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Tetraaqua(5-fluorouracil-1-acetato-*O*)copper(II) tetrahydrate

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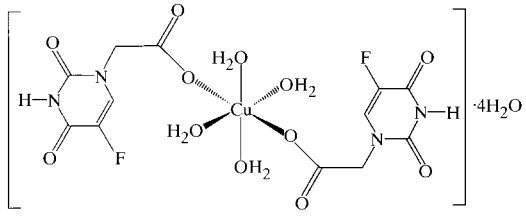
In the title complex, $[Cu(C_6H_4FN_2O_4)_2(H_2O)_4]\cdot 4H_2O$, the Cu atom is located in the centre of a distorted octahedral geometry. The coordination atoms are six O atoms provided by two carboxylate groups [coordinated in a monodentate mode, with Cu—O = 1.9551 (10) Å] and four water molecules [Cu—O = 1.9241 (13) and 2.5771 (14) Å]. In addition, one intramolecular hydrogen bond and ten intermolecular hydrogen bonds make up a three-dimensional network.

Comment

It was reported that some metal complexes with 5-fluorouracil-1-acetic acid (5-FUAA) have antitumor activity (Qin *et al.*, 1989; Wang *et al.*, 1993). However, the assignments of coordination mode of 5-FUAA are not easy due to its several potential donor atoms. We report here the crystal structure of one of these complexes. The complex, tetraaqua(5-fluorouracil-1-acetato-*O*)copper(II) tetrahydrate, (I), has an inversion centre at the Cu atom which is located in the centre of a distorted octahedral geometry. The coordination atoms are six



(I)

O atoms provided by two carboxylate groups (coordinated in a monodentate mode) and four water molecules. The Cu—O(carboxylate) bond length is 1.9551 (10) Å, while the Cu—O(water) bond lengths are 2.5771 (14) and 1.9241 (13) Å. In the carboxylate group, the two C—O bond lengths are almost equal [1.2519 (17) and 1.2409 (17) Å] and the O—C—O angle is 126.84 (13)°, which indicate that C—O has partial double-bond character (Leandro *et al.*, 1997). With four water molecules in the asymmetric unit, one intramolecular hydrogen bond and ten intermolecular hydrogen bonds are formed (Table 2), and make up a three-dimensional network.

Experimental

5-FUAA was prepared according to the method of Tada (1975). The title complex was synthesized as follows: a water–ethanol (1:1) solution of 5-FUAA (1 mmol) was added to an aqueous ethanol (1:1) solution of $Cu(OAc)_2\cdot 2H_2O$ (0.05 mmol) with stirring at reflux temperature. After the mixture had been stirred continuously for 8 h, a blue precipitate was obtained; this was filtered off, washed with 50% ethanol and dried *in vacuo*. Crystals suitable for diffraction studies were obtained from the mother solution by slow evaporation.

Crystal data

$[Cu(C_6H_4FN_2O_4)_2(H_2O)_4]\cdot 4H_2O$	D_x = 1.745 Mg m^{-3}
M_r = 581.89	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 150 reflections
a = 8.304 (1) Å	θ = 16.06–16.94°
b = 12.044 (2) Å	μ = 1.090 mm^{-1}
c = 11.082 (2) Å	T = 293 (2) K
β = 92.47 (1)°	Prismatic, blue
V = 1107.3 (3) Å^3	0.30 × 0.15 × 0.10 mm
Z = 2	

Data collection

Enraf-Nonius CAD-4 diffractometer	1838 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	θ_{max} = 24.96°
Absorption correction: ψ scan (*TEXSAN*; Molecular Structure Corporation, 1989)	$h = -9 \rightarrow 9$
T_{min} = 0.851, T_{max} = 0.897	$k = 0 \rightarrow 14$
1941 measured reflections	$l = 0 \rightarrow 13$
1941 independent reflections	3 standard reflections every 400 reflections
	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0200P)^2 + 0.4440P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.020	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.056	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
S = 1.029	$\Delta\rho_{min}$ = −0.34 e Å^{-3}
1941 reflections	
160 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cu—O2	1.9241 (13)	N1—C6	1.374 (2)
Cu—O2^i	1.9241 (13)	N1—C3	1.378 (2)
Cu—O3^i	1.9551 (10)	N1—C2	1.4580 (18)
Cu—O3	1.9551 (10)	N2—C4	1.373 (2)
Cu—O1	2.5771 (14)	N2—C3	1.3847 (19)
O3—C1	1.2519 (17)	F—C5	1.3391 (18)
O4—C1	1.2409 (17)	C1—C2	1.5295 (19)
O5—C3	1.2144 (18)	C4—C5	1.435 (2)
O6—C4	1.229 (2)	C5—C6	1.323 (2)
O2—Cu—O2^i	180.00 (4)	C6—N1—C2	119.33 (12)
O2—Cu—O3^i	90.35 (5)	C3—N1—C2	119.02 (12)
O2^i—Cu—O3^i	89.65 (5)	C4—N2—C3	127.30 (13)
O2—Cu—O3	89.65 (5)	O4—C1—O3	126.84 (13)
O2^i—Cu—O3	90.35 (5)	O4—C1—C2	116.26 (12)
O3^i—Cu—O3	180.00 (8)	O3—C1—C2	116.90 (12)
O2—Cu—O1	89.95 (5)	N1—C2—C1	112.85 (11)
O2^i—Cu—O1	90.05 (5)	O5—C3—N1	123.29 (13)
O3^i—Cu—O1	96.58 (4)	O5—C3—N2	121.89 (13)
O3—Cu—O1	83.42 (4)	N1—C3—N2	114.81 (12)
C1—O3—Cu	126.21 (9)	O6—C4—N2	121.38 (14)
C6—N1—C3	121.16 (12)	O6—C4—C5	125.38 (15)

