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Bis(2,2':6',2''-terpyridine)copper(II) Hexafluorophosphate

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Abstract. $[\text{Cu}(\text{C}_{15}\text{H}_{11}\text{N}_3)_2](\text{F}_6\text{P})_2$, $M_r = 820.0$, tetragonal, $P4_2/c$, $a = 8.930(3)$, $c = 20.623(5)$ Å, $V = 1644.6$ Å³, $Z = 2$, $D_m = D_c = 1.66$ g cm⁻³, $F(000) = 882$, $\mu(\text{Mo } K\alpha) = 8.97$ cm⁻¹. The $[\text{Cu}(\text{tpy})_2](\text{F}_6\text{P})_2$ structure was solved by the Patterson method and refined to $R = 0.048$ for 559 independent reflections. This structure consists of isolated $[M(\text{tpy})]^{2+}$ groups and isolated $(\text{F}_6\text{P})^-$ octahedra. It is compared with $[\text{Cu}(\text{tpy})_2](\text{NO}_3)_2$.

Introduction. Crystal structures of various M^{2+} terpyridine complexes have recently been reported (Maslen, Raston & White, 1974; Allmann, Henke & Reinen, 1978), but so far no data are available for similar compounds with the $(\text{F}_6\text{P})^-$ ligand.

$[\text{Cu}(\text{tpy})_2](\text{F}_6\text{P})_2$ was first synthesized by one of us (TR) starting from a 2,2':6',2''-terpyridine solution in hot water and a $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ solution. A KF_6P solution was added to the Cu-tpy chloride complex until a light-green precipitate appeared. This precipitate was washed with chloroform and crystallized from acetone to give green crystals, which were dried *in vacuo* over P_2O_5 .

Elemental analysis: calculated for $[\text{Cu}(\text{C}_{15}\text{H}_{11}\text{N}_3)_2](\text{F}_6\text{P})_2$: C 43.9, N 10.2, H 2.7, Cu 7.8%; experimental: C 44.1, N 10.4, H 2.8, Cu (determined by atomic absorption spectroscopy) 7.9%.

A crystal of $[\text{Cu}(\text{tpy})_2](\text{F}_6\text{P})_2$, $0.3 \times 0.27 \times 0.25$ mm, was mounted on a Syntex $P2_1$ four-circle diffractometer. Graphite-monochromated Mo $K_{\alpha 1}$ radiation ($\lambda = 0.7107$ Å) was used. 720 reflections with $2\theta \leq 47^\circ$ were collected. Of these, 559 with $I \geq 2.5\sigma(I)$ were considered as observed. The relative

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Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters
$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B_{eq} (\AA^2)
Cu	0	0	0	5.59 (3)
N(1)	0	0	959 (5)	5.81 (3)
C(2)	1198 (11)	-556 (9)	1280 (5)	5.98 (1)
C(3)	1234 (11)	-562 (10)	1947 (6)	7.26 (2)
C(4)	0	0	2293 (7)	8.26 (3)
C(5)	2407 (10)	-1095 (9)	877 (6)	5.59 (2)
N(6)	2179 (9)	-947 (8)	240 (4)	5.22 (2)
C(7)	3259 (11)	-1386 (10)	-156 (5)	5.98 (1)
C(8)	4605 (11)	-1992 (11)	63 (6)	7.36 (3)
C(9)	4810 (15)	-2157 (12)	709 (6)	8.32 (4)
C(10)	3727 (11)	-1709 (11)	1134 (5)	6.47 (1)
P(11)	0	5000	1889 (2)	7.59 (3)
F(12)	1255 (9)	3740 (7)	1883 (4)	11.12 (2)
F(13)	844 (12)	5882 (9)	2434 (4)	14.63 (8)
F(14)	-855 (7)	4120 (7)	1351 (4)	9.59 (1)

intensities were corrected for the usual Lorentz and polarization factors. No corrections were made for absorption.

The structure of $[\text{Cu}(\text{tpy})_2](\text{F}_6\text{P})_2$ was solved by the Patterson method.

The refinement was carried out by the program *SHELX 76* (Sheldrick, 1976) with anisotropic thermal parameters for the non-hydrogen atoms. At the end of the refinement process the final R value was 0.048 for all observed reflections.

The final positional and thermal parameters, with their standard deviations, are listed in Table 1.*

Discussion. Fig. 1 is a stereoview of the $[\text{Cu}(\text{tpy})_2](\text{F}_6\text{P})_2$ molecule. Bond distances and angles are given in Tables 2 and 3. The crystal structure is in perfect agreement with the elemental analysis of the complex, corresponding to the formula $[\text{Cu}(\text{C}_{15}\text{H}_{11}\text{N}_3)_2](\text{F}_6\text{P})_2$.

