Table 2. *Bond lengths* (Å) *and selected bond angles* (°)

Cp denotes the cyclopentadienyl ring centroid.

Fe(1)—Fe(1^{i})	2·534 (1)	O(2)—C(2)	1·137 (5)
Fe(1)—C(1)	1·933 (5)	O(3)—C(9)	1·416 (6)
Fe(1)—C(1^{i})	1·897 (5)	C(3)—C(4)	1·418 (7)
Fe(1)—C(2)	1·759 (5)	C(3)—C(7)	1·407 (6)
Fe(1)—C(3)	2·128 (4)	C(4)—C(5)	1·388 (7)
Fe(1)—C(4)	2·119 (4)	C(5)—C(6)	1·406 (7)
Fe(1)—C(5)	2·119 (5)	C(6)—C(7)	1·420 (7)
Fe(1)—C(6)	2·097 (5)	C(7)—C(8)	1·499 (6)
Fe(1)—C(7)	2·144 (4)	C(8)—C(9)	1·497 (7)
O(1)—C(1)	1·183 (5)	Fe(1)···Cp	1·771 (4)
O(3)···O(3^{ii})	2·656 (4)		
Fe(1)—Fe(1^{i})—C(1)	48·0 (1)	C(4)—C(3)—C(7)	108·0 (4)
Fe(1)—Fe(1^{i})—C(1^{i})	49·2 (1)	C(3)—C(4)—C(5)	109·2 (4)
C(1)—Fe(1)—C(1^{i})	97·2 (2)	C(4)—C(5)—C(6)	107·0 (4)
C(1)—Fe(1)—C(2)	93·6 (2)	C(5)—C(6)—C(7)	109·5 (4)
C(1^{i})—Fe(1)—C(2)	90·8 (2)	C(3)—C(7)—C(6)	106·4 (4)
Fe(1)—C(1)—Fe(1^{i})	82·8 (2)	C(3)—C(7)—C(8)	125·5 (5)
Fe(1)—C(1)—O(1)	136·4 (4)	C(6)—C(7)—C(8)	128·1 (4)
Fe(1^{i})—C(1)—O(1)	140·8 (4)	C(7)—C(8)—C(9)	114·3 (4)
Fe(1)—C(2)—O(2)	177·7 (5)	O(3)—C(9)—C(8)	112·2 (5)

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

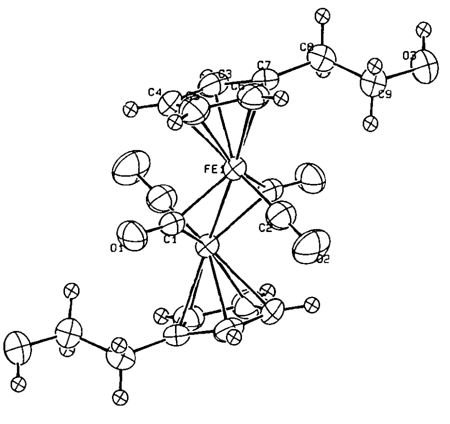

Fig. 1. The molecular structure of $Fe_2(CO)_4(C_5H_4CH_2CH_2OH)_2$ showing thermal ellipsoids and numbering of atoms.

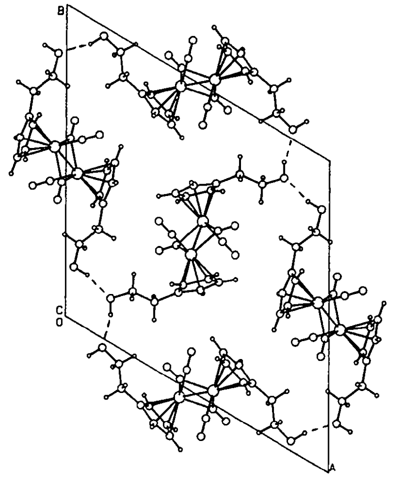

Fig. 2. Part of one hydrogen-bonded net viewed along **c**.

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Structure of Aquabis(2,2′-bipyrimidine-*N*,*N*′)copper(II) Perchlorate Dihydrate

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Abstract. $[Cu(C_8H_6N_4)_2(H_2O)](ClO_4)_2\cdot 2H_2O$, $M_r = 632.86$, monoclinic, $C2/c$, $a = 18.232\ (3)$, $b = 7.613\ (1)$, $c = 16.842\ (4)$ Å, $\beta = 96.27\ (2)°$, $V = 2323.6\ (8)$ Å^3, $Z = 4$, $D_x = 1.82$ g cm^{-3}, Zr-filtered Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 12.54$ cm^{-1}, $F(000) = 1284$, $T = 294\ (1)$ K, $R = 0.037$ for 1211 observed

