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5-Fluorouracil–1,4-dioxane (4/1)

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.114
Data-to-parameter ratio = 11.9

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

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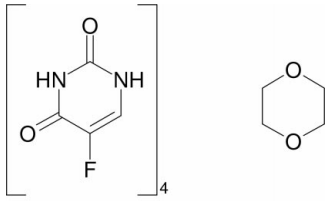
A solvate of 5-fluorouracil with 1,4-dioxane, $4C_4H_3FN_2O_2\cdot C_4H_8O_2$, is reported. It crystallizes in the triclinic space group $P\overline{1}$. Two molecules of 5-fluorouracil are present in the asymmetric unit, together with one-half molecule of 1,4-dioxane, which lies on a centre of symmetry. In the crystal structure, ribbons of 5-fluorouracil molecules are joined by 1,4-dioxane-mediated interactions, forming sheets parallel to the $(2\overline{1}1)$ planes.

Comment

In the course of a polymorph screen performed on 5-fluorouracil, three solvates were discovered; the crystal structure of one of these solvates is reported here.



(I)

The title compound, (I), crystallizes in the space group $P\overline{1}$ with two molecules of 5-fluorouracil and one-half molecule of 1,4-dioxane in the asymmetric unit (Fig. 1). The 1,4-dioxane molecule is located on a crystallographic centre of symmetry.

Four distinct N—H···O hydrogen bonds occur in the crystal structure (Table 1). Both the crystallographically independent 5-fluorouracil molecules are present as centrosymmetric hydrogen-bonded dimers. One dimer contains the hydrogen bond N3—H3···O7^{ii} (symmetry codes are given in Table 1), with a donor–acceptor distance of 2.857 (2) Å, while the other dimer contains the hydrogen bond N13—H13···O18^{iii} [2.824 (2) Å]. These dimers are linked, forming ribbon-like structures, by N1—H1···O17^{i} hydrogen bonds. Adjacent

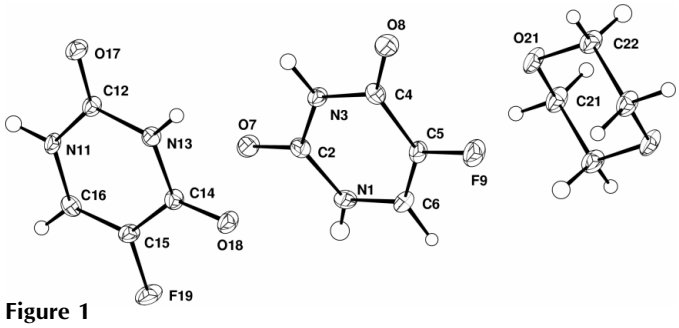


Figure 1
View (Watkin *et al.*, 1996) of the asymmetric unit of the title compound and the other half of the dioxane molecule, with atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

ribbons of 5-fluorouracil molecules are linked, forming sheets parallel to the $(2\bar{1}1)$ planes *via* 1,4-dioxane molecules which act as N11—H11···O21 [N···O = 2.746 (2) Å] hydrogen-bond bridges (Fig. 2).

Experimental

5-Fluorouracil was obtained from the Aldrich Chemical Company Inc. The crystals were grown by solvent evaporation of a saturated solution of 5-fluorouracil in 1,4-dioxane.

Crystal data

$4C_4H_3FN_2O_2\cdot C_4H_8O_2$
M_r = 608.44
Triclinic, $P\bar{1}$
a = 7.0847 (11) Å
b = 8.4733 (13) Å
c = 10.2291 (15) Å
α = 98.128 (3)°
β = 96.913 (3)°
γ = 99.785 (3)°
V = 592.45 (16) Å^3
Z = 1
D_x = 1.705 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1082 reflections
θ = 2.5–26.7°
μ = 0.16 mm^{-1}
T = 150 (2) K
Plate, colourless
0.35 × 0.24 × 0.03 mm

Data collection

Bruker SMART APEX diffractometer
Narrow-frame ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.947, T_{max} = 0.995
5320 measured reflections
2741 independent reflections
2131 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 28.3°
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.114
S = 1.08
2741 reflections
230 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.1655P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O17^{i}	0.83 (3)	1.98 (3)	2.798 (2)	167 (2)
N3—H3···O7^{ii}	0.91 (2)	1.95 (2)	2.857 (2)	176 (2)
N11—H11···O21	0.91 (2)	1.84 (2)	2.746 (2)	171 (2)
N13—H13···O18^{iii}	0.85 (2)	1.98 (2)	2.824 (2)	175 (2)

Symmetry codes: (i) $x, 1+y, z$; (ii) $-x, 1-y, 1-z$; (iii) $1-x, 1-y, 1-z$.

All H atoms were located in a difference map and were refined isotropically. C—H distances were in the range 0.93 (2)–1.00 (2) Å and N—H distances were in the range 0.83 (3)–0.91 (2) Å.

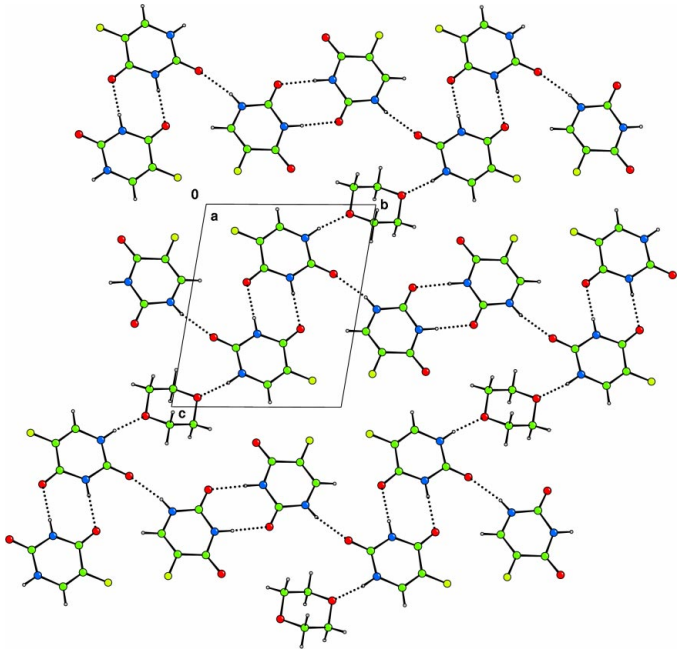


Figure 2
The hydrogen-bonded sheet structure, viewed along the *a* axis. Ribbons of 5-fluorouracil molecules are joined by 1,4-dioxane-mediated interactions, forming the sheet structure. Dashed lines indicate hydrogen bonds.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *SHELXL*97.

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