C10—C1—B1	114.93 (14)	C20—C2—B2	115.73 (14)
C10—C1—B2	115.81 (14)	C20—C2—B1	113.97 (14)
C10—C1—B7	121.77 (14)	C20—C2—B8	122.67 (14)
C10—C1—B5	118.18 (14)	C20—C2—B6	120.46 (15)
C10—C1—B3	119.68 (14)	C20—C2—B4	117.71 (14)
O10—C10—N10	124.8 (2)	O20—C20—N20	125.1 (2)
O10—C10—C1	120.4 (2)	O20—C20—C2	120.4 (2)
N10—C10—C1	114.8 (2)	N20—C20—C2	114.5 (2)
C10—N10—C11	125.3 (2)	C20—N20—C21	128.0 (2)
C12—C11—C16	120.5 (2)	C22—C21—C26	120.5 (2)
C12—C11—N10	121.1 (2)	C22—C21—N20	123.2 (2)
C16—C11—N10	118.3 (2)	C26—C21—N20	116.4 (2)
B1—C1—C10—O10	−18.3 (4)	B1—C2—C20—O20	32.6 (4)
B2—C1—C10—O10	52.8 (4)	B2—C2—C20—O20	−38.1 (4)
C11—N10—C10—O10	−2.3 (5)	C21—N20—C20—O20	−3.0 (5)

Program used to solve structure: *SHELXTL-Plus* (Sheldrick, 1989). Program used to refine structure: *SHELXL93* (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1141). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Brisson, J. & Brisse, F. (1985). *Can. J. Chem.* **63** 3390–3397.

Brisson, J. & Brisse, F. (1986). *Macromolecules*, **19**, 2632–2639.

Leites, L. A., Kats, G. A., Bukalov S. S. & Komarova, L. G. (1991). *Izv. Akad. Nauk SSSR Ser. Khim.* pp. 371–376.

Ruede, D. R. & Fayos, J. (1982). *Eur. Polym. J.* **18**, 1–4.

Sheldrick, G. M. (1989). *SHELXTL-Plus*. PC version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93*. *Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.

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Bis(triphenylphosphine)iminium Hexafluorophosphate Dichloromethane Solvate (1/1)

ALEXANDER J. BLAKE, GIDEON FREEMAN,
BRIAN F. G. JOHNSON AND GORDON S. MCDOUGALL

Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

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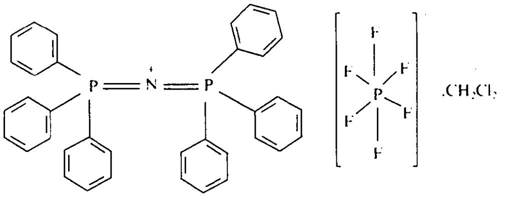
Abstract

Bis(triphenylphosphine)iminium hexafluorophosphate [or bis(phosphoranylidene)ammonium hexafluorophosphate] crystallizes from dichloromethane/hexane as a (1/1) solvate, $[C_{36}H_{30}NP_2][PF_6].CH_2Cl_2$, in which the CH_2Cl_2 molecule is extensively disordered. The cation and anion are essentially ions linked by some short intermolecular contacts.

Comment

The bis(triphenylphosphine)iminium cation $[Ph_3P{=}N^+{=}PPh_3]$ has found extensive use as a large non-interacting organic cation for the crystallization of anionic inorganic species: overwhelmingly these anions are metal carbonyl clusters (*e.g.* Bailey, Conole, Johnson, Lewis, McPartlin, Moule & Wilkinson, 1991) but there are also examples of boranes (Meyer, Muller, Paetzold & Boese, 1992), sulfur–nitrogen species (Chivers, Laidlaw, Oakley & Trsic, 1980) and chloro complexes (Fanwick, King, Tetrick & Walton, 1985).

In bis(triphenylphosphine)iminium hexafluorophosphate dichloromethane solvate, (I), the ions are essentially isolated but linked by relatively short intermolecular contacts [H216···F1(at x, $1+y$, z) = 2.25 (1) Å]. Hexafluorophosphate (PF_6^-) anions typically exhibit a range of disorder modes at ambient temperature involving marked libration or free rotation of the F atoms about one or more axes passing through the P atom. A study of a large number of PF_6^- salts at temperatures below *ca* 180 K has shown that the F atoms then usu-



(I)

