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N,*N*,2,4,6-Pentamethylanilinium hexafluorophosphate

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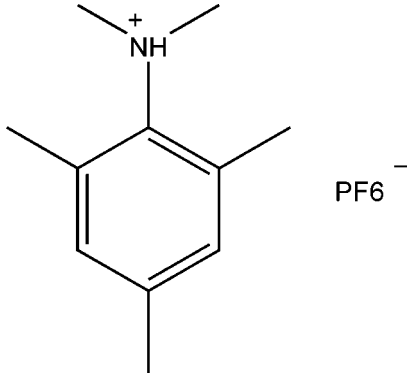
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.063; wR factor = 0.186; data-to-parameter ratio = 18.9.

In the crystal structure of the title salt, $C_{11}H_{18}N^+\cdot PF_6^-$, the cation and anion are connected *via* an N—H···F hydrogen bond; weak C—H···F hydrogen bonding also occurs between the cations and anions.

Related literature

For the background to the title salt, see: Haertling *et al.* (1999); Homes *et al.* (2001).



Experimental

Crystal data

$C_{11}H_{18}N^+\cdot PF_6^-$
M_r = 309.23
Monoclinic, $P2_1/c$
a = 11.466 (2) Å
b = 8.2439 (16) Å
c = 15.559 (3) Å
β = 97.82 (3)°
V = 1457.0 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 K
0.20 × 0.19 × 0.18 mm

Data collection

Rigaku Mercury2 diffractometer
14679 measured reflections
3340 independent reflections
1942 reflections with $I > 2\sigma(I)$
R_{int} = 0.074

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.186
S = 1.03
3340 reflections
177 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···F1	0.91	2.27	2.979 (3)	134
C11—H11A···F4^{i}	0.96	2.47	3.399 (4)	162

Symmetry code: (i) $-x+1, -y+2, -z+1$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5658).

References

Haertling, G. H. (1999). *J. Am. Ceram. Soc.* A**82**, 797–810.
Homes, C. C., Vogt, T., Shapiro, S. M., Wakimoto, S. & Ramirez, A. P. (2001). *Science*, **293**, 673–676.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

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S1. Comment

At present, much attention in ferroelectric material field is focused on developing ferroelectric pure organic or inorganic compounds (Haertling *et al.* 1999; Homes *et al.* 2001). In order to find more dielectric ferroelectric materials, we investigate the physical properties of the title compound (Fig. 1). The dielectric constant of the title compound as a function of temperature indicates that the permittivity is basically temperature-independent (dielectric constant equaling to 3.7 to 5.2), suggesting that this compound should be not a real ferroelectrics or there may be no distinct phase transition occurred within the measured temperature range. Similarly, below the melting point (453 K) of the compound, the dielectric constant as a function of temperature also goes smoothly, and there is no dielectric anomaly observed (dielectric constant equaling to 3.7 to 5.2). Herein, we report the synthesis and crystal structure of the title compound.

Molecules of the title compound have normal geometric parameters. The bond lengths and angles are within their normal ranges. In the crystal, the cation and anion are connected *via* N—H···F and weak C—H···F hydrogen bonds (Table 1).

S2. Experimental

A mix of *N*,*N*,2,4,6-pentamethylbenzenamine (1.36 g, 0.01 mol) and hexafluorophosphoric acid (1.90 g, 0.01 mol) in methanol (20 ml) was stirred until clear. After several days, the title compound was formed and recrystallized from methanol solution to afford colourless prismatic crystals suitable for X-ray analysis.

S3. Refinement

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.96 and N—H = 0.91 Å and U_{iso}(H) = 1.2$_{eq}$(C,N).

