

## 5-Fluorouracil–2,2,2-trifluoroethanol (1/1)

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The title compound,  $C_4H_3FN_2O_2 \cdot C_2H_3F_3O$ , crystallizes with one 5-fluorouracil and one 2,2,2-trifluoroethanol molecule in the asymmetric unit. The 5-fluorouracil molecules are linked into a chain primarily *via*  $N-H \cdots O$  hydrogen bonds, with the 2,2,2-trifluoroethanol molecules attached to this *via*  $O-H \cdots O$  hydrogen bonds.

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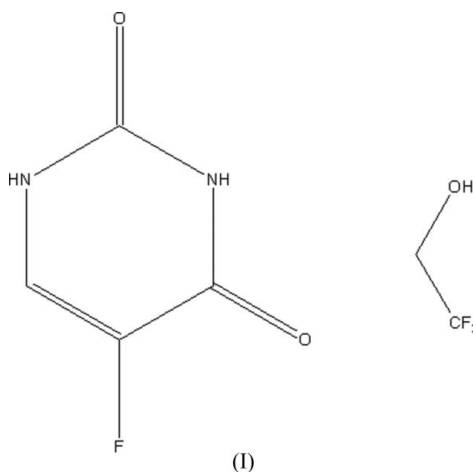
## Key indicators

Single-crystal X-ray study  
 $T = 150$  K  
 Mean  $\sigma(C-C) = 0.003$  Å  
 R factor = 0.028  
 wR factor = 0.074  
 Data-to-parameter ratio = 6.8

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

## Comment

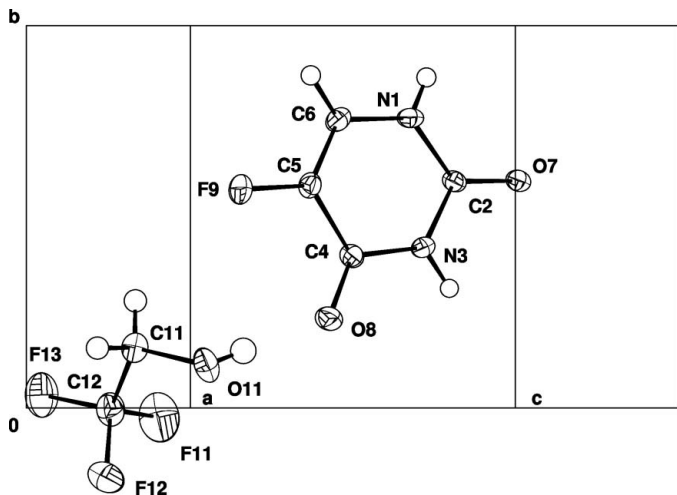
The title compound, (I), is the fourth solvate of 5-fluorouracil obtained in the course of a polymorph screen. The previously published structures contained 1,4-dioxane (Hulme & Tocher, 2004a), dimethylformamide (Hulme & Tocher, 2004b) and dimethylsulfoxide (Hulme & Tocher, 2004c).



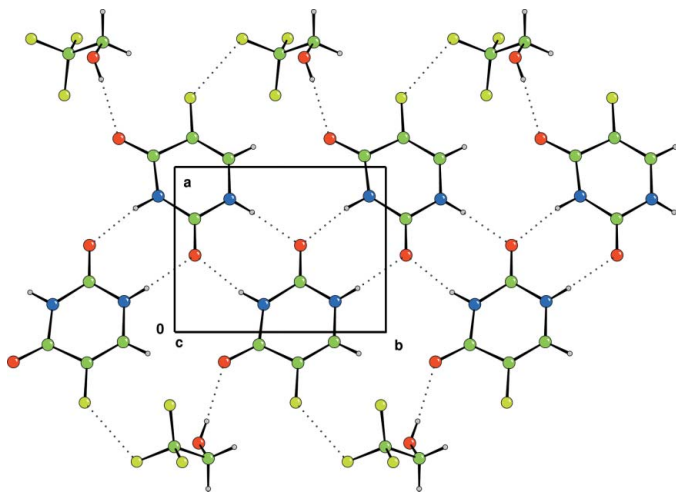
One fluorouracil molecule and one 2,2,2-trifluoroethanol molecule are present in the asymmetric unit of (I) (Fig. 1). This structure bears no similarity to any of the previously reported solvate structures of 5-fluorouracil.