* Lists of structure factors, anisotropic thermal parameters and atomic coordinates of hydrogen atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36589 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

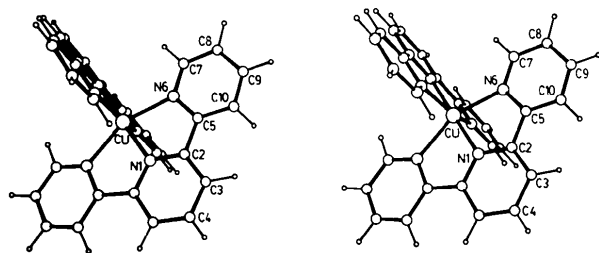


Fig. 1. Stereoscopic view of a molecule of $[\text{Cu}(\text{tpy})_2](\text{F}_6\text{P})_2$ (*PLUTO*, Motherwell & Clegg, 1978), with the atom numbering.

Table 2. Interatomic distances (\AA)

N(1)—Cu	1.977 (8)	C(7)—N(6)	1.322 (9)
N(6)—Cu	2.179 (7)	C(8)—C(7)	1.394 (12)
C(2)—N(1)	1.353 (9)	C(9)—C(8)	1.352 (11)
C(3)—C(2)	1.376 (13)	C(10)—C(9)	1.366 (12)
C(5)—C(2)	1.445 (11)	F(12)—P(11)	1.589 (6)
C(4)—C(3)	1.405 (10)	F(13)—P(11)	1.566 (7)
N(6)—C(5)	1.336 (10)	F(14)—P(11)	1.560 (6)
C(10)—C(5)	1.404 (11)		

Table 3. Bond angles ($^\circ$)

The superscripts refer to the following symmetry operations: (i) \bar{x}, \bar{y}, z ; (ii) y, x, \bar{z} ; (iii) y, \bar{x}, \bar{z} .

N(6)—Cu—N(1)	76.9 (0.2)	C(7)—N(6)—C(5)	117.8 (0.7)
C(2)—N(1)—Cu	119.3 (0.5)	N(6)—Cu—N(6 ⁱⁱ)	93.0 (0.1)
C(3)—C(2)—N(1)	120.6 (0.9)	N(6)—Cu—N(6 ⁱⁱⁱ)	93.0 (0.1)
C(5)—C(2)—N(1)	115.5 (0.8)	C(8)—C(7)—N(6)	123.0 (0.8)
C(5)—C(2)—C(3)	123.8 (0.9)	C(9)—C(8)—C(7)	118.6 (1.0)
C(2)—N(1)—C(2 ⁱ)	121.3 (1.0)	C(10)—C(9)—C(8)	120.3 (1.1)
C(4)—C(3)—C(2)	119.2 (1.0)	C(9)—C(10)—C(5)	117.8 (0.9)
C(3)—C(4)—C(3 ⁱ)	119.0 (1.2)	F(13)—P(11)—F(12)	91.3 (0.4)
N(6)—C(5)—C(2)	114.8 (0.8)	F(14)—P(11)—F(12)	89.0 (0.3)
C(10)—C(5)—C(2)	122.7 (0.9)	F(14)—P(11)—F(13)	179.4 (0.5)
C(10)—C(5)—N(6)	122.5 (0.8)	F(12)—P(11)—F(12 ⁱ)	179.1 (0.3)
C(5)—N(6)—Cu	113.4 (0.6)	F(13)—P(11)—F(13 ⁱ)	88.2 (0.7)
C(7)—N(6)—Cu	128.8 (0.6)	F(14)—P(11)—F(14 ⁱ)	89.2 (0.5)

The octahedral coordination of Cu confirms the +2 oxidation state of the metal. The Cu—N distances in the CuN_6 octahedron are in agreement with the structural data reported by Allmann *et al.* (1978).

The adaptation of the terpyridine rings to the octahedral geometry of CuN_6 polyhedra gives rise to distinct deformations of the ligand framework. Within the ligand rings C—C distances of 1.35–1.44 \AA and C—N bond distances of 1.32–1.35 \AA are found. They are within the range of the previously observed values (Maslen *et al.*, 1974; Allmann *et al.*, 1978) as are the inner bond angles of 120 (2°).

The $(\text{F}_6\text{P})^-$ ions also show octahedral coordination and are arranged along the z axis at approximately $z = 0, \frac{1}{4}, \frac{1}{2}, \frac{3}{4}$ between the $[\text{Cu}(\text{tpy})_2]^{2+}$ ions; this is in agreement with what has been reported elsewhere (Allmann *et al.*, 1978; Bernal, LaPlaca, Korp, Brunner & Herrmann, 1978).

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