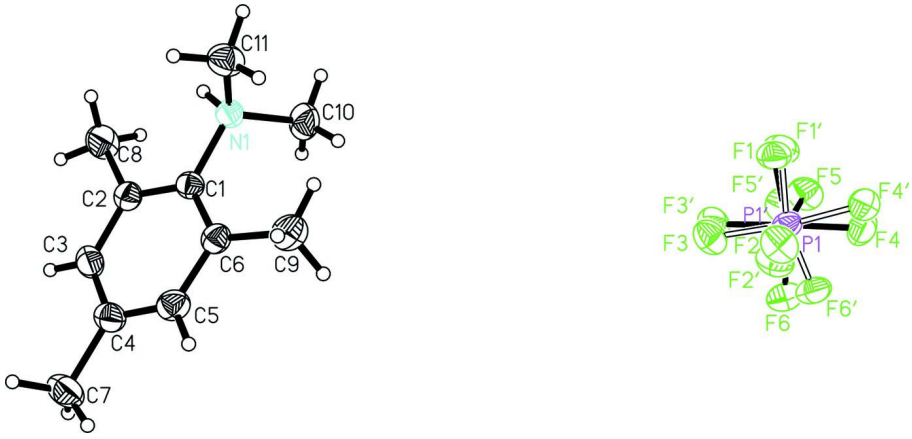

Figure 1

Perspective structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

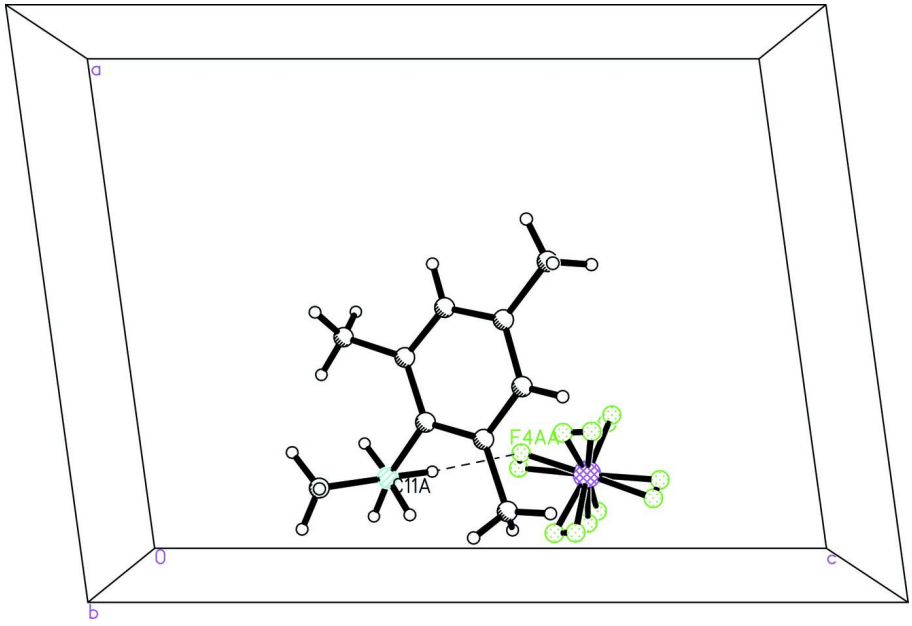

Figure 2

The crystal packing of the title compound viewed along the*b* axis showing the hydrogen bondings network.

N,*N*,2,4,6-Pentamethylanilinium hexafluorophosphate

Crystal data

$C_{11}H_{18}N^{+}\cdot PF_6^{-}$
M_r = 309.23
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 11.466 (2) Å
b = 8.2439 (16) Å
c = 15.559 (3) Å
β = 97.82 (3)°
V = 1457.0 (5) Å^3
Z = 4
$F(000)$ = 640
D_x = 1.410 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3340 reflections
θ = 2.7–27.5°
μ = 0.24 mm^{-1}

T = 293 K
Prism, colorless
0.20 × 0.19 × 0.18 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
14679 measured reflections
3340 independent reflections
1942 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −14→14
k = −10→10
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.186
S = 1.03
3340 reflections
177 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0788P)^2 + 0.4571P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
P1	0.32971 (7)	0.80605 (11)	0.35021 (6)	0.0588 (3)
N1	0.68536 (19)	0.8547 (3)	0.37739 (14)	0.0466 (6)
H1	0.6213	0.8201	0.4009	0.056*
F1	0.43410 (18)	0.9123 (3)	0.39786 (18)	0.1138 (9)
F2	0.2284 (2)	0.6948 (3)	0.30436 (15)	0.0956 (8)
F3	0.3564 (2)	0.8663 (3)	0.25858 (15)	0.1075 (8)
F4	0.3043 (2)	0.7382 (4)	0.44062 (14)	0.1104 (9)
F5	0.4216 (2)	0.6632 (3)	0.34402 (15)	0.0948 (7)
F6	0.2418 (2)	0.9486 (3)	0.3564 (2)	0.1270 (11)
C1	0.7884 (2)	0.7941 (3)	0.43803 (17)	0.0432 (6)
C2	0.7626 (2)	0.7091 (3)	0.51013 (18)	0.0482 (7)
C3	0.8564 (3)	0.6512 (4)	0.56766 (19)	0.0559 (8)
H3	0.8409	0.5922	0.6158	0.067*
C4	0.9722 (3)	0.6782 (4)	0.5560 (2)	0.0560 (8)
C5	0.9930 (3)	0.7638 (4)	0.4838 (2)	0.0568 (8)

