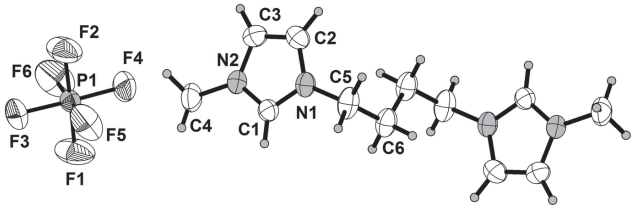
Xiong Wan-Ming, Chen Jing, Peng Da-Yong, Nie Xu-Liang* and Chang-gan Huang

Crystal structure of 1,1′-butanebis(3-methyl-1*H*-imidazol-3-ium)bis(hexafluorophosphate), $C_{12}H_{20}F_{12}N_4P_2$



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Abstract

$C_{12}H_{20}F_{12}N_4P_2$, triclinic, $P\bar{1}$ (no. 2), $a = 7.388(4)$ Å, $b = 8.499(4)$ Å, $c = 9.347(4)$ Å, $\alpha = 90.198(5)°$, $\beta = 106.496(5)°$, $\gamma = 113.042(5)°$, $V = 513.4(4)$ Å^3, $Z = 1$, $R_{gt}(F) = 0.0652$, $wR_{ref}(F^2) = 0.2037$, $T = 296(2)$ K.

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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-Methylimidazole (22.99 g, 0.28 mol) and 1,4-dichlorobutane (17.77 g, 0.14 mol) were quickly added in 40 mL acetonitrile, then stirred well for 10 h at 80 °C. After the reaction completed (monitored by TLC), a white solid was produced. The resulting suspension was filtered, crushed and washed with ethylacetate and diethyl ether five times, respectively. The intermediate were dried *in vacuo* with the yield 90%. Then the intermediate (1.90 g, 0.005 mol) and potassium hexafluorophosphate (2 g, 0.011 mol) were dissolved in deionized water (30 mL). The mixture was stirred for 10 h at 70 °C and then

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.22 × 0.20 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.33 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω-scans
$2\theta_{max}$, completeness:	25.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3943, 1902, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1652
$N(param)_{refined}$:	138
Programs:	Bruker programs [1], SHELX [2]

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

Atom	x	y	z	U_{iso}*/U_{eq}
P1	0.41723(14)	0.25954(11)	0.71920(9)	0.0541(4)
F1	0.4215(6)	0.1032(4)	0.8039(4)	0.1337(15)
F2	0.4080(7)	0.4167(4)	0.6365(4)	0.1322(14)
F3	0.4754(6)	0.3674(4)	0.8758(3)	0.1096(11)
F4	0.3621(5)	0.1575(4)	0.5596(3)	0.1047(11)
F5	0.6543(5)	0.3271(6)	0.7406(4)	0.1254(14)
F6	0.1799(5)	0.1942(7)	0.6945(4)	0.1503(18)
N1	1.0245(4)	0.2575(4)	0.1802(3)	0.0535(7)
N2	0.8571(4)	0.2861(4)	0.3243(3)	0.0542(7)
C1	1.0056(5)	0.2416(4)	0.3156(4)	0.0562(8)
H1	1.0845	0.2048	0.3929	0.067*
C2	0.8804(6)	0.3131(5)	0.0986(4)	0.0646(10)
H2	0.8590	0.3344	-0.0010	0.077*
C3	0.7763(6)	0.3310(5)	0.1886(4)	0.0634(9)
H3	0.6689	0.3673	0.1635	0.076*
C4	0.7815(8)	0.2785(6)	0.4550(5)	0.0796(12)
H4A	0.6591	0.1744	0.4402	0.119*
H4B	0.7496	0.3763	0.4658	0.119*
H4C	0.8864	0.2801	0.5443	0.119*
C5	1.1740(6)	0.2197(5)	0.1257(5)	0.0743(12)
H5A	1.3022	0.2521	0.2075	0.089*
H5B	1.2038	0.2894	0.0465	0.089*
C6	1.0968(6)	0.0327(5)	0.0662(5)	0.0689(11)
H6A	1.2048	0.0159	0.0375	0.083*
H6B	1.0710	−0.0361	0.1467	0.083*

[a]Occupancy: 0.854(7).

***Corresponding author: Nie Xu-Liang,** Key Laboratory of Natural, Product Research and Development/College of Sciences, Jiangxi Agricultural University, Nanchang 330045, People's Republic of China, e-mail: niexuliang1981@163.com; windhcg@163.com
Xiong Wan-Ming and Peng Da-Yong: Collaborative Innovation Center of Jiangxi Typical Trees Cultivation and Utilization/College of Sciences, Jiangxi Agricultural University, Nanchang 330045, People's Republic of China
Chen Jing and Chang-gan Huang: College of Sciences, Jiangxi Agricultural University, Nanchang 330045, People's Republic of China

cooled slowly. The obtained colorless crystals were washed with deionized water. The final product was obtained after air drying with the yield 64%.

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C—H = 0.96-0.97 Å with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and 1.2 $U_{eq}(C)$ for all other H atoms.

Discussion

Ionic liquid, as a new type of environmentally friendly solvent, owing to the advantages of adjustable structure, high catalytic efficiency, mild conditions, and can be recycled, etc, has been widely used in catalytic science, electrochemistry, environmental science, extraction and separation, biomass energy, resource conversion and other fields [3–5]. Because of the unique physical and chemical properties of ionic liquids, ionic liquids has the unique potential advantages of biodiesel preparation. In recent years, various functional ionic liquids have been synthesised, and have been used to produce biodiesel highly efficiently and environmental friendly [6, 7]. It was found that dinuclear alkaline ionic liquid bis-(3-methyl-1-imidazolium-)-ethlyene dihydroxide shows excellent catalytic efficiency, the highest conversion rate of cotton seed oil was up to 98.5%, and the stability of and separation effect of the catalyst was very ideal [8]. Recently, our group still focused on the preparation of biodiesel catalyzed by ionic liquid [9, 10] and reported a crystal structure of 1,1′-(hexane-1,6-diyl)bis(3-methyl-1*H*-imidazol-3-ium) bis(hexafluorophosphate) [11]. In order to find the ionic liquid catalyst with better catalytic efficiency we were engaged in synthesising novel ionic liquid catalysts with imidazole. Bond lengths and angles within the imidazole ring are very similar to those given in the literature for diimidazoles [12]. The title structure consists of one half of a $C4M^{2+}$ cation (1,1′-butanebis(3-methyl-1*H*-imidazol-3-ium)) and one PF_6^- anion (*cf*. the figure). Two cationic 1-ethylimidazolium rings were bound to the both sides of butyl group. The two imidazole rings are crystallographically dependant planar and parallel to each other. The torsion angle of C5–N1–C2–C3 and C6–C5–N1–C2 is 179.7° and 93.6°, respectively.

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