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Tetrapyridinecopper(I) hexafluoridophosphate(V)

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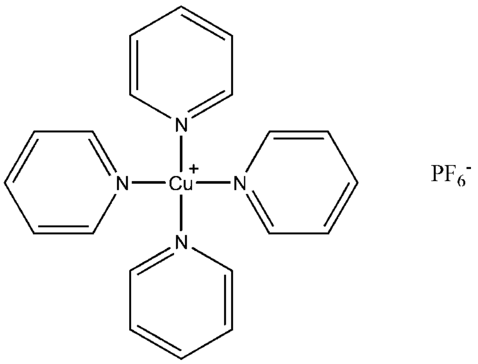
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.007 Å; R factor = 0.044; wR factor = 0.118; data-to-parameter ratio = 16.6.

The crystal structure of the title compound, $[Cu(C_5H_5N)_4]PF_6$, consists of discrete $[Cu(C_5H_5N)_4]^+$ and PF_6^- ions. Both the cations and the anions are located on $\overline{4}$ axes. The pyridine ligands are bonded to the Cu^+ ion in an almost perfectly tetrahedral coordination geometry, with Cu—N = 2.061 (3) Å and N—Cu—N angles of 110.00 (9) and 108.42 (17)°.

Related literature

The only previously described structure containing tetrakis(pyridine)copper(I) cations is the perchlorate salt described by Nilsson & Oskarsson (1981, 1982). It crystallized in the same space group as the title compound with analogous placement of the ions. The Cu atom was almost perfectly tetrahedrally coordinated.



Experimental

Crystal data

$[Cu(C_5H_5N)_4]PF_6$
M_r = 524.91
Tetragonal, $I\overline{4}$
a = 12.600 (2) Å
c = 7.0983 (14) Å
V = 1126.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.10 mm^{-1}
T = 295 (2) K
0.39 × 0.19 × 0.14 mm

Data collection

Oxford Diffraction Xcalibur CCD area-detector diffractometer
Absorption correction: analytical (Alcock, 1970)
T_{min} = 0.616, T_{max} = 0.791
4610 measured reflections
1212 independent reflections
1074 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.118
S = 1.09
1212 reflections
73 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), with 551 Friedel pairs
Flack parameter: −0.01 (3)

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST95* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2014).

References

Alcock, N. W. (1970). *Alcock, N. W. (1970). Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, p. 271–278. Copenhagen: Munksgaard.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Nilsson, K. & Oskarsson, A. (1981). *Acta Cryst.* A**37**, C-227.
Nilsson, K. & Oskarsson, A. (1982). *Acta Chem. Scand. A*, **36**, 605–610.
Oxford Diffraction (2003). *CrysAlis CCD* and *CrysAlis RED*. Versions 1.170. Oxford Diffraction Ltd., Wrocław, Poland.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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Tetrapyridinecopper(I) hexafluoridophosphate(V)

G. Horvat, **T. Portada**, **V. Stilinovic** and **V. Tomisic**

Comment

The crystal structure of (I) consists of discrete tetrakis(pyridine)copper(I) and hexafluorophosphate(V) ions. The copper and phosphorus atoms are placed in 2a and 2c special positions of the space group respectively. The coordination of copper is almost perfectly tetrahedral with N—Cu—N angles 110.00 (9)° and 108.42 (17)°. The planes of the pyridine rings are at angles of 79.38° and 65.93°.

The hexafluorophosphate anions are octahedral and slightly elongated along *z* axes with F—P—F angles in the range 89.4 (3)° − 90.6 (3)° and P—F bond lengths 1.527 (4) Å and 1.553 (5) Å.

The shortest intermolecular distance is a C—H···F contact of 3.445 Å

Experimental

The title compound was obtained unintentionally while attempting to crystallize a coordination compound of copper(I) with a bisoxazoline ligand. Tetrakis(acetonitrile)copper(I) hexafluorophosphate(V) (10 mg) and bisoxazoline (20 mg) were dissolved in a mixture of pyridine and toluene (10 ml, 1:1) and heated to boiling. The resulting solution was cooled to room temperature and left to slowly evaporate. Colourless crystals of (I) were obtained after 4 days.

Refinement

All H atoms were placed geometrically and included in the refinement in the riding-model approximation, with C—H distances 0.93 with $U_{\mathrm{iso}} = 1.2U_{\mathrm{eq}}$ of the carrier atom.

Figures

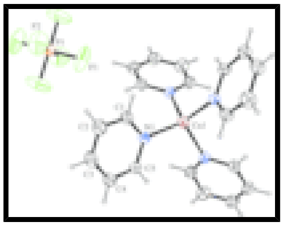

Fig. 1. View of (I) with the atom labeling scheme. Displacement ellipsoids are shown at 20% probability. Hydrogen atoms are shown as spheres of arbitrary radius.

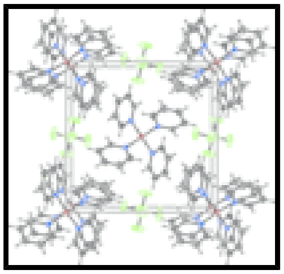

Fig. 2. Crystal packing of (I) viewed along the *z* axis.

