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Bis(4,4′-bipyridin-1-ium) bis(5-fluorouracil-1-acetate) monohydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.076
wR factor = 0.189
Data-to-parameter ratio = 11.6

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

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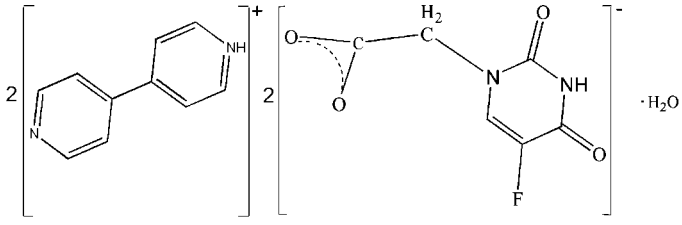
The chemical structural unit of the title compound [systematic name: bis(4,4′-bipyridin-1-ium) bis(5-fluoro-1,2,3,6-tetrahydro-2,6-dioxopyrimidine-3-acetate) monohydrate], $2C_{10}H_9N_2^+\cdot 2C_6H_4FN_2O_4^-\cdot H_2O$, comprises two ion pairs and one water molecule, which lies on a twofold rotation axis. O—H···O, N—H···O and N—H···N hydrogen-bond interactions connect the cations, anions and water molecules to produce a ribbon-like double-chain along the [101] direction. The hydrogen-bonding pattern can be described in graph-set notation as $R_6^8(24)C_2^2(16)$.

Comment

The 5-fluorouracil-1-acetate anion is an excellent candidate for the development of supramolecular motifs in crystals, as it possesses not only acceptor atoms (carboxylate O and carbonyl O) but also a donor atom (the uracil N). 4,4′-Bipyridine is a basic amine spacer which readily forms a monocation or dication, where the N—H bonds are generally active in hydrogen-bond formation (Zhu *et al.*, 2003). In order to better understand the behavior of proton transfer and hydrogen-bond motifs between 5-fluorouracil-1-acetic acid and 4,4′-bipyridine molecules, the synthesis and crystal structure of the title compound, (I), have been investigated.



(I)

The chemical structural unit of (I) comprises two 5-fluorouracil-1-acetate anions, two 4,4′-bipyridin-1-ium monocations and one water molecule, which lies on a twofold rotation axis. The bond distances and angles of the 5-fluorouracil-1-acetate anion in (I) are unexceptional and compare well with the coordinated 5-fluorouracil-1-acetate anion in $Ni(C_6H_4N_2O_4F)_2(C_7H_6N_2)_2(H_2O)_2$ (Hu & Wang, 2005) (Table 1 and Fig. 1). The identification of the protonated and unprotonated rings of the 4,4′-bipyridin-1-ium monocation can be confirmed by the C—C and C—N distances [1.368 (5)–1.392 (4) Å and 1.314 (4)–1.331 (4) Å, respectively], which are between single and double bonds (Table 1 and Fig. 1). The two rings are linked by a single bond [C9—C12 = 1.481 (5) Å], with the property of rotation, and are not in the same plane, with a dihedral angle 8.7 (2)°. Moreover, O5—H5···O2, O5—

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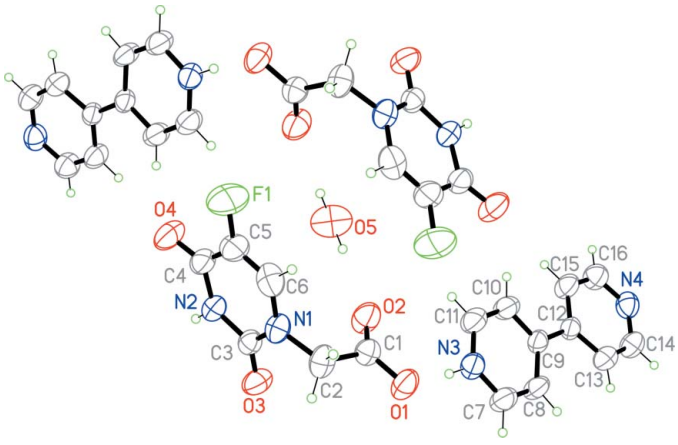

Figure 1
The chemical structural unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code for unlabeled atoms: $2-x, y, \frac{3}{2}-z$.]

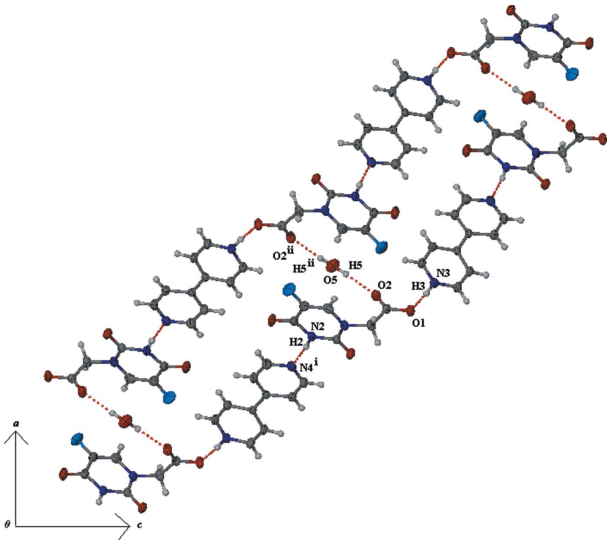

Figure 2
The ribbon-like double chain in (I) along the [101] direction, formed by O—H···O, N—H···O and N—H···N hydrogen-bond interactions, which are shown as dashed lines. [Symmetry codes: (i) $1+x, -y, \frac{1}{2}+z$; (ii) $2-x, y, \frac{3}{2}-z$.]