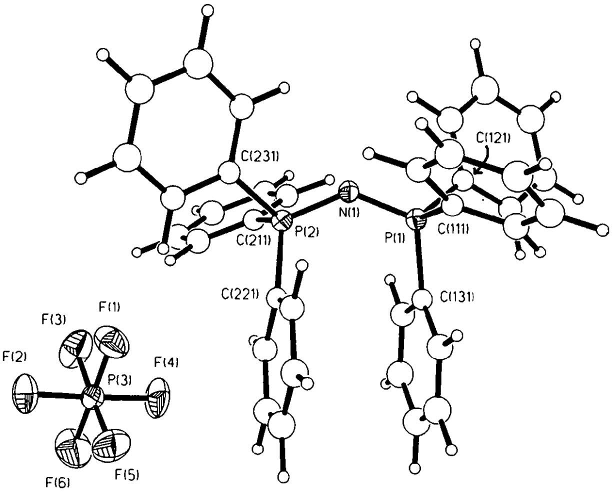


Fig. 1. A view of the ion pair with the atom-numbering scheme employed. Displacement ellipsoids for P, F and N atoms are drawn at the 30% probability level.

ally appear to be ordered (Blake, 1994). In this room-temperature study the anion is essentially ordered but the dichloromethane solvate molecule is extensively disordered.

Experimental

Crystal data

$C_{36}H_{30}NP_2^+.PF_6^-.CH_2Cl_2$
$M_r = 768.45$
Triclinic
$P\bar{1}$
$a = 12.044$ (5) Å
$b = 12.270$ (5) Å
$c = 14.282$ (6) Å
$\alpha = 68.14$ (2)°
$\beta = 66.968$ (13)°
$\gamma = 71.043$ (14)°
$V = 1762.6$ (13) Å^3
$Z = 2$
$D_x = 1.448$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 12 reflections
$\theta = 11$–12°
$\mu = 0.381$ mm^{-1}
$T = 293$ (1) K
Column
0.9 × 0.5 × 0.5 mm
Colourless

Data collection

Stoe Stadi-4 four-circle diffractometer
$\omega/2\theta$ scans [width $(0.9 + 0.35\tan\theta)°$ in ω]
Absorption correction: semi-empirical (ψ scans)
$T_{\min} = 0.185$, $T_{\max} = 0.255$
4551 measured reflections
4551 independent reflections