Tetrapyridinecopper(I) hexafluoridophosphate(V)

Crystal data

$[Cu(C_5H_5N)_4]PF_6$
$M_r = 524.91$
Tetragonal, $I\bar{4}$
Hall symbol: I -4
$a = 12.600$ (2) Å
$b = 12.600$ (2) Å
$c = 7.0983$ (14) Å
$\alpha = 90°$
$\beta = 90°$
$\gamma = 90°$
$V = 1126.9$ (3) Å^3

$Z = 2$
$F_{000} = 532$
$D_x = 1.547$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 1485 reflections
$\theta = 4.6–32.0°$
$\mu = 1.10$ mm^{-1}
$T = 295$ (2) K
Prism, colourless
0.39 × 0.19 × 0.14 mm

Data collection

Oxford Diffraction Xcalibur CCD area-detector diffractometer
Radiation source: fine–focus sealed tube
Monochromator: graphite
$T = 295(2)$ K
ω scans
Absorption correction: analytical (Alcock, 1970)
$T_{min} = 0.616$, $T_{max} = 0.791$
4610 measured reflections

1212 independent reflections
1074 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 27.0°$
$\theta_{min} = 4.6°$
$h = -16\rightarrow16$
$k = -16\rightarrow15$
$l = -9\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.118$
$S = 1.09$

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0713P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

1212 reflections	Extinction correction: none
73 parameters	Absolute structure: Flack (1983), with 551 Friedel pairs
1 restraint	Flack parameter: −0.01 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0	0	0	0.0670 (3)
P1	0.5	0	0.25	0.0556 (4)
C5	0.0527 (3)	0.2144 (3)	0.1582 (6)	0.0663 (9)
H5	0.007	0.2382	0.0643	0.08*
N1	0.0734 (2)	0.1106 (2)	0.1698 (4)	0.0594 (7)
C4	0.0961 (3)	0.2872 (3)	0.2790 (7)	0.0770 (12)
H4	0.0789	0.3587	0.2691	0.092*
C3	0.1657 (4)	0.2524 (4)	0.4157 (7)	0.0828 (12)
H3	0.1956	0.2998	0.501	0.099*
C2	0.1896 (5)	0.1477 (4)	0.4225 (7)	0.0879 (13)
H2	0.2386	0.1225	0.5096	0.105*
C1	0.1414 (3)	0.0798 (3)	0.3012 (6)	0.0702 (10)
H1	0.1569	0.0079	0.3109	0.084*
F2	0.5	0	0.4688 (6)	0.156 (2)
F1	0.3816 (3)	−0.0255 (6)	0.2479 (10)	0.196 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0591 (4)	0.0591 (4)	0.0828 (6)	0	0	0
P1	0.0555 (6)	0.0555 (6)	0.0558 (9)	0	0	0
C5	0.061 (2)	0.0560 (19)	0.082 (2)	0.0073 (17)	−0.0066 (18)	−0.0021 (18)
N1	0.0593 (16)	0.0497 (14)	0.0694 (16)	−0.0001 (12)	0.0014 (13)	0.0025 (12)
C4	0.078 (3)	0.058 (2)	0.095 (3)	−0.0017 (19)	0.010 (2)	−0.012 (2)
C3	0.085 (3)	0.083 (3)	0.080 (2)	−0.016 (2)	−0.001 (2)	−0.024 (2)
C2	0.101 (3)	0.091 (3)	0.072 (2)	−0.010 (3)	−0.020 (2)	−0.001 (2)
C1	0.078 (2)	0.059 (2)	0.074 (2)	−0.0019 (19)	−0.0111 (18)	0.0089 (17)
F2	0.178 (6)	0.227 (7)	0.062 (3)	−0.019 (5)	0	0
F1	0.0681 (19)	0.323 (8)	0.198 (5)	−0.030 (3)	−0.012 (3)	−0.014 (5)

Geometric parameters (Å, °)

Cu1—N1	2.061 (3)	C5—C4	1.369 (6)

Cu1—N1^{i}	2.061 (3)	C5—H5	0.93
Cu1—N1^{ii}	2.061 (3)	N1—C1	1.324 (5)
Cu1—N1^{iii}	2.061 (3)	C4—C3	1.379 (7)
P1—F1^{iv}	1.526 (4)	C4—H4	0.93
P1—F1	1.526 (4)	C3—C2	1.354 (7)
P1—F1^{v}	1.526 (4)	C3—H3	0.93
P1—F1^{vi}	1.526 (4)	C2—C1	1.357 (7)
P1—F2^{iv}	1.553 (5)	C2—H2	0.93
P1—F2	1.553 (5)	C1—H1	0.93
C5—N1	1.337 (5)		

