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1-Benzylimidazolium hexafluorophosphate–1-benzylimidazole (1/1)

Yan Zang,[a] Xiu-Mei Wu,[a] Zhan-Ying Zheng,[b] Hai-Bin Song[b] and Qing-Xiang Liu[a]*

[a]Key Laboratory of Molecular Structure and Materials Performance, College of Chemistry and Life Science, Tianjin Normal University, Tianjin 300387, People's Republic of China, and [b]State Key Laboratory of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China
Correspondence e-mail: tjnulqx@163.com

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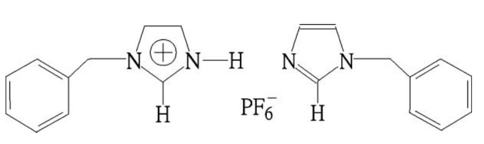
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; disorder in solvent or counterion; R factor = 0.037; wR factor = 0.117; data-to-parameter ratio = 11.2.

In the title compound, $C_{10}H_{11}N_2^+\cdot PF_6^-\cdot C_{10}H_{10}N_2$, the H atom involved in protonation is disordered equally between the cation and the neutral molecule. The dihedral angle between the phenyl and imidazole rings is 82.6 (2)°. In the crystal structure, there are head-to-tail π–π stacking interactions between imidazole rings; the interplanar separation is 3.295 (1) Å and the centroid–centroid separation is 3.448 (3) Å. In the centrosymmetric anion, two F atoms are disordered over two positions; the refined site-occupancy factors are 0.855 (11) and 0.145 (11).

Related literature

For background to the chemistry of imidazolium compounds, see: Hunter & Sanders (1990); Sundberg & Martin (1974); Kurdziel & Glowiak (2000). For related literature, see: Liu *et al.* (2003).



Experimental

Crystal data

$C_{10}H_{11}N_2^+\cdot PF_6^-\cdot C_{10}H_{10}N_2$
M_r = 462.38
Monoclinic, $P2_1/n$
a = 6.6459 (4) Å
b = 6.9825 (4) Å
c = 22.3558 (12) Å
β = 97.109 (1)°
V = 1029.45 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 296 (2) K
0.25 × 0.24 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.951, T_{max} = 0.957
5057 measured reflections
1820 independent reflections
1585 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.116
S = 1.05
1820 reflections
162 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Data collection: *APEX2* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2226).

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supporting information

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1-Benzylimidazolium hexafluorophosphate–1-benzylimidazole (1/1)

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S1. Comment

Some weak interactions in crystal engineering, such as *π*-*π* stacking interactions (Hunter & Sanders, 1990), often affect the structure of complexes, and they can link discrete sub-units or low-dimensional entities into high-dimensional supramolecular networks. The imidazole ring is a structural component of many compounds occurring in living organisms (Sundberg & Martin, 1974). In the mechanisms of the majority of enzymatic reactions, an important role is played by the formation of coordination compounds with ions (Kurdziel & Glowiak, 2000.). We are interested in imidazole compounds with *π*-*π* stacking interactions and here report the synthesis and crystal structure of 1-benzyl-imidazolium hexafluoridophosphate.

In the title compound, $C_{10}H_{10}N_2.C_{10}H_{11}N_2^+.F_6P^-$, atom H2 is disordered equally between the cation and neutral molecule. The dihedral angle between the phenyl and imidazole rings is 82.6 (2)° (Fig. 1). The N1A—C1A, N1A—C3A, N2A—C1A and N2A—C2A bond distances are 1.312 (2), 1.368 (3), 1.308 (3) and 1.355 (3) Å, respectively, and the N1A—C1A—N2A bond angle is 108.78 (18)°; these values are similar to those observed in 1-(9-anthracenylmethyl)-3-ethyl-imidazolium iodide (Liu *et al.*, 2003).

In the crystal structure of the title compound (Fig. 2), there are head-to-tail *π*-*π* stacking interactions between imidazole rings; the interplanar separation is 3.295 (1) Å and the centroid-to-centroid separation is 3.448 (3) Å.

