

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-[5-[N-(2-Pyridyl)carbamoyl]pentan-amido}pyridinium hexafluorophosphate

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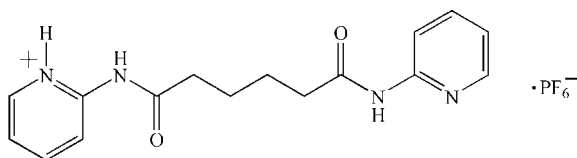
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Received 17 June 2009; accepted 7 July 2009

 Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.036; wR factor = 0.096; data-to-parameter ratio = 12.1.

In the crystal structure of the title compound, $\text{C}_{16}\text{H}_{19}\text{N}_4\text{O}_2^+ \cdot \text{PF}_6^-$, the cations and anions are situated on centres of inversion. Thus, the N—H H atom is disordered over both N atoms due to symmetry. In the crystal, molecules are connected *via* N—H \cdots F and N—H \cdots O hydrogen bonds. The cation adopts the $\cdots\text{AAA}\cdots$ *trans* conformation in the solid state.

Related literature

 For similar structures, see: Chen *et al.* (2007).


Experimental

Crystal data

 $\text{C}_{16}\text{H}_{19}\text{N}_4\text{O}_2^+ \cdot \text{PF}_6^-$
 $M_r = 444.32$

 Monoclinic, $P2_1/c$
 $a = 6.2119$ (18) Å

 $b = 12.9265$ (11) Å

 $c = 11.439$ (2) Å

 $\beta = 96.415$ (10) $^\circ$
 $V = 912.8$ (3) Å³
 $Z = 2$

 Mo $K\alpha$ radiation

 $\mu = 0.23$ mm⁻¹
 $T = 295$ K

 $0.5 \times 0.2 \times 0.2$ mm

Data collection

 Bruker P4 diffractometer
 Absorption correction: multi-scan
 (*XSCANS*; Siemens, 1995)
 $T_{\min} = 0.945$, $T_{\max} = 0.962$
 2288 measured reflections
 1612 independent reflections

 1334 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 3 standard reflections
 every 97 reflections
 intensity decay: none

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.096$
 $S = 1.07$
 1612 reflections

 133 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.30$ e Å⁻³
Table 1

 Hydrogen-bond geometry (Å, $^\circ$).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots F1	0.86	1.98	2.737 (2)	145
N1—H1A \cdots O	0.86	2.10	2.674 (2)	124
N2—H2A \cdots F3 ⁱ	0.86	1.95	2.774 (2)	161
N2—H2A \cdots F1 ⁱ	0.86	2.40	3.050 (2)	133

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

Data collection: *XSCANS* (Siemens, 1995); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We are grateful to the National Science Council of the Republic of China for support. This research was also supported by the project of specific research fields in Chung-Yuan Christian University, Taiwan, under grant CYCU-97-CR-CH.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2150).

References

- Chen, H.-C., Hu, H.-L., Chan, Z.-K., Yeh, C.-W., Jia, H.-W., Wu, C.-P., Chen, J.-D. & Wang, J.-C. (2007). *Cryst. Growth Des.* **7**, 698–704.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Siemens (1995). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supporting information

Acta Cryst. (2009). E65, o1825 [doi:10.1107/S1600536809026488]

2-{5-[*N*-(2-Pyridyl)carbamoyl]pentanamido}pyridinium hexafluorophosphate

Pei-Chi Cheng, Chia-Jun Wu, Huan-Ching Chen, Jhy-Der Chen and Ju-Chun Wang

S1. Comment

The compound *N*¹,*N*²-di(2-pyridyl)adipoamide has been used as bridging ligand in coordination chemistry (Chen *et al.*, 2007). In the present work the structure of the title compound (Fig. 1) has been determined to investigate the role of the cation-anion interaction on the ligand conformation. The molecules are connected *via* N—H···F and N—H···O hydrogen bonds (Tab. 1). The cation adopts the AAA *trans* conformation in the solid state. This conformation is the same as that found for the neutral *N*¹,*N*²-di(2-pyridyl)adipoamide ligand which cocrystallize with water (Chen *et al.*, 2007).