2518 observed reflections $[I > 2\sigma(I)]$
$\theta_{\max} = 22.51°$
$h = -11 \rightarrow 12$
$k = -11 \rightarrow 13$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity variation: 3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0644$
$wR(F^2) = 0.1682$
$S = 1.165$
4524 reflections
377 parameters
H-atom parameters not refined
Calculated weights $w = 1/[\sigma^2(F_o^2) + (0.1054P)^2 + 2.3325P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.036$
$\Delta\rho_{\max} = 0.74$ e Å^{-3}
$\Delta\rho_{\min} = -0.50$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
P1	0.42324 (15)	0.21064 (14)	0.18293 (13)	0.0381 (5)
P2	0.65493 (15)	0.16838 (15)	0.22587 (13)	0.0379 (5)
N1	0.5639 (4)	0.1528 (4)	0.1783 (4)	0.0417 (13)
C111	0.3276 (3)	0.1047 (3)	0.2706 (3)	0.039 (2)
C112	0.3798 (3)	−0.0108 (4)	0.3216 (3)	0.046 (2)
C113	0.3055 (4)	−0.0939 (3)	0.3872 (3)	0.059 (2)
C114	0.1789 (4)	−0.0617 (4)	0.4017 (3)	0.063 (2)
C115	0.1266 (3)	0.0538 (4)	0.3507 (4)	0.060 (2)
C116	0.2010 (3)	0.1370 (3)	0.2851 (3)	0.050 (2)
C121	0.4090 (4)	0.2429 (4)	0.0542 (3)	0.044 (2)
C122	0.3188 (4)	0.3375 (4)	0.0195 (3)	0.055 (2)
C123	0.3066 (4)	0.3573 (4)	−0.0791 (4)	0.073 (2)
C124	0.3846 (5)	0.2827 (5)	−0.1430 (3)	0.080 (3)
C125	0.4748 (5)	0.1882 (5)	−0.1083 (3)	0.072 (2)
C126	0.4870 (4)	0.1683 (4)	−0.0097 (4)	0.061 (2)
C131	0.3622 (4)	0.3472 (3)	0.2204 (3)	0.040 (2)
C132	0.2698 (4)	0.3566 (3)	0.3155 (3)	0.051 (2)
C133	0.2347 (4)	0.4622 (4)	0.3450 (3)	0.061 (2)
C134	0.2919 (5)	0.5585 (3)	0.2793 (4)	0.066 (2)
C135	0.3842 (4)	0.5490 (3)	0.1842 (4)	0.064 (2)
C136	0.4194 (4)	0.4434 (4)	0.1548 (3)	0.052 (2)
C211	0.7646 (3)	0.2546 (4)	0.1251 (3)	0.044 (2)
C212	0.7637 (4)	0.2955 (4)	0.0202 (3)	0.053 (2)
C213	0.8532 (4)	0.3569 (4)	−0.0589 (2)	0.064 (2)
C214	0.9436 (4)	0.3773 (4)	−0.0331 (3)	0.066 (2)
C215	0.9446 (4)	0.3364 (4)	0.0718 (4)	0.061 (2)
C216	0.8551 (4)	0.2750 (4)	0.1509 (3)	0.051 (2)
C221	0.5796 (4)	0.2314 (4)	0.3351 (3)	0.039 (2)
C222	0.5868 (4)	0.3446 (3)	0.3305 (3)	0.050 (2)
C223	0.5164 (4)	0.3924 (3)	0.4158 (4)	0.058 (2)
C224	0.4389 (4)	0.3269 (4)	0.5057 (3)	0.063 (2)
C225	0.4318 (4)	0.2137 (4)	0.5104 (3)	0.056 (2)
C226	0.5021 (4)	0.1660 (3)	0.4251 (3)	0.049 (2)
C231	0.7438 (4)	0.0220 (3)	0.2747 (3)	0.043 (2)
C232	0.7925 (4)	0.0006 (4)	0.3554 (3)	0.048 (2)
C233	0.8645 (4)	−0.1116 (4)	0.3901 (3)	0.061 (2)
C234	0.8878 (4)	−0.2024 (3)	0.3442 (4)	0.066 (2)
C235	0.8390 (5)	−0.1810 (3)	0.2635 (4)	0.066 (2)
C236	0.7670 (4)	−0.0688 (4)	0.2287 (3)	0.052 (2)
P3	0.8861 (2)	−0.5938 (2)	0.3722 (2)	0.0564 (6)
F1	0.8623 (5)	−0.7125 (4)	0.3702 (4)	0.098 (2)
F2	0.9737 (4)	−0.6751 (4)	0.4424 (4)	0.090 (2)
F3	1.0008 (5)	−0.5983 (5)	0.2696 (4)	0.112 (2)
F4	0.8010 (5)	−0.5139 (4)	0.2990 (4)	0.101 (2)
F5	0.7712 (5)	−0.5953 (6)	0.4755 (4)	0.116 (2)
F6	0.9086 (7)	−0.4779 (5)	0.3759 (6)	0.138 (2)
C1*S*†	0.9535 (23)	0.0064 (26)	0.9611 (19)	0.115 (8)
C2*S*†	0.8827 (25)	0.0994 (25)	0.9217 (22)	0.133 (8)
Cl1*S*‡	0.8000 (4)	−0.0873 (4)	0.9592 (3)	0.133 (2)
Cl2*S*§	0.9259 (17)	−0.0564 (17)	0.9755 (15)	0.125 (6)
Cl3*S*¶	0.9029 (12)	0.0028 (12)	0.8689 (10)	0.128 (4)
Cl4*S*‡	0.8148 (4)	0.1399 (4)	0.8239 (4)	0.154 (2)

† Site occupancy factor = 0.5.
‡ Site occupancy factor = 0.78.
§ Site occupancy factor = 0.19.
¶ Site occupancy factor = 0.26.

Table 2. *Selected geometric parameters* (Å, °)

P1—N1	1.595 (5)	P2—C231	1.805 (3)
P1—C131	1.787 (3)	P3—F1	1.585 (5)
P1—C111	1.794 (3)	P3—F2	1.585 (5)
P1—C121	1.797 (4)	P3—F3	1.577 (5)
P2—N1	1.587 (5)	P3—F4	1.590 (5)
P2—C221	1.787 (3)	P3—F5	1.578 (5)
P2—C211	1.795 (3)	P3—F6	1.554 (5)
N1—P1—C131	114.6 (2)	C212—C211—P2	120.2 (2)
N1—P1—C111	110.5 (2)	C222—C221—P2	123.3 (2)
C131—P1—C111	109.2 (2)	C226—C221—P2	116.4 (2)
N1—P1—C121	108.9 (2)	C236—C231—P2	119.6 (2)
C131—P1—C121	107.4 (2)	C232—C231—P2	120.4 (2)
C111—P1—C121	105.8 (2)	F6—P3—F3	91.2 (4)
N1—P2—C221	114.4 (2)	F6—P3—F5	91.3 (4)
N1—P2—C211	111.2 (2)	F3—P3—F5	177.4 (4)
C221—P2—C211	109.9 (2)	F6—P3—F1	179.2 (4)
N1—P2—C231	108.5 (2)	F3—P3—F1	89.5 (3)
C221—P2—C231	106.4 (2)	F5—P3—F1	87.9 (3)
C211—P2—C231	106.1 (2)	F6—P3—F2	91.0 (3)
P2—N1—P1	134.3 (3)	F3—P3—F2	89.2 (3)
C116—C111—P1	119.8 (2)	F5—P3—F2	90.2 (3)
C112—C111—P1	120.2 (2)	F1—P3—F2	88.7 (3)
C122—C121—P1	121.3 (3)	F6—P3—F4	89.8 (3)
C126—C121—P1	118.7 (3)	F3—P3—F4	89.3 (3)
C132—C131—P1	122.8 (2)	F5—P3—F4	91.3 (3)
C136—C131—P1	116.9 (2)	F1—P3—F4	90.6 (3)
C216—C211—P2	119.7 (2)	F2—P3—F4	178.3 (3)

