 (4)	4091 (4)	9652 (4)
C(6)	2336 (4)	2946 (3)	10459 (3)
C(7)	2559 (3)	2036 (2)	6518 (2)
C(8)	3463 (3)	1369 (3)	6180 (3)
C(9)	3619 (4)	1293 (3)	5072 (3)
C(10)	2919 (4)	1873 (3)	4314 (3)
C(11)	2041 (3)	2553 (3)	4648 (3)
C(12)	1853 (3)	2630 (3)	5747 (3)
C(13)	1139 (3)	1140 (2)	8194 (2)
C(14)	-29 (3)	1365 (3)	7801 (3)
C(15)	-903 (3)	602 (4)	7922 (4)
C(16)	-612 (4)	-382 (4)	8416 (4)
C(17)	541 (4)	-606 (3)	8816 (3)
C(18)	1424 (3)	159 (3)	8712 (3)

Table 2. Fractional coordinates of H atoms ($\times 10^3$) with e.s.d.'s in parentheses

	x	y	z
H(1)C(2)	126 (3)	358 (3)	862 (3)
H(2)C(2)	228 (4)	397 (3)	799 (3)
H(3)C(4)	423 (4)	227 (4)	866 (3)
H(4)C(4)	360 (3)	154 (3)	947 (3)
H(5)C(5)	420 (4)	419 (4)	900 (3)
H(6)C(5)	345 (4)	476 (3)	990 (4)
H(7)C(5)	442 (3)	384 (3)	19 (3)
H(8)C(6)	162 (3)	236 (3)	19 (3)
H(9)C(6)	190 (4)	358 (4)	83 (4)
H(10)C(6)	284 (3)	260 (3)	106 (3)
H(11)C(8)	385 (3)	102 (3)	669 (3)
H(12)C(9)	424 (4)	91 (4)	488 (4)
H(13)C(10)	317 (4)	177 (4)	359 (4)
H(14)C(11)	159 (4)	297 (4)	414 (3)
H(15)C(12)	127 (3)	305 (3)	599 (3)
H(16)C(14)	-22 (3)	203 (3)	759 (3)
H(17)C(15)	-168 (4)	73 (4)	775 (4)
H(18)C(16)	-123 (4)	-79 (4)	845 (4)
H(19)C(17)	73 (3)	-128 (3)	914 (3)
H(20)C(18)	225 (3)	0 (3)	901 (3)

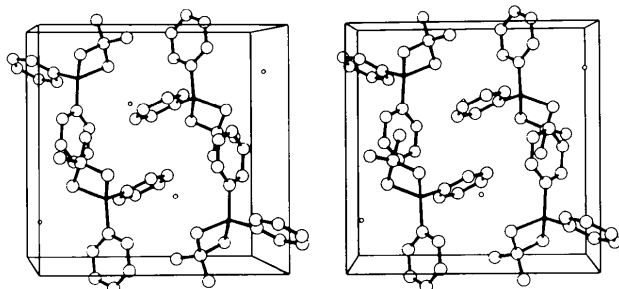


Fig. 2. Stereopair showing the packing of molecules.

P(1)-C(2)	1.804 (3)	C(2)-H(1)	0.92 (3)
P(1)-C(4)	1.802 (3)	C(2)-H(2)	0.90 (4)
P(1)-C(7)	1.781 (3)	C(4)-H(3)	0.92 (4)
P(1)-C(13)	1.786 (3)	C(4)-H(4)	0.97 (4)
C(2)-C(3)	1.565 (4)	C(5)-H(5)	0.91 (4)
C(3)-C(4)	1.576 (5)	C(5)-H(6)	1.01 (5)
C(3)-C(5)	1.512 (5)	C(5)-H(7)	0.92 (4)
C(3)-C(6)	1.514 (5)	C(6)-H(8)	1.10 (4)
C(7)-C(8)	1.395 (4)	C(6)-H(9)	1.04 (5)
C(7)-C(12)	1.388 (4)	C(6)-H(10)	0.99 (4)
C(8)-C(9)	1.380 (6)	C(8)-H(11)	0.84 (4)
C(9)-C(10)	1.366 (6)	C(9)-H(12)	0.89 (4)
C(10)-C(11)	1.380 (6)	C(10)-H(13)	0.95 (4)
C(11)-C(12)	1.377 (5)	C(11)-H(14)	0.82 (4)
C(13)-C(14)	1.391 (5)	C(12)-H(15)	0.90 (3)
C(13)-C(18)	1.386 (4)	C(14)-H(16)	0.88 (3)
C(14)-C(15)	1.376 (5)	C(15)-H(17)	0.90 (4)
C(15)-C(16)	1.377 (6)	C(16)-H(18)	0.86 (5)
C(16)-C(17)	1.378 (6)	C(17)-H(19)	0.93 (4)
C(17)-C(18)	1.381 (5)	C(18)-H(20)	0.99 (4)