The 5-fluorouracil molecules of (I) form a ribbon propagated by the screw axis, with trifluoroethanol molecules attached to the outer edges of the ribbon. Each 5-fluorouracil molecule forms two  $R_2^2(8)$  hydrogen bonds with adjacent 5-fluorouracil molecules, as shown in Fig. 2; details are given in Table 1. A further hydrogen bond joins the 5-fluorouracil carbonyl O atom, unused in forming the ribbon, with the hydroxyl group of the trifluoroethanol molecule (Fig. 2 and Table 1).

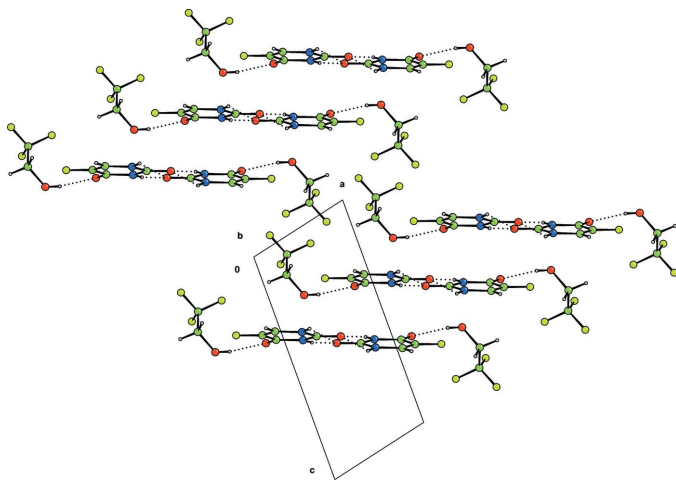
The ribbons stack upon one another parallel to [001] (Fig. 3). Close  $F \cdots F$  contacts are an interesting feature present in this structure. There is a short  $F \cdots F$  contact within the ribbon,  $F9 \cdots F12^{iv}$  [2.891 (2) Å; symmetry code: (iv)  $x, y + 1, z$ ], which acts as a weak stabilizing interaction for the



**Figure 1**  
A view of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.



**Figure 2**  
The structure of the ribbon, showing  $R_2^2(8)$  hydrogen-bonded dimers and the hydrogen bonds (dotted lines) between 5-fluorouracil and 2,2,2-trifluoroethanol.



**Figure 3**  
The stacking of the ribbons side-by-side into layers. Hydrogen bonds are shown as dotted lines.

ribbon motif. A short contact is also present between trifluoromethyl groups in ribbons of adjacent layers, viz.  $F12 \cdots F13^v$  [3.001 (2) Å; symmetry code: (v)  $-x, y - \frac{1}{2}, -z$ ]. A third short  $F \cdots F$  contact,  $F9 \cdots F13^{vi}$  [2.906 (2) Å; symmetry code: (vi)  $1 - x, \frac{1}{2} + y, -z$ ], also links ribbons in adjacent layers. These interlayer  $F \cdots F$  contacts are the only interactions between the layers.

## Experimental

Typically, crystals of length 2–5 mm were grown from a solution of 5-fluorouracil in 2,2,2-trifluoroethanol by solvent evaporation. Attempts to cut crystals to a suitable size for X-ray diffraction led to shattering. Consequently, a large crystal with a longest dimension of 1.49 mm was mounted and used for the experiment.

### Crystal data

$C_4H_3FN_2O_2 \cdot C_2H_3F_3O$   
 $M_r = 230.13$   
 Monoclinic,  $P2_1$   
 $a = 5.3976$  (6) Å  
 $b = 6.7062$  (8) Å  
 $c = 12.1098$  (14) Å  
 $\beta = 102.807$  (2)°  
 $V = 427.44$  (9) Å<sup>3</sup>  
 $Z = 2$

$D_x = 1.788$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 287 reflections  
 $\theta = 3.5$ – $28.1^\circ$   
 $\mu = 0.19$  mm<sup>-1</sup>  
 $T = 150$  (2) K  
 Lath, colourless  
 $1.49 \times 0.34 \times 0.17$  mm

### Data collection

Bruker SMART APEX diffractometer  
 $\omega$  rotation scans with narrow frames  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{min} = 0.760$ ,  $T_{max} = 0.968$   
 2634 measured reflections

1090 independent reflections  
 1060 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.017$   
 $\theta_{max} = 28.2^\circ$   
 $h = -7 \rightarrow 6$   
 $k = -8 \rightarrow 8$   
 $l = -15 \rightarrow 15$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.028$   
 $wR(F^2) = 0.074$   
 $S = 1.04$   
 1090 reflections  
 160 parameters  
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.0688P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.31$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -0.24$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N3-H3 \cdots O7^i$	0.87 (3)	1.92 (3)	2.786 (2)	173 (2)
$N1-H1 \cdots O7^{ii}$	0.82 (3)	2.20 (3)	2.924 (2)	147 (2)
$N1-H1 \cdots O11^{iii}$	0.82 (3)	2.43 (3)	3.037 (2)	132 (2)
$O11-H11 \cdots O8$	0.76 (3)	2.00 (3)	2.7507 (19)	171 (3)