S2. Experimental

A 1,4-dioxane solution (20 ml) of imidazole (1.420 g, 20.8 mmol) was added to a suspension of oil-free sodium hydride (0.500 g, 20.8 mmol) in 1,4-dioxane (20 ml) and stirred for 1 h at 90°C. A 1,4-dioxane (20 ml) solution of benzyl bromide (3.240 g, 19 mmol) was then added dropwise to the above solution. The mixture was stirred for 22 h at 90°C, and a brown solution was obtained. The solvent was removed with a rotary evaporator and H_2O (50 ml) was added to the residue. The solution was then extracted with CH_2Cl_2 (50 ml), and the solution was dried with anhydrous $MgSO_4$. After removing CH_2Cl_2, 1-benzylimidazole, as a pale yellow liquid was obtained yield: 2.7 g (89.7%).

1-Benzylimidazole (2.000 g, 12.6 mmol) was reacted with hydrochloric acid (8 ml, 6 mol l^{-1}) to afford 1-benzyl-imidazolium chloride as a pale yellow solid (2.618 g, 94%). NH_4PF_6 (5.050 g, 31 mmol) was added to a methanol solution (50 ml) of 1-benzylimidazolium chloride (2.000 g, 10.3 mmol). A pale yellow precipitate formed immediately, which was collected by filtration, washed with small portions of methanol, and dried in a vacuum to give 1-benzyl-imidazolium hexafluoridophosphate as a pale yellow powder (3.3 g, 95%). m.p. 202–204°C. Crystals suitable for X-ray diffraction were obtained by evaporating slowly a CH_3OH solution at room temperature.

S3. Refinement

All H atoms were initially located in a difference Fourier map. They were then placed in geometrically idealized positions and constrained to ride on their parent atoms, with C*sp*^{3}—H = 0.97 Å, C*sp*^{2}—H = 0.93 Å and N—H = 0.86 Å and U_{iso}(H)

= $1.2U_{eq}$(C,*N*). In the centrosymmetric anion, atoms F2 and F3 are disordered over two positions; the refined site occupancy factors are 0.855 (11) and 0.145 (11). Atom H2 is disordered equally between the cation and the neutral molecule.

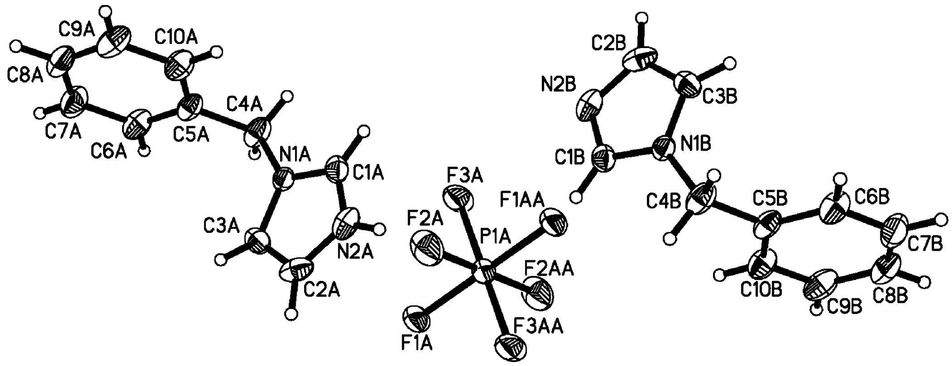

Figure 1

Perspective view of the title compound, with displacement ellipsoids drawn at the 30% probability level. The organic moiety with A atom labels is the cation; that with B atom labels is the centrosymmetrically related neutral molecule. In the centrosymmetric anion, only one disorder component is shown.

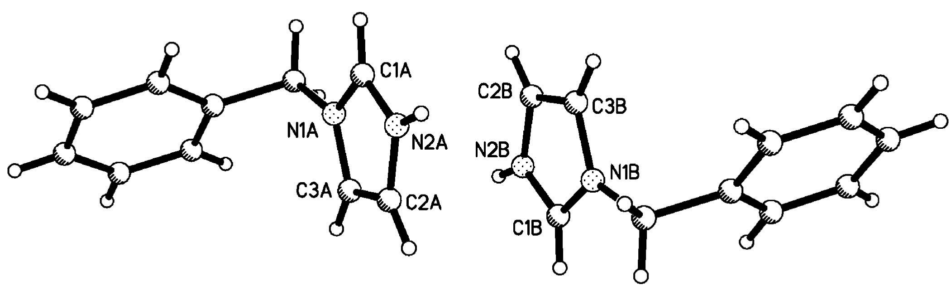

Figure 2

The *π*-*π* stacking interaction between imidazole rings in the title compound.

