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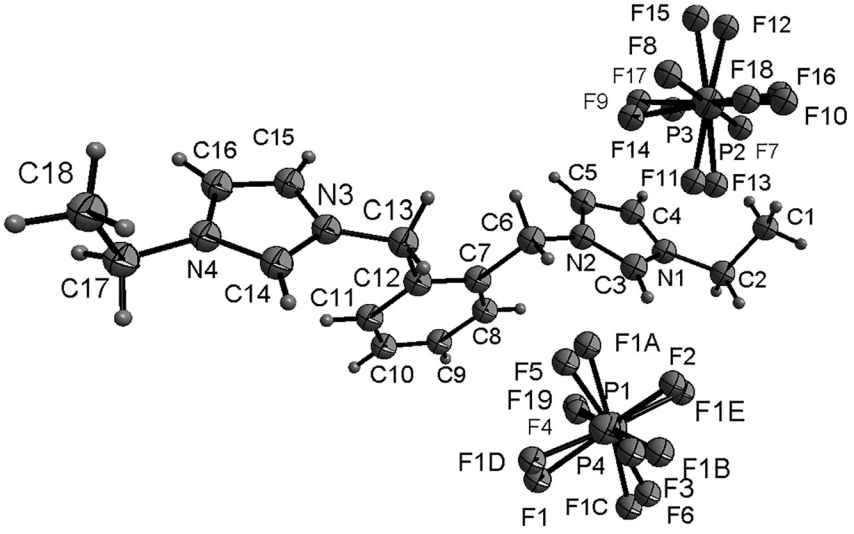
Crystal structure of 3,3′-(1,2-phenylene-bis(methylene))bis(1-ethyl-1*H*-imidazol-3-ium) bis(hexafluorophosphate), $C_{18}H_{24}F_{12}N_4P_2$



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Abstract

$C_{18}H_{24}F_{12}N_4P_2$, monoclinic, $P2_1/n$ (no. 14), a = 8.3185(9) Å, b = 21.883(2) Å, c = 13.4345(15) Å, β = 103.4730(10)°, V = 2378.3(5) Å^3, Z = 4, $R_{gt}(F)$ = 0.0531, $wR_{ref}(F^2)$ = 0.1424, T = 296(2) K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size	0.20 × 0.14 × 0.13 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}:	12957, 4654, 0.044
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2189
$N(param)_{refined}$:	454
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Source of material

To a stirred solution of 1-ethylimidazole (1.92 g, 0.02 mol) in acetonitrile (25 mL) at 75 °C was added dropwise *α*,*α*′-dibromo-*o*-xylene (2.64 g, 0.01 mol). The mixture was vigorously stirred at 75 °C for about 24 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 diethyl ether 3 times respectively. Then residual solvent was removed, and the product was dried in vacuo at 60 °C for 1 h to give a white powder solid in 98.68% yield. The intermediate compound (1.14 g, 2.5 mmol) and

