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5-Fluorouracil–1,4-dioxane (4/1)

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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.

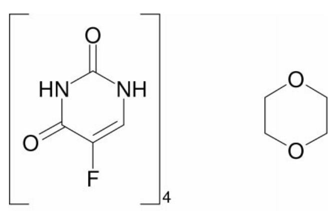
Key indicators

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.

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

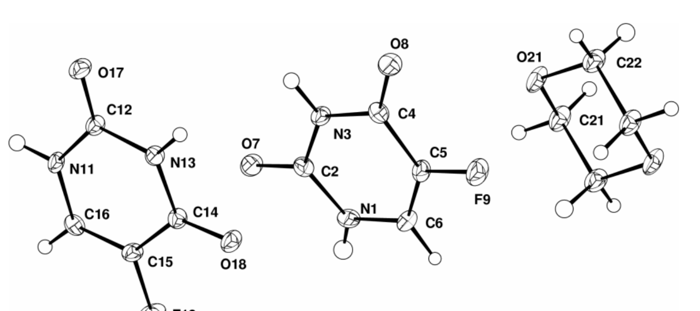


Figure 1
View (Watkin *et al.*, 1996) of the asymmetric unit of the title compound and the other half of the dioxane molecule, with atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

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

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

Data collection

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

Refinement

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

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.

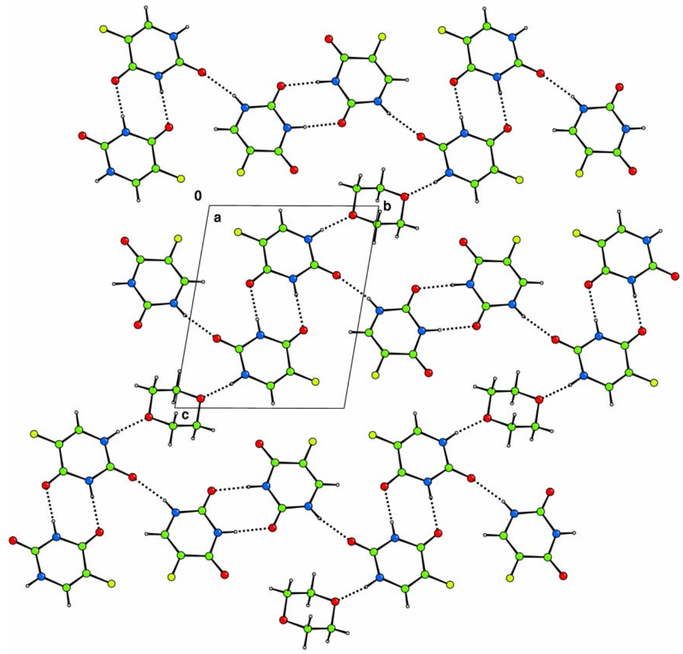


Figure 2
The hydrogen-bonded sheet structure, viewed along the *a* axis. Ribbons of 5-fluorouracil molecules are joined by 1,4-dioxane-mediated interactions, forming the sheet structure. Dashed lines indicate hydrogen bonds.

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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supporting information

