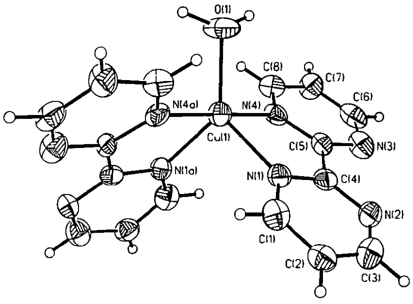

Fig. 1. Molecular structure and numbering scheme, thermal ellipsoids at 50% probability level. H atoms have arbitrary radius.

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).

Support of this work by the Office of Basic Energy Sciences, Department of Energy, is gratefully acknowledged.

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Acta Cryst. (1992). C**48**, 165–167

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

By Connie K. Cho and Anthony S. Secco*

Department of Chemistry, University of Manitoba, Winnipeg, Manitoba, Canada R3T 2N2

(*Received* 7 *May* 1991; *accepted* 16 *July* 1991)

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; $R_{int} = 0.017$. 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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* To whom all correspondence should be addressed.

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Table 1. *Final positional parameters* (*fractional* × 10^5 *for* Fe, × 10^4 *for other atoms*) *and isotropic thermal parameters* ($Å^2$ × 10^3) *with e.s.d.'s in parentheses*

U_{eq} is defined as one third of the trace of the diagonalized and anisotropic temperature-factor matrix.

	x	*y*	*z*	U_{eq}
Fe	85829 (6)	10895 (4)	22362 (4)	44
N(11)	7209 (4)	369 (2)	1864 (2)	49
N(12)	9598 (4)	447 (2)	1619 (2)	51
N(21)	8876 (4)	421 (2)	3095 (2)	53
N(22)	7463 (4)	1628 (2)	2879 (2)	50
N(31)	8313 (4)	1836 (2)	1437 (2)	47
N(32)	9998 (4)	1816 (2)	2522 (2)	51
C(11)	5977 (5)	362 (3)	2042 (3)	61
C(12)	5099 (5)	−181 (4)	1778 (3)	76
C(13)	5476 (6)	−716 (4)	1303 (4)	89
C(14)	6732 (7)	−725 (4)	1106 (3)	80
C(15)	7581 (5)	−176 (3)	1406 (3)	55
C(16)	8947 (5)	−131 (3)	1255 (3)	53
C(17)	9512 (6)	−620 (3)	796 (3)	65
C(18)	10796 (7)	−527 (4)	708 (3)	73
C(19)	11474 (5)	48 (4)	1051 (3)	72
C(110)	10860 (5)	530 (3)	1507 (3)	62
C(21)	9641 (5)	−210 (3)	3162 (3)	65
C(22)	9748 (6)	−662 (4)	3758 (4)	75
C(23)	9034 (7)	−470 (4)	4313 (4)	87
C(24)	8244 (6)	165 (4)	4279 (3)	75
C(25)	8189 (5)	609 (3)	3659 (3)	57
C(26)	7408 (5)	1311 (3)	3536 (3)	53
C(27)	6640 (6)	1618 (4)	4042 (3)	68
C(28)	5896 (6)	2249 (4)	3851 (4)	77
C(29)	5935 (6)	2581 (3)	3195 (4)	74
C(210)	6741 (5)	2252 (3)	2714 (3)	63
C(31)	7407 (5)	1812 (3)	879 (3)	55
C(32)	7318 (5)	2356 (4)	344 (3)	65
C(33)	8180 (6)	2953 (4)	355 (3)	68
C(34)	9118 (5)	2989 (3)	914 (3)	61
C(35)	9158 (5)	2437 (3)	1448 (3)	49
C(36)	10112 (5)	2424 (3)	2081 (3)	49
C(37)	11050 (5)	2985 (3)	2211 (3)	63
C(38)	11887 (5)	2887 (3)	2810 (3)	68
C(39)	11809 (5)	2287 (4)	3262 (3)	67
C(310)	10846 (5)	1759 (3)	3110 (3)	62
F(13)	4535 (8)	−1163 (6)	995 (6)	138
F(18)	11470 (7)	−941 (4)	291 (4)	97
F(23)	9251 (12)	−924 (7)	4915 (7)	176
F(28)	5034 (5)	2548 (3)	4270 (3)	63
F(38)	12835 (4)	3420 (2)	2940 (3)	117
P(1)	3625 (1)	174 (1)	3754 (1)	64
F(1*a*)	2862 (4)	−195 (3)	3074 (2)	118
F(1*b*)	2323 (3)	524 (2)	4014 (2)	94
F(1*c*)	3415 (4)	−581 (2)	4204 (2)	99
F(1*d*)	4927 (3)	−180 (2)	3502 (2)	99
F(1*e*)	4379 (4)	548 (2)	4433 (2)	109
F(1*f*)	3861 (4)	929 (2)	3299 (3)	111
P(2)	3519 (2)	2090 (1)	759 (1)	74
F(2*a*)	2273 (4)	1766 (3)	327 (3)	134
F(2*b*)	2827 (4)	2872 (3)	878 (3)	137
F(2*c*)	4786 (4)	2408 (3)	1184 (3)	116
F(2*d*)	4215 (5)	1306 (3)	634 (3)	134
F(2*e*)	3049 (4)	1759 (3)	1482 (3)	129
F(2*f*)	3992 (5)	2395 (3)	39 (3)	145

