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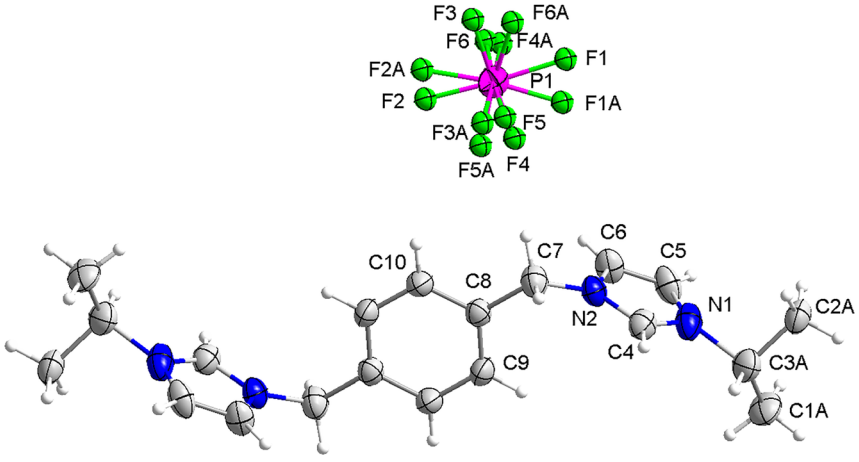
Crystal structure of 3,3′-(1,4-phenylenebis(methylene))bis(1-isopropyl-1*H*-imidazol-3-ium) bis(hexafluorophosphate(V)), $C_{10}H_{14}F_6N_2P$



https://doi.org/10.1515/ncrs-2023-0440
Received October 9, 2023; accepted November 23, 2023;
published online December 6, 2023

Abstract

$C_{10}H_{14}F_6N_2P$, orthorhombic, *Pbca* (no. 61), a = 9.5929(10) Å, b = 15.9201(16) Å, c = 17.8640(19) Å, V = 2728.2(5) Å^3, Z = 8, $R_{gt}(F)$ = 0.0587, $wR_{ref}(F^2)$ = 0.1735, T = 296(2) K.

CCDC no.: 2309863

The molecular 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.

1 Source of materials

1,4-Bis(chloromethyl)benzene (2.63 g, 0.015 mol) and 1-isopropylimidazole (3.30 g, 0.03 mol) were added to a round-bottomed flask respectively, and 30 mL of acetonitrile was added as a solvent. The mixtures were stirred vigorously for 24 h at 75 °C. After the reaction has been completed, it was poured out the upper layer of acetonitrile, the intermediate was then washed three times with ethyl acetate and anhydrous ether, respectively. The intermediate product was

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.30 × 0.20 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.26 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}:	19,825, 2534, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1681
$N(param)_{refined}$:	240
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

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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}
C1B[a]	0.795 (3)	0.382 (2)	0.6181 (14)	0.128 (5)
H1BA[a]	0.846217	0.330437	0.616431	0.192*
H1BB[a]	0.856549	0.427309	0.631172	0.192*
H1BC[a]	0.754228	0.392947	0.569982	0.192*
C1A[b]	0.824 (3)	0.384 (2)	0.6436 (13)	0.128 (5)
H1AA[b]	0.855962	0.347135	0.605070	0.192*
H1AB[b]	0.902210	0.406003	0.670888	0.192*
H1AC[b]	0.773320	0.430089	0.621392	0.192*
C2B[a]	0.7279 (13)	0.3557 (7)	0.7572 (7)	0.110 (3)
H2BA[a]	0.648396	0.340182	0.786809	0.165*
H2BB[a]	0.771247	0.404460	0.778589	0.165*
H2BC[a]	0.793439	0.310086	0.756555	0.165*
C2A[b]	0.6658 (13)	0.3969 (7)	0.7521 (7)	0.110 (3)
H2AA[b]	0.594824	0.430349	0.728655	0.165*
H2AB[b]	0.734697	0.433095	0.773970	0.165*
H2AC[b]	0.624550	0.362734	0.790424	0.165*
C3B[a]	0.6806 (9)	0.3754 (5)	0.6762 (5)	0.0830 (16)
H3B[a]	0.619437	0.424721	0.674884	0.100*
C3A[b]	0.7352 (9)	0.3401 (5)	0.6931 (5)	0.0830 (16)
H3A[b]	0.788911	0.296196	0.718475	0.100*
C4	0.5966 (3)	0.2185 (2)	0.66835 (15)	0.0684 (8)
H4	0.654622	0.189675	0.701257	0.082*
C5	0.5032 (5)	0.3165 (2)	0.6013 (2)	0.1079 (14)
H5	0.485713	0.368605	0.579695	0.129*
C6	0.4342 (5)	0.2463 (2)	0.5879 (2)	0.0984 (12)
H6	0.359116	0.239993	0.555474	0.118*
C7	0.4487 (4)	0.09738 (19)	0.63449 (16)	0.0744 (8)
H7A	0.349873	0.095542	0.645849	0.089*
H7B	0.497900	0.070006	0.675176	0.089*
C8	0.4750 (3)	0.04942 (16)	0.56303 (14)	0.0575 (7)
C9	0.5932 (3)	0.06091 (18)	0.52018 (15)	0.0663 (7)
H9	0.656931	0.102310	0.533507	0.080*
C10	0.3817 (3)	−0.01204 (17)	0.54203 (16)	0.0655 (7)
H10	0.301275	−0.020592	0.570051	0.079*
F1[b]	−0.0096 (16)	0.1992 (5)	0.6824 (6)	0.157 (4)
F1A[a]	0.0752 (15)	0.1956 (8)	0.6549 (7)	0.213 (7)
F2[b]	−0.0085 (16)	0.0441 (7)	0.5794 (7)	0.179 (6)
F2A[a]	−0.0693 (16)	0.0403 (7)	0.5947 (9)	0.189 (6)
F3[b]	−0.1367 (12)	0.0838 (6)	0.6725 (7)	0.125 (4)
F3A[a]	0.0921 (12)	0.0567 (8)	0.6733 (6)	0.141 (5)
F4[b]	0.1155 (10)	0.1569 (10)	0.5854 (8)	0.180 (6)
F4A[a]	−0.0969 (11)	0.1789 (6)	0.5791 (7)	0.118 (4)
F5[b]	0.0925 (14)	0.0825 (9)	0.6881 (5)	0.140 (5)
F5A[a]	0.1121 (10)	0.1173 (7)	0.5648 (5)	0.124 (4)
F6[b]	−0.1109 (8)	0.1611 (7)	0.5735 (6)	0.112 (4)
F6A[a]	−0.1090 (11)	0.1288 (10)	0.6916 (6)	0.152 (5)
N1	0.6052 (3)	0.29870 (17)	0.65260 (15)	0.0871 (9)
N2	0.4932 (2)	0.18509 (15)	0.63032 (11)	0.0617 (6)
P1	−0.00459 (9)	0.11970 (6)	0.62776 (4)	0.0735 (3)

