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Bis(2,2′-dipyridylamine)dimethanolcopper(II) hexafluorosilicate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.032
wR factor = 0.074
Data-to-parameter ratio = 14.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[Cu(C_{10}H_9N_3)_2(CH_3OH)_2](SiF_6)$, was obtained in low yield in the reaction of dipyridylamine with $Cu(BF_4)_2$ using thermal conditions in a sealed pressure tube. The Cu-based cation lies at the centre of the triclinic unit cell, the hexafluorosilicate anions lying at the corners. Cations and anions, which are centrosymmetric, are held together by hydrogen bonds.

Comment

Hexafluorosilicate-containing coordination compounds can be prepared either starting from $M^{II}SiF_6$ salts (Subramanian & Zaworotko, 1995) or from solutions containing $M^{II}(BF_4)_2$, where gradual ligand-assisted BF_4^- decomposition followed by attack of the fluoride ions on the glass surface of the reaction vessel leads to the slow formation of hexafluorosilicate ions (van Koningsbruggen *et al.*, 1993). The title compound was prepared using the latter procedure by reaction, under thermal conditions in a sealed pressure tube, of dipyridylamine and $Cu(BF_4)_2$. An *ORTEPII* (Johnson, 1976) drawing of the ion pair of the title compound is depicted in Fig. 1. As shown in Fig. 2, the cationic Cu-based core lies at the centre of the triclinic unit cell, eight hexafluorosilicate dianions being at the corners of a distorted cube. The asymmetric unit contains one-half of the monometallic cation with the inversion centre located at the Cu atom. The copper(II) ion is six-coordinated by four N atoms from two dipyridylamine ligands [Cu—N = 2.0061 (17)–2.0165 (17) Å] and two O atoms from two methanol molecules [Cu—O(MeOH) = 2.4812 (17) Å]. The Cu^{II} centre is in a Jahn–Teller distorted octahedral environment. The Cu—N bond distances are

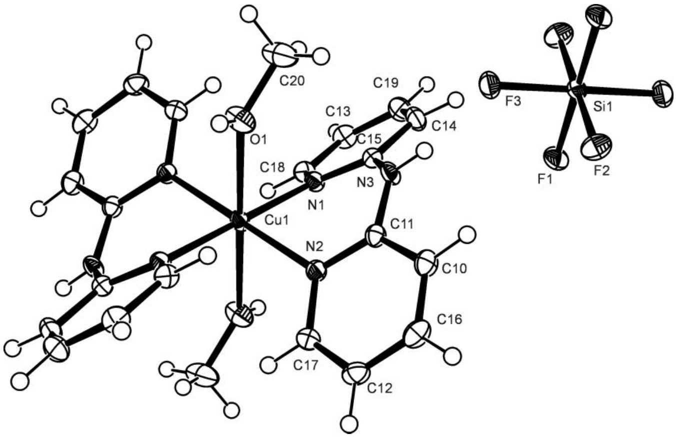
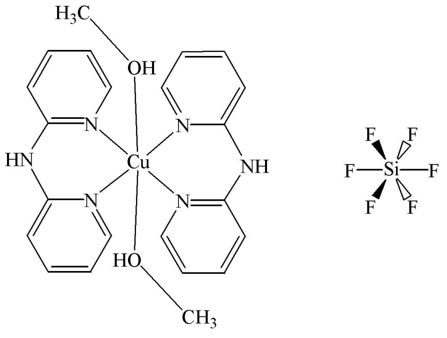


Figure 1
ORTEPII plot (Johnson, 1976) of the structure of the title compound (40% displacement ellipsoids). Unlabelled atoms of the cation are related to labelled atoms by (−*x*, −*y*, −*z*).

unexceptional, as well as the Cu—O distances for Cu—O elongated bonds. The dipyridylamine ligand is not planar, with a dihedral angle of 146.3 (2)° between the two rings. The structure is stabilized by numerous hydrogen bonds. As a main feature, SiF_6^{2-} is a connector for amine groups and MeOH molecules through a three-dimensional hydrogen-bonding network, as shown in Fig. 3. The $D\cdots A$ distances are 2.786 (2) Å for the solvent–F interactions and 2.726 (2) Å for the amine–F interactions.



