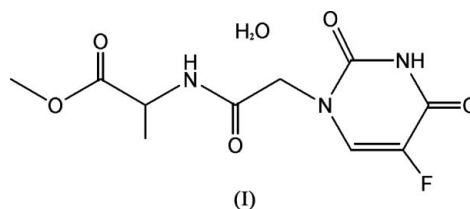


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Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.043
 wR factor = 0.107
Data-to-parameter ratio = 7.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Methyl (2*S*)-2-[2-(5-fluoro-2,4-dioxo-
1,2,3,4-tetrahydropyrimidin-1-yl)acetamido]-
propionate monohydrateIn the title compound, $\text{C}_{10}\text{H}_{12}\text{FN}_3\text{O}_5 \cdot \text{H}_2\text{O}$, the crystal packing
is stabilized by an extensive three-dimensional network of
intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds.Received 22 March 2006
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Comment

5-Fluorouracil has been increasingly employed, alone or in
combination with other drugs and hormones, in the treatment
of several tumours (Markova *et al.*, 2005; Akgerman & Guney,
2000). However, it exhibits strong side effects, such as
gastrointestinal toxicity and delivery problems (Nichifor *et al.*,
1997; Hulme *et al.*, 2005; Hu & Wang, 2005). It is expected that
a dipeptide derivative of 5-fluorouracil can reduce the side
effects and have a longer duration of activity (Li *et al.*, 2000).
In view of these expectations, we have synthesized the title
compound, (I), and determined its crystal structure.In (I) (Fig. 1), the bond lengths and angles (Table 1) show
normal values, although the $\text{N3}-\text{C6}$ bond distance of
 $1.324(3)\text{ \AA}$ is shorter than a typical $\text{C}-\text{N}$ bond length (*ca*
 1.44 \AA ; Jin *et al.*, 2004), but longer than a typical double $\text{C}=\text{N}$
bond (*ca* 1.27 \AA), indicating electron delocalization of atoms
 O3 , C6 and N3 . In the crystal structure, an extensive three-
dimensional network of intermolecular $\text{O}-\text{H} \cdots \text{O}$ and $\text{N}-$
 $\text{H} \cdots \text{O}$ hydrogen bonds (Table 2) involving the uncoordinated
water molecule stabilizes the crystal packing (Fig. 2).

Experimental

The starting material 5-fluorouracil-1-acetic acid (5-fluoro-2,4-dioxo-
1,2,3,4-tetrahydropyrimidine-1-acetic acid) was prepared from 5-
fluorouracil and bromoacetic acid following a known method (Liu *et al.*,
2002). The title compound was synthesized from 5-fluorouracil-1-
acetic acid, dicyclohexyl carbodiimide (DCC), and 1-hydroxy-
benzotriazole (HOBT). A solution (25 ml) of *N,N*-dimethyl-
formamide (DMF) with DCC (0.024 mol) was added dropwise to a
DMF solution (75 ml) of 5-fluorouracil-1-acetic acid (0.02 mol) and
HOBT (0.02 mol) at 273 K and left to stand for 5 h at room
temperature. 2(*S*)-Amino-2-methyl acetate (0.02 mol) and triethyl-
amine (0.02 mol) were then added to the mixture. After stirring for
4 h, a white solid was obtained after filtration, reduced pressure
distillation of DMF, and column chromatography separation. The
purified product was dissolved in 95% ethanol and single crystals
were obtained after 10 d.

Crystal data

C₁₀H₁₂FN₃O₅·H₂O

M_r = 291.24

Orthorhombic, *P*2₁2₁2₁

a = 4.7472 (6) Å

b = 11.2103 (14) Å

c = 25.139 (3) Å

V = 1337.8 (3) Å³

Z = 4

D_x = 1.446 Mg m⁻³

Mo *K*α radiation

Cell parameters from 2999

reflections

θ = 2.5–24.2°

μ = 0.13 mm⁻¹

T = 298 (2) K

Block, colourless

0.43 × 0.28 × 0.20 mm

Data collection

Bruker APEX area-detector

diffractometer

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2002)

T_{min} = 0.947, *T_{max}* = 0.970

1455 measured reflections

1455 independent reflections

1410 reflections with *I* > 2σ(*I*)

R_{int} = 0.000

θ_{max} = 25.2°

h = 0 → 5

k = 0 → 13

l = 0 → 30

Refinement

Refinement on *F*²

R [*F*² > 2σ(*F*²)] = 0.043

wR (*F*²) = 0.107

S = 1.19

1455 reflections

189 parameters

H atoms treated by a mixture of

independent and constrained

refinement

$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.3758P]$

where $P = (F_o^2 + 2F_c^2)/3$

($\Delta\sigma$)_{max} < 0.001

$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O3—C6	1.224 (3)	N3—C6	1.324 (3)
N1—C4	1.373 (4)	N3—C7	1.444 (3)
N1—C1	1.373 (4)	C2—C3	1.435 (4)
N1—C5	1.456 (3)	C3—C4	1.325 (4)
N2—C1	1.371 (4)	C5—C6	1.520 (4)
N2—C2	1.385 (4)		
C6—N3—C7	120.7 (2)	N3—C6—C5	116.5 (2)
N1—C5—C6	111.2 (2)	N3—C7—C10	111.8 (3)
O3—C6—N3	123.1 (3)	N3—C7—C8	110.6 (2)
O3—C6—C5	120.3 (3)		