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Crystal data

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M_r = 608.44
Triclinic, $P\overline{1}$
Hall symbol: -P 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
F(000) = 312
D_x = 1.705 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1082 reflections
θ = 2.5–26.7°
μ = 0.16 mm^{-1}
T = 150 K
Plate, colourless
0.35 × 0.24 × 0.03 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω rotation with narrow frames 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°, θ_{min} = 2.0°
h = −9→9
k = −11→11
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.114
S = 1.08
2741 reflections
230 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: found from delta F
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}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
F9	0.37304 (19)	0.84841 (15)	0.16434 (12)	0.0278 (3)
O7	0.0566 (2)	0.69895 (17)	0.59096 (14)	0.0232 (4)
O8	0.1991 (2)	0.53627 (17)	0.17616 (14)	0.0234 (4)
N1	0.2094 (3)	0.8886 (2)	0.48334 (18)	0.0196 (4)
H1	0.207 (4)	0.966 (3)	0.543 (3)	0.035 (7)*
N3	0.1319 (2)	0.6192 (2)	0.38430 (16)	0.0172 (4)
H3	0.075 (3)	0.515 (3)	0.389 (2)	0.025 (6)*
C2	0.1276 (3)	0.7343 (2)	0.4933 (2)	0.0175 (4)
C4	0.2065 (3)	0.6469 (2)	0.2685 (2)	0.0172 (4)
C5	0.2912 (3)	0.8144 (2)	0.2720 (2)	0.0182 (4)
C6	0.2897 (3)	0.9295 (3)	0.3745 (2)	0.0206 (5)
H6	0.341 (3)	1.040 (3)	0.377 (2)	0.016 (5)*
F19	0.47754 (18)	0.80432 (14)	0.86893 (12)	0.0258 (3)
O17	0.2285 (2)	0.17889 (17)	0.65744 (14)	0.0222 (4)
O18	0.5329 (2)	0.68229 (17)	0.61498 (14)	0.0214 (3)
N11	0.2653 (2)	0.3825 (2)	0.83458 (16)	0.0162 (4)
H11	0.203 (3)	0.311 (3)	0.881 (2)	0.026 (6)*
N13	0.3790 (3)	0.4320 (2)	0.63923 (17)	0.0167 (4)
H13	0.399 (3)	0.398 (3)	0.561 (2)	0.021 (6)*
C12	0.2873 (3)	0.3209 (2)	0.70817 (19)	0.0158 (4)
C14	0.4501 (3)	0.5943 (2)	0.6848 (2)	0.0161 (4)
C15	0.4153 (3)	0.6458 (2)	0.8187 (2)	0.0169 (4)
C16	0.3266 (3)	0.5434 (2)	0.8892 (2)	0.0171 (4)
H16	0.304 (3)	0.576 (3)	0.976 (2)	0.020 (6)*
O21	0.0777 (2)	0.14345 (17)	0.95410 (15)	0.0241 (4)
C21	0.1347 (3)	−0.0087 (3)	0.9117 (2)	0.0230 (5)
H21A	0.162 (3)	−0.009 (3)	0.819 (2)	0.021 (6)*
H21B	0.255 (3)	−0.013 (3)	0.973 (2)	0.021 (6)*
C22	0.0271 (4)	0.1472 (3)	1.0864 (2)	0.0237 (5)
H22A	0.143 (3)	0.138 (3)	1.150 (2)	0.031 (7)*
H22B	−0.012 (3)	0.249 (3)	1.109 (2)	0.019 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F9	0.0363 (8)	0.0252 (7)	0.0238 (7)	0.0018 (6)	0.0137 (6)	0.0079 (6)
O7	0.0332 (9)	0.0184 (8)	0.0172 (8)	0.0007 (6)	0.0091 (7)	0.0006 (6)
O8	0.0327 (9)	0.0190 (8)	0.0178 (8)	0.0029 (6)	0.0092 (7)	−0.0015 (6)
N1	0.0261 (10)	0.0149 (9)	0.0155 (9)	0.0021 (7)	0.0035 (8)	−0.0032 (7)
N3	0.0236 (10)	0.0126 (9)	0.0150 (9)	0.0014 (7)	0.0056 (7)	0.0010 (7)

C2	0.0201 (11)	0.0174 (10)	0.0141 (10)	0.0034 (8)	0.0020 (8)	0.0009 (8)
C4	0.0176 (10)	0.0194 (10)	0.0149 (10)	0.0055 (8)	0.0022 (8)	0.0018 (8)
C5	0.0208 (11)	0.0190 (11)	0.0160 (10)	0.0021 (8)	0.0060 (8)	0.0059 (8)
C6	0.0242 (12)	0.0141 (10)	0.0231 (11)	0.0001 (8)	0.0030 (9)	0.0062 (9)
F19	0.0354 (8)	0.0137 (6)	0.0255 (7)	−0.0024 (5)	0.0111 (6)	−0.0034 (5)
O17	0.0314 (9)	0.0136 (7)	0.0202 (8)	0.0001 (6)	0.0071 (7)	0.0006 (6)
O18	0.0297 (8)	0.0166 (8)	0.0183 (7)	0.0001 (6)	0.0109 (6)	0.0031 (6)
N11	0.0221 (9)	0.0147 (9)	0.0132 (8)	0.0016 (7)	0.0077 (7)	0.0047 (7)
N13	0.0233 (9)	0.0150 (9)	0.0116 (8)	0.0022 (7)	0.0064 (7)	0.0002 (7)
C12	0.0171 (10)	0.0150 (10)	0.0162 (10)	0.0025 (8)	0.0060 (8)	0.0034 (8)
C14	0.0173 (10)	0.0161 (10)	0.0157 (10)	0.0032 (8)	0.0041 (8)	0.0036 (8)
C15	0.0200 (11)	0.0130 (10)	0.0166 (10)	0.0020 (8)	0.0039 (8)	−0.0005 (8)
C16	0.0194 (11)	0.0197 (11)	0.0123 (10)	0.0052 (8)	0.0038 (8)	0.0004 (8)
O21	0.0348 (9)	0.0160 (8)	0.0251 (8)	0.0031 (6)	0.0163 (7)	0.0078 (6)
C21	0.0301 (13)	0.0190 (11)	0.0232 (12)	0.0046 (9)	0.0138 (10)	0.0058 (9)
C22	0.0344 (13)	0.0158 (11)	0.0214 (12)	0.0023 (9)	0.0109 (10)	0.0017 (9)

Geometric parameters (Å, º)