H5	1.0706	0.7832	0.4757	0.068*
C6	0.9034 (2)	0.8228 (3)	0.42212 (18)	0.0487 (7)
C7	1.0726 (3)	0.6148 (4)	0.6206 (2)	0.0763 (11)
H7A	1.1392	0.5919	0.5912	0.114*
H7B	1.0484	0.5172	0.6468	0.114*
H7C	1.0940	0.6949	0.6647	0.114*
C8	0.6383 (3)	0.6771 (5)	0.5279 (2)	0.0735 (10)
H8A	0.6403	0.6149	0.5802	0.110*
H8B	0.5964	0.6177	0.4803	0.110*
H8C	0.5991	0.7784	0.5344	0.110*
C9	0.9394 (3)	0.9078 (5)	0.3437 (2)	0.0707 (10)
H9A	0.9402	0.8312	0.2973	0.106*
H9B	1.0166	0.9534	0.3584	0.106*
H9C	0.8842	0.9928	0.3256	0.106*
C10	0.6719 (3)	0.7799 (5)	0.2888 (2)	0.0751 (10)
H10A	0.7327	0.8200	0.2576	0.113*
H10B	0.5963	0.8079	0.2580	0.113*
H10C	0.6783	0.6642	0.2940	0.113*
C11	0.6748 (3)	1.0353 (4)	0.3754 (2)	0.0693 (9)
H11A	0.6797	1.0758	0.4336	0.104*
H11B	0.6005	1.0656	0.3434	0.104*
H11C	0.7375	1.0806	0.3480	0.104*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0438 (5)	0.0666 (6)	0.0658 (6)	−0.0018 (4)	0.0060 (4)	−0.0077 (4)
N1	0.0410 (12)	0.0534 (14)	0.0461 (13)	−0.0011 (10)	0.0077 (10)	0.0047 (10)
F1	0.0631 (13)	0.121 (2)	0.154 (2)	−0.0277 (14)	0.0021 (14)	−0.0462 (17)
F2	0.0882 (15)	0.1006 (17)	0.0926 (16)	−0.0308 (13)	−0.0073 (12)	−0.0137 (12)
F3	0.126 (2)	0.1015 (18)	0.0994 (18)	0.0058 (15)	0.0303 (15)	0.0358 (14)
F4	0.1209 (19)	0.148 (2)	0.0674 (15)	−0.0243 (18)	0.0314 (13)	−0.0149 (14)
F5	0.0918 (15)	0.0934 (16)	0.1004 (17)	0.0358 (13)	0.0172 (13)	0.0080 (12)
F6	0.0688 (14)	0.0922 (18)	0.222 (3)	0.0201 (13)	0.0262 (16)	−0.0326 (19)
C1	0.0427 (14)	0.0416 (15)	0.0451 (15)	0.0000 (12)	0.0057 (11)	−0.0008 (12)
C2	0.0487 (15)	0.0503 (17)	0.0466 (16)	0.0005 (13)	0.0096 (12)	0.0003 (13)
C3	0.064 (2)	0.0586 (19)	0.0435 (16)	0.0016 (15)	0.0034 (14)	0.0015 (13)
C4	0.0576 (18)	0.0516 (18)	0.0548 (18)	0.0065 (14)	−0.0069 (14)	−0.0121 (14)
C5	0.0431 (15)	0.0622 (19)	0.065 (2)	−0.0031 (14)	0.0062 (14)	−0.0076 (16)
C6	0.0448 (15)	0.0517 (17)	0.0504 (17)	−0.0029 (13)	0.0092 (12)	−0.0023 (13)
C7	0.076 (2)	0.074 (2)	0.070 (2)	0.0143 (19)	−0.0217 (18)	−0.0071 (18)
C8	0.059 (2)	0.096 (3)	0.068 (2)	−0.0054 (18)	0.0193 (16)	0.0252 (19)
C9	0.0540 (18)	0.086 (2)	0.076 (2)	−0.0051 (18)	0.0245 (16)	0.0151 (19)
C10	0.067 (2)	0.105 (3)	0.051 (2)	−0.005 (2)	−0.0001 (15)	−0.0204 (18)
C11	0.069 (2)	0.060 (2)	0.079 (2)	0.0075 (17)	0.0088 (17)	0.0133 (17)

