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Crystal structure of 3-(2-ethoxy-2-oxoethyl)-1-ethyl-1*H*-imidazol-3-ium hexafluoridophosphate(V), C₉H₁₅F₆N₂O₂P

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.21 × 0.17 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.26 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11050, 2724, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2028
$N(\text{param})_{\text{refined}}$:	205
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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Abstract

C₉H₁₅F₆N₂O₂P, monoclinic, $P2_1/n$ (no. 14), $a = 8.3781(15)$ Å, $b = 13.970(3)$ Å, $c = 12.904(2)$ Å, $\beta = 104.500(2)^\circ$, $V = 1462.3(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0677$, $wR_{\text{ref}}(F^2) = 0.1977$, $T = 296(2)$ K.

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The molecular structure is shown in the figure (Atoms are shown with arbitrary radii). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

Ethyl bromoacetate (7.5 mL, 0.067 mol) was added dropwise to a stirred solution of 1-ethylimidazole (5.77 g, 0.06 mol) in

THF (60 mL). The mixture was stirred vigorously at -5°C for 1.5 h, then at room temperature for 5–8 h. After the reaction completed (monitored by TLC), the THF top phase was decanted and the product washed with ethyl acetate and diethyl ether three 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 85.5% yield. Then the intermediate (3-(2-ethoxy-2-oxoethyl)-1-ethyl-1*H*-imidazol-3-ium bromide) (1.32 g, 0.005 mol) and KPF₆ (1.08 g, 0.0058 mol) were dissolved in water (25 mL). The mixture stirred at 80°C for 14 h, and then cooled slowly. The crystals were obtained in 25% 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 $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2 U_{\text{eq}}(\text{C})$ for all other H atoms. The ethyl group is disordered (see Table 2 and the Figure).

Comment

As a room temperature molten salt, some ionic liquids have been recognized as a new type of substance [5]. Most of them exist in liquid form at relatively low temperatures (below 100°C) or room temperature [6]. Due to their wide

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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} */ <i>U</i> _{eq}
C1	−0.215 (2)	0.6235 (18)	0.341 (2)	0.107 (8)
H1A	−0.2679	0.6186	0.4000	0.128*
H1B	−0.2162	0.5615	0.3074	0.128*
C2	−0.294 (3)	0.7010 (18)	0.2611 (17)	0.112 (8)
H2A	−0.2258	0.7131	0.2130	0.168*
H2B	−0.3060	0.7588	0.2988	0.168*
H2C	−0.4009	0.6797	0.2212	0.168*
C3	0.0885 (5)	0.6878 (3)	0.3473 (3)	0.0744 (10)
H3	0.0904	0.7073	0.2787	0.089*
C4	0.1674 (6)	0.6492 (3)	0.5167 (3)	0.0874 (12)
H4	0.2332	0.6380	0.5851	0.105*
C5	0.0042 (7)	0.6381 (4)	0.4832 (5)	0.1069 (17)
H5	−0.0651	0.6171	0.5246	0.128*
C6	0.3859 (4)	0.7006 (2)	0.4253 (3)	0.0654 (9)
H6A	0.3879	0.7139	0.3518	0.078*
H6B	0.4534	0.6444	0.4486	0.078*
C7	0.4584 (5)	0.7843 (3)	0.4944 (3)	0.0683 (9)
C8	0.6852 (7)	0.8905 (4)	0.5296 (5)	0.1161 (18)
H8A	0.6118	0.9450	0.5239	0.139*
H8B	0.7240	0.8738	0.6047	0.139*
C9	0.8191 (10)	0.9145 (6)	0.4887 (7)	0.186 (4)
H9A	0.8884	0.8595	0.4910	0.280*
H9B	0.8810	0.9650	0.5310	0.280*
H9C	0.7798	0.9357	0.4160	0.280*
N1	−0.0432 (4)	0.6629 (3)	0.3780 (3)	0.0974 (12)
N2	0.2178 (4)	0.6806 (2)	0.4296 (2)	0.0625 (7)
O1	0.4026 (4)	0.8196 (2)	0.5618 (2)	0.0909 (9)
O2	0.5963 (3)	0.80970 (19)	0.4704 (2)	0.0791 (8)
P1	0.21382 (14)	0.92432 (7)	0.20331 (8)	0.0741 (4)
F1	0.3934 (4)	0.9605 (3)	0.2159 (4)	0.1680 (16)
F2	0.0353 (4)	0.8861 (3)	0.1873 (3)	0.1488 (14)
F3	0.1626 (4)	1.02882 (19)	0.2258 (2)	0.1221 (11)
F4	0.2675 (5)	0.8199 (2)	0.1817 (3)	0.1370 (13)
F5	0.2497 (5)	0.9010 (2)	0.3253 (2)	0.1282 (12)
F6	0.1809 (5)	0.9463 (3)	0.0801 (2)	0.1323 (12)
C2A	−0.2120 (9)	0.6706 (8)	0.3010 (8)	0.111 (3)
H2AA	−0.2871	0.7049	0.3340	0.133*
H2AB	−0.2049	0.7045	0.2367	0.133*
C1A	−0.2678 (12)	0.5757 (7)	0.2759 (10)	0.155 (4)
H1AA	−0.3757	0.5770	0.2279	0.233*
H1AB	−0.2723	0.5428	0.3404	0.233*
H1AC	−0.1931	0.5430	0.2425	0.233*

^aOccupancy: 0.265(15), ^bOccupancy: 0.735(15).

electrochemical window [7], they have a wide application prospect in extraction [8], biocatalysis [9], and material synthesis [10] compared to traditional solvents. In recent years, some hexafluorophosphate ionic liquids have been extensively studied due to their superior physical and chemical properties [11, 12]. Therefore, in order to keep up with the pace of green chemistry, we are committed to find

hexafluorophosphate ionic liquids with better catalytic and recycled utilization efficiency [5, 13–17].

In the title structure bond lengths and angles within 3-(2-ethoxy-2-oxoethyl)-1-ethyl-1*H*-imidazol-3-ium and in the counter anion hexafluoridophosphate(V) are very similar to those given in the literature for 3-(2-ethoxy-2-oxoethyl)-1-vinyl-1*H*-imidazol-3-ium hexafluoridophosphate(V) [5, 18]. The atoms of the imidazole moiety are coplanar, and the dihedral angle of the imidazole ring and the carboxylate group is 58.8(2)°. The torsion angles of C3–N2–C6–C7, N2–C6–C7–O2, C6–C7–O2–C8, and C7–O2–C8–C9 are 116.0(3)°, −167.8(2)°, −179.9(3)° and 174.3(4)°, respectively.

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