S2. Experimental

*N*¹,*N*²-Di(2-pyridyl)adipoamide (0.30 g, 1.00 mmol) and AgPF₆ (0.25 g, 1.00 mmol) were placed in a flask containing 20 ml of CH₂Cl₂. The mixture was refluxed for 8 h to give a white precipitate, which was then filtered and dried under vacuum. By dissolving the solid in dichloromethane, followed by allowing the solvent to evaporate slowly under air, plate colorless crystals suitable for X-ray crystallography were obtained.

S3. Refinement

All the hydrogen atoms were situated into idealized positions and constrained by the riding atom approximation with C—H = 0.93 — 0.97 Å, N—H = 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$. The occupancy of the H atom H1A was set to be 0.5 to balance the charge. Because of the disorder of the N-H H atom, the structure was also refined in space group *Pc* and *P2*₁. However, even in these cases the disorder is still present and therefore, space group *P2*₁/*c* was selected.

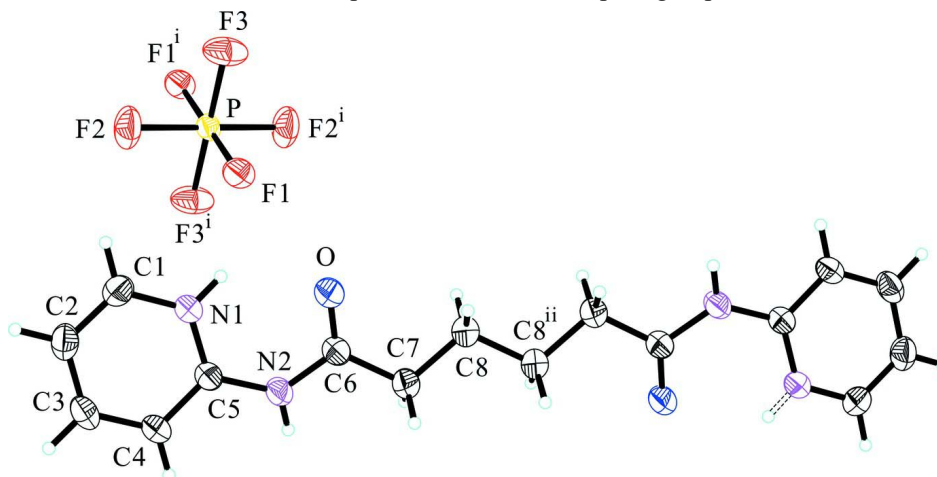


Figure 1

The title molecule with the labelling scheme. The bond to the disordered H atom is indicated by dashed open lines. The displacement ellipsoids are drawn at the 30% probability level. Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x-1, -y, -z$.

2-[5-[N-(2-Pyridyl)carbamoyl]pentanamido]pyridinium hexafluorophosphate

*Crystal data*C₁₆H₁₉N₄O₂⁺·PF₆⁻ $M_r = 444.32$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 6.2119 (18) \text{ \AA}$ $b = 12.9265 (11) \text{ \AA}$ $c = 11.439 (2) \text{ \AA}$ $\beta = 96.415 (10)^\circ$ $V = 912.8 (3) \text{ \AA}^3$ $Z = 2$ $F(000) = 456$ $D_x = 1.617 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 27 reflections

 $\theta = 5.1\text{--}12.5^\circ$ $\mu = 0.23 \text{ mm}^{-1}$ $T = 295 \text{ K}$

Plate, colorless

 $0.5 \times 0.2 \times 0.2 \text{ mm}$ *Data collection*

Bruker P4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ω scans

Absorption correction: multi-scan

(XSCANS; Siemens, 1995)

 $T_{\min} = 0.945$, $T_{\max} = 0.962$

2288 measured reflections

1612 independent reflections

1334 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.020$ $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 2.4^\circ$ $h = -1 \rightarrow 7$ $k = -15 \rightarrow 1$ $l = -13 \rightarrow 13$

3 standard reflections every 97 reflections

intensity decay: none

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.036$ $wR(F^2) = 0.096$ $S = 1.07$

