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Crystal structure of 1,1′-(hexane-1,6-diyl)bis(3-ethyl-1H-imidazol-3-ium) bis(hexafluorido phosphate), C₁₆H₂₈F₁₂N₄P₂

Table 1: Data collection and handling.

| Crystal: | Colourless block |
| Size: | 0.20 × 0.13 × 0.12 mm |
| Wavelength: | Mo Ka radiation (0.71073 Å) |
| µ: | 0.28 mm⁻¹ |
| Diffractometer, scan mode: | Bruker APEX-II, φ and ω |
| θmax, completeness: | 25.0°, >99% |
| N(hkl) measured, N(hkl) unique: | 8792, 2125, 0.025 |
| Criterion for Iobs, R(hkl)gt: | Iobs > 2 σ(Iobs), 1691 |
| N(param) refined: | 214 |
| Programs: | Bruker [1], SHELX [2] |

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material
1-Ethylimidazole (9.61 g, 0.1 mol) was dissolved in methylbenzene (20 mL), 1,6-dibromohexane (12.1 g, 0.05 mol) was quickly added under stirring. The mixture was reacted at 80 °C for 10 min, and then heated to 90 °C for 8 hours. After the reaction completed (monitored by TLC), a white solid was produced after cooling. The resulting suspension was filtered, crushed and washed with ethylacetate and diethyl ether 3 times respectively. The white powder intermediate (C₆EM—Br) was dried in vacuo (19.32 g, yield 89%). Then the intermediate (C₆EM—Br)(2.17 g, 0.005 mol), potassium hexafluorophosphate (2.48 g, 0.012 mol) was dissolved in water (36 mL) and methanol (4 mL). The mixture stirred well for 12 h at 95 °C and then cooled slowly. The crystals suitable for X-ray analysis were obtained.

Experimental details
All H atoms were included in calculated positions and refined as riding atoms, with C—H = 0.90—0.97 Å with Uiso(H) = 1.5Ueq(C) for methyl H atoms and 1.2Ueq(C) for all other H atoms. The [PF₆]⁻ anion is disordered over two positions (cf. the figure, Table 2), which is typical for many hexafluoridophosphates.
Ionic liquids have been synthesized, and have been used in biodiesel preparation. In recent years, various functionalized ionic liquids have the unique potential advantages of the unique physical and chemical properties of ionic liquids. Dicationic ionic liquid, with a large variety of tunable interactions, has been explored in the past decade [3–5]. Because of the unique physical and chemical properties of ionic liquids, ionic liquids have the unique potential advantages of biodiesel preparation. In recent years, various functional ionic liquids have been synthesized, and have been used to prepare biodiesel highly efficiently and environmentally friendly [6, 7]. It was found that dinuclear alkaline liquid bis-(3-methyl-1-imidazolium)-ethylene dihydroxide (MC2OH) shows excellent catalytic efficiency. The highest conversion rate of cotton seed oil was up to 98.5%, and the stability and separation effect of the catalyst was ideal [8].

Recently, our group focused on the preparation of biodiesel catalyzed by ionic liquid [9, 10] and reported three crystal structures of 1,1′-(butane-1,4-diyl)bis(3-methyl-1H-imidazol-3-ium)-bis(hexafluorophosphate), 1,1′-(hexane-1,6-diyl)bis(3-methyl-1H-imidazol-3-ium)-bis(hexafluorophosphate) and 1,1′-(ethane-1,2-diyl)bis(3-ethyl-1H-imidazol-3-ium)-bis(hexafluorophosphate) [11–13]. In order to find the ionic liquid catalyst with better catalytic efficiency, we were engaged in synthesising novel ionic liquid catalysts with imidazole.

Herein, we report the synthesis and structure of the bisimidazoles ionic liquid. Bond lengths and angles within the imidazoles ring are very similar to those given in the literature for diimidazole ionic liquid [14]. The title structure consists of one half of C Stick2 + cation (1,1′-(ethane-1,2-diyl)bis(3-ethyl-1H-imidazol-3-ium)) and one [PF6]− anion (cf. the figure). Two cationic 1-ethylimidazolium rings were bound to the both sides of ethyl group. The two imidazole rings are crystallographically dependent planar and parallel to each other. The torsion angle of C4—N1—C3—C2, C3—N1—C4—C5 and N1—C4—C5—C6 is 175.1(2)°, 156.7(2)° and 67.5(3)°, respectively.

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