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Crystal structure of 3,3'-(1,2-phenylene-bis(methylene))bis(1-vinyl-1*H*-imidazol-3-ium) bis(hexafluoro phosphate)(V), C₁₈H₂₀F₁₂N₄P₂

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Abstract

C₁₈H₂₀F₁₂N₄P₂, monoclinic, *P*₂/*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) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0470, *wR*_{ref}(*F*²) = 0.1305, *T* = 296(2) K.

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The asymmetric unit of the title crystal structure is shown in the figure. 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 × 0.15 × 0.13 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.29 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	25.5°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	18161, 4415, 0.034
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3052
<i>N</i> (<i>param</i>) _{refined} :	455
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Source of material

To a stirred solution of 1-vinylimidazole (1.88 g, 0.02 mol) in toluene (25 mL) at 105 °C was added dropwise 1,2-bis(bromomethyl)benzene (2.64 g, 0.01 mol). The mixture was vigorously stirred at 105 °C for about 16–18 h, and then allowed to stand at room temperature for about 3 h. After the reaction has been completed (monitored by TLC), the toluene 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 vacuum at 60 °C for 1 h to give a white powder solid in 97.57% yield. The intermediate compound (1.13 g, 0.0025 mol) and KPF₆ (0.92 g, 0.005 mol) 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 93.47% yield.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.4763 (5)	1.18599 (15)	0.7142 (3)	0.0979 (11)
H1A	0.5396	1.1783	0.7797	0.118*
H1B	0.4658	1.2257	0.6888	0.118*
C2	0.4047 (4)	1.14248 (14)	0.6595 (3)	0.0761 (8)
H2	0.3424	1.1517	0.5943	0.091*
C3	0.3557 (3)	1.03284 (13)	0.63230 (19)	0.0590 (7)
H3	0.3090	1.0347	0.5624	0.071*
C4	0.4709 (3)	1.05921 (12)	0.78921 (19)	0.0592 (6)
H4	0.5177	1.0827	0.8463	0.071*
C5	0.4476 (3)	0.99907 (12)	0.78741 (18)	0.0582 (6)
H5	0.4753	0.9727	0.8431	0.070*
C6	0.3380 (3)	0.92022 (11)	0.6526 (2)	0.0582 (6)
H6A	0.2606	0.9212	0.5864	0.070*
H6B	0.2853	0.8990	0.6999	0.070*
C7	0.4908 (3)	0.88509 (11)	0.64350 (17)	0.0508 (6)
C8	0.6405 (3)	0.91448 (12)	0.64981 (19)	0.0579 (6)
H8	0.6469	0.9565	0.6599	0.069*
C9	0.7798 (3)	0.88285 (13)	0.6414 (2)	0.0651 (7)
H9	0.8790	0.9034	0.6459	0.078*
C10	0.7716 (4)	0.82093 (13)	0.6266 (2)	0.0708 (8)
H10	0.8651	0.7991	0.6212	0.085*
C11	0.6235 (4)	0.79127 (12)	0.6197 (2)	0.0693 (8)
H11	0.6181	0.7492	0.6096	0.083*
C12	0.4829 (3)	0.82245 (12)	0.62748 (19)	0.0582 (6)
C13	0.3210 (4)	0.78849 (13)	0.6203 (3)	0.0797 (9)
H13A	0.3139	0.7755	0.6882	0.096*
H13B	0.2293	0.8159	0.5940	0.096*
C14	0.3294 (3)	0.67763 (13)	0.5819 (2)	0.0663 (7)
H14	0.3528	0.6636	0.6491	0.080*
C15	0.2761 (4)	0.73690 (13)	0.4491 (2)	0.0693 (7)
H15	0.2561	0.7717	0.4083	0.083*
C16	0.2795 (4)	0.67949 (15)	0.4170 (2)	0.0733 (8)
H16	0.2621	0.6668	0.3493	0.088*
C17 ^a	0.3244 (6)	0.5773 (2)	0.5097 (4)	0.0722 (14)
H17 ^a	0.3266	0.5596	0.5730	0.087*
C18 ^a	0.3318 (6)	0.5427 (3)	0.4348 (4)	0.0873 (18)
H18A ^a	0.3298	0.5596	0.3710	0.105*
H18B ^a	0.3393	0.5006	0.4438	0.105*
C19 ^b	0.332 (3)	0.5859 (7)	0.4476 (18)	0.080 (7)
H19 ^b	0.3595	0.5821	0.3847	0.096*
C20 ^b	0.304 (3)	0.5424 (10)	0.5032 (18)	0.086 (7)
H20A ^b	0.2768	0.5507	0.5652	0.103*
H20B ^b	0.3101	0.5022	0.4821	0.103*
N1	0.4133 (2)	1.08000 (9)	0.69139 (15)	0.0544 (5)
N2	0.3753 (2)	0.98288 (9)	0.68843 (14)	0.0525 (5)
N3	0.3077 (3)	0.73505 (9)	0.55351 (16)	0.0579 (5)
N4	0.3127 (3)	0.64320 (9)	0.50010 (19)	0.0631 (6)
P1	0.90678 (9)	0.08208 (4)	0.65747 (6)	0.0633 (2)
P2	0.29913 (10)	0.84651 (4)	0.94041 (6)	0.0691 (3)
F1 ^c	0.3022 (16)	0.9144 (3)	0.9527 (8)	0.126 (4)
F2 ^d	0.8909 (9)	0.1495 (3)	0.7115 (4)	0.126 (2)
F3 ^d	0.9184 (10)	0.0265 (3)	0.5948 (4)	0.142 (3)
F4 ^d	0.8313 (10)	0.0582 (5)	0.7422 (7)	0.125 (3)
F5 ^d	0.7291 (6)	0.0986 (5)	0.5879 (3)	0.129 (3)
F6 ^d	1.0827 (7)	0.0783 (4)	0.7256 (5)	0.110 (3)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
F7 ^c	0.1848 (13)	0.8394 (6)	1.0094 (10)	0.133 (4)
F8 ^c	0.4255 (12)	0.8549 (6)	0.8650 (9)	0.105 (4)
F9 ^e	0.1752 (16)	0.7940 (5)	0.9278 (6)	0.177 (4)
F10 ^e	0.4209 (12)	0.9020 (4)	0.9698 (4)	0.138 (3)
F11 ^e	0.4231 (15)	0.8107 (6)	1.0214 (8)	0.148 (5)
F12 ^e	0.1753 (9)	0.8905 (5)	0.8669 (5)	0.130 (3)
F13 ^c	0.4560 (14)	0.8351 (7)	1.0257 (6)	0.114 (5)
F14 ^e	0.2214 (10)	0.8689 (4)	1.0347 (5)	0.120 (3)
F15 ^c	0.1509 (10)	0.8481 (6)	0.8427 (7)	0.135 (3)
F16 ^e	0.3736 (14)	0.8291 (6)	0.8540 (7)	0.151 (5)
F17 ^c	0.3058 (18)	0.7761 (3)	0.9201 (9)	0.155 (4)
F18 ^d	0.9838 (10)	0.1046 (5)	0.5689 (6)	0.150 (3)
F19 ^f	0.9702 (17)	0.0133 (5)	0.6425 (13)	0.145 (4)
F20 ^f	0.8443 (14)	0.1289 (7)	0.7000 (16)	0.240 (10)
F21 ^f	0.7385 (11)	0.0552 (5)	0.5951 (6)	0.102 (3)
F22 ^f	0.928 (2)	0.1246 (5)	0.5722 (11)	0.136 (6)
F23 ^f	1.0792 (16)	0.0972 (8)	0.7206 (12)	0.155 (7)
F24 ^f	0.875 (2)	0.0354 (9)	0.7402 (13)	0.161 (7)