C(2)-P(1)-C(4)	82.2 (1)	P(1)-C(7)-C(8)	120.7 (2)
C(2)-P(1)-C(7)	115.4 (1)	P(1)-C(7)-C(12)	119.3 (2)
C(2)-P(1)-C(13)	115.1 (1)	C(8)-C(7)-C(12)	120.0 (3)
C(4)-P(1)-C(7)	118.3 (1)	C(7)-C(8)-C(9)	118.8 (3)
C(4)-P(1)-C(13)	114.6 (1)	C(7)-C(8)-H(11)	115 (3)
C(7)-P(1)-C(13)	109.3 (1)	C(9)-C(8)-H(11)	126 (3)
P(1)-C(2)-C(3)	88.6 (2)	C(8)-C(9)-C(10)	121.1 (4)
P(1)-C(2)-H(1)	113 (2)	C(8)-C(9)-H(12)	117 (3)
P(1)-C(2)-H(2)	111 (3)	C(10)-C(9)-H(12)	121 (3)
C(3)-C(2)-H(1)	117 (2)	C(9)-C(10)-C(11)	120.3 (4)
C(3)-C(2)-H(2)	114 (3)	C(9)-C(10)-H(13)	111 (3)
H(1)-C(2)-H(2)	112 (3)	C(11)-C(10)-H(13)	129 (3)
C(2)-C(3)-C(4)	97.9 (2)	C(10)-C(11)-C(12)	119.9 (3)
C(2)-C(3)-C(5)	112.9 (3)	C(10)-C(11)-H(14)	121 (3)
C(2)-C(3)-C(6)	110.0 (3)	C(12)-C(11)-H(14)	120 (3)
C(4)-C(3)-C(5)	112.5 (3)	C(11)-C(12)-C(7)	120.0 (3)
C(4)-C(3)-C(6)	111.4 (3)	C(11)-C(12)-H(15)	122 (2)
C(5)-C(3)-C(6)	111.4 (3)	C(7)-C(12)-H(15)	118 (2)
P(1)-C(4)-C(3)	88.4 (2)	P(1)-C(13)-C(14)	118.2 (2)
P(1)-C(4)-H(3)	113 (3)	P(1)-C(13)-C(18)	121.2 (2)
P(1)-C(4)-H(4)	117 (2)	C(14)-C(13)-C(18)	120.6 (3)
C(3)-C(4)-H(3)	112 (3)	C(13)-C(14)-C(15)	119.5 (3)
C(3)-C(4)-H(4)	114 (2)	C(13)-C(14)-H(16)	119 (2)
H(3)-C(4)-H(4)	111 (4)	C(15)-C(14)-H(16)	120 (2)
C(3)-C(5)-H(5)	105 (3)	C(14)-C(15)-C(16)	119.8 (4)
C(3)-C(5)-H(6)	108 (3)	C(14)-C(15)-H(17)	123 (3)
C(3)-C(5)-H(7)	106 (2)	C(16)-C(15)-H(17)	117 (3)
H(5)-C(5)-H(6)	112 (4)	C(15)-C(16)-C(17)	121.0 (4)
H(5)-C(5)-H(7)	113 (4)	C(15)-C(16)-H(18)	112 (3)
H(6)-C(5)-H(7)	112 (4)	C(17)-C(16)-H(18)	127 (4)
C(3)-C(6)-H(8)	105 (2)	C(16)-C(17)-C(18)	119.8 (4)
C(3)-C(6)-H(9)	118 (3)	C(16)-C(17)-H(19)	120 (2)
C(3)-C(6)-H(10)	115 (2)	C(18)-C(17)-H(19)	120 (2)
H(8)-C(6)-H(9)	105 (3)	C(17)-C(18)-C(13)	119.3 (3)
H(8)-C(6)-H(10)	108 (3)	C(17)-C(18)-H(20)	120 (2)
H(9)-C(6)-H(10)	105 (4)	C(13)-C(18)-H(20)	121 (2)