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.2587 (11)	0.0640 (5)	0.0084 (2)	0.191 (3)
H1A	−0.1472	0.0790	−0.0081	0.287*
H1B	−0.3653	0.1015	−0.0063	0.287*
H1C	−0.2841	−0.0099	0.0071	0.287*
C2	−0.2277 (8)	0.0965 (4)	0.05750 (19)	0.1330 (19)
H2A	−0.1208	0.0578	0.0723	0.160*
H2B	−0.3392	0.0790	0.0742	0.160*
C3	−0.0359 (6)	0.2543 (4)	0.08282 (13)	0.0770 (11)
H3	0.0665	0.2176	0.0973	0.092*
C4	−0.3035 (6)	0.2891 (4)	0.04575 (15)	0.0909 (13)
H4	−0.4223	0.2812	0.0296	0.109*
C5	−0.2171 (6)	0.3798 (4)	0.05631 (14)	0.0831 (12)
H5	−0.2637	0.4465	0.0490	0.100*
C6	0.0991 (5)	0.4301 (3)	0.09713 (13)	0.0759 (11)
H6A	0.1112	0.4841	0.0733	0.091*
H6B	0.2199	0.3937	0.1013	0.091*
C7	0.0581 (5)	0.4814 (3)	0.14343 (12)	0.0589 (9)
C8	−0.0909 (5)	0.4497 (3)	0.16896 (13)	0.0669 (10)
H8	−0.1683	0.3947	0.1578	0.080*
C9	−0.1266 (5)	0.4992 (3)	0.21120 (14)	0.0748 (11)
H9	−0.2287	0.4776	0.2280	0.090*
C10	−0.0134 (6)	0.5793 (3)	0.22832 (13)	0.0749 (11)
H10	−0.0376	0.6121	0.2567	0.090*
C11	0.1370 (5)	0.6113 (3)	0.20330 (13)	0.0712 (10)
H11	0.2148	0.6656	0.2150	0.085*
C12	0.1736 (4)	0.5630 (3)	0.16064 (12)	0.0619 (9)
C13	0.3395 (5)	0.5978 (3)	0.13295 (14)	0.0861 (12)
H13A	0.4430	0.5481	0.1385	0.103*
H13B	0.3007	0.5965	0.0993	0.103*
C14	0.5630 (5)	0.7254(3)	0.17234 (14)	0.0741 (11)
H14	0.6494	0.6771	0.1860	0.089*
C15	0.3200 (6)	0.7932 (4)	0.13238 (16)	0.0922 (13)
H15	0.2075	0.8000	0.1134	0.111*
C16	0.4249 (7)	0.8706 (4)	0.15166 (17)	0.0943 (13)
H16	0.3991	0.9419	0.1486	0.113*
C17	0.7331 (7)	0.8837 (4)	0.20233 (16)	0.1075 (15)
H17A	0.6850	0.9495	0.2140	0.129*
H17B	0.7782	0.8424	0.2297	0.129*
C18	0.8873 (7)	0.9050 (5)	0.1748 (2)	0.161 (3)
H18A	0.8412	0.9359	0.1451	0.241*
H18B	0.9529	0.8409	0.1688	0.241*
H18C	0.9729	0.9528	0.1916	0.241*
N1	−0.1893 (5)	0.2108 (3)	0.06249 (11)	0.0823 (10)
N2	−0.0468 (4)	0.3561 (3)	0.07993 (10)	0.0682 (8)
N3	0.4082 (4)	0.7021 (3)	0.14572 (11)	0.0729 (9)
N4	0.5761 (5)	0.8274 (3)	0.17660 (11)	0.0771 (9)
P1[a]	0.4597 (7)	0.2120 (4)	0.1793 (2)	0.0593 (15)
P2[a]	0.7963 (12)	0.7189 (8)	0.0430 (3)	0.108 (3)
F1[a]	0.5375 (13)	0.2689 (8)	0.2241 (3)	0.121 (4)
F2[a]	0.3941 (19)	0.1501 (12)	0.1348 (4)	0.145 (5)
F3[a]	0.6654 (14)	0.1857 (15)	0.1722 (6)	0.120 (4)
F4[a]	0.266 (2)	0.2451 (13)	0.1886 (6)	0.178 (6)
F5[a]	0.488 (2)	0.3069 (10)	0.1497 (5)	0.126 (4)
F6[a]	0.4387 (19)	0.1105 (9)	0.2069 (6)	0.163 (6)
F7[a]	0.9713 (13)	0.7899 (10)	0.0442 (4)	0.148 (4)
F8[a]	0.6270 (11)	0.6403 (8)	0.0423 (4)	0.127 (4)
F9[a]	0.9218 (18)	0.6240 (9)	0.0272 (5)	0.146 (5)
F10[a]	0.6697 (18)	0.8043 (8)	0.0610 (4)	0.136 (4)
F11[a]	0.7517 (14)	0.7496 (11)	−0.0088 (3)	0.129 (6)
F12[a]	0.8503 (19)	0.6903 (10)	0.0958 (4)	0.129 (4)
P3[b]	0.7935 (7)	0.7147 (6)	0.04360 (18)	0.051 (2)
F13[b]	0.786 (2)	0.7824 (12)	−0.0036 (5)	0.138 (7)
F14[b]	0.722 (2)	0.6223 (6)	0.0103 (4)	0.137 (5)
F15[b]	0.786 (2)	0.6421 (13)	0.0889 (5)	0.144 (7)
F16[b]	0.866 (3)	0.8113 (9)	0.0731 (5)	0.175 (6)
F17[b]	1.0006 (16)	0.6802 (16)	0.0354 (5)	0.150 (7)
F18[b]	0.5795 (13)	0.7496 (16)	0.0481 (4)	0.147 (7)
P4[b]	0.4495 (15)	0.2036 (10)	0.1771 (4)	0.124 (4)
F19[b]	0.2389 (17)	0.2502 (9)	0.1871 (4)	0.084 (3)
F1A[b]	0.459 (3)	0.2895 (17)	0.1347 (7)	0.172 (10)
F1B[b]	0.649 (2)	0.1511 (18)	0.1656 (8)	0.156 (9)
F1C[b]	0.4338 (16)	0.1244 (13)	0.2212 (5)	0.119 (5)
F1D[b]	0.548 (2)	0.2884 (13)	0.2137 (7)	0.190 (9)
F1E[b]	0.335 (2)	0.1257 (13)	0.1395 (6)	0.163 (8)

[a]Occupancy: 0.558(12). [b]Occupancy: 0.442(12).

KPF_6 (0.92 g, 5 mmol) were dissolved in distilled water (25 mL). The mixture stirred for 6–8 h at 95 °C and then cooled slowly. The crystals of the target compound were obtained in 94.36% 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.

Comment

Ionic liquids (ILs) generally refer to a class of low-melting compounds composed of adjustable organic cations and inorganic or organic anions, and have different functions [5, 6]. ILs as a new type of environmentally friendly solvent, owing high catalytic efficiency at mild conditions, and can be recycled, etc., has been widely used in catalytic science, extraction and separation, biomass energy, resource conversion and other fields [7–9]. It is worth mentioning that various functional ionic liquids have been used to produce biodiesel highly efficiently and environmental friendly

[10–12]. Our group is still focusing on the preparation of ionic liquid that are able to catalyze the reaction to biodiesel [13, 14]. We have demonstrated that diimidazole-based hexafluorophosphate exhibits the temperature-controlled characteristics in methanol and water [15]. In order to find an ionic liquid to catalyze the reaction with better catalytic and recycled utilization efficiency, we were engaged in synthesizing the novel ionic liquid catalyst with imidazole.

In the cation of the title compound bond lengths and angles are very similar to those given in the literature [16]. The atoms of imidazole ring are coplanar, and the dihedral angle of two imidazole rings and the phenyl group are 6.9(1)°, 82.0(1)° and 84.0(1)°, respectively. The torsion angles of C1–C2–N1–C3, C7–C6–N2–C3, C12–C13–N3–C14 and C18–C17–N4–C14 are –120.4(4)°, –102.4(4)°, 103.7(4)° and 86.8(6)°, respectively.

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