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

All H atoms were located in a difference map and were refined isotropically, with C–H distances between 0.89 (3) and 0.97 (2) Å. See Table 1 for N–H and O–H bond distances.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: CAMERON (Watkin *et al.*, 1996); software used to prepare material for publication: SHELXL97.

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## supporting information

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Monoclinic,  $P2_1$

Hall symbol: P2yb

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$c = 12.1098$  (14) Å

$\beta = 102.807$  (2)°

$V = 427.44$  (9) Å<sup>3</sup>

$Z = 2$

$F(000) = 232$

$D_x = 1.788$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2027 reflections

$\theta = 3.5$ – $28.1$ °

$\mu = 0.19$  mm<sup>-1</sup>

$T = 150$  K

Plate, colourless

$1.49 \times 0.34 \times 0.17$  mm

*Data collection*

Bruker SMART APEX  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  rotation with narrow frames scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

$T_{\min} = 0.760$ ,  $T_{\max} = 0.968$

2634 measured reflections

1090 independent reflections

1060 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.017$

$\theta_{\max} = 28.2$ °,  $\theta_{\min} = 1.7$ °

$h = -7 \rightarrow 6$

$k = -8 \rightarrow 8$

$l = -15 \rightarrow 15$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.074$

$S = 1.04$

1090 reflections

160 parameters

1 restraint

Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.0688P]$

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

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

$\Delta\rho_{\max} = 0.31$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.24$  e Å<sup>-3</sup>

*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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
F9	0.57881 (18)	0.57165 (18)	0.24396 (9)	0.0274 (3)
O7	1.5295 (2)	0.59410 (19)	0.49255 (11)	0.0208 (3)
O8	0.8249 (2)	0.2336 (2)	0.34213 (11)	0.0248 (3)
N1	1.1899 (3)	0.7582 (2)	0.38580 (12)	0.0209 (3)
H1	1.271 (5)	0.862 (5)	0.393 (2)	0.037 (7)*
N3	1.1724 (3)	0.4198 (2)	0.41781 (12)	0.0191 (3)
H3	1.254 (4)	0.313 (4)	0.446 (2)	0.020 (5)*
C2	1.3099 (3)	0.5927 (3)	0.43551 (13)	0.0176 (3)
C4	0.9293 (3)	0.3969 (3)	0.35374 (13)	0.0186 (3)
C5	0.8192 (3)	0.5820 (3)	0.30489 (14)	0.0200 (3)
C6	0.9461 (3)	0.7542 (3)	0.32056 (14)	0.0216 (3)
H6	0.881 (4)	0.870 (4)	0.2910 (18)	0.017 (5)*
F11	0.5628 (3)	-0.0249 (3)	0.08309 (13)	0.0571 (5)
F12	0.2214 (3)	-0.1821 (2)	0.08921 (13)	0.0506 (4)
F13	0.2185 (3)	0.0332 (3)	-0.04178 (11)	0.0533 (4)
O11	0.3333 (3)	0.1118 (2)	0.25622 (11)	0.0299 (3)
H11	0.466 (5)	0.155 (5)	0.275 (2)	0.033 (7)*
C11	0.2408 (4)	0.1569 (3)	0.14144 (16)	0.0289 (4)
H12	0.056 (5)	0.155 (4)	0.126 (2)	0.030 (6)*
H13	0.316 (5)	0.277 (5)	0.121 (2)	0.041 (7)*
C12	0.3116 (4)	-0.0040 (4)	0.06811 (17)	0.0323 (4)

