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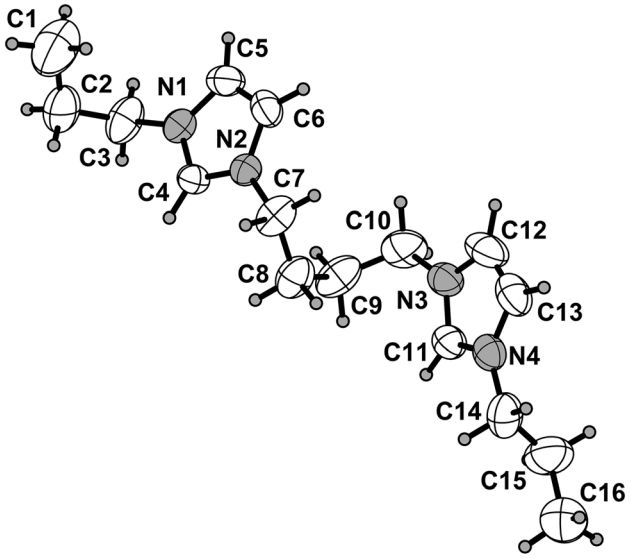
Crystal structure of 1,1′-(butane-1,4-diyl) bis(3-propyl-1*H*-imidazol-3-ium) bis(hexafluoridophosphate), $C_{32}H_{56}F_{24}N_8P_4$



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Abstract

$C_{32}H_{56}F_{24}N_8P_4$, triclinic, $P\bar{1}$ (no. 2), a = 8.6769(13) Å, b = 9.8858(14) Å, c = 14.510(2) Å, α = 102.564(2)°, β = 94.461(3)°, γ = 92.152(3)°, V = 81.710(2) Å^3, Z = 1, $R_{gt}(F)$ = 0.0692, $wR_{ref}(F^2)$ = 0.2245, T = 296(2) K.

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The molecular structure is shown in the figure (The anions were omitted for clarity). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.14 × 0.06 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.28 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9680, 4556, 0.053
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1874
$N(param)_{refined}$:	419
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Source of material

1-Propylimidazole (4.41 g, 0.040 mol) and 1,4-dibromobutane (4.43 g, 0.020 mol) were added into a flask containing toluene (20 mL). The mixture was stirred vigorously at 110 °C for 8 h. After the reaction has been completed (monitored by TLC), the acetonitrile top phase was decanted and the product was washed with ethyl acetate and anhydrous diethyl ether three times respectively. The intermediate product was dried in vacuo at 40 °C for 1 h to give a white powder in 99.2% yield. During the anion exchange reaction, the intermediate (2.18 g, 0.005 mol) and KPF_6 (1.84 g, 0.01 mol) were dissolved in water (25 mL), and the reaction proceeded at 85 °C for 6 h before the reaction mixture was allowed to cool slowly to room temperature. After deionized water washing and air drying, the final product was obtained in 81.27% yield.