ratio; given the wide range of thermal parameters displayed in the structure and the fact that all F atoms experienced different environments, a uniform temperature factor for each pair of disordered F atoms could not be assumed; in the absence of any internal evidence as to the occupancies of the disordered F atoms, the solution ratio of 3:1 (*mer*:*fac*) was chosen and F(13), F(18), F(23) and F(28) were assigned half occupancies; F(38) was assigned full occupancy as no excess electron density was observed around C(33). All atoms were initially refined isotropically by full-matrix least-squares refinement using 3345 reflections with $I \geq 3\sigma(I)$; all

Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

Fe—N(11)	1.981 (4)	C(23)—C(24)	1.371 (9)
Fe—N(12)	1.965 (4)	C(23)—F(23)	1.371 (12)
Fe—N(21)	1.975 (4)	C(24)—C(25)	1.381 (8)
Fe—N(22)	1.969 (4)	C(25)—C(26)	1.470 (8)
Fe—N(31)	1.971 (4)	C(26)—C(27)	1.388 (7)
Fe—N(32)	1.978 (4)	C(27)—C(28)	1.368 (9)
N(11)—C(11)	1.351 (6)	C(28)—C(29)	1.352 (9)
N(11)—C(15)	1.347 (6)	C(28)—F(28)	1.339 (8)
N(12)—C(16)	1.359 (6)	C(29)—C(210)	1.395 (8)
N(12)—C(110)	1.355 (6)	C(31)—C(32)	1.367 (7)
N(21)—C(21)	1.351 (7)	C(32)—C(33)	1.368 (8)
N(21)—C(25)	1.355 (6)	C(33)—C(34)	1.370 (8)
N(22)—C(26)	1.343 (6)	C(34)—C(35)	1.376 (7)
N(22)—C(210)	1.337 (6)	C(35)—C(36)	1.478 (7)
N(31)—C(31)	1.346 (6)	C(36)—C(37)	1.385 (7)
N(31)—C(35)	1.361 (6)	C(37)—C(38)	1.368 (8)
N(32)—C(36)	1.345 (6)	C(38)—C(39)	1.341 (8)
N(32)—C(310)	1.352 (6)	C(38)—F(38)	1.359 (6)
C(11)—C(12)	1.373 (8)	C(39)—C(310)	1.369 (7)
C(12)—C(13)	1.358 (9)	P(1)—F(1*a*)	1.574 (4)
C(13)—C(14)	1.387 (9)	P(1)—F(1*b*)	1.595 (4)
C(13)—F(13)	1.341 (9)	P(1)—F(1*c*)	1.575 (4)
C(14)—C(15)	1.385 (8)	P(1)—F(1*d*)	1.592 (4)
C(15)—C(16)	1.474 (7)	P(1)—F(1*e*)	1.571 (4)
C(16)—C(17)	1.368 (7)	P(1)—F(1*f*)	1.586 (4)
C(17)—C(18)	1.370 (8)	P(2)—F(2*a*)	1.575 (4)
C(18)—C(19)	1.351 (8)	P(2)—F(2*b*)	1.556 (4)
C(18)—F(18)	1.301 (8)	P(2)—F(2*c*)	1.581 (4)
C(19)—C(110)	1.381 (8)	P(2)—F(2*d*)	1.565 (4)
C(21)—C(22)	1.353 (8)	P(2)—F(2*e*)	1.573 (5)
C(22)—C(23)	1.360 (9)	P(2)—F(2*f*)	1.555 (5)

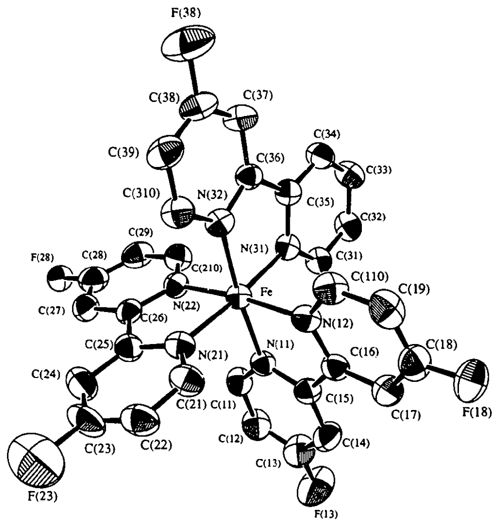

Fig. 1. *ORTEP* plot (Johnson, 1976) of the title cation with ellipsoids at 40% probability level. H atoms and counterions are omitted for clarity.