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
F9	0.0195 (5)	0.0296 (6)	0.0290 (5)	0.0011 (5)	-0.0037 (4)	0.0016 (5)
O7	0.0193 (5)	0.0154 (5)	0.0257 (6)	-0.0009 (5)	0.0005 (4)	-0.0018 (5)
O8	0.0202 (6)	0.0184 (6)	0.0335 (6)	-0.0034 (5)	0.0009 (5)	-0.0011 (5)
N1	0.0224 (7)	0.0126 (7)	0.0267 (7)	-0.0009 (6)	0.0034 (5)	0.0009 (6)
N3	0.0192 (6)	0.0140 (6)	0.0226 (6)	0.0014 (5)	0.0015 (5)	0.0019 (6)
C2	0.0199 (7)	0.0144 (7)	0.0181 (7)	-0.0015 (7)	0.0038 (5)	-0.0022 (6)
C4	0.0189 (7)	0.0174 (8)	0.0192 (7)	-0.0004 (6)	0.0036 (6)	-0.0015 (6)
C5	0.0179 (7)	0.0217 (8)	0.0194 (7)	0.0020 (7)	0.0020 (6)	0.0005 (6)
C6	0.0226 (8)	0.0187 (8)	0.0227 (7)	0.0041 (7)	0.0031 (6)	0.0042 (7)
F11	0.0388 (7)	0.0803 (13)	0.0542 (9)	0.0108 (8)	0.0149 (6)	-0.0146 (8)
F12	0.0700 (10)	0.0305 (7)	0.0444 (8)	-0.0085 (7)	-0.0023 (7)	-0.0050 (6)
F13	0.0697 (9)	0.0655 (11)	0.0216 (6)	-0.0002 (8)	0.0032 (6)	-0.0015 (6)
O11	0.0266 (6)	0.0391 (8)	0.0227 (6)	-0.0110 (6)	0.0026 (5)	-0.0024 (6)
C11	0.0284 (8)	0.0287 (10)	0.0270 (8)	0.0003 (8)	0.0003 (7)	0.0012 (8)
C12	0.0347 (9)	0.0361 (10)	0.0242 (8)	0.0000 (9)	0.0025 (7)	-0.0022 (8)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

F9—C5	1.3448 (19)	C5—C6	1.335 (3)
O7—C2	1.233 (2)	C6—H6	0.89 (3)
O8—C4	1.225 (2)	F11—C12	1.335 (3)
N1—C2	1.357 (2)	F12—C12	1.336 (3)
N1—C6	1.377 (2)	F13—C12	1.338 (2)
N1—H1	0.82 (3)	O11—C11	1.402 (2)
N3—C2	1.368 (2)	O11—H11	0.76 (3)
N3—C4	1.377 (2)	C11—C12	1.500 (3)
N3—H3	0.87 (3)	C11—H12	0.97 (2)
C4—C5	1.445 (2)	C11—H13	0.96 (3)
C2—N1—C6	122.69 (16)	C5—C6—H6	123.5 (14)
C2—N1—H1	118 (2)	N1—C6—H6	116.9 (14)
C6—N1—H1	119.7 (19)	C11—O11—H11	108 (2)
C2—N3—C4	126.78 (15)	O11—C11—C12	110.50 (17)
C2—N3—H3	115.3 (16)	O11—C11—H12	108.0 (15)
C4—N3—H3	117.7 (16)	C12—C11—H12	105.2 (16)
O7—C2—N1	123.20 (16)	O11—C11—H13	111.0 (17)
O7—C2—N3	121.01 (16)	C12—C11—H13	106.0 (18)
N1—C2—N3	115.79 (14)	H12—C11—H13	116 (2)
O8—C4—N3	121.37 (16)	F11—C12—F12	106.4 (2)
O8—C4—C5	125.73 (15)	F11—C12—F13	107.51 (17)
N3—C4—C5	112.90 (15)	F12—C12—F13	106.53 (18)
C6—C5—F9	121.65 (16)	F11—C12—C11	112.29 (18)
C6—C5—C4	122.26 (14)	F12—C12—C11	112.26 (17)
F9—C5—C4	116.08 (15)	F13—C12—C11	111.48 (18)
C5—C6—N1	119.57 (16)		
C6—N1—C2—O7	178.79 (15)	O8—C4—C5—F9	1.7 (2)
C6—N1—C2—N3	-0.6 (2)	N3—C4—C5—F9	-178.07 (13)
C4—N3—C2—O7	-178.17 (15)	F9—C5—C6—N1	178.49 (14)
C4—N3—C2—N1	1.2 (2)	C4—C5—C6—N1	-0.4 (2)
C2—N3—C4—O8	178.87 (15)	C2—N1—C6—C5	0.2 (2)
C2—N3—C4—C5	-1.3 (2)	O11—C11—C12—F11	-61.2 (2)
O8—C4—C5—C6	-179.36 (17)	O11—C11—C12—F12	58.7 (2)
N3—C4—C5—C6	0.8 (2)	O11—C11—C12—F13	178.12 (16)

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N3—H3 $\cdots$ O7 <sup>i</sup>	0.87 (3)	1.92 (3)	2.786 (2)	173 (2)
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