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

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.028
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.

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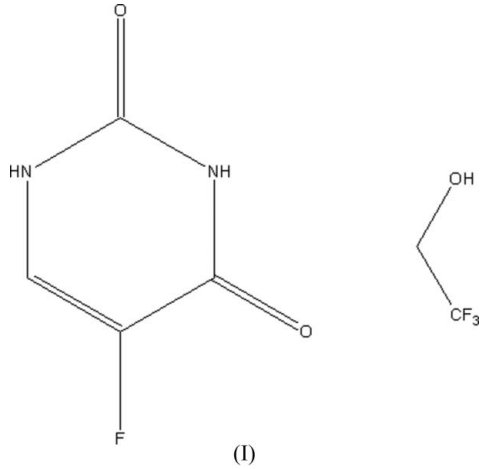
The title compound, $C_4H_3FN_2O_2\cdot C_2H_3F_3O$, crystallizes with one 5-fluorouracil and one 2,2,2-trifluoroethanol molecule in the asymmetric unit. The 5-fluorouracil molecules are linked into a chain primarily *via* N—H···O hydrogen bonds, with the 2,2,2-trifluoroethanol molecules attached to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

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, 2004*a*), dimethylformamide (Hulme & Tocher, 2004*b*) and dimethylsulfoxide (Hulme & Tocher, 2004*c*).



(I)

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···F contacts are an interesting feature present in this structure. There is a short F···F contact within the ribbon, F9···F12^{iv} [2.891 (2) Å; symmetry code: (iv) x, $y+1$, z], which acts as a weak stabilizing interaction for the

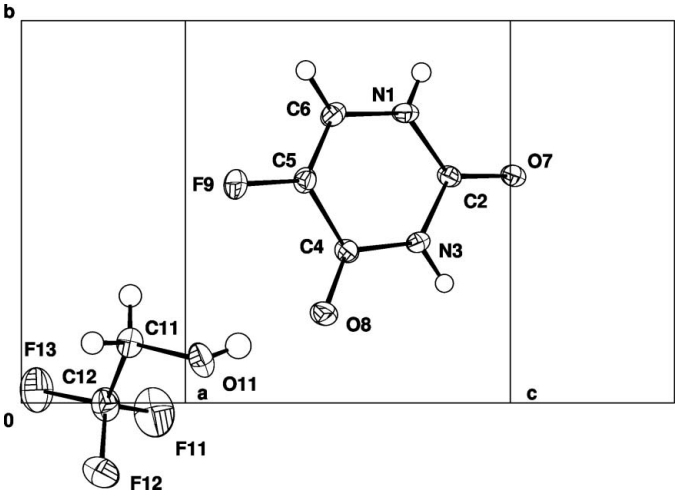


Figure 1
A view of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.

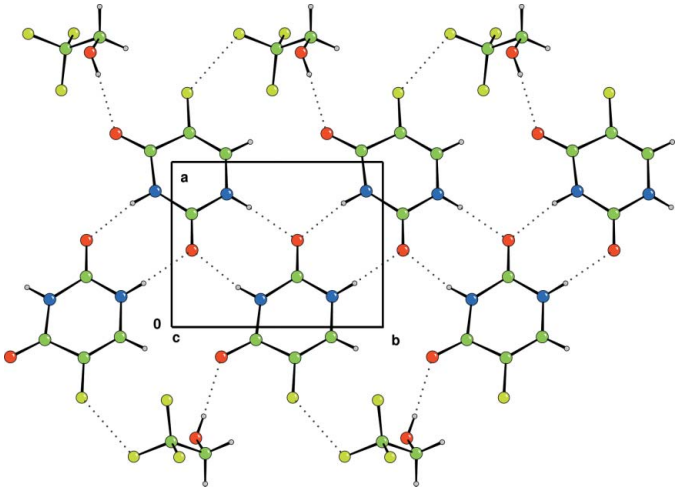


Figure 2
The structure of the ribbon, showing $R_2^2(8)$ hydrogen-bonded dimers and the hydrogen bonds (dotted lines) between 5-fluorouracil and 2,2,2-trifluoroethanol.

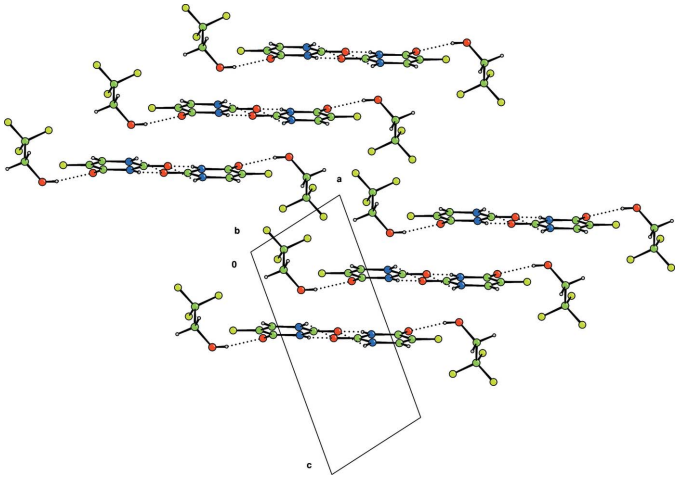


Figure 3
The stacking of the ribbons side-by-side into layers. Hydrogen bonds are shown as dotted lines.

ribbon motif. A short contact is also present between trifluoromethyl groups in ribbons of adjacent layers, *viz*. F12···F13^{v} [3.001 (2) Å; symmetry code: (v) $-x, y-\frac{1}{2}, -z$]. A third short F···F contact, F9···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···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^3
$Z = 2$
$D_x = 1.788$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2027 reflections
$\theta = 3.5$–28.1°
$\mu = 0.19$ mm^{-1}
$T = 150$ (2) K
Lath, colourless
1.49 × 0.34 × 0.17 mm

Data collection

Bruker SMART APEX diffractometer
ω 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 $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 28.2°$
$h = -7 \rightarrow 6$
$k = -8 \rightarrow 8$
$l = -15 \rightarrow 15$

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^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O7^{i}	0.87 (3)	1.92 (3)	2.786 (2)	173 (2)
N1—H1···O7^{ii}	0.82 (3)	2.20 (3)	2.924 (2)	147 (2)
N1—H1···O11^{iii}	0.82 (3)	2.43 (3)	3.037 (2)	132 (2)
O11—H11···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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