1-Benzylimidazolium hexafluorophosphate–1-benzylimidazole (1/1)

Crystal data

$C_{10}H_{11}N_2^+\cdot PF_6^-\cdot C_{10}H_{10}N_2$
M_r = 462.38
Monoclinic, $P2_1/n$
a = 6.6459 (4) Å
b = 6.9825 (4) Å
c = 22.3558 (12) Å
β = 97.109 (1)°
V = 1029.45 (10) Å^3
Z = 2
$F(000)$ = 476
D_x = 1.492 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3167 reflections
θ = 3.1–27.8°
μ = 0.20 mm^{-1}
T = 296 K
Block, colorless
0.25 × 0.24 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.951, T_{max} = 0.957
5057 measured reflections
1820 independent reflections
1585 reflections with $I > 2\sigma(I)$

$R_{int} = 0.014$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.8°$
$h = -6\rightarrow7$
$k = -8\rightarrow8$
$l = -26\rightarrow21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.116$
$S = 1.05$
1820 reflections
162 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0633P)^2 + 0.4527P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.013 (3)

Special details

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
P1	0.5000	0.5000	0.0000	0.0381 (3)	
F1	0.25151 (17)	0.51481 (17)	0.00618 (6)	0.0514 (4)	
F2	0.5364 (5)	0.4064 (5)	0.06870 (9)	0.0695 (10)	0.855 (11)
F3	0.5370 (4)	0.7209 (3)	0.02900 (16)	0.0629 (9)	0.855 (11)
F2′	0.553 (3)	0.310 (5)	0.042 (2)	0.152 (18)	0.145 (11)
F3′	0.542 (3)	0.648 (6)	0.0573 (15)	0.160 (17)	0.145 (11)
N1	0.0699 (2)	0.1368 (2)	0.09486 (6)	0.0371 (4)	
N2	0.1897 (3)	−0.1141 (3)	0.05810 (8)	0.0527 (5)	
H2	0.2730	−0.1922	0.0444	0.063*	0.50
C1	0.2320 (3)	0.0610 (3)	0.07629 (9)	0.0445 (5)	
H1	0.3564	0.1218	0.0761	0.053*	
C2	−0.0066 (4)	−0.1526 (3)	0.06429 (9)	0.0550 (6)	
H2A	−0.0753	−0.2664	0.0543	0.066*	
C3	−0.0827 (3)	0.0043 (3)	0.08755 (10)	0.0479 (5)	
H3	−0.2141	0.0202	0.0969	0.058*	
C4	0.0549 (4)	0.3309 (3)	0.11951 (9)	0.0549 (6)	
H4A	−0.0577	0.3979	0.0969	0.066*	
H4B	0.1780	0.4014	0.1152	0.066*	
C5	0.0237 (3)	0.3236 (3)	0.18532 (9)	0.0456 (5)	
C6	−0.1565 (4)	0.3843 (3)	0.20279 (10)	0.0594 (6)	

