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Key indicators

Single-crystal X-ray study

$T = 294$ K

Mean $\sigma(\text{C}-\text{C}) = 0.001$ Å

Disorder in solvent or counterion

R factor = 0.054

wR factor = 0.179

Data-to-parameter ratio = 11.3

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

3(5,10)-Anthracena-7(1,3)-benzena-1,5(1,3)- diimidazolacyclooctaphane-1,3-dium bis(hexafluorophosphate) acetonitrile trisolvate

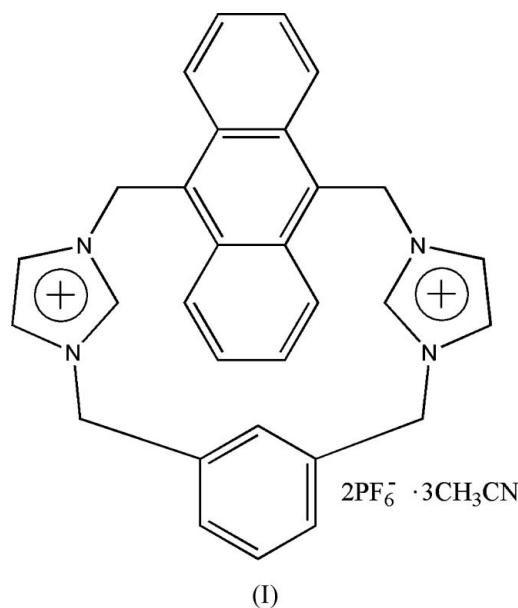
In the title compound, $\text{C}_{30}\text{H}_{26}\text{N}_4^{2+} \cdot 2\text{PF}_6^- \cdot 3\text{CH}_3\text{CN}$, the two imidazolium rings in the cation adopt a *syn* configuration with respect to the anthracene group. The crystal packing shows weak intermolecular $\text{C}-\text{H} \cdots \text{N}$ hydrogen bonds between the cations and acetonitrile molecules.

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Comment

In recent years, rigid cyclic *N*-heterocyclic carbene (NHC) precursors have attracted considerable attention, due to their diverse coordination capabilities to transition metals and main-group elements, along with an increasing use in organometallic chemistry and homogeneous catalysis (Herrmann & Kocher, 1997; Bourissou *et al.*, 2000; Barnard *et al.*, 2004; Lee *et al.*, 2004; Yoon *et al.*, 2004; Baker *et al.*, 2004, 2006). We present here the title compound, (I), which is a new macrocyclic NHC precursor.



The asymmetric unit of (I) contains one $\text{C}_{30}\text{H}_{26}\text{N}_4^{2+}$ cation, two PF_6^- anions and three acetonitrile solvent molecules. The cation consists of the two imidazolium rings (N1/N2/C1–C3, *A*, and N3/N4/C12–C14, *B*), a benzene ring (C5–C10, *C*) and an anthracene fragment (C16–C29, *D*), which make the following dihedral angles: $A/C = 87.1(1)^\circ$, $B/C = 83.9(1)^\circ$, $B/D = 73.7(1)^\circ$ and $A/D = 75.43(1)^\circ$. The two imidazolium rings adopt a *syn* configuration with respect to the anthracene unit.

In the crystal structure, weak intermolecular $\text{C}-\text{H} \cdots \text{N}$ hydrogen bonds (Table 1) link the cations and acetonitrile solvent molecules.

Experimental

The title compound was prepared by the reaction of bis(imidazol-1-yl)-*m*-xylene with 9,10-bis(chloromethyl)anthracene in dioxane and anion exchange with NH_4PF_6 in methanol. The residue was purified by silica-gel flash chromatography using dichloromethane–methanol (10:1) as eluent to give the title compound and another 2+2 cycloaddition compound. Yellow single crystals suitable for X-ray diffraction were obtained by recrystallization from diethyl ether–acetonitrile (1:1).

Crystal data

$\text{C}_{30}\text{H}_{26}\text{N}_4^{2+} \cdot 2\text{PF}_6^- \cdot 3\text{C}_2\text{H}_3\text{N}$	$Z = 4$
$M_r = 855.65$	$D_x = 1.415 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.643 (2) \text{ \AA}$	$\mu = 0.20 \text{ mm}^{-1}$
$b = 22.391 (6) \text{ \AA}$	$T = 294 (2) \text{ K}$
$c = 20.983 (5) \text{ \AA}$	Block, yellow
$\beta = 98.506 (5)^\circ$	$0.40 \times 0.22 \times 0.18 \text{ mm}$
$V = 4015.9 (18) \text{ \AA}^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	20280 measured reflections
φ and ω scans	7086 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3197 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.949$, $T_{\max} = 0.965$	$R_{\text{int}} = 0.058$
	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0813P)^2 + 0.3116P]$
$R[F^2 > 2\sigma(F^2)] = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.179$	$(\Delta/\sigma)_{\text{max}} = 0.005$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$
7086 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
627 parameters	
H-atom parameters constrained	

Table 1

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{C1}-\text{H1} \cdots \text{N5}$	0.93	2.52	3.407 (3)	160
$\text{C14}-\text{H14} \cdots \text{N5}$	0.93	2.47	3.387 (3)	167

Each hexafluorophosphate anion is disordered over two different orientations, with refined occupancies of 0.3571 (3) and 0.6429 (3) for the first anion, and 0.3422 (4) and 0.6578 (4) for the second anion. The P–F distances were restrained to 1.56 (4) \AA . H atoms were placed in calculated positions, with C–H = 0.93 or 0.96 \AA and N–H = 0.85 \AA , and included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$.

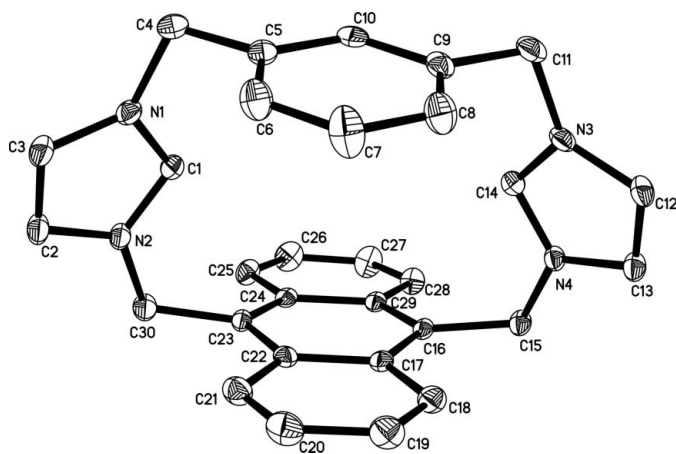


Figure 1

View of the cation of the title compound with displacement ellipsoids drawn at the 40% probability level. H atoms have been omitted for clarity.

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

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