[a]Occupancy: 0.493 (6), [b]occupancy: 0.507 (6).

vacuum dried at 60 °C for 0.5 h to obtain a white powder with 93.22 % yield. During anion exchange, the intermediate (1.43 g, 0.005 mol) and potassium hexafluorophosphate (1.84 g, 0.01 mol) were dissolved in deionized water (50 mL) and stirred vigorously at 85 °C for 6 h. After the reaction time was over the resulting mixture was slowly cooled to room temperature. After washing with deionized water and drying with air, the yield was 76.76 %.

2 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_{iso}(H) = 1.5 U_{eq}(C) for methyl H atoms and 1.2 U_{eq}(C) for all other H atoms.

3 Comment

Ionic liquids (ILs) are a kind of molten salts with melting points typically below 100 °C, usually consisting of bulky and unpaired organic cations with organic or inorganic anions [5]. They have been studied with wide interest because of the unique physicochemical properties such as low toxicity, high thermal stability, and low volatility [6–9]. Hexafluorophosphate ionic liquids have also gained a great deal of attention and research due to their excellent characteristics such as high oxidative stability [10]. As far as we know, previous work has focused on the synthesis of diimidazole hexafluorophosphates in which two imidazoles are linked by a flexible carbon chain [11]. Among them, a series of diimidazole hexafluorophosphates with different crystal structures were synthesized by altering the length of the flexible carbon chain, and the R group of the sidechain of the imidazole ring. However, there are relatively few reports on the synthesis of diimidazole ionic liquids with two imidazole rings linked by a phenyl-substituted flexible carbon chain. The introduction of a benzene ring combined with nitrogen to form an sp^2 hybridization improves thermal stability, additional electronic effects and spatial site resistance, as well as improves solubility and facilitates catalytic extraction [12]. In view of this, equally considering the excellent properties of diimidazole hexafluorophosphate, it is of great significance to seek a diimidazole hexafluorophosphate that is coupled by phenyl [13, 14].

In the cation of the title compound, bond lengths and angles are very similar to those given in the literature [14, 15]. Both hexafluorophosphate ion and isopropyl in the molecule are disordered. All atoms of imidazole ring are on the same plane, and the two imidazole rings are parallel to each other. The dihedral angle of imidazole rings and the phenyl group is 88.6(1)°. The torsion angles of C1A–C3A–N1–C4, C3A–N1–C4–N2, C4–N2–C7–C8 and N2–C7–C8–C9 are 126.8(13)°, –176.1(4)°, –112.7(3)°, and 38.6(4)°, respectively.

Acknowledgment: X-ray data were collected at Instrumental Analysis Center Nanchang Hangkong University, Nanchang, 330063, People's Republic of China

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by the Natural Science Foundation of Jiangxi Province of China (No. 20202BABL205003), the Research Foundation of Education Department of Jiangxi Province of China (No. GJJ210430) and National College Students Innovation and Entrepreneurship Training Program (No. S202310410048 and 202310410313).

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