Table 2

Hydrogen-bond 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>
O6—H6B...O5 ⁱ	0.84 (2)	2.01 (2)	2.845 (4)	171 (4)
O6—H6A...O3 ⁱⁱ	0.83 (2)	2.17 (3)	2.936 (4)	153 (4)
N3—H3...O3 ⁱⁱⁱ	0.86	2.23	2.981 (3)	147
N2—H2...O6	0.86	1.92	2.781 (3)	173

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

H atoms of the water molecule were located in difference density maps and refined with O—H distances restrained to 0.82 (2) Å and with *U*_{iso}(H) = 1.2*U*_{eq}(parent atom). Other H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of *Csp*²—H = 0.93 Å with *U*_{iso}(H) = 1.2*U*_{eq}(parent atom), *Csp*³—H = 0.96 or 0.97 Å with *U*_{iso}(H) = 1.5*U*_{eq}(parent atom) and N—H = 0.86 Å with *U*_{iso}(H) = 1.2*U*_{eq}(parent atom). Due to the absence of any significant anomalous scatterers, Friedel pairs were merged.

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

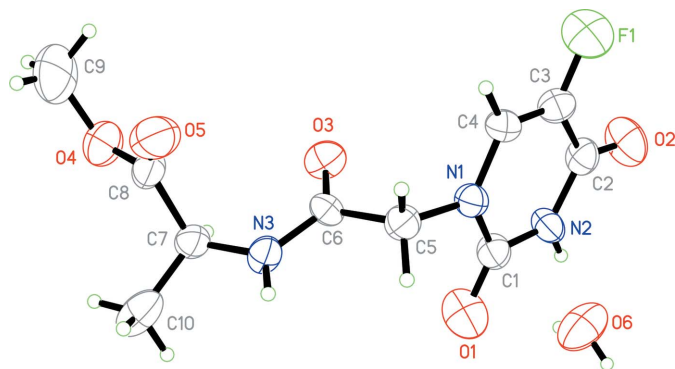


Figure 1

View of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

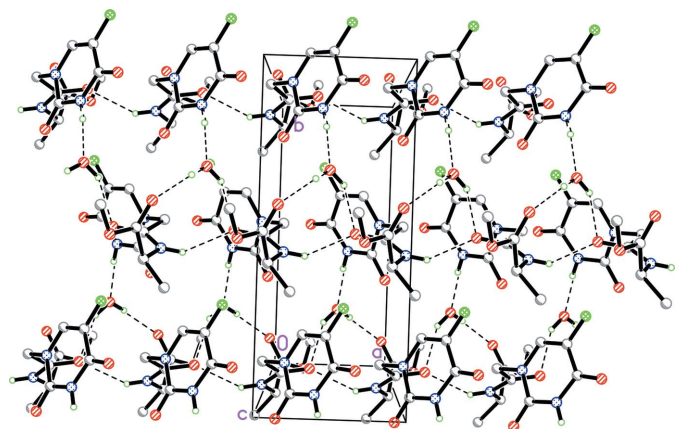


Figure 2

The packing, viewed approximately along the *c* axis. Dashed lines indicate intermolecular O—H...O and N—H...O hydrogen bonds. H atoms not involved in hydrogen bonds have been omitted for clarity.

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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References

- Akgerman, A. & Guney, O. (2000). *J. Chem. Eng. Data*, **45**, 1049–1052.
- Bruker (2002). *SADABS* (Version 2.03), *SAINT* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
- Hulme, A. T., Price, S. L. & Tocher, D. A. (2005). *J. Am. Chem. Soc.* **127**, 1116–1117.
- Hu, M.-L. & Wang, W.-D. (2005). *Acta Cryst.* **E61**, m975–m977.
- Jin, Z.-M., Li, L., Li, M.-C., Hu, M. L. & Shen, L. (2004). *Acta Cryst.* **C60**, o642–o643.
- Li, C.-Z., Wang, L.-F., Li, Y.-Z., Xia, C.-G. & Dai, R.-B. (2000). *Acta Cryst.* **C56**, e376–e377.
- Liu, X. J., Chen, R. Y. & Liu, Y. Y. (2002). *Chem. J. Chin. Univ.* **23**, 1299–1303.
- Markova, N., Enchev, V. & Timtcheva, I. (2005). *J. Phys. Chem. A*, **109**, 1981–1988.
- Nichifor, M., Schacht, E. H. & Seymour, L. W. (1997). *J. Cont. Rel.* **48**, 165–178.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.