H6	−0.2584	0.4301	0.1741	0.071*
C7	−0.1878 (4)	0.3780 (4)	0.26278 (11)	0.0674 (7)
H7	−0.3096	0.4214	0.2743	0.081*
C8	−0.0405 (4)	0.3081 (4)	0.30527 (10)	0.0619 (7)
H8	−0.0632	0.3011	0.3454	0.074*
C9	0.1401 (4)	0.2487 (3)	0.28843 (10)	0.0637 (7)
H9	0.2413	0.2030	0.3174	0.076*
C10	0.1734 (4)	0.2560 (3)	0.22857 (10)	0.0566 (6)
H10	0.2967	0.2154	0.2174	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0353 (4)	0.0392 (4)	0.0410 (4)	−0.0034 (3)	0.0103 (3)	−0.0044 (3)
F1	0.0343 (7)	0.0547 (7)	0.0675 (8)	−0.0048 (5)	0.0155 (5)	−0.0146 (6)
F2	0.0586 (14)	0.100 (2)	0.0487 (13)	−0.0140 (13)	0.0031 (8)	0.0211 (12)
F3	0.0463 (10)	0.0459 (13)	0.101 (2)	−0.0107 (7)	0.0257 (14)	−0.0320 (11)
F2′	0.055 (8)	0.13 (2)	0.28 (4)	0.032 (11)	0.052 (16)	0.16 (3)
F3′	0.059 (8)	0.31 (4)	0.117 (19)	−0.061 (17)	0.031 (11)	−0.17 (2)
N1	0.0446 (9)	0.0352 (8)	0.0325 (8)	−0.0022 (7)	0.0089 (6)	−0.0015 (6)
N2	0.0710 (13)	0.0487 (11)	0.0388 (9)	0.0183 (9)	0.0087 (8)	−0.0018 (8)
C1	0.0425 (11)	0.0539 (12)	0.0380 (10)	0.0016 (9)	0.0091 (8)	0.0034 (9)
C2	0.0813 (17)	0.0370 (11)	0.0422 (11)	−0.0110 (11)	−0.0107 (10)	0.0007 (9)
C3	0.0407 (11)	0.0527 (13)	0.0506 (12)	−0.0064 (9)	0.0067 (9)	0.0048 (9)
C4	0.0907 (17)	0.0326 (10)	0.0429 (11)	0.0006 (10)	0.0152 (11)	−0.0022 (9)
C5	0.0699 (14)	0.0296 (9)	0.0378 (10)	0.0031 (9)	0.0084 (9)	−0.0033 (8)
C6	0.0710 (15)	0.0586 (14)	0.0474 (12)	0.0162 (12)	0.0026 (10)	−0.0073 (10)
C7	0.0746 (17)	0.0755 (17)	0.0541 (14)	0.0086 (13)	0.0162 (12)	−0.0162 (12)
C8	0.0926 (19)	0.0541 (13)	0.0402 (11)	−0.0015 (12)	0.0132 (11)	−0.0085 (10)
C9	0.0926 (19)	0.0507 (13)	0.0441 (12)	0.0114 (12)	−0.0068 (12)	−0.0023 (10)
C10	0.0686 (15)	0.0487 (12)	0.0522 (12)	0.0126 (11)	0.0068 (11)	−0.0071 (10)

Geometric parameters (Å, º)

P1—F2′^{i}	1.636 (8)	C2—C3	1.338 (3)
P1—F2′	1.636 (9)	C2—H2A	0.9300
P1—F3′	1.641 (9)	C3—H3	0.9300
P1—F3′^{i}	1.641 (9)	C4—C5	1.512 (3)
P1—F2^{i}	1.659 (2)	C4—H4A	0.9700
P1—F2	1.659 (2)	C4—H4B	0.9700
P1—F1	1.6773 (11)	C5—C6	1.372 (3)
P1—F1^{i}	1.6773 (11)	C5—C10	1.382 (3)
P1—F3	1.679 (2)	C6—C7	1.383 (3)
P1—F3^{i}	1.679 (2)	C6—H6	0.9300
N1—C1	1.312 (2)	C7—C8	1.367 (4)
N1—C3	1.368 (3)	C7—H7	0.9300
N1—C4	1.471 (3)	C8—C9	1.366 (4)
N2—C1	1.308 (3)	C8—H8	0.9300