N1—Cu1—N1^{i}	110.00 (9)	F2^{iv}—P1—F2	180
N1—Cu1—N1^{ii}	110.00 (9)	N1—C5—C4	122.6 (4)
N1^{i}—Cu1—N1^{ii}	108.42 (17)	N1—C5—H5	118.7
N1—Cu1—N1^{iii}	108.42 (17)	C4—C5—H5	118.7
N1^{i}—Cu1—N1^{iii}	110.00 (9)	C1—N1—C5	117.1 (3)
N1^{ii}—Cu1—N1^{iii}	110.00 (9)	C1—N1—Cu1	120.3 (2)
F1^{iv}—P1—F1	90.006 (6)	C5—N1—Cu1	122.6 (3)
F1^{iv}—P1—F1^{v}	90.006 (6)	C5—C4—C3	118.8 (4)
F1—P1—F1^{v}	178.9 (6)	C5—C4—H4	120.6
F1^{iv}—P1—F1^{vi}	178.9 (6)	C3—C4—H4	120.6
F1—P1—F1^{vi}	90.006 (6)	C2—C3—C4	118.4 (4)
F1^{v}—P1—F1^{vi}	90.006 (6)	C2—C3—H3	120.8
F1^{iv}—P1—F2^{iv}	90.6 (3)	C4—C3—H3	120.8
F1—P1—F2^{iv}	89.4 (3)	C3—C2—C1	119.5 (5)
F1^{v}—P1—F2^{iv}	89.4 (3)	C3—C2—H2	120.3
F1^{vi}—P1—F2^{iv}	90.6 (3)	C1—C2—H2	120.3
F1^{iv}—P1—F2	89.4 (3)	N1—C1—C2	123.5 (4)
F1—P1—F2	90.6 (3)	N1—C1—H1	118.2
F1^{v}—P1—F2	90.6 (3)	C2—C1—H1	118.2
F1^{vi}—P1—F2	89.4 (3)		

Symmetry codes: (i) $-y, x, -z$; (ii) $y, -x, -z$; (iii) $-x, -y, z$; (iv) $y+1/2, -x+1/2, -z+1/2$; (v) $-x+1, -y, z$; (vi) $-y+1/2, x-1/2, -z+1/2$.

Fig. 1

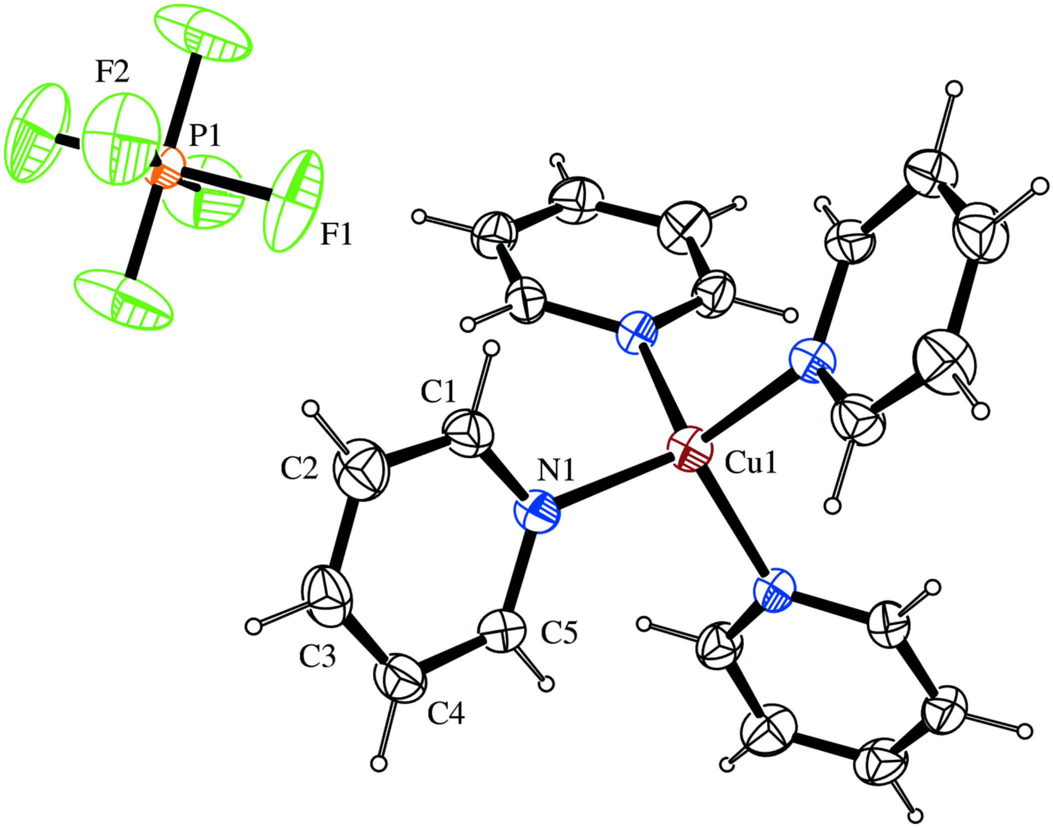

Fig. 2