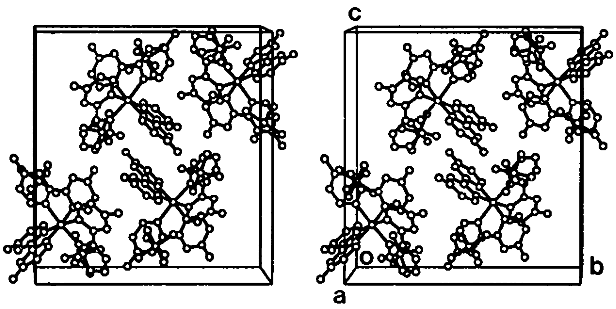

Fig. 2. Packing diagram of the unit cell viewed down the *a* axis (into page).

H atoms were inserted in calculated positions and assigned isotropic temperature factors equal to those of their attached atoms plus 10%. After the final cycle of refinement $R = 0.065$ and $wR = 0.055$ for all 4388 reflections while $R = 0.048$ and $wR = 0.055$ for the 3345 observed reflections [$w = A + BF_o + CF_o^2 + DF_o^3$, where $A = 4.9106$, $B = -0.05777$, $C = -0.000178$, $D = 0.000017$; function minimized, $w(|F_o| - k|F_c|)^2$]; goodness of fit $S = 0.805$; maximum $\Delta/\sigma = 0.002$. The final difference Fourier map was essentially featureless with maximum and minimum residuals of 0.547 and -0.280 e Å^{-3}, respectively. Atomic scattering factors for non-H atoms were from Cromer & Mann (1968) and from Stewart, Davidson & Simpson (1965) for H atoms.* Final atomic coordinates and thermal factors are given in Table 1, bond lengths in Table 2.† The structure of the title cation (without counterions) is shown in Fig. 1, and the entire unit-cell contents are shown in Fig. 2.

Related literature. The title compound was synthesized as part of a study on ML_n compounds with trigonal bipyramidal and octahedral symmetry (Marat & Janzen, 1977, 1980; Secco, Alam, Blackburn & Janzen, 1986, and references therein). The 3:1 *mer:fac* isomer ratio observed in ^{19}F NMR solution studies (Janzen, Nguyen, Qu & Marat, 1988) could also exist in the solid state; however, intermolecular contacts [< 3.2 Å between C(33) and neighboring atoms] would not allow an F atom to occupy the area around C(33), permitting only four of the eight possible combinations. Bond distances and bond angles (deposited) were generally normal, except for an unusually short C(18)—F(18) bond.

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* Computation was carried out on the University of Manitoba Computer Services Department's Amdahl 5870 mainframe computer using locally written programs for processing and modified versions of the following programs for structure solution, refinement and calculations: *FORDAP* (A. Zalkin, unpublished); *ORFLS* (Busing, Martin & Levy, 1962); *ORFFE* (Busing, Martin & Levy, 1964); *ORTEP*II (Johnson, 1976).

† Lists of bond angles, anisotropic thermal parameters, H-atom positional and thermal parameters, bond distances and angles involving H atoms, torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54544 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BR0002]

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Acta Cryst. (1992). C**48**, 167–169

Structure of *trans*-Diaquabis[(2-oxopyrrolidin-1-yl)acetamide]copper(II) Perchlorate

By X. Delacruz, A. Martínez-Balbás, J. Tormo and N. Verdaguer

Unitat de Química Macromolecular, Escola Tècnica Superior d'Enginyers Industrials, Diagonal 647, 08028 Barcelona, Spain

(*Received* 6 *July* 1990; *accepted* 18 *July* 1991)

Abstract. $[Cu(C_6H_{10}N_2O_2)_2(H_2O)_2](ClO_4)_2$, $M_r = 582.78$, monoclinic, $P2_1/c$, $a = 7.231$ (10), $b = 10.633$ (3), $c = 15.767$ (3) Å, $\beta = 103.94$ (5)°, $V = 1176.57$ Å^3, $Z = 2$, $D_x = 1.645$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 12.08$ mm^{-1}, $F(000) = 598$, room temperature, $R = 0.067$ for 1561 observed reflections. The only important structural difference between the oxopyrrolidine ligand in this Cu complex and the free molecule [Pritzkow (1983). *Cryst. Res. Technol.* **18**(8), 1069–1074] is in the orientation of the acetamide group relative to the pyrrolidine ring. In the centrosymmetric cation the CuO_6 octahedron is tetragonally distorted, with long Cu—O(H_2O) distances of 2.450 (5) Å. The perchlorate anions are disordered.

Experimental. Crystals were grown by mixing equal volumes of aqueous solutions of 0.5 *M* 2-oxo-1-

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