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL* (Sheldrick, 1994). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1992).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bailey, P. J., Conole, G. C., Johnson, B. F. G., Lewis, J., McPartlin, M., Moule, A. & Wilkinson, D. A. (1991). *Angew. Chem. Int. Ed. Engl.* **30**, 1706–1707.
Blake, A. J. (1994). Unpublished observations.
Chivers, T., Laidlaw, W. G., Oakley, R. T. & Trsic, M. (1980). *J. Am. Chem. Soc.* **102**, 5773–5781.
Fanwick, P. E., King, M. K., Tetrick, S. M. & Walton, R. A. (1985). *J. Am. Chem. Soc.* **107**, 5009–5011.
Meyer, F., Muller, J., Paetzold, P. & Boese, R. (1992). *Angew. Chem. Int. Ed. Engl.* **31**, 1227–1229.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1992). *SHELXTL/PC User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1994). *J. Appl. Cryst.* In preparation.
Stoe & Cie (1988*a*). *DIF*4. *Diffractometer Control Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1988*b*). *REDU*4. *Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.

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1,7,9-Trimethyl-2,6-purinedione Monohydrate, $C_8H_{10}N_4O_2.H_2O$

MASOOD PARVEZ

Department of Chemistry, University of Calgary,
2500 University Drive N.W., Calgary, Alberta,
Canada T2N 1N4

GEORGE FERGUSON

Department of Chemistry and Biochemistry,
University of Guelph, Guelph, Ontario,
Canada N1G 2W1

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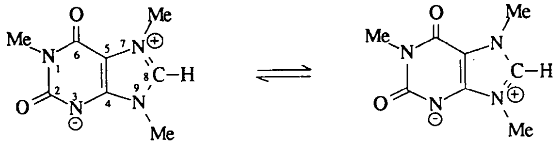
Abstract

The fused-ring system is essentially planar. Both N atoms of the five-membered ring are methylated with a delocalized positive charge (resulting in a zwitterionic structure, 1,7,9-trimethyl-2,6-dioxo-1,3-dihydropurin-(7/9)-ium-3-ide). One N atom of the six-membered ring is also methylated and the other has a formal negative charge. The crystal contains three types of hydrogen bonding, O—H···O, O—H···N and C—H···O with O···O 2.918 (3), O···N 2.896 (3) and C···O 3.009 (3) Å, and H···acceptor 2.05 (4)–2.06(4) Å.

Comment

The analysis established the structure of the title compound, (1), to be as shown in the scheme below and in Fig. 1. The bond lengths are consistent with a significant contribution from the resonance form shown with a formal negative charge on N(3) and the positive charge in the five-membered ring delocalized over N(7) and N(9), resulting in a zwitterionic structure.



(1)

In the six-membered ring, bonds involving N(3) (which has a formal negative charge) are the shortest [N(3)—C(2) 1.351 (3), N(3)—C(4) 1.334 (3) Å] and those involving N(1) are longer [N(1)—C(2) 1.434 (3), N(1)—C(6) 1.391 (3) Å]. The distances involving C(5) [C(4)—C(5) 1.368 (3), C(5)—C(6) 1.421 (3) Å] are also consistent with a major contribution of the ground-state form with C(4)=C(5) a

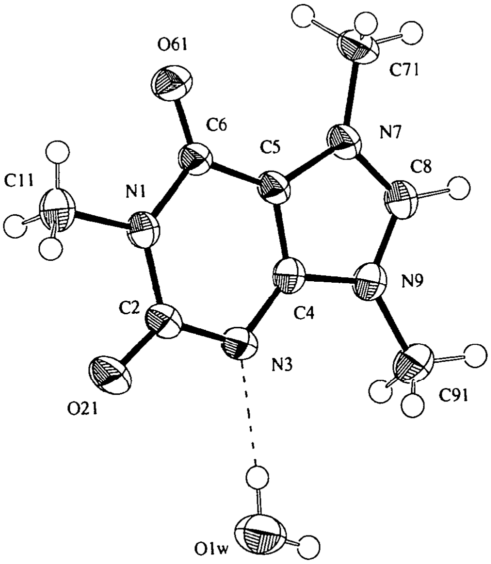


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the crystallographic numbering scheme.