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Crystal structure of 1,1'-(methane-1,1-diyl)bis(3-methyl-1*H*-imidazol-3-ium) bis(hexafluoridophosphate), $C_9H_{14}F_{12}N_4P_2$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.17 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.37 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	6290, 1635, 0.028
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1345
$N(\text{param})_{\text{refined}}$:	158
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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Abstract

$C_9H_{14}F_{12}N_4P_2$, monoclinic, $P2_1/m$ (no. 11), $a = 5.8968(9)$ Å, $b = 11.5554(18)$ Å, $c = 12.994(2)$ Å, $\beta = 94.577(2)^\circ$, $V = 882.6(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0676$, $wR_{\text{ref}}(F^2) = 0.2020$, $T = 296(2)$ 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.

Source of material

Dibromomethane (1.74 g, 0.02 mol) and 1-methylimidazole (1.64 g, 0.02 mol) were added into a 10 mL reaction tubes of a

microwave oven, and then the reaction proceeded under stirring at 70 °C for 8 min. A white solid was obtained after the reaction was completed and cooled down to room temperature; and the solid was washed with ethyl acetate and diethyl ether three times, respectively. The residual solvent was removed using a rotary evaporator and dried *in vacuo* at 50 °C for 30 min, and the white powder solid dimethylimidazole bromide was obtained with a yield of 76.62%. In the ion-exchanging reaction, the intermediate product (0.25 g, 0.0007 mol), potassium hexafluorophosphate (0.28 g, 0.0015 mol) and H₂O (10 mL) were added to the reaction tube. Subsequently, the mixture was stirred at 85 °C for 8 min in a microwave oven; then, a colorless crystal was obtained with a yield of 43.40%.

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 (ILs), as a class of organic ionic salt compounds with low melting point, are generally called room temperature ILs [5], and have the advantages of high

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

Atom	x	y	z	<i>U</i> _{iso} ^a / <i>U</i> _{eq}
P1	0.1006 (2)	0.2500	0.61407 (10)	0.0605 (5)
F1	0.3674 (7)	0.2500	0.6214 (5)	0.1334 (19)
F2	0.0933 (7)	0.3455 (4)	0.5280 (3)	0.1516 (16)
F3	−0.1652 (6)	0.2500	0.5999 (4)	0.1196 (17)
F4	0.1036 (6)	0.1523 (4)	0.6956 (3)	0.1388 (14)
P2	0.1201 (2)	0.2500	0.04918 (10)	0.0589 (5)
F5 ^a	−0.1333 (11)	0.2204 (15)	0.0491 (6)	0.170 (7)
F6 ^a	0.050 (2)	0.3636 (7)	0.0950 (9)	0.156 (4)
F7 ^a	0.3711 (10)	0.2825 (19)	0.0529 (6)	0.179 (10)
F8 ^a	0.190 (3)	0.1336 (8)	0.0085 (11)	0.190 (5)
F9 ^a	0.1523 (16)	0.2038 (12)	0.1611 (5)	0.201 (9)
F10 ^a	0.0882 (13)	0.2905 (13)	−0.0588 (5)	0.179 (9)
N1	0.4653 (6)	0.0018 (2)	0.2353 (3)	0.0657 (9)
N2	0.6369 (5)	0.1470 (3)	0.3087 (2)	0.0572 (8)
C1	0.6139 (6)	0.0828 (3)	0.2237 (3)	0.0586 (9)
H1	0.6921	0.0940	0.1651	0.070*
C2	0.3880 (9)	0.0130 (4)	0.3310 (4)	0.0858 (14)
H2	0.2806	−0.0340	0.3590	0.103*
C3	0.4935 (9)	0.1032 (4)	0.3773 (3)	0.0825 (13)
H3	0.4739	0.1310	0.4431	0.099*
C4	0.7751 (9)	0.2500	0.3232 (4)	0.0679 (14)
H4A	0.8911	0.2500	0.2743	0.081*
H4B	0.8508	0.2500	0.3923	0.081*
C5	0.3919 (10)	−0.0859 (4)	0.1587 (4)	0.0988 (16)
H5A	0.2809	−0.1355	0.1862	0.148*
H5B	0.3259	−0.0487	0.0974	0.148*
H5C	0.5206	−0.1312	0.1421	0.148*

^aOccupancy: 0.5.

selectivity and recyclability. After nearly 20 years of research and development, ILs have formed a wide variety and a large number of systems, which provides a good basis for the green catalytic reaction process [6–9], and have been widely used Functional ILs containing hexafluorophosphate, have been widely studied due to their superior physical and chemical properties [10]. They are used in functional materials such as electric contact lubricants, gel electrolyte, and other functional materials [11, 12]. So far, most of the research work on hexafluorophosphate containing ILs has focused on mono-imidazole based ILs [13]. Therefore, some diimidazole hexafluorophosphate ILs were synthesized by conventional oil bath heating in our group [14–18]. In order to explore a wider range of diimidazole ionic compounds and keep up with the pace of green chemistry, the synthesis of IL was carried out in microwave, and a crystalline compound was obtained in this experiment.

There is one half of a cation and two half anions in the asymmetric unit (shown in the figure). In the ions of the title compound bond lengths and angles are very similar to

those given in the literature [18, 19]. The atoms of the imidazole ring are coplanar. The torsion angles of C3–C2–N1–C5, C2–C3–N2–C4, C3–N2–C4–N2' are −179.5(4)°, 175.7(4)° and −75.4(4)°, respectively.

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