There are eight structures determined by X-ray analysis which contain a phosphetane ring (Swank & Caughlan, 1968; Moret & Trefonas, 1969; Mazhar-ul-Haque, 1970, 1971; Mazhar-ul-Haque *et al.*, 1978; Fitzgerald *et al.*, 1978). In seven of these the amount of pucker in the four-membered ring is $16.7\text{--}29.8^\circ$ and

Table 4. Intermolecular contacts $<4.0 \text{ \AA}$ involving non-hydrogen atoms (\AA)

Roman numeral superscripts denote the following equivalent positions, relative to the reference molecule at x, y, z :

(I)	$\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$	(IV)	$\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$
(II)	$x - \frac{1}{2}, \frac{1}{2} - y, \frac{1}{2} + z$	(V)	$-x, -y, 2 - z$
(III)	$1 - x, 1 - y, 2 - z$		

I...C(8 ^I)	3.970 (3)	C(8)...C(17 ^I)	3.879 (5)
I...C(10 ^{II})	3.991 (4)	C(10)...C(14 ^{IV})	3.756 (5)
C(2)...C(8 ^I)	3.649 (4)	C(10)...C(15 ^{IV})	3.823 (6)
C(2)...C(18 ^I)	3.881 (5)	C(11)...C(15 ^{IV})	3.963 (6)
C(5)...C(5 ^{III})	3.421 (6)	C(11)...C(17 ^I)	3.901 (6)
C(5)...C(8 ^I)	3.924 (5)	C(12)...C(17 ^I)	3.652 (5)
C(5)...C(9 ^I)	3.938 (6)	C(12)...C(18 ^I)	3.692 (5)
C(5)...C(14 ^{IV})	3.975 (6)	C(15)...C(17 ^V)	3.966 (6)
C(5)...C(15 ^{IV})	3.992 (6)	C(16)...C(17 ^V)	3.581 (6)
C(6)...C(15 ^{IV})	3.896 (6)	C(16)...C(18 ^V)	3.700 (6)
C(6)...C(16 ^V)	3.999 (6)	C(17)...C(17 ^V)	3.549 (6)
C(7)...C(17 ^I)	3.643 (5)	C(17)...C(18 ^V)	3.918 (6)

one of them is puckered with an angle of 46.6° (Mazhar-ul-Haque *et al.*, 1978). This dihedral angle is almost twice the average puckering angle found in other phosphetanes, an increase which is probably due to the geometric restrictions imposed by the tetracyclic system. The average puckering angle for the five symmetrically substituted compounds is 22.9° (Swank & Caughlan, 1968; Moret & Trefonas, 1969; Mazhar-ul-Haque, 1970, 1971). The two unsymmetrical structures have puckering angles of 16.9 and 29.8° (Fitzgerald *et al.*, 1978). In this compound C(2) and C(4) are unsubstituted; therefore, it is expected to exhibit less puckering than the more-substituted phosphetane ring systems. The puckering angle of 18.6° does show the expected result.

The P—C lengths of 1.804 (3) and 1.802 (3) \AA agree reasonably well with the 1.788 (5) and 1.799 (5) \AA found for unsubstituted and monosubstituted C atoms, respectively, of unsymmetrically substituted phosphetane rings, but are significantly shorter than the 1.83 – 1.94 \AA reported for symmetrically substituted phosphetane rings. The C—C distances in the phosphetane ring, 1.565 (4) and 1.576 (5) \AA , are a little shorter than some and comparable to others found in previous phosphetane ring systems. The P—C(Ph) distances, 1.781 and 1.786 (3) \AA , are similar to those in *N,N*-dimethyldiphenylphosphinamide (Mazhar-ul-Haque & Caughlan, 1976) and are not significantly different from those in other phosphetanes (Swank & Caughlan, 1968; Moret & Trefonas, 1969; Mazhar-ul-Haque, 1970, 1971; Fitzgerald *et al.*, 1978).

In each of the two phenyl rings, the C—C distances agree well with each other; the mean (1.381 and 1.382

\AA) in each phenyl group is not significantly shorter than the standard value [1.395 (3) \AA] (Kennard *et al.*, 1972). Both phenyl groups are planar. The dihedral angle between the two phenyl groups is 77.2° .

Most of the chemically equivalent bond lengths and angles are almost identical, although crystallography does not require this.

The I^- ion is in contact with two C atoms, the closer one at 3.970 \AA . There are 22 C...C contacts <4.0 \AA , the shortest being 3.421 \AA . None of these contacts can be considered close. Therefore, the packing is somewhat loose.

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