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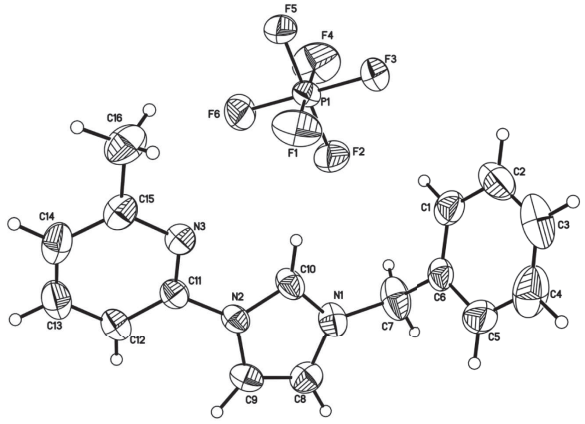
Crystal structure of 1-benzyl-3-(4-methylpyridin-2-yl)-1H-imidazol-3-ium hexafluorophosphate, $C_{16}H_{16}F_6N_3P$



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Abstract

$C_{16}H_{16}F_6N_3P$, monoclinic, $P2_1/n$, $a = 8.6414(8)$ Å, $b = 20.755(2)$ Å, $c = 10.1157(8)$ Å, $\beta = 104.686(9)°$, $V = 1755.0(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0547$, $wR_{ref}(F^2) = 0.1496$, $T = 291(2)$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colorless blocks, size 0.17×0.18×0.2 mm
Wavelength:	Cu K_α radiation (1.54184 Å)
μ:	20.14 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Eos, Gemini, ω scans
$2\theta_{max}$:	134.15°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	6326, 3133
$N(param)_{refined}$:	237
Programs:	SHELX [5–7]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.2556	0.7912	0.2813	0.097
H(2)	4*e*	0.0665	0.8399	0.1102	0.123
H(3)	4*e*	−0.0612	0.9285	0.1518	0.143
H(4)	4*e*	−0.0013	0.9739	0.3644	0.148
H(5)	4*e*	0.1840	0.9249	0.5419	0.110
H(7A)	4*e*	0.4377	0.7960	0.4927	0.118
H(7B)	4*e*	0.4399	0.8552	0.5893	0.118
H(8)	4*e*	0.4108	0.8180	0.8237	0.088
H(9)	4*e*	0.2813	0.7293	0.9109	0.083
H(10)	4*e*	0.1783	0.7145	0.5082	0.080
H(12)	4*e*	0.2076	0.6207	0.9084	0.086
H(13)	4*e*	0.0529	0.5284	0.9025	0.103
H(14)	4*e*	−0.1303	0.5014	0.7039	0.102
H(16A)	4*e*	−0.2805	0.5938	0.4351	0.152
H(16B)	4*e*	−0.2549	0.5202	0.4683	0.152
H(16C)	4*e*	−0.1465	0.5559	0.3887	0.152

Source of material

The title compound was obtained by the follow method: the mixture of benzyl chloride (2.52 g, 20 mmol) and 2-(imidazol-1-yl)pyridine (1.45 g, 10 mmol) was refluxed for 40 h in ethyl acetate (30 mL). After the solvent was evaporated, the residue was dissolved in acetone (50 mL), then KPF_6 (2.76 g, 15 mmol) was added and reacted for 3 h. After the precipitate was filtrated, the organic solvent was evaporated and a white powder was obtained (2.7 g, yield 71%). Single crystals suitable for X-ray diffraction were obtained by crystallization from methanol.