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cu(1)	0	889 (1)	2500	34 (1)
Cl(1)	2508 (1)	120 (2)	3604 (1)	45 (1)
O(1)	0	−1729 (8)	2500	54 (3)
O(2)	3065 (5)	−1094 (11)	3808 (4)	136 (4)
O(3)	2741 (5)	1241 (10)	3005 (5)	137 (4)
O(4)	2357 (3)	1153 (7)	4275 (3)	62 (2)
O(5)	1845 (4)	−697 (11)	3271 (4)	116 (3)
O(6)	−436 (3)	−3756 (8)	3676 (3)	51 (2)
N(1)	−664 (3)	2661 (6)	1794 (3)	31 (2)
N(2)	−802 (3)	3824 (7)	482 (3)	39 (2)
N(3)	425 (3)	1873 (7)	170 (3)	38 (2)
N(4)	522 (2)	912 (6)	1520 (3)	31 (2)
C(1)	−1209 (3)	3677 (9)	1995 (4)	37 (2)
C(2)	−1568 (3)	4831 (9)	1447 (4)	42 (2)
C(3)	−1358 (4)	4838 (9)	681 (4)	43 (2)
C(4)	−470 (3)	2826 (7)	1053 (3)	30 (2)
C(5)	192 (3)	1815 (8)	889 (3)	30 (2)
C(6)	1033 (3)	939 (9)	80 (4)	42 (2)
C(7)	1405 (4)	−45 (9)	676 (4)	42 (2)
C(8)	1133 (3)	−22 (8)	1417 (4)	39 (2)

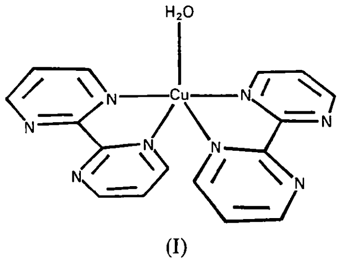
Table 2. *Bond distances* (Å) *and angles* (°)

Cu(1)—O(1)	1.993 (4)	Cu(1)—N(1)	2.094 (4)
Cu(1)—N(4)	1.993 (4)	Cl(1)—O(2)	1.388 (5)
Cl(1)—O(3)	1.421 (6)	Cl(1)—O(4)	1.428 (4)
Cl(1)—O(5)	1.419 (6)	N(1)—C(1)	1.332 (7)
N(1)—C(4)	1.340 (7)	N(2)—C(3)	1.345 (7)
N(2)—C(4)	1.320 (7)	N(3)—C(5)	1.327 (7)
N(3)—C(6)	1.339 (7)	N(4)—C(5)	1.351 (6)
N(4)—C(8)	1.349 (7)	C(1)—C(2)	1.386 (8)
C(2)—C(3)	1.385 (8)	C(4)—C(5)	1.483 (7)
C(6)—C(7)	1.371 (9)	C(7)—C(8)	1.392 (8)

O(1)—Cu(1)—N(1)	130.1 (1)	O(1)—Cu(1)—N(4)	90.5 (1)
N(1)—Cu(1)—N(4)	79.8 (1)	N(1)—Cu(1)—N(1a)	99.8 (1)
N(1)—Cu(1)—N(4a)	99.5 (1)	N(4)—Cu(1)—N(4a)	179.0 (1)
O(2)—Cl(1)—O(3)	107.9 (5)	O(2)—Cl(1)—O(4)	111.7 (3)
O(2)—Cl(1)—O(5)	112.0 (5)	O(3)—Cl(1)—O(4)	109.2 (4)
O(3)—Cl(1)—O(5)	106.9 (5)	O(4)—Cl(1)—O(5)	108.9 (3)
Cu(1)—N(1)—C(1)	129.1 (4)	Cu(1)—N(1)—C(4)	113.2 (4)
C(1)—N(1)—C(4)	117.6 (4)	C(3)—N(2)—C(4)	116.5 (5)
C(5)—N(3)—C(6)	115.9 (6)	Cu(1)—N(4)—C(5)	116.4 (3)
Cu(1)—N(4)—C(8)	125.3 (4)	C(5)—N(4)—C(8)	118.1 (4)
N(1)—C(1)—C(2)	120.7 (5)	C(1)—C(2)—C(3)	117.7 (5)
N(2)—C(3)—C(2)	121.5 (6)	N(1)—C(4)—N(2)	125.9 (5)
N(1)—C(4)—C(5)	115.3 (5)	N(2)—C(4)—C(5)	118.8 (5)
N(3)—C(5)—N(4)	125.1 (5)	N(3)—C(5)—C(4)	120.1 (5)
N(4)—C(5)—C(4)	114.8 (4)	N(3)—C(6)—C(7)	123.8 (5)
C(6)—C(7)—C(8)	116.9 (5)	N(4)—C(8)—C(7)	120.1 (6)

reflections. The title compound represents one of the first examples of a five-coordinate bis(bipyrimidine)-copper(II) complex. The cation is situated about a crystallographic twofold rotation axis which is coincident with the Cu—O bond. The coordination geometry about the metal center is approximately trigonal bipyramidal. Strong hydrogen bonding links the two water molecules of hydration to the coordinated aqua ligand; no other significant interactions were observed.

Experimental. Blue–green, parallelepiped crystal of the title compound (I), dimensions 0.20 × 0.20 × 0.30 mm, formed by reaction of stoichiometric amounts of copper(II) perchlorate and bipyrimidine in ethanol/water solution.



