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Crystal structure of 3-(2-ethoxy-2-oxoethyl)-1-methyl-1*H*-imidazol-3-ium hexafluoridophosphate(V), $C_8H_{13}F_6N_2O_2P$

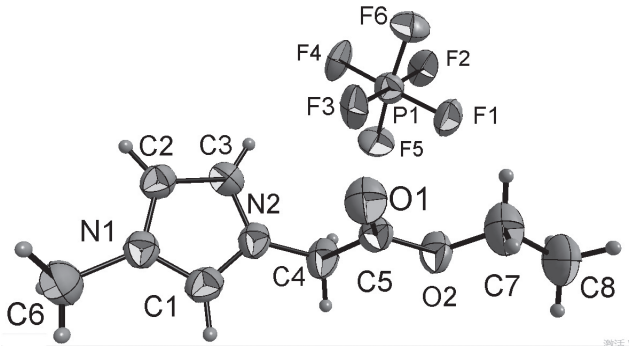

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.12 × 0.14 × 0.18 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%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9660, 2377, 0.015
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2026
$N(param)_{refined}$:	174
Programs:	Bruker [1], SHELX [2]

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Abstract

$C_8H_{13}F_6N_2O_2P$, monoclinic, $P2_1/c$ (no. 14), $a = 9.0228(12)$ Å, $b = 13.6501(18)$ Å, $c = 11.2771(15)$ Å, $\beta = 112.904(1)°$, $V = 1279.4(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0773$, $wR_{ref}(F^2) = 0.2385$, $T = 296(2)$ 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.

Source of material

To a stirred solution of l-methylimidazole (4.1 g, 4.0 mL) in THF (50 mL) at −5 °C under a nitrogen atmosphere was added dropwise ethyl bromoacetate (10.0 g, 6.7 mL). The mixture was stirred vigorously at −5 °C for 1 h, then at room temperature for 3 h. After the reaction completed (monitored by TLC), the THF top phase was decanted and the product washed with ethyl acetate and diethyl ether 3 times respectively, then residual solvent removed, and the product was dried *in vacuo* at 60 °C for 72 h to give a clear viscous oil in 95.3% yield. Then the intermediate (3-(2-ethoxy-2-oxoethyl)-1-methyl-1*H*-imidazol-3-ium bromide) (0.55 g, 2.2 mmol) and KPF_6 (0.41 g, 2.2 mmol) were dissolved in water (40 mL). The mixture stirred for 12 h at 90 °C and then cooled slowly. The crystals were obtained in 76.8% yield.

Experimental details

All H atoms were included in calculated positions and refined as riding atoms. Difference electron density peaks (Q1–Q10; 0.7–0.3 eA^3) are located near the PF_6^- counterion. The non-ideal R-factors are caused by the disorder of the counterion.

Comment

Ionic liquids are a subset of molten salts (melting points at or below 100 °C) with huge potential to have an impact across many areas of scientific and engineering research [3]. Due to their low volatility, they are considered as promising alternative solvents to replace traditional organic compounds (VOCs), spurred by the green chemistry movement. Nowadays, various exciting applications of ionic liquids have been developed continuously [4–7]. Temperature-dependant systems not only showed excellent catalytic performance,

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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	−0.1616(5)	0.8914(3)	0.2941(3)	0.0521(8)
H1	−0.2505	0.8501	0.2658	0.062*
C2	−0.0098(4)	1.0227(3)	0.3497(3)	0.0490(8)
H2	0.0221	1.0878	0.3655	0.059*
C3	0.0872(4)	0.9448(3)	0.3687(3)	0.0507(8)
H3	0.1990	0.9457	0.4002	0.061*
C4	0.0430(5)	0.7621(3)	0.3378(3)	0.0599(10)
H4A	0.1282	0.7584	0.3058	0.072*
H4B	−0.0461	0.7225	0.2821	0.072*
C5	0.1033(4)	0.7212(3)	0.4725(3)	0.0475(8)
C6	−0.3118(5)	1.0476(3)	0.2653(4)	0.0676(11)
H6A	−0.4016	1.0061	0.2561	0.101*
H6B	−0.3309	1.0796	0.1848	0.101*
H6C	−0.2989	1.0959	0.3304	0.101*
C7	0.2197(7)	0.5809(4)	0.5938(5)	0.0773(13)
H7A	0.1446	0.5834	0.6361	0.093*
H7B	0.3188	0.6128	0.6486	0.093*
C8	0.2521(7)	0.4774(4)	0.5703(6)	0.0921(16)
H8A	0.3306	0.4757	0.5324	0.138*
H8B	0.1543	0.4473	0.5130	0.138*
H8C	0.2924	0.4425	0.6505	0.138*
N1	−0.1657(4)	0.9883(2)	0.3025(3)	0.0478(7)
N2	−0.0096(4)	0.8635(2)	0.3327(3)	0.0498(7)
O1	0.1050(3)	0.7639(2)	0.5645(2)	0.0600(7)
O2	0.1520(3)	0.62990(18)	0.4698(2)	0.0587(7)
P1	0.57699(11)	0.79854(8)	0.50128(10)	0.0564(4)
F1	0.5472(5)	0.6916(3)	0.5400(5)	0.1315(15)
F2	0.7427(6)	0.7608(5)	0.5085(8)	0.192(3)
F3	0.4204(6)	0.8346(4)	0.5040(7)	0.185(3)
F4	0.6175(8)	0.8994(3)	0.4628(6)	0.175(2)
F5	0.5005(10)	0.7673(5)	0.3625(4)	0.216(3)
F6	0.6634(9)	0.8222(5)	0.6458(4)	0.194(3)

but also provided a convenient way to reuse and avoid the leaching of catalysts from the solvents [8, 9]. The catalytic system with a thermoregulated property, namely, the monophasic reaction combined with a biphasic separation, which facilitated the fast reaction of the substrates in monophasic phase, the simple separation of the product from the catalytic phase and the recycling of the catalytic system. Recently, our group has demonstrated that ionic liquids of [RMIM]-PF_6 are responsible for the thermomorphic properties of the ionic liquid in water or toluene. And our group still focused on the preparation of thermoregulated ionic liquid [10–12]. In order to find an ionic liquid catalyst with better catalytic and recycled utilization efficiency, we were engaged in synthesizing the novel ionic liquid catalyst with imidazole.

In the molecule of the title compound bond lengths and angles within methyl 4-acetoxybenzoate are very similar to those given in the literature [13–15]. In the title structure, the part of 2-ethoxy-2-oxoethyl is approximately planar. The dihedral angle formed by the imidazole ring with the 2-ethoxy-2-oxoethyl group plane is 79.3(3)°.

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