N2—C2	1.355 (3)	C9—C10	1.384 (3)
N2—H2	0.8600	C9—H9	0.9300
C1—H1	0.9300	C10—H10	0.9300

F2′^{i}—P1—F2′	180.0 (16)	F1—P1—F3^{i}	89.41 (10)
F2′^{i}—P1—F3′	86.7 (15)	F1^{i}—P1—F3^{i}	90.59 (10)
F2′—P1—F3′	93.3 (15)	F3—P1—F3^{i}	180.0 (2)
F2′^{i}—P1—F3′^{i}	93.3 (15)	C1—N1—C3	108.37 (18)
F2′—P1—F3′^{i}	86.7 (15)	C1—N1—C4	125.90 (19)
F3′—P1—F3′^{i}	180.0 (11)	C3—N1—C4	125.74 (18)
F2′^{i}—P1—F2^{i}	32 (2)	C1—N2—C2	109.05 (17)
F2′—P1—F2^{i}	148 (2)	C1—N2—H2	125.5
F3′—P1—F2^{i}	117.8 (19)	C2—N2—H2	125.5
F3′^{i}—P1—F2^{i}	62.2 (19)	N2—C1—N1	108.78 (18)
F2′^{i}—P1—F2	148 (2)	N2—C1—H1	125.6
F2′—P1—F2	32 (2)	N1—C1—H1	125.6
F3′—P1—F2	62.2 (19)	C3—C2—N2	106.97 (19)
F3′^{i}—P1—F2	117.8 (19)	C3—C2—H2A	126.5
F2^{i}—P1—F2	180.0 (2)	N2—C2—H2A	126.5
F2′^{i}—P1—F1	81.6 (6)	C2—C3—N1	106.84 (19)
F2′—P1—F1	98.4 (6)	C2—C3—H3	126.6
F3′—P1—F1	88.3 (7)	N1—C3—H3	126.6
F3′^{i}—P1—F1	91.7 (7)	N1—C4—C5	110.92 (16)
F2^{i}—P1—F1	91.13 (12)	N1—C4—H4A	109.5
F2—P1—F1	88.87 (12)	C5—C4—H4A	109.5
F2′^{i}—P1—F1^{i}	98.4 (6)	N1—C4—H4B	109.5
F2′—P1—F1^{i}	81.6 (6)	C5—C4—H4B	109.5
F3′—P1—F1^{i}	91.7 (7)	H4A—C4—H4B	108.0
F3′^{i}—P1—F1^{i}	88.3 (7)	C6—C5—C10	119.0 (2)
F2^{i}—P1—F1^{i}	88.87 (12)	C6—C5—C4	119.8 (2)
F2—P1—F1^{i}	91.13 (12)	C10—C5—C4	121.3 (2)
F1—P1—F1^{i}	180.00 (9)	C5—C6—C7	120.5 (2)
F2′^{i}—P1—F3	59 (2)	C5—C6—H6	119.8
F2′—P1—F3	121 (2)	C7—C6—H6	119.8
F3′—P1—F3	28.2 (18)	C8—C7—C6	120.3 (2)
F3′^{i}—P1—F3	151.8 (18)	C8—C7—H7	119.8
F2^{i}—P1—F3	89.61 (12)	C6—C7—H7	119.8
F2—P1—F3	90.39 (12)	C9—C8—C7	119.7 (2)
F1—P1—F3	90.59 (10)	C9—C8—H8	120.2
F1^{i}—P1—F3	89.41 (10)	C7—C8—H8	120.2
F2′^{i}—P1—F3^{i}	121 (2)	C8—C9—C10	120.4 (2)
F2′—P1—F3^{i}	59 (2)	C8—C9—H9	119.8
F3′—P1—F3^{i}	151.8 (18)	C10—C9—H9	119.8
F3′^{i}—P1—F3^{i}	28.2 (18)	C5—C10—C9	120.1 (2)
F2^{i}—P1—F3^{i}	90.39 (12)	C5—C10—H10	119.9
F2—P1—F3^{i}	89.61 (12)	C9—C10—H10	119.9

C2—N2—C1—N1	0.8 (2)	N1—C4—C5—C10	66.8 (3)

C3—N1—C1—N2	−0.6 (2)	C10—C5—C6—C7	0.0 (4)
C4—N1—C1—N2	179.22 (17)	C4—C5—C6—C7	179.8 (2)
C1—N2—C2—C3	−0.7 (2)	C5—C6—C7—C8	−1.0 (4)
N2—C2—C3—N1	0.3 (2)	C6—C7—C8—C9	1.6 (4)
C1—N1—C3—C2	0.2 (2)	C7—C8—C9—C10	−1.0 (4)
C4—N1—C3—C2	−179.65 (18)	C6—C5—C10—C9	0.6 (3)
C1—N1—C4—C5	−113.5 (2)	C4—C5—C10—C9	−179.3 (2)
C3—N1—C4—C5	66.3 (3)	C8—C9—C10—C5	0.0 (4)
N1—C4—C5—C6	−113.0 (2)		

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