(I)

Picker diffractometer automated with *PCXTL* (Bennett, 1990), $\omega/2\theta$ scans of 2° $\min^{-1}$, $2\theta_{max} = 48°$, $h = -21$ to 21, $k = 0$ to 8, $l = 0$ to 18, 2340 measurements, 2086 unique ($R_{int} = 0.020$), 1211 observed [$I > 3\sigma(I)$]. Unit-cell dimensions determined by least-squares fit to settings of four symmetry-equivalent centering measurements for 12 unique reflections ($18 < 2\theta < 30°$), using a simplex optimization technique (Weinrach & Bennett, 1991). Empirical absorption correction, transmission factors 0.75–1.00; three standards monitored (002, 004, 204; ±1%), 44.8 h of X-ray exposure. Solved by Patterson method; full-matrix least-squares refinement on F, $R = 0.0370$, $wR = 0.0420$, $S = 1.50$, $(\Delta/\sigma)_{max} = 0.01$, 173 variables including positional and anisotropic thermal parameters for the non-H atoms. H atoms were located by difference Fourier techniques and included at optimized positions ($d_{O—H} = 0.99$; $d_{C—H} = 1.08$ Å) with fixed isotropic thermal parameters ($U = 0.06$ Å^2). Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2(F_o)$, $\sigma(F_o)$ based on counting statistics. Final difference Fourier peaks ranged from -0.25 to 0.25 e Å^{-3}. Computer programs for structure solution (Sheldrick, 1986), for refinement (Sheldrick, 1976); scattering factors from Cromer & Waber (1974), real and imaginary anomalous-dispersion corrections from Cromer (1974). Final atomic coordinates are given in Table 1,* and selected distances and angles in Table 2; the molecule is displayed in Fig. 1.

Related literature. A number of related five-coordinate complexes with bis(2,2′-bipyridyl)copper(II) bound to a monodentate ligand, L, have been structurally characterized: for $L = H_2O$, Cl^- see Harrison, Hathaway & Kennedy (1979); for $L = NCCH_3$ see Tyagi & Hathaway (1983); for $L =$

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters, as well as a summary of crystallographic details have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54167 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0503]

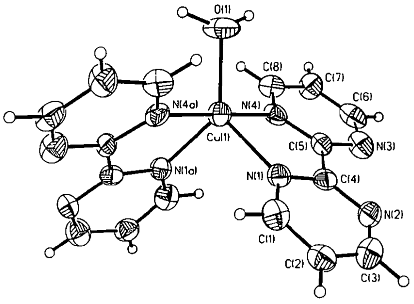

Fig. 1. Molecular structure and numbering scheme, thermal ellipsoids at 50% probability level. H atoms have arbitrary radius.

NCS^- see Tyagi & Hathaway (1981); for $L = NH_3$ see Stephens (1972); for $L = Cl^-$ (different counterions than above) see Harrison, Kennedy, Power, Sheahan & Hathaway (1981). The structure of aquabis(2,2′-bipyrimidine-*N*,*N*′)copper(II) hexafluorophosphate dihydrate has been reported recently (De Munno, Bruno, Julve & Romeo, 1990).

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Tris(4-fluoro-2,2′-bipyridyl)iron(II) Hexafluorophosphate

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Abstract. $[Fe(C_{10}H_7FN_2)_3][PF_6]_2$, $M_r = 868.31$, monoclinic, $P2_1/n$, $a = 10.422\ (1)$, $b = 17.294\ (2)$, $c = 18.581\ (3)$ Å, $\beta = 94.56\ (1)°$, $V = 3338.3\ (7)$ Å^3, $Z = 4$, $D_x = 1.728$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.659$ mm^{-1}, $F(000) = 1736$, $T = 294$ K, $R = 0.048$ for 3345 reflections with $I \geq 3\sigma(I)$. The solid-state results are consistent with the isomeric ratio of 3:1 *mer*:*fac* observed in solution, although only four of the eight possible isomers are permitted.

Experimental. The title compound was prepared by T. Nguyen (Janzen, Nguyen, Qu & Marat, 1988). A shiny black crystal of dimensions 0.4 × 0.2 × 0.2 mm was used for data collection. Data were collected on a Nicolet *R*3*m* diffractometer with the $\omega/2\theta$ scan mode, using graphite-monochromated Mo $K\alpha$ radiation; unit-cell parameters determined by least-squares refinement of 24 reflections in the 2θ range 25–39°; 4735 reflections ($-12 \leq h \leq 12$, $0 \leq k \leq 19$, $0 \leq l \leq 21$) collected in the range $2 \leq 2\theta \leq 50°$; data corrected for Lorentz–polarization effects, resulting in 4388 unique reflections; no absorption correction applied; $R_{\rm int} = 0.017$. Three standard reflections monitored during data collection showed no significant intensity variation after 48 h. Fe, P and F atoms were located on Patterson maps, other atoms were revealed in subsequent difference Fourier maps; reasonable temperature factors and occupancies for F(13), F(18), F(23), F(28) and F(38) could be adjusted (either with temperature factors fixed and occupancies refined, or with occupancies fixed and temperature factors refined) so as to achieve *mer*:*fac* ratios anywhere from 1:0 to 5:1; agreement indicators did not discriminate in favor of one particular

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