N2—C4—C5	113.23 (14)	F—C5—C4	116.67 (14)
C6—C5—F	121.79 (15)	C5—C6—N1	121.87 (14)
C6—C5—C4	121.54 (14)		
O2—Cu—O3—C1	102.87 (13)	C2—N1—C3—N2	175.26 (11)
$O2^{i}$—Cu—O3—C1	−77.13 (13)	C4—N2—C3—O5	178.02 (15)
$O3^{i}$—Cu—O3—C1	−116.00 (10)	C4—N2—C3—N1	−3.3 (2)
O1—Cu—O3—C1	−167.14 (12)	C3—N2—C4—O6	−178.49 (15)
Cu—O3—C1—O4	−2.8 (2)	C3—N2—C4—C5	2.0 (2)
Cu—O3—C1—C2	177.08 (9)	O6—C4—C5—C6	179.79 (17)
C6—N1—C2—C1	80.72 (16)	N2—C4—C5—C6	−0.7 (2)
C3—N1—C2—C1	−91.41 (15)	O6—C4—C5—F	−0.1 (3)
O4—C1—C2—N1	−178.05 (13)	N2—C4—C5—F	179.47 (15)
O3—C1—C2—N1	2.05 (18)	F—C5—C6—N1	−179.18 (15)
C6—N1—C3—O5	−178.02 (14)	C4—C5—C6—N1	1.0 (3)
C2—N1—C3—O5	−6.0 (2)	C3—N1—C6—C5	−2.4 (2)
C6—N1—C3—N2	3.28 (19)	C2—N1—C6—C5	−174.35 (15)

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

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7A···O5	0.732 (14)	2.319 (18)	2.9228 (16)	141 (2)
O1—H1A···$O4^{i}$	0.734 (14)	2.075 (18)	2.6971 (17)	143 (3)
O1—H1B···$O6^{ii}$	0.675 (14)	2.232 (16)	2.8753 (17)	160 (3)
O2—H2A···F^{iii}	0.705 (14)	2.59 (2)	3.0096 (19)	120 (2)
O2—H2B···$O7^{i}$	0.725 (14)	2.26 (2)	2.7128 (17)	121 (2)
N2—H2···$O7^{iv}$	0.86	1.97	2.8300 (17)	176
O7—H7A···$O8^{v}$	0.732 (14)	2.540 (17)	3.0402 (18)	127.4 (19)
O7—H7B···$O4^{vi}$	0.698 (14)	1.974 (14)	2.6706 (16)	176 (2)
O8—H8A···$O6^{ii}$	0.718 (14)	2.184 (19)	2.8350 (19)	151 (3)
O8—H8A···$O7^{vii}$	0.718 (14)	2.65 (2)	3.0402 (18)	116 (2)
O8—H8B···$O1^{vii}$	0.738 (14)	2.20 (2)	2.783 (2)	137 (3)

Symmetry codes: (i) $1-x, -y, -z$; (ii) $1-x, -y, 1-z$; (iii) $x, -\frac{1}{2}-y, z-\frac{1}{2}$; (iv) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (v) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (vi) $2-x, -y, -z$; (vii) $1-x, y-\frac{1}{2}, \frac{1}{2}-z$.

All water H atoms were refined and those attached to C and N atoms were treated as riding atoms (C—H = 0.93 and 0.97 Å; N—H = 0.86 Å)

Data collection: *CAD*-4/*PC* (Enraf–Nonius, 1989); cell refinement: *CAD*-4/*PC*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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