Discussion

Since the isolation of the thermally stable, free N-heterocyclic carbenes (NHCs) by Arduengo et al. in 1991, metal complexes based on NHCs have been extensively reported for their intriguing structures and numerous applications in catalysis,

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.2040(5)	0.8281(2)	0.3001(4)	0.088(2)	0.062(2)	0.095(2)	−0.014(2)	0.029(2)	0.001(2)
C(2)	4*e*	0.0909(6)	0.8573(3)	0.1978(4)	0.109(3)	0.124(4)	0.068(2)	−0.037(3)	0.012(2)	0.015(2)
C(3)	4*e*	0.0161(6)	0.9096(3)	0.2223(6)	0.086(3)	0.135(5)	0.132(4)	0.001(3)	0.019(3)	0.069(4)
C(4)	4*e*	0.0502(7)	0.9364(2)	0.3488(7)	0.120(4)	0.093(3)	0.179(5)	0.040(3)	0.077(4)	0.035(4)
C(5)	4*e*	0.1624(5)	0.9075(2)	0.4544(4)	0.116(3)	0.078(2)	0.093(3)	−0.002(2)	0.050(2)	−0.005(2)
C(6)	4*e*	0.2419(4)	0.8530(1)	0.4303(3)	0.062(2)	0.058(2)	0.069(2)	−0.016(1)	0.012(1)	0.012(1)
C(7)	4*e*	0.3723(4)	0.8219(2)	0.5371(4)	0.067(2)	0.114(3)	0.104(3)	−0.024(2)	0.006(2)	0.040(2)
C(8)	4*e*	0.3476(4)	0.7861(2)	0.7720(3)	0.061(2)	0.076(2)	0.075(2)	−0.007(2)	0.003(2)	−0.008(2)
C(9)	4*e*	0.2766(4)	0.7375(2)	0.8196(3)	0.066(2)	0.089(2)	0.050(1)	0.000(2)	0.010(1)	−0.005(2)
C(10)	4*e*	0.2192(4)	0.7291(2)	0.5971(3)	0.061(2)	0.083(2)	0.050(1)	−0.015(2)	0.002(1)	0.011(1)
C(11)	4*e*	0.1049(3)	0.6440(1)	0.7108(3)	0.054(1)	0.063(2)	0.057(2)	0.003(1)	0.017(1)	0.003(1)
C(12)	4*e*	0.1318(4)	0.6085(2)	0.8296(3)	0.075(2)	0.080(2)	0.061(2)	0.005(2)	0.021(2)	0.013(2)
C(13)	4*e*	0.0407(5)	0.5542(2)	0.8252(4)	0.098(3)	0.079(2)	0.089(2)	0.008(2)	0.038(2)	0.023(2)
C(14)	4*e*	−0.0677(5)	0.5381(2)	0.7072(4)	0.084(2)	0.061(2)	0.123(3)	−0.003(2)	0.049(2)	0.007(2)
C(15)	4*e*	−0.0857(4)	0.5763(2)	0.5914(4)	0.058(2)	0.066(2)	0.091(2)	−0.003(2)	0.023(2)	−0.006(2)
C(16)	4*e*	−0.2023(5)	0.5601(2)	0.4590(4)	0.085(3)	0.094(3)	0.115(3)	−0.023(2)	0.008(2)	−0.018(2)
N(1)	4*e*	0.3103(3)	0.7803(1)	0.6324(3)	0.054(1)	0.083(2)	0.069(2)	−0.014(1)	−0.002(1)	0.017(1)
N(2)	4*e*	0.1949(3)	0.7018(1)	0.7093(2)	0.054(1)	0.068(1)	0.046(1)	−0.001(1)	0.0086(9)	0.005(1)
N(3)	4*e*	0.0020(3)	0.6300(1)	0.5938(2)	0.053(1)	0.064(1)	0.061(1)	−0.002(1)	0.014(1)	0.001(1)
F(1)	4*e*	0.1954(3)	0.6561(2)	0.3122(3)	0.082(1)	0.178(3)	0.106(2)	0.020(2)	0.035(1)	−0.027(2)
F(2)	4*e*	0.4490(3)	0.6849(1)	0.3512(2)	0.100(2)	0.098(2)	0.093(1)	−0.021(1)	−0.002(1)	−0.014(1)
F(3)	4*e*	0.2853(4)	0.6900(1)	0.1399(2)	0.206(3)	0.080(1)	0.071(1)	−0.014(2)	0.003(2)	0.017(1)
F(4)	4*e*	0.4795(4)	0.6160(2)	0.1904(4)	0.130(2)	0.182(3)	0.178(3)	−0.001(2)	0.098(2)	−0.043(3)
F(5)	4*e*	0.2233(3)	0.5867(1)	0.1512(2)	0.128(2)	0.082(1)	0.068(1)	−0.016(1)	−0.009(1)	−0.005(1)
F(6)	4*e*	0.3857(3)	0.5806(1)	0.3610(2)	0.119(2)	0.093(2)	0.087(1)	−0.003(1)	−0.017(1)	0.024(1)
P(1)	4*e*	0.33791(9)	0.63520(4)	0.25089(7)	0.0625(5)	0.0682(5)	0.0512(4)	0.0029(4)	0.0105(3)	−0.0002(3)