Geometric parameters (Å, º)

P1—F6	1.560 (2)	C5—C6	1.394 (4)
P1—F2	1.573 (2)	C5—H5	0.9300
P1—F3	1.579 (2)	C6—C9	1.512 (4)
P1—F4	1.577 (3)	C7—H7A	0.9600
P1—F1	1.584 (2)	C7—H7B	0.9600
P1—F5	1.591 (2)	C7—H7C	0.9600
N1—C11	1.494 (4)	C8—H8A	0.9600
N1—C1	1.494 (3)	C8—H8B	0.9600
N1—C10	1.499 (4)	C8—H8C	0.9600
N1—H1	0.9100	C9—H9A	0.9600
C1—C2	1.388 (4)	C9—H9B	0.9600
C1—C6	1.394 (4)	C9—H9C	0.9600
C2—C3	1.387 (4)	C10—H10A	0.9600
C2—C8	1.512 (4)	C10—H10B	0.9600
C3—C4	1.382 (4)	C10—H10C	0.9600
C3—H3	0.9300	C11—H11A	0.9600
C4—C5	1.374 (4)	C11—H11B	0.9600
C4—C7	1.515 (4)	C11—H11C	0.9600
F6—P1—F2	91.36 (14)	C6—C5—H5	118.4
F6—P1—F3	91.42 (16)	C1—C6—C5	116.4 (3)
F2—P1—F3	89.77 (14)	C1—C6—C9	126.2 (3)
F6—P1—F4	90.74 (17)	C5—C6—C9	117.4 (3)
F2—P1—F4	89.02 (14)	C4—C7—H7A	109.5
F3—P1—F4	177.55 (15)	C4—C7—H7B	109.5
F6—P1—F1	90.47 (14)	H7A—C7—H7B	109.5
F2—P1—F1	177.89 (15)	C4—C7—H7C	109.5
F3—P1—F1	91.24 (15)	H7A—C7—H7C	109.5
F4—P1—F1	89.91 (15)	H7B—C7—H7C	109.5
F6—P1—F5	178.83 (14)	C2—C8—H8A	109.5
F2—P1—F5	89.71 (14)	C2—C8—H8B	109.5
F3—P1—F5	88.11 (13)	H8A—C8—H8B	109.5
F4—P1—F5	89.76 (15)	C2—C8—H8C	109.5
F1—P1—F5	88.47 (13)	H8A—C8—H8C	109.5
C11—N1—C1	113.6 (2)	H8B—C8—H8C	109.5
C11—N1—C10	113.1 (3)	C6—C9—H9A	109.5
C1—N1—C10	114.6 (2)	C6—C9—H9B	109.5
C11—N1—H1	104.7	H9A—C9—H9B	109.5
C1—N1—H1	104.7	C6—C9—H9C	109.5
C10—N1—H1	104.7	H9A—C9—H9C	109.5
C2—C1—C6	122.7 (2)	H9B—C9—H9C	109.5
C2—C1—N1	116.2 (2)	N1—C10—H10A	109.5
C6—C1—N1	121.1 (2)	N1—C10—H10B	109.5
C1—C2—C3	117.6 (3)	H10A—C10—H10B	109.5
C1—C2—C8	123.0 (3)	N1—C10—H10C	109.5
C3—C2—C8	119.3 (3)	H10A—C10—H10C	109.5

C4—C3—C2	122.3 (3)	H10B—C10—H10C	109.5
C4—C3—H3	118.9	N1—C11—H11A	109.5
C2—C3—H3	118.9	N1—C11—H11B	109.5
C5—C4—C3	117.8 (3)	H11A—C11—H11B	109.5
C5—C4—C7	121.3 (3)	N1—C11—H11C	109.5
C3—C4—C7	120.9 (3)	H11A—C11—H11C	109.5
C4—C5—C6	123.2 (3)	H11B—C11—H11C	109.5
C4—C5—H5	118.4		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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