(I)

Experimental

Copper(II) tetrafluoroborate (20 mg, 0.084 mmol) was added to an equimolar methanol solution (6 ml) of dipyridylamine. The resulting green limpid solution was allowed to stand at 373 K for 12 h in a sealed tube to form the side product [Cu(dipyridylamine)$_2$-(CH_3OH)$_2$](SiF_6) as prismatic light-yellow crystals. The crystals could be easily separated from the main product.

Crystal data

[$Cu(C_{10}H_9N_3)_2(CH_4O)_2$]($SiF_6$)
M_r = 612.12
Triclinic, $P\bar{1}$
a = 8.0593 (3) Å
b = 8.4334 (3) Å
c = 9.8099 (4) Å
α = 111.688 (2)°
β = 91.079 (2)°
γ = 99.634 (2)°
V = 608.44 (4) Å^3
Z = 1
D_x = 1.671 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2188 reflections
θ = 2.6–27.5°
μ = 1.03 mm^{-1}
T = 150 (2) K
Prism, yellow
0.25 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.783, T_{max} = 0.904
4671 measured reflections
2684 independent reflections
2327 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.074
S = 1.06
2684 reflections
181 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + 0.7099P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

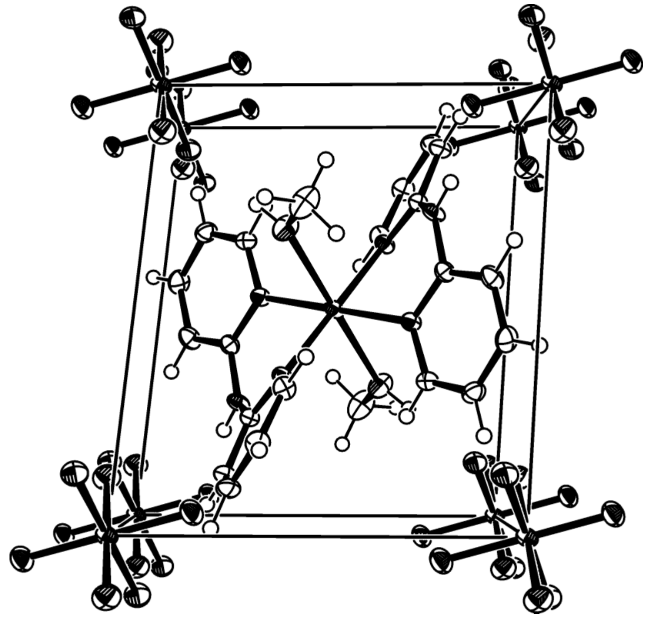


Figure 2
ORTEPII (Johnson, 1976) view of the unit cell contents of the title compound.

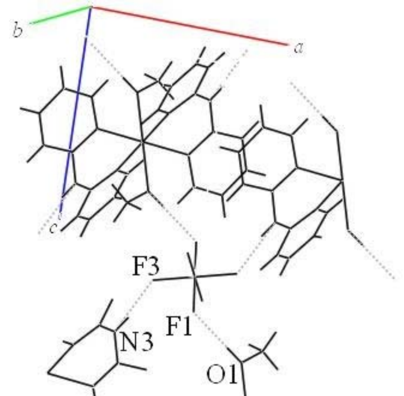


Figure 3
View of the hydrogen bonds (dotted lines) in the title compound [O1$\cdots$F1 = 2.786 (2) Å and N3$\cdots$F3 = 2.726 (2) Å].

Table 1
Selected geometric parameters (Å, °).

Cu1—N1	2.0061 (17)	Cu1—N2	2.0165 (17)
Cu1—O1	2.4812 (17)		
N1^i—Cu1—N1	180	N1—Cu1—N2	85.95 (7)
N1^i—Cu1—N2	94.05 (7)		

Symmetry code: (i) $-x+1, -y+1, -z+1$.

H atoms on C and O atoms were placed in geometrically calculated positions and refined using a riding model [C—H = 0.95 and 0.98 Å, O—H = 0.84 Å, and U_{iso}(H) = 1.5U_{eq}(C_{methyl},O), or 1.2U_{eq}(C) for other H atoms].

Data collection: *KappaCCD* (Nonius, 1998); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SIR92* (Giacov-

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azzo, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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