F9—C5	1.349 (2)	N11—C16	1.372 (3)
O7—C2	1.223 (2)	N11—H11	0.91 (2)
O8—C4	1.223 (2)	N13—C14	1.374 (3)
N1—C2	1.360 (3)	N13—C12	1.376 (3)
N1—C6	1.371 (3)	N13—H13	0.85 (2)
N1—H1	0.83 (3)	C14—C15	1.441 (3)
N3—C2	1.380 (3)	C15—C16	1.330 (3)
N3—C4	1.387 (3)	C16—H16	0.93 (2)
N3—H3	0.91 (2)	O21—C22	1.439 (2)
C4—C5	1.437 (3)	O21—C21	1.440 (3)
C5—C6	1.330 (3)	C21—C22^{i}	1.499 (3)
C6—H6	0.94 (2)	C21—H21A	0.99 (2)
F19—C15	1.346 (2)	C21—H21B	1.00 (2)
O17—C12	1.222 (2)	C22—C21^{i}	1.499 (3)
O18—C14	1.230 (2)	C22—H22A	1.00 (2)
N11—C12	1.362 (2)	C22—H22B	0.96 (2)
C2—N1—C6	123.56 (18)	O17—C12—N13	121.69 (18)
C2—N1—H1	120.8 (18)	N11—C12—N13	114.89 (17)
C6—N1—H1	115.6 (18)	O18—C14—N13	121.64 (18)
C2—N3—C4	126.62 (18)	O18—C14—C15	125.45 (18)
C2—N3—H3	115.6 (15)	N13—C14—C15	112.90 (17)
C4—N3—H3	117.7 (15)	C16—C15—F19	121.57 (18)
O7—C2—N1	123.09 (19)	C16—C15—C14	122.27 (19)
O7—C2—N3	122.19 (19)	F19—C15—C14	116.16 (17)
N1—C2—N3	114.72 (18)	C15—C16—N11	119.84 (19)
O8—C4—N3	121.43 (19)	C15—C16—H16	122.7 (14)
O8—C4—C5	125.64 (19)	N11—C16—H16	117.5 (14)
N3—C4—C5	112.93 (17)	C22—O21—C21	109.42 (16)

C6—C5—F9	121.62 (18)	O21—C21—C22^{i}	110.12 (18)
C6—C5—C4	122.52 (19)	O21—C21—H21A	107.1 (13)
F9—C5—C4	115.86 (17)	C22^{i}—C21—H21A	109.0 (13)
C5—C6—N1	119.60 (19)	O21—C21—H21B	107.8 (12)
C5—C6—H6	123.6 (13)	C22^{i}—C21—H21B	112.2 (12)
N1—C6—H6	116.8 (13)	H21A—C21—H21B	110.5 (17)
C12—N11—C16	122.96 (17)	O21—C22—C21^{i}	110.23 (18)
C12—N11—H11	116.2 (15)	O21—C22—H22A	109.5 (14)
C16—N11—H11	120.8 (14)	C21^{i}—C22—H22A	109.2 (14)
C14—N13—C12	127.10 (18)	O21—C22—H22B	106.3 (13)
C14—N13—H13	115.1 (15)	C21^{i}—C22—H22B	110.9 (13)
C12—N13—H13	117.7 (15)	H22A—C22—H22B	110.6 (19)
O17—C12—N11	123.41 (18)		

C6—N1—C2—O7	179.8 (2)	C16—N11—C12—N13	1.0 (3)
C6—N1—C2—N3	0.3 (3)	C14—N13—C12—O17	179.6 (2)
C4—N3—C2—O7	179.2 (2)	C14—N13—C12—N11	0.7 (3)
C4—N3—C2—N1	−1.3 (3)	C12—N13—C14—O18	178.74 (19)
C2—N3—C4—O8	−178.30 (19)	C12—N13—C14—C15	−1.7 (3)
C2—N3—C4—C5	2.3 (3)	O18—C14—C15—C16	−179.2 (2)
O8—C4—C5—C6	178.1 (2)	N13—C14—C15—C16	1.3 (3)
N3—C4—C5—C6	−2.4 (3)	O18—C14—C15—F19	0.9 (3)
O8—C4—C5—F9	−1.8 (3)	N13—C14—C15—F19	−178.66 (17)
N3—C4—C5—F9	177.60 (17)	F19—C15—C16—N11	−179.98 (18)
F9—C5—C6—N1	−178.30 (19)	C14—C15—C16—N11	0.1 (3)
C4—C5—C6—N1	1.7 (3)	C12—N11—C16—C15	−1.3 (3)
C2—N1—C6—C5	−0.6 (3)	C22—O21—C21—C22^{i}	58.7 (3)
C16—N11—C12—O17	−178.0 (2)	C21—O21—C22—C21^{i}	−58.8 (3)

Symmetry code: (i) −*x*, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O17^{ii}	0.83 (3)	1.98 (3)	2.798 (2)	167 (2)
N3—H3···O7^{iii}	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^{iv}	0.85 (2)	1.98 (2)	2.824 (2)	175 (2)

Symmetry codes: (ii) *x*, *y*+1, *z*; (iii) −*x*, −*y*+1, −*z*+1; (iv) −*x*+1, −*y*+1, −*z*+1.