1612 reflections

133 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

 $w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.4314P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.40 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.30 \text{ e \AA}^{-3}$ *Special details*

Experimental. 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 $F^2 > \sigma(F^2)$ 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.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
P	0.0000	0.5000	0.0000	0.0275 (2)	
F1	0.0589 (2)	0.37673 (9)	-0.03443 (10)	0.0367 (3)	
F2	0.2624 (2)	0.52790 (12)	0.01505 (15)	0.0549 (4)	
F3	-0.0162 (3)	0.53230 (11)	-0.14390 (11)	0.0517 (4)	
O	-0.0438 (3)	0.16414 (14)	0.01248 (14)	0.0526 (5)	
N1	0.2814 (3)	0.26186 (14)	0.14024 (15)	0.0331 (4)	
H1A	0.1777	0.2752	0.0861	0.040*	0.50
N2	0.0760 (3)	0.12150 (15)	0.20013 (15)	0.0352 (4)	
H2A	0.0473	0.0858	0.2599	0.042*	
C1	0.4629 (4)	0.32123 (18)	0.1488 (2)	0.0387 (5)	
H1B	0.4739	0.3749	0.0957	0.046*	
C2	0.6283 (4)	0.30293 (19)	0.2341 (2)	0.0410 (5)	
H2B	0.7533	0.3430	0.2392	0.049*	
C3	0.6078 (4)	0.22297 (19)	0.3140 (2)	0.0386 (5)	
H3A	0.7187	0.2102	0.3739	0.046*	
C4	0.4248 (4)	0.16336 (18)	0.30451 (18)	0.0347 (5)	
H4A	0.4107	0.1102	0.3579	0.042*	
C5	0.2600 (3)	0.18256 (16)	0.21465 (17)	0.0300 (5)	
C6	-0.0652 (4)	0.11249 (17)	0.09912 (19)	0.0336 (5)	
C7	-0.2390 (4)	0.03259 (18)	0.10327 (19)	0.0365 (5)	
H7A	-0.3056	0.0409	0.1755	0.044*	
H7B	-0.1743	-0.0357	0.1041	0.044*	
C8	-0.4124 (4)	0.04088 (18)	-0.0011 (2)	0.0375 (5)	
H8A	-0.4784	0.1089	-0.0012	0.045*	
H8B	-0.3451	0.0339	-0.0733	0.045*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P	0.0269 (4)	0.0280 (4)	0.0265 (4)	-0.0007 (3)	-0.0013 (3)	-0.0024 (3)
F1	0.0443 (7)	0.0275 (6)	0.0364 (6)	0.0055 (6)	-0.0032 (5)	-0.0043 (5)
F2	0.0268 (7)	0.0474 (8)	0.0887 (12)	-0.0045 (6)	-0.0011 (7)	-0.0043 (8)
F3	0.0854 (11)	0.0420 (8)	0.0276 (7)	0.0119 (7)	0.0052 (7)	0.0018 (6)
O	0.0577 (12)	0.0553 (11)	0.0398 (9)	-0.0212 (9)	-0.0163 (8)	0.0162 (8)
N1	0.0326 (10)	0.0340 (10)	0.0319 (9)	-0.0024 (8)	-0.0009 (8)	0.0032 (8)
N2	0.0315 (10)	0.0448 (11)	0.0282 (9)	-0.0073 (9)	-0.0020 (7)	0.0074 (8)
C1	0.0428 (13)	0.0332 (12)	0.0395 (12)	-0.0057 (10)	0.0023 (10)	0.0022 (10)
C2	0.0328 (12)	0.0410 (13)	0.0482 (13)	-0.0077 (10)	-0.0002 (10)	-0.0053 (11)
C3	0.0307 (11)	0.0445 (14)	0.0381 (12)	0.0037 (10)	-0.0077 (9)	-0.0038 (10)
C4	0.0333 (12)	0.0385 (12)	0.0305 (10)	0.0014 (10)	-0.0040 (9)	0.0043 (9)
C5	0.0277 (11)	0.0336 (11)	0.0281 (10)	0.0009 (9)	0.0007 (8)	0.0005 (9)
C6	0.0325 (11)	0.0342 (11)	0.0329 (11)	0.0008 (9)	-0.0022 (9)	0.0012 (9)
C7	0.0367 (12)	0.0382 (12)	0.0330 (11)	-0.0049 (10)	-0.0023 (9)	0.0004 (10)
C8	0.0353 (11)	0.0362 (12)	0.0394 (12)	-0.0046 (10)	-0.0024 (10)	0.0009 (10)

