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trans-Tetraaquabis(5-fluorouracil-1-acetato-*O*)zinc(II) tetrahydrate

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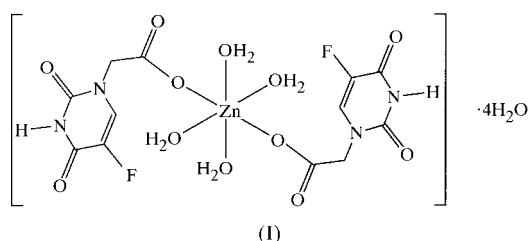
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In the title complex, $[\text{Zn}(\text{C}_6\text{H}_4\text{FN}_2\text{O}_4)_2(\text{H}_2\text{O})_4]\cdot 4\text{H}_2\text{O}$, the Zn atom is located at a center of symmetry and has a distorted octahedral geometry, with Zn—O distances in the range 2.052 (2)–2.181 (2) Å. Hydrogen bonds involving the water molecules make up a three-dimensional network.

Comment

5-Fluorouracil is a normal antitumor drug used in clinics and it can be used to treat breast cancer, gastric carcinoma and bladder cancer. However, the toxic side effects, such as marrow inhibition, limits its wider applicability. A search for highly effective and low toxicity novel antitumor drugs is an active research field and an urgent task for scientists. In order to reduce the side effects, many derivatives of 5-fluorouracil have been synthesized and some compounds have better biological activity. 5-Fluoro-1-uracilacetic acid (5-FUAA) is a member of this family. Its metal complexes had been reported.



However, assignment of the coordination mode are very confused due to the fact that it contains several potential donor atoms; it is therefore necessary to clarify the structures of these complexes.

5-FUAA was synthesized according to literature methods (Tada, 1975) and the title complex, (I), was synthesized by a new method. The physicochemical properties were characterized by elemental analysis, TG-DTA, IR and X-ray diffraction.

The antitumor activity against K562 and L1210 was investigated. In the title complex, the Zn atom is located in a center of symmetry and has a distorted octahedral geometry with Zn—O distances in the range 2.052 (2)–2.181 (2) Å. Because there are four coordinated water and four crystal water molecules, three intramolecular hydrogen bonds and nine intermolecular hydrogen bonds are formed (Table 2). These hydrogen bonds make up of a network, which may be the reason for the stability of the crystal.

Experimental

The ligand 5-fluorouracil-1-acetic acid (5-FUAA) was prepared according to literature methods (Tada, 1975). The complex was synthesized as follows: a water–ethanol (1:1) solution of 5-FUAA (1 mmol, 0.1891 g) was added to an aqueous ethanol (1:1) solution of $\text{Zn}(\text{OAc})_2\cdot 2\text{H}_2\text{O}$ (0.05 mmol, 0.1096 g) with stirring. The mixture was stirred continuously for 8 h and the white precipitate obtained was suction-filtrated and washed with 50% ethanol and finally dried *in vacuo*. Colorless crystals suitable for diffraction studies were obtained from an aqueous ethanol solution by slow evaporation.

Crystal data

$[\text{Zn}(\text{C}_6\text{H}_4\text{FN}_2\text{O}_4)_2(\text{H}_2\text{O})_4]\cdot 4\text{H}_2\text{O}$	$D_x = 1.729 \text{ Mg m}^{-3}$
$M_r = 583.72$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
$a = 8.571 (2) \text{ \AA}$	$\theta = 16.12\text{--}16.92^\circ$
$b = 12.102 (2) \text{ \AA}$	$\mu = 1.198 \text{ mm}^{-1}$
$c = 10.814 (2) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 91.53 (2)^\circ$	Prismatic, colorless
$V = 1121.3 (4) \text{ \AA}^3$	$0.35 \times 0.15 \times 0.10 \text{ mm}$
$Z = 2$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.030$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 24.97^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = 0 \rightarrow 10$
$T_{\text{min}} = 0.856$, $T_{\text{max}} = 0.887$	$k = 0 \rightarrow 14$
1986 measured reflections	$l = -12 \rightarrow 12$
1971 independent reflections	5 standard reflections every 390 reflections
1804 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0200P)^2 + 0.3500P]$
$R[F^2 > 2\sigma(F^2)] = 0.030$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.110$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.884$	$\Delta\rho_{\text{max}} = 0.55 \text{ e \AA}^{-3}$
1971 reflections	$\Delta\rho_{\text{min}} = -0.48 \text{ e \AA}^{-3}$
160 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

Zn—O1	2.0521 (17)	Zn—O2	2.1806 (16)
Zn—O3	2.0668 (15)		
O1—Zn—O3 ⁱ	89.42 (7)	O3 ⁱ —Zn—O2 ⁱ	86.58 (6)
O1 ⁱ —Zn—O3 ⁱ	90.58 (7)	O3—Zn—O2 ⁱ	93.42 (6)
O1—Zn—O3	90.58 (7)	O1—Zn—O2	90.88 (7)
O1 ⁱ —Zn—O3	89.42 (7)	O1 ⁱ —Zn—O2	89.12 (7)
O1—Zn—O2 ⁱ	89.12 (7)	O3 ⁱ —Zn—O2	93.42 (6)
O1 ⁱ —Zn—O2 ⁱ	90.88 (7)	O3—Zn—O2	86.58 (6)

Symmetry code: (i) 1 − x, −y, −z.

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N2—H2...O8	0.90	1.92	2.818 (3)	176
O7—H7A...O6	0.96	1.89	2.847 (3)	180
O8—H8A...O7	0.96	2.21	3.070 (3)	148
O1—H1A...F ⁱ	0.96	2.45	2.999 (3)	116
O1—H1A...O7 ⁱⁱ	0.96	2.49	3.288 (3)	140
O1—H1B...O8 ⁱⁱ	0.96	2.03	2.747 (2)	130
O2—H2A...O7 ⁱⁱⁱ	0.96	2.29	2.879 (3)	119
O2—H2B...O4 ^{iv}	0.96	1.97	2.645 (2)	125
O7—H7B...O1 ^v	0.96	1.83	2.681 (2)	146
O7—H7B...F ^{vi}	0.96	2.55	3.232 (3)	128
O8—H8A...O5 ^{vii}	0.96	2.40	2.890 (2)	111
O8—H8B...O4 ^{viii}	0.96	1.98	2.651 (2)	125

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iii) $x, -\frac{1}{2} - y, z - \frac{1}{2}$; (iv) $1 - x, -y, -z$; (v) $1 - x, -y, 1 - z$; (vi) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (vii) $x, -\frac{1}{2} - y, \frac{1}{2} + z$; (viii) $2 - x, y - \frac{1}{2}, \frac{1}{2} - z$.

The positions of all H atoms were fixed geometrically and distances to H atoms were set by the refinement program (O—H/C—H = 0.96 Å and N—H = 0.90 Å).

Data collection: *CAD-4 SDP/VAX* (Enraf–Nonius, 1989); cell refinement: *CAD-4 SDP/VAX*; data reduction: *TEXSAN* (Molecular

Structure Corporation, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

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