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.90–0.97 Å with U_{iso}(H) = 1.5 U_{eq}(C) for methyl H atoms and 1.2 U_{eq}(C) for all other H atoms.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.0033 (11)	0.5243 (9)	−0.3639 (5)	0.175 (4)
H1A	−0.0434	0.5299	−0.3026	0.263*
H1B	−0.0871	0.5195	−0.4059	0.263*
H1C	0.0571	0.6039	−0.3848	0.263*
C2	0.0946 (10)	0.4007 (8)	−0.3612 (4)	0.128 (3)
H2A	0.1237	0.3909	−0.4246	0.153*
H2B	0.0328	0.3219	−0.3370	0.153*
C3	0.2371 (8)	0.3965 (7)	−0.3052 (4)	0.107 (2)
H3A	0.2950	0.3140	−0.3120	0.128*
H3B	0.3007	0.4742	−0.3289	0.128*
C4	0.1599 (6)	0.2916 (5)	−0.1464 (4)	0.0668 (13)
H4	0.1440	0.2048	−0.1619	0.080*
C5	0.2136 (6)	0.5052 (5)	−0.1562 (5)	0.0812 (16)
H5	0.2408	0.5944	−0.1796	0.097*
C6	0.1777 (6)	0.4582 (6)	−0.0680 (4)	0.0800 (16)
H6	0.1770	0.5088	−0.0187	0.096*
C7	0.0922 (6)	0.2368 (6)	0.0217 (4)	0.0885 (17)
H7A	0.0920	0.2894	0.0732	0.106*
H7B	−0.0128	0.2086	0.0133	0.106*
C8	0.1875 (7)	0.1160 (6)	0.0459 (4)	0.0985 (19)
H8A	0.1731	0.0576	−0.0016	0.118*
H8B	0.1475	0.0670	0.1039	0.118*
C9	0.3559 (8)	0.1316 (7)	0.0572 (4)	0.107 (2)
H9A	0.4009	0.0406	0.0686	0.129*
H9B	0.3969	0.1754	−0.0020	0.129*
C10	0.4120 (8)	0.2080 (6)	0.1298 (5)	0.108 (2)
H10A	0.3790	0.3026	0.1150	0.130*
H10B	0.5238	0.2064	0.1287	0.130*
C11	0.3485 (6)	0.0255 (6)	0.2617 (4)	0.0760 (15)
H11	0.3749	−0.0476	0.2307	0.091*
C12	0.3122 (8)	0.2264 (7)	0.2933 (6)	0.109 (2)
H12	0.3082	0.3212	0.2883	0.131*
C13	0.2739 (7)	0.1402 (8)	0.3676 (5)	0.102 (2)
H13	0.2377	0.1628	0.4241	0.122*
C14	0.2674 (8)	−0.1125 (7)	0.4078 (4)	0.104 (2)
H14A	0.2110	−0.1737	0.3740	0.124*
H14B	0.2034	−0.0936	0.4597	0.124*
C15	0.4143 (9)	−0.1821 (7)	0.4447 (5)	0.133 (3)
H15A	0.4800	−0.1952	0.3921	0.160*
H15B	0.4675	−0.1214	0.4803	0.160*
C16	0.3954 (11)	−0.3119 (8)	0.5023 (5)	0.162 (3)
H16A	0.3212	−0.3029	0.5506	0.243*
H16B	0.4924	−0.3416	0.5297	0.243*
H16C	0.3600	−0.3779	0.4650	0.243*
N1	0.2033 (5)	0.3992 (5)	−0.2056 (3)	0.0695 (11)
N2	0.1424 (4)	0.3249 (4)	−0.0625 (3)	0.0620 (10)
N3	0.3582 (5)	0.1546 (5)	0.2257 (3)	0.0798 (13)
N4	0.2967 (5)	0.0140 (5)	0.3467 (3)	0.0759 (12)
P1	0.32433 (16)	0.65806 (14)	0.15320 (10)	0.0685 (5)
P2	0.82607 (18)	0.06239 (16)	0.28031 (11)	0.0802 (5)
F1[a]	0.4619 (19)	0.7628 (16)	0.1611 (8)	0.078 (6)
F2[a]	0.202 (4)	0.541 (3)	0.153 (2)	0.091 (7)
F3[a]	0.219 (2)	0.738 (3)	0.2104 (18)	0.143 (10)
F4[a]	0.428 (3)	0.585 (3)	0.091 (2)	0.117 (7)
F5[a]	0.381 (4)	0.566 (3)	0.244 (2)	0.148 (11)
F6[a]	0.273 (2)	0.746 (2)	0.0649 (15)	0.119 (6)
F7[b]	0.7616 (17)	0.0407 (18)	0.1850 (10)	0.162 (6)
F8[b]	0.9025 (14)	0.0791 (16)	0.3769 (5)	0.130 (5)
F9[b]	0.7216 (14)	−0.0471 (13)	0.3250 (16)	0.199 (7)
F10[b]	0.9441 (12)	0.1769 (7)	0.2342 (8)	0.118 (4)
F11[b]	0.709 (2)	0.1842 (14)	0.2836 (11)	0.123 (4)
F12[b]	0.953 (3)	−0.051 (2)	0.2736 (14)	0.117 (6)
F9A[c]	0.813 (3)	−0.039 (2)	0.3742 (9)	0.180 (7)
F7A[c]	0.7079 (12)	−0.0393 (13)	0.2466 (12)	0.102 (4)
F8A[c]	0.9232 (17)	0.1530 (17)	0.3268 (19)	0.171 (8)
F10A[c]	0.818 (2)	0.1475 (18)	0.1841 (11)	0.165 (7)
F11A[c]	0.678 (2)	0.1379 (19)	0.3133 (12)	0.131 (7)
F12A[c]	0.961 (4)	−0.020 (3)	0.2426 (18)	0.115 (7)
F1A[d]	0.457 (2)	0.757 (2)	0.1666 (15)	0.142 (10)
F2A[d]	0.177 (4)	0.567 (3)	0.135 (2)	0.079 (6)
F3A[d]	0.204 (2)	0.765 (2)	0.174 (3)	0.178 (12)
F4A[d]	0.443 (3)	0.546 (3)	0.127 (3)	0.162 (13)
F5A[d]	0.336 (5)	0.603 (4)	0.255 (2)	0.182 (16)
F6A[d]	0.316 (4)	0.709 (4)	0.0441 (12)	0.172 (11)

[a]Occupancy: 0.51 (3), [b]Occupancy: 0.569 (13), [c]Occupancy: 0.431 (13), [d]Occupancy: 0.49 (3).

Comment

Ionic liquids are composed of organic cations and inorganic or organic anions [5]. Its melting point is 100 °C or lower [6], and it has the advantages of low vapor pressure, non-flammability, good thermal and chemical stability, and great structural adjustability [7–11]. Hexafluorophosphate as an anion makes ionic liquids to show good hydrophobicity and solubility is more sensitive to temperature. This ionic liquid can reflect the function of "low temperature multiphase, high temperature homogeneous" in the solvent, which has a wide range of applications in synthesis, extraction, etc. [12, 13]. Based on our earlier research on aliphatic chain hydrocarbons and methyl imidazole [14–19], we here report the title compound.

There are two anions and one cation in the asymmetric unit (only the cation is shown in the figure). In the molecule of the title compound bond lengths and angles are very similar to those reported in the literature [17–19]. The atoms of imidazole ring are coplanar, and the dihedral angles between the two imidazole rings are 17.6(2)°. The torsion angles of C1–C2–C3–N1, C2–C3–N1–C4, C4–N2–C7–C8, N2–C7–C8–C9, C11–N4–C14–C15, and N4–C14–C15–C16 are 62.6(8)°, 79.5(7)°, 57.5(7)°, 54.94(7)°, 74.7(8)°, and −177.0(6)°, respectively.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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