^aOccupancy: 0.810 (10), ^bOccupancy: 0.190 (10), ^cOccupancy: 0.436 (12),

^dOccupancy: 0.640 (10), ^eOccupancy: 0.564 (12), ^fOccupancy: 0.360 (10).

Experimental details

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

Comment

Ionic liquids generally refer to a class of low-melting salt composed of adjustable organic cations and inorganic or organic anions, and have different functions [5]. They have high chemical stability and thermodynamic stability, and have been widely applied in many fields [6–9]. A series of new ionic liquids have thermal response characteristics in organic solvents or water. They provide new ideas for recycling catalysts and avoiding their leaching from solvents [10, 11]. The novel ionic liquid as catalysts can realize the catalytic process of thermally regulated phase separation catalysis, and have the characteristics of homogeneous reaction at high temperature and phase separation at low temperature [12]. Our group is still continuously synthesizing such ionic liquids in order to find a temperature-controllable ionic liquid with better catalytic performance and recovery efficiency [13–16]. Based on previous research, our team is committed to synthesizing new temperature-adjusting ionic liquids by adjusting the different compositions of anions and

cations; and further studying the thermal stability of such ionic liquids in water or toluene.

In the molecule 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 between the two imidazole rings and the phenyl group are $18.3(2)^\circ$, $81.0(2)^\circ$ and $87.6(2)^\circ$, respectively. The torsion angles of C1-C2-N1-C3, C7-C6-N2-C3, C12-C13-N3-C14 and C18-C17-N4-C14 are $-171.6(6)$, $100.9(5)$, $103.0(5)$ and $-160.8(7)^\circ$, respectively.

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