medicine, and materials chemistry [1–3]. It has been known that imidazolium salt precursors are suitable for the synthesis of NHCs complexes [4]. So we focus on new imidazolium salts with different substituents that can provide metal complexes with interesting properties.

The asymmetric unit of the title compound consists of one 1-benzyl-3-(4-methylpyridin-2-yl)-1H-imidazol-3-ium cation and one PF_6^- anion. In the cation, the dihedral angles between the benzene ring (C1—C6), the imidazolium ring (C8—C10/N1/N2), and the pyridine ring (C11—C15/N3) are 71.71(4)°, 20.48(3)°, and 66.76(3)°, respectively. The bond of C11—N2 (1.433(4) Å) is slightly shorter than a single C—N bond, indicating a small double bond character. In the imidazolium group, the bonding pattern presents two short C—N bonds (C10—N1 = 1.316(4) Å, C10—N2 = 1.332(3) Å), one short C—C bond (C8—C9 = 1.332(5) Å), and two longer C—N bonds (C8—N1 = 1.371(4) Å, C9—N2 = 1.376(4) Å).

Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors $U_{iso}(H) = 1.2$ times U_{eq}(C, aromatic ring), $U_{iso}(H) = 1.2$ times U_{eq}(C, methylene) and $U_{iso}(H) = 1.5$ times U_{eq}(C, methyl). The final refinement was executed using geometrical restraints, with C–H = 0.93 Å (aromatic ring), C–H = 0.96 Å (methyl), and C–H = 0.97 Å (methylene).

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References

1. Pugh, D.; Danopoulos, A. A.: Metal complexes with 'pincer'-type ligands incorporating N-Heterocyclic carbene functionalities. Coord. Chem. Rev. **251** (2007) 610–641.
2. Catalano, V. J.; Munro, L. B.; Strasser, C. E.; Samin, A. F.: Modulation of metal-metal separations in a series of Ag(I) and intensely blue photoluminescent Cu(I) NHC-Bridged triangular clusters. Inorg. Chem. **50** (2011) 8465–8476.
3. Gimeno, M.C.; Laguna, A.; Visbal, R.: N-Heterocyclic carbene coinage metal complexes as intense blue-green emitters. Organometallics. **31** (2012) 7146–7157.
4. Gründemann, S.; Kovacevic, A.; Albrecht, M.; Faller, J. W.; Crabtree, R. H.: Abnormal ligand binding and reversible ring hydrogenation in the reaction of imidazolium salts with $IrH_5(PPh_3)_2$. J. Am. Chem. Soc. **124** (2002) 10473–10481.
5. Sheldrick, G. M.: SHELXS-97. Program for the solution of crystal structures. University of Göttingen, Germany 1997.
6. Sheldrick, G. M.: SHELXL-97. Program for the refinement of crystal structures. University of Göttingen, Germany 1997.
7. Sheldrick, G. M.: SHELXTL. Structure determination software suite. Version 5.10. Bruker AXS, Madison, Wisconsin, USA 1998.