Geometric parameters (Å, °)

P—F2 ⁱ	1.6596 (14)	C1—H1B	0.9300
P—F2	1.6596 (14)	C2—C3	1.395 (3)
P—F3	1.6902 (13)	C2—H2B	0.9300
P—F3 ⁱ	1.6902 (13)	C3—C4	1.368 (3)
P—F1	1.6912 (12)	C3—H3A	0.9300
P—F1 ⁱ	1.6913 (12)	C4—C5	1.389 (3)
O—C6	1.214 (3)	C4—H4A	0.9300
N1—C5	1.348 (3)	C6—C7	1.499 (3)
N1—C1	1.358 (3)	C7—C8	1.520 (3)
N1—H1A	0.8600	C7—H7A	0.9700
N2—C6	1.375 (3)	C7—H7B	0.9700
N2—C5	1.383 (3)	C8—C8 ⁱⁱ	1.519 (4)
N2—H2A	0.8600	C8—H8A	0.9700
C1—C2	1.356 (3)	C8—H8B	0.9700
F2 ⁱ —P—F2	180.0	C3—C2—H2B	120.5
F2 ⁱ —P—F3	90.09 (8)	C4—C3—C2	120.1 (2)
F2—P—F3	89.91 (8)	C4—C3—H3A	120.0
F2 ⁱ —P—F3 ⁱ	89.91 (8)	C2—C3—H3A	120.0
F2—P—F3 ⁱ	90.09 (8)	C3—C4—C5	119.7 (2)
F3—P—F3 ⁱ	180.00 (9)	C3—C4—H4A	120.2
F2 ⁱ —P—F1	90.39 (7)	C5—C4—H4A	120.2
F2—P—F1	89.61 (7)	N1—C5—N2	119.76 (18)
F3—P—F1	89.82 (6)	N1—C5—C4	119.09 (19)
F3 ⁱ —P—F1	90.18 (6)	N2—C5—C4	121.14 (19)
F2 ⁱ —P—F1 ⁱ	89.61 (7)	O—C6—N2	121.4 (2)
F2—P—F1 ⁱ	90.39 (7)	O—C6—C7	123.3 (2)
F3—P—F1 ⁱ	90.18 (6)	N2—C6—C7	115.21 (18)
F3 ⁱ —P—F1 ⁱ	89.82 (6)	C6—C7—C8	112.12 (18)
F1—P—F1 ⁱ	180.00 (8)	C6—C7—H7A	109.2
C5—N1—C1	121.58 (19)	C8—C7—H7A	109.2
C5—N1—H1A	119.2	C6—C7—H7B	109.2
C1—N1—H1A	119.2	C8—C7—H7B	109.2
C6—N2—C5	126.15 (18)	H7A—C7—H7B	107.9
C6—N2—H2A	116.9	C8 ⁱⁱ —C8—C7	112.6 (2)
C5—N2—H2A	116.9	C8 ⁱⁱ —C8—H8A	109.1
C2—C1—N1	120.6 (2)	C7—C8—H8A	109.1
C2—C1—H1B	119.7	C8 ⁱⁱ —C8—H8B	109.1
N1—C1—H1B	119.7	C7—C8—H8B	109.1
C1—C2—C3	118.9 (2)	H8A—C8—H8B	107.8
C1—C2—H2B	120.5		

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x-1, -y, -z$.

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···F1	0.86	1.98	2.737 (2)	145
N1—H1A···O	0.86	2.10	2.674 (2)	124
N2—H2A···F3 ⁱⁱⁱ	0.86	1.95	2.774 (2)	161
N2—H2A···F1 ⁱⁱⁱ	0.86	2.40	3.050 (2)	133

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