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[(Triglycol ditolylene)imidazolium]-2,6-dimethylpyridine bis(hexafluorophosphate)

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Key indicators

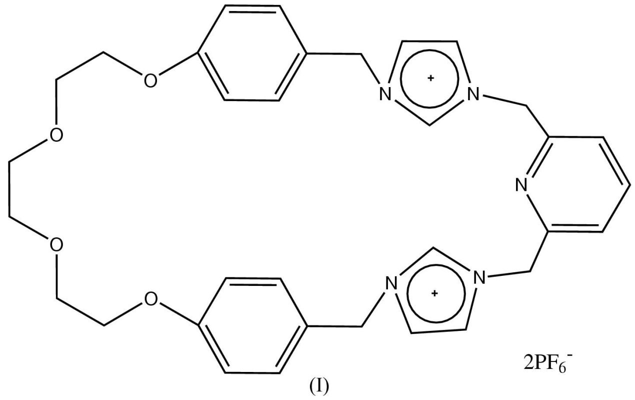
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.056
wR factor = 0.188
Data-to-parameter ratio = 11.7

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

In the title compound, 6,16,38-triaza-3,13-diazonia-22,25,28,-31-tetraoxaoctacyclo[30.2.2.2^{18,21}.1^{3,6}.1^{8,12}.1^{13,16}]hentetraconta-1(34),3,8,10,12(38),13,18,20,32,35,40-undecaene bis(hexafluorophosphate), $C_{33}H_{37}N_5O_4^{2+}\cdot 2PF_6^-$, the two imidazolium rings adopt a *cis* configuration with respect to the 2,6-dimethylpyridine group. The crystal packing is stabilized by C—H$\cdots$F hydrogen bonds.

Comment

In recent years, numerous cyclic *N*-heterocyclic carbene (NHC) precursors have been synthesized and structurally investigated. They have attracted considerable attention owing to their ability to coordinate very strongly to transition metals and main-group elements and an increasing use in organometallic chemistry, homogeneous catalysis and anion recognition (Herrmann & Kocher, 1997; Bourissou *et al.*, 2000; Barnard *et al.*, 2004; Lee *et al.*, 2004; Yoon *et al.*, 2004; Baker *et al.*, 2004). We report here the synthesis and crystal structure of a new macrocyclic NHC precursor, the title compound, (I).



(I)

The asymmetric unit of (I) is shown in Fig. 1. It consists of one $C_{33}H_{37}N_5O_4^{2+}$ cation and two PF_6^- anions. The heterocyclic cation contains a pyridine ring [*A* (N1,C1–C5)], two imidazolium rings [*B* (N2,N3,C7–C9) and *C* (N4,N5,C30–C32)] and two benzene rings [*D* (C11–C16) and *E* (C23–C28)]. The two imidazolium rings adopt a *cis* configuration with respect to the 2,6-dimethylpyridine group. The dihedral angles *A*/*B*, *A*/*C*, *B*/*C*, *B*/*D*, *C*/*E*, *D*/*E* are 52.8 (1), 82.3 (1), 32.2 (2), 85.4 (1), 70.6 (1) and 19.1 (2)°, respectively. The O—C—C—O and C—O—C—C torsion angles in the triglycol linkage are given in Table 1.

In the crystal structure, the $C_{33}H_{37}N_5O_4^{2+}$ cations and PF_6^- anions are linked by C—H$\cdots$F hydrogen bonds; the C$\cdots$F distances range from 2.949 (7) to 3.399 (5) Å (Table 2).

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Experimental

Compound (I) was prepared according to the reported precedure of Garrison *et al.* (2001). Colourless single crystals of (I) were obtained by recrystallization from diethyl ether and acetonitrile (1: 1, *v*/*v*).

Crystal data

$C_{33}H_{37}N_5O_4{}^{2+}\cdot 2PF_6{}^-$
$M_r = 857.62$
Triclinic, $P\bar{1}$
$a = 9.8299\ (11)$ Å
$b = 10.1415\ (11)$ Å
$c = 19.486\ (2)$ Å
$\alpha = 83.183\ (2)^\circ$
$\beta = 89.886\ (2)^\circ$
$\gamma = 76.646\ (2)^\circ$
$V = 1876.0\ (4)$ Å^3
$Z = 2$
$D_x = 1.518$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3160 reflections
$\theta = 2.2$–21.9°
$\mu = 0.22$ mm^{-1}
$T = 293\ (2)$ K
Block, colourless
$0.32 \times 0.24 \times 0.22$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.922$, $T_{\max} = 0.953$
10269 measured reflections
6554 independent reflections
4429 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.017$
$\theta_{\max} = 25.0^\circ$
$h = -11 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -23 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.188$
$S = 1.08$
6554 reflections
560 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1116P)^2 + 0.2928P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.44$ e Å^{-3}
$\Delta\rho_{\min} = -0.24$ e Å^{-3}

Table 1
Selected torsion angles (°).

C14—O1—C17—C18	−113.1 (4)	O2—C19—C20—O3	−66.8 (4)
C19—O2—C18—C17	−161.6 (4)	C20—O3—C21—C22	−165.7 (3)
O1—C17—C18—O2	73.2 (4)	C23—O4—C22—C21	−180.0 (3)
C18—O2—C19—C20	−179.6 (3)	O3—C21—C22—O4	−171.8 (3)
C21—O3—C20—C19	108.8 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6*B*···F3$^{\text{i}}$	0.97	2.51	3.399 (5)	153
C7—H7···F7$^{\text{ii}}$	0.93	2.19	2.949 (7)	138
C9—H9···F8$^{\text{i}}$	0.93	2.51	3.299 (9)	142
C25—H25···F10	0.93	2.51	3.347 (9)	150
C29—H29*B*···F10	0.97	2.46	3.301 (7)	145
C30—H30···F5	0.93	2.44	3.252 (4)	146

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

One of the hexafluorophosphate groups is disordered over two different orientations, with occupancies of 0.568 (15) and 0.432 (15). The P—F distances were restrained to 1.58 (1) Å. H atoms were placed in calculated positions, with C—H distances of 0.93 or 0.96 Å, and included in the final cycles of refinement using a riding-model approximation, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$(carrier atom).

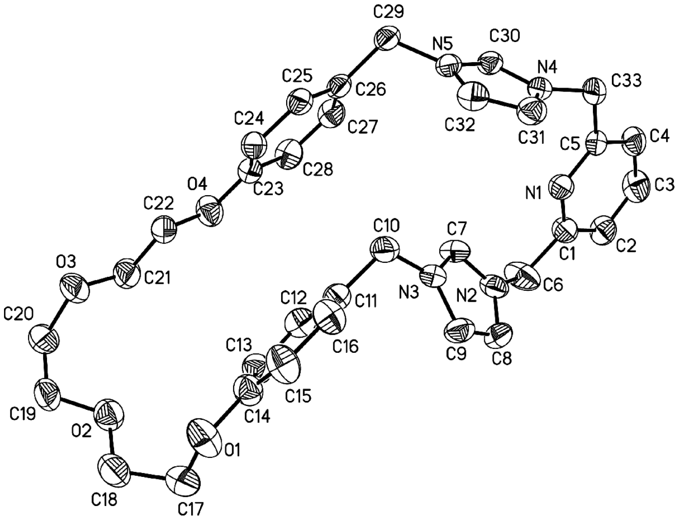

Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 40% probability level. H atoms and counter-ions have been omitted for clarity.

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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