H5^{ii}···O2^{ii}, N3—H3···O1 and N2—H2···N4^{i} [symmetry codes: (i) $1+x, -y, \frac{1}{2}+z$; (ii) $2-x, y, \frac{3}{2}-z$] hydrogen-bond interactions connect the above monocations, anions and water molecules to produce a ribbon-like double chain along the [101] direction (Table 2 and Fig. 2). The hydrogen bonding pattern, as shown in Fig. 2, can be described in graph-set notation (Etter, 1990; Grell *et al.*, 2000) as $R_6^8(24)C_2^2(16)$.

Experimental

5-Fluorouracil-1-acetic acid (1 mmol, 0.19 g) was dissolved in a mixed solvent of water (5 ml) and dimethylformamide (5 ml). The solution was added dropwise to a stirred ethanol solution (10 ml) of 4,4′-bipyridine (1 mmol, 0.16 g). The resulting solution was filtered and allowed to evaporate slowly at room temperature. After three weeks, colorless crystals of (I) appeared.

Crystal data

$2C_{10}H_9N_2^+\cdot 2C_6H_4FN_2O_4^-\cdot H_2O$
$M_r = 706.62$
Monoclinic, $P2/c$
$a = 14.2530\ (15)$ Å
$b = 4.9385\ (5)$ Å
$c = 25.868\ (2)$ Å
$\beta = 119.826\ (2)^\circ$
$V = 1579.6\ (3)$ Å^3
$Z = 2$
$D_x = 1.486$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1246 reflections
$\theta = 2.8$–24.1°
$\mu = 0.12$ mm^{-1}
$T = 298\ (2)$ K
Block, colorless
0.28 × 0.22 × 0.10 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.971$, $T_{max} = 0.990$
7788 measured reflections
2783 independent reflections
2156 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 25.0^\circ$
$h = -10 \rightarrow 16$
$k = -5 \rightarrow 5$
$l = -30 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.076$
$wR(F^2) = 0.189$
$S = 1.17$
2783 reflections
240 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0742P)^2 + 0.8989P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

F1—C5	1.338 (4)	N1—C2	1.455 (4)
O1—C1	1.280 (4)	N2—C4	1.371 (4)
O2—C1	1.209 (4)	N2—C3	1.371 (4)
O3—C3	1.219 (4)	N3—C11	1.314 (4)
O4—C4	1.229 (4)	N3—C7	1.325 (4)
N1—C3	1.371 (4)	N4—C14	1.323 (5)
N1—C6	1.374 (4)	N4—C16	1.331 (4)
C3—N1—C6	121.0 (3)	N2—C3—N1	115.3 (3)
C3—N1—C2	117.8 (3)	O4—C4—N2	121.7 (4)
C6—N1—C2	120.3 (3)	O4—C4—C5	125.7 (4)
C4—N2—C3	127.6 (3)	N2—C4—C5	112.6 (3)
C11—N3—C7	119.3 (3)	C6—C5—F1	120.3 (4)
C14—N4—C16	116.3 (3)	F1—C5—C4	117.3 (4)
O2—C1—O1	125.9 (3)	C5—C6—N1	121.1 (4)
O2—C1—C2	120.7 (3)	N3—C7—C8	121.9 (3)
O1—C1—C2	113.5 (3)	N3—C11—C10	121.6 (3)
N1—C2—C1	112.4 (3)	N4—C14—C13	123.9 (3)
O3—C3—N2	122.1 (3)	N4—C16—C15	123.6 (4)
O3—C3—N1	122.6 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O2	0.90 (2)	1.95 (3)	2.821 (3)	162 (5)
N2—H2···N4^{i}	0.88 (2)	1.96 (2)	2.836 (4)	174 (3)
N3—H3···O1	0.91 (2)	1.65 (2)	2.564 (4)	176 (3)

Symmetry code: (i) $x+1, -y, z+\frac{1}{2}$.

H atoms of the water molecule and N atoms were located in difference density maps and refined with O—H and N—H distances restrained to 0.82 (2) and 0.86 (2) Å, respectively. The other H atoms were positioned geometrically and allowed to ride on their parent

atoms at distances of Csp^2—H = 0.93 Å and Csp^3–H = 0.97 Å. For all H atoms, U_{iso}(H) = 1.2U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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