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9,10-Bis[3-(2-pyridylmethyl)imidazolium-1-ylmethyl]anthracene bis(hexafluorophosphate)

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Key indicators
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.06 Å
Disorder in solvent or counterion
R factor = 0.065
wR factor = 0.219
Data-to-parameter ratio = 12.0

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

In the title centrosymmetric compound, $C_{34}H_{30}N_6^{2+}\cdot 2PF_6^-$, the planes through the pyridine and anthracene ring systems form dihedral angles of 78.8 (1) and 73.5 (1)°, respectively, with the imidazole ring. The crystal packing is stabilized by π–π stacking interactions between the anthracene ring system and pyridine rings of adjacent molecules, and also by C—H···N hydrogen bonds.

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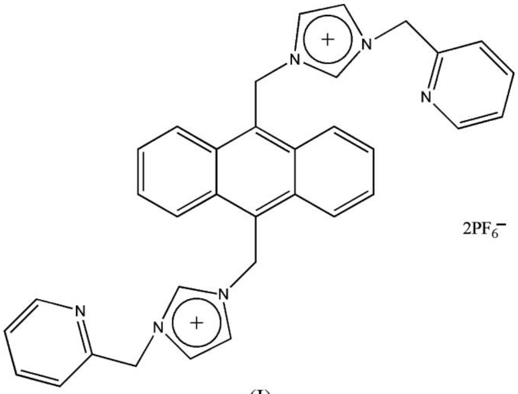
Comment

Since the discovery of stable *N*-heterocyclic carbenes (NHC), numerous flexible or rigid *N*-heterocyclic carbene precursors have been synthesized and studied because of their diverse coordination capabilities and the important catalytic properties of their metal complexes (Bourissou *et al.*, 2000; Herrmann & Kocher, 1997; Herrmann, 2002). In addition, the photophysical properties of these complexes have also been reported by our group (Liu *et al.*, 2003, 2004). As a continuation of our systematic studies of the various *N*-heterocyclic carbene ligands and the photophysical properties of their metal complexes, a new biscarbene precursor with a fluorescent anthracene group and a pyridyl ring, the title compound, (I), has been synthesized and its crystal structure is reported here.



(I)

The cation of (I) lies across a crystallographic inversion centre; the asymmetric unit therefore contains one-half of the cation and a PF_6^- anion (Fig. 1). The planes through the pyridine and anthracene ring systems form dihedral angles of 78.8 (1) and 73.5 (1)°, respectively, with the imidazole ring plane.

As shown in Fig. 2, π–π stacking interactions are observed between the anthracene ring system and the pyridine rings of two adjacent molecules, which are almost parallel [dihedral angle = 5.3 (2)°], with a centroid–centroid separation of

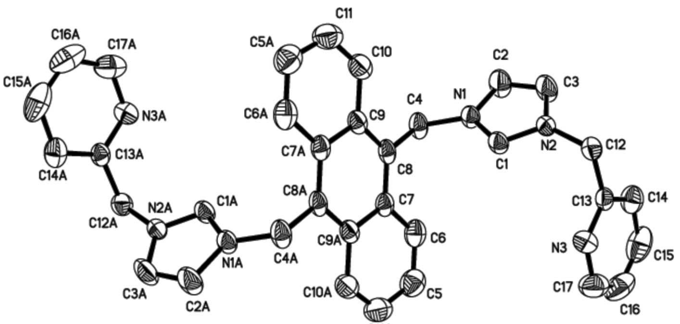


Figure 1
The cation of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Atoms labeled with the suffix A are generated by the symmetry operation $(1 - x, 1 - y, 1 - z)$. H atoms have been omitted for clarity.

3.701 (3) Å. In addition, the crystal packing is stabilized by C—H···N hydrogen bonds (Table 1).

Experimental

9,10-Bis(chloromethy1)anthracene (2.572 g, 0.01 mol) and 2-(imidazolylmethyl)pyridine (3.343 g, 0.021 mol) were mixed and stirred at 368 K in 1,4-dioxane (160 ml) for 48 h. The precipitate that formed was collected by filtration. The solid was dissolved in methanol and an aqueous solution of ammonium hexafluorophosphate (3.423 g, 0.021 mmol) was added. The precipitate that formed was collected by filtration and recrystallized from CH_3CN and diethyl ether (1:6, *v*/*v*) to give the title compound (yield 92%). Analysis found: C 50.19, H 3.72, N 10.33%; calculated for $C_{34}H_{30}F_{12}N_6P_2$: C 50.26, H 3.72, N, 10.34%. ^{1}H NMR (400 *M*, DMSO-d_6): δ 9.17 (*s*, 2H), 8.62 (*d*, 4H), 8.46 (*d*, 2H), 7.83 (*t*, 2H), 7.77 (*d*, 4H), 7.73 (*s*, 2H), 7.59 (*s*, 2H), 7.37 (*t*, 4H), 6.62 (*s*, 4H), 5.46 (s, 4H).

Crystal data

$C_{34}H_{30}N_6^{2+}\cdot 2PF_6^-$
$M_r = 812.58$
Triclinic, $P\overline{1}$
$a = 9.2564$ (15) Å
$b = 9.7534$ (15) Å
$c = 11.4425$ (18) Å
$\alpha = 65.621$ (2)°
$\beta = 75.971$ (3)°
$\gamma = 76.993$ (2)°
$V = 903.7$ (2) Å^3
$Z = 1$
$D_x = 1.493$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1905 reflections
$\theta = 2.3$–25.0°
$\mu = 0.22$ mm^{-1}
$T = 294$ (2) K
Block, yellow
0.32 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.934$, $T_{\max} = 0.958$
5051 measured reflections
3594 independent reflections
2248 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\max} = 26.3°$
$h = -10 \rightarrow 11$
$k = -12 \rightarrow 11$
$l = -14 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.219$
$S = 1.03$
3594 reflections
299 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1121P)^2 + 0.4256P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.39$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}

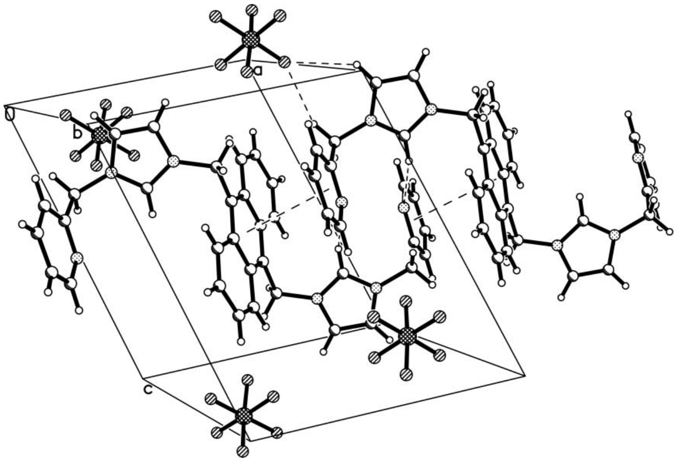


Figure 2
Molecular packing diagram, showing the intermolecular hydrogen-bonding and π–π stacking interactions.

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···N3$^{\rm i}$	0.93	2.42	3.233 (5)	146

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

The hexafluorophosphate group is disordered over two different orientations, with occupancies of 0.589 (12) and 0.411 (12). The P—F and F···F distances were restrained to 1.56 (1) and 2.21 (1) Å, respectively. The H atoms were placed in calculated positions, with C—H = 0.93 (aromatic) or 0.97 Å (methylene), and included in the final cycles of refinement using a riding-model approximation, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(carrier atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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