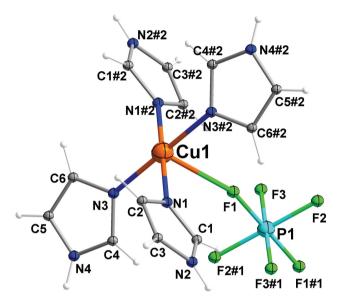
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The crystal structure of tetrakis(imidazole)-copper(I) hexafluorophosphate, C₁₂H₁₆CuF₆PN₈



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Abstract

C₁₂H₁₆CuF₆PN₈, monoclinic, *C*2/*c* (no. 15), a = 13.102(2) Å, b = 9.7902(18) Å, c = 14.727(3) Å, $\beta = 102.336(6)^{\circ}$, V = 1845.3(6) Å³, Z = 4, $R_{gt}(F) = 0.0291$, $wR_{ref}(F^2) = 0.0733$, T = 150(2) K.

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The molecular structure is shown in the figure (#1 = 2 - x, -y, 1 - z; #2 = 3/2 - x, 1/2 - y, 1 - z). Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Blue block
Size:	$0.10\times0.09\times0.05~\text{mm}$
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	$1.35 \; \text{mm}^{-1}$
Diffractometer, scan mode:	SMART-CCD, $arphi$ and ω
$\theta_{\sf max}$, completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	12617, 2117, 0.041
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{\rm obs} > 2 \ \sigma(I_{\rm obs})$, 1819
N(param) _{refined} :	130
Programs:	SHELX [1], Diamond [2], Bruker [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\mathring{A}^2).

Atom	x	у	Z	$m{U}_{iso}$ * $/m{U}_{eq}$
Cu1	0.7500	0.2500	0.5000	0.01249(11)
P1	1.0000	0.0000	0.5000	0.01608(17)
F1	0.91577(9)	0.12743(12)	0.50604(8)	0.0213(3)
F2	1.10077(9)	0.11204(12)	0.52407(9)	0.0237(3)
F3	0.98707(9)	0.03018(12)	0.38507(8)	0.0190(3)
N1	0.76290(13)	0.20315(17)	0.63424(11)	0.0144(3)
C1	0.83501(16)	0.1270(2)	0.68815(14)	0.0201(4)
H1	0.8877	0.0769	0.6672	0.024*
N2	0.82319(14)	0.13044(19)	0.77608(12)	0.0234(4)
H20	0.8623	0.0873	0.8234	0.028*
C3	0.74002(18)	0.2123(2)	0.77962(15)	0.0253(5)
Н3	0.7133	0.2335	0.8331	0.030*
C2	0.70283(16)	0.2577(2)	0.69154(14)	0.0190(4)
H2	0.6449	0.3172	0.6726	0.023*
N3	0.66010(13)	0.08440(17)	0.46104(12)	0.0167(4)
C4	0.66951(18)	-0.0373(2)	0.50006(16)	0.0264(5)
H4	0.7231	-0.0617	0.5518	0.032*
N4	0.59424(16)	-0.12227(19)	0.45787(14)	0.0288(5)
H40	0.5863	-0.2080	0.4728	0.035*
C5	0.5324(2)	-0.0518(3)	0.38771(19)	0.0353(6)
H5	0.4722	-0.0850	0.3457	0.042*
C6	0.57341(18)	0.0752(2)	0.38949(17)	0.0316(6)
Н6	0.5466	0.1470	0.3478	0.038*

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Source of material

2-Naphthylacetic acid (0.056 g, 0.3 mmol) was dissolved in a methanol solution (6 mL) of $Cu(CH_3CN)_4PF_4$ (0.112 g, 0.3 mmol) under ultrasonication. To the resulting green solution imidazole (IM) (0.04 g, 0.6 mmol) was added under ultrasonication. Afterwards the mixture was sealed and heated to

70 °C for 20 h. After cooled to room temperature, it was filtered, and than the cyan filtrate was evaporated at 5 °C. After 2 weeks, blue block crystals of title complex formed.

Experimental details

All hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms.

Comment

The coordination chemistry between copper and imidazolates are so colorful that versatile structures and functions such as adsorption and separation, catalysis, magnetism etc. have been reported in many studies [4]. Copper with different valences mainly like +1 and +2 has flexible coordination geometries and is an excellent candidate for constructing various metal-ligand frameworks. For imidazolates, they are widely used to construct organic-inorganic hybrid compounds due to their different coordination modes acting as a terminal and/or bridging ligand [5, 6]. Furthermore, in the copper/imidazolate system the types of counter ions [7], solvents types [4] and the used secondary ligands [8], as well as the reaction stoichiometry [9], temperature [4, 10] and pH [11] are key factors for the formation of final structures. Therefore the structure construction between copper and imidazolates has been far beyond prediction and control.

In the mononuclear title complex, the Cu^I ion, is located on a crystallographic inversion centre, and is six coordinated by two F atoms from two symmetry related PF_6^- and four N atoms from four imidazoles. This coordination is very similar to the complex tetrakis(N-methylimidazole-N)copper(I) perchlorate, $[Cu(C_4H_6N_2)_4][ClO_4]$ [12]. Four imidazole ligands act as terminal ligands with Cu(I)-N distances from 2.0011(16) to 2.0140(17) Å, whereas the PF_6^- anions act as bridging ligand linking two nearby copper atoms to form a one-dimensional coordination polymer.

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