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1,1,4,7,7-Pentamethyldiethylenetriammonium bis(hexafluorophosphate)

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.105
Data-to-parameter ratio = 17.2

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

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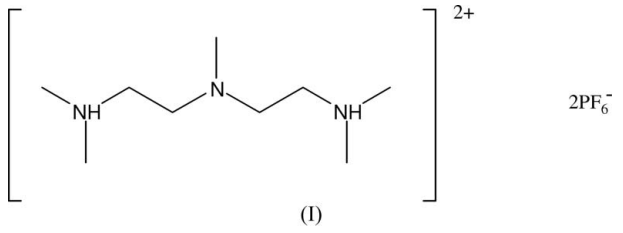
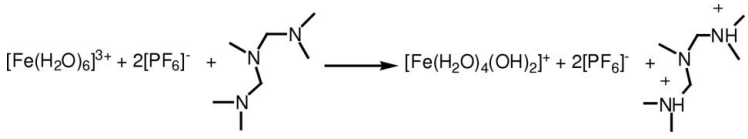
The title compound, $C_9H_{25}N_3^{2+}\cdot 2PF_6^-$, consists of discrete hexafluorophosphate anions and 1,1,4,7,7-pentamethyldiethylenetriamine cations. The geometric parameters are in the usual ranges. Only one PF_6^- cation forms hydrogen bonds with both NH donors of the cation.

Comment

We are interested in the synthesis of transition metal complexes with 1,1,4,7,7-pentamethyldiethylenetriamine (PMDTA) as a ligand (Margraf *et al.*, 2005). In an attempt to prepare an iron(III) complex with PMDTA and hexafluorophosphate as counter-ion, as shown in the reaction scheme below, we obtained the title compound, (I).



(I)



A perspective view of (I) is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.6 plus three updates; *MOGUL* Version 1.0; Allen, 2002).

The title compound crystallizes with discrete hexafluorophosphate anions and 1,1,4,7,7-pentamethyldiethylenetriamine cations. Both $N-CH_2-CH_2-N$ linkages of the cation adopt a *gauche* conformation. Only one PF_6^- cation forms hydrogen bonds with both NH donors of the cation. The P—F bonds in this anion are significantly different (Table 1). The F atom forming two hydrogen bonds shows the longest P—F bond. The P—F bonds involving the F atoms forming only one hydrogen bond are slightly longer than the remaining three. In the other cation, however, all the P—F bonds are of almost the same length.

Experimental

PMDTA (1.52 mmol) was added to a solution of $Fe(PF_6)_3$ (1.52 mmol) in acetonitrile. Colourless crystals of the title compound

suitable for X-ray diffraction were grown by slow diffusion of diethyl ether into an acetonitrile solution at ambient temperature.

Crystal data

$C_9H_{25}N_3^{2+}\cdot 2PF_6^-$
M_r = 465.26
Monoclinic, $P2_1/c$
a = 8.7310 (5) Å
b = 12.7348 (5) Å
c = 17.2366 (10) Å
β = 99.440 (5)°
V = 1890.54 (17) Å^3
Z = 4
D_x = 1.635 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 26656 reflections
θ = 2.0–27.5°
μ = 0.34 mm^{-1}
T = 173 (2) K
Block, colourless
0.49 × 0.48 × 0.38 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
T_{min} = 0.850, T_{max} = 0.881
26656 measured reflections
4201 independent reflections
4042 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 27.2°
$h = -11 \rightarrow 11$
$k = -15 \rightarrow 16$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.11
4201 reflections
244 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.8541P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.021 (3)

Table 1
Selected geometric parameters (Å, °).

P1—F11	1.5842 (13)	P2—F24	1.5989 (10)
P1—F14	1.5874 (11)	P2—F22	1.6026 (11)
P1—F15	1.5957 (11)	P2—F23	1.6029 (11)
P1—F13	1.5987 (12)	P2—F26	1.6050 (10)
P1—F12	1.6062 (12)	P2—F25	1.6055 (10)
P1—F16	1.6262 (10)	P2—F21	1.6087 (11)
N1—C2—C3—N4	52.69 (18)	N4—C5—C6—N7	−52.33 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···F16	0.88 (2)	2.18 (2)	2.9590 (16)	146 (2)
N1—H1···F12	0.88 (2)	2.49 (2)	3.1482 (18)	132 (2)
N7—H7···F16	0.85 (2)	2.21 (2)	2.9502 (16)	145 (2)
N7—H7···F13	0.85 (2)	2.40 (2)	3.0883 (17)	139 (2)

H atoms were located in a difference map, but those bonded to C were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C)] using a riding model, with C—H = 0.98 and 0.99 Å, for methyl and methylene H atoms, respectively. H atoms bonded to N were refined isotropically.

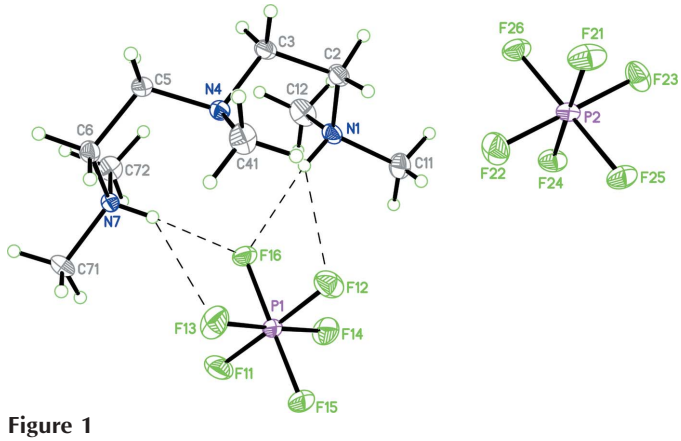


Figure 1
Perspective view of the title compound, with the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level; hydrogen bonds are shown as dashed lines.

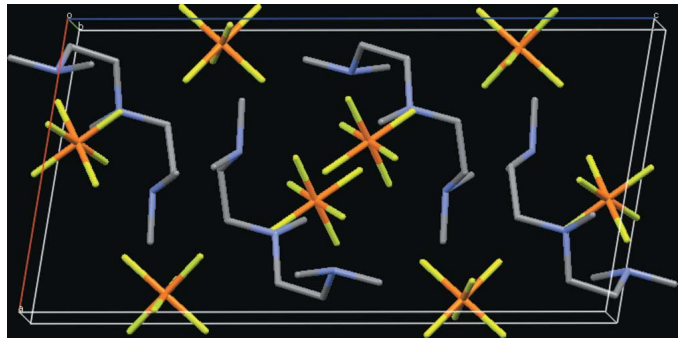


Figure 2
Packing diagram of the title compound, viewed on to the *ac* plane; H atoms have been omitted for clarity.

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) and *MERCURY* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97*.

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