

5-Fluorouracil–dimethylformamide (2/1)

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A solvate of 5-fluorouracil with dimethylformamide (DMF), $2C_4H_3FN_2O_2 \cdot C_3H_7NO$, is reported. It crystallizes in the monoclinic space group $P2_1/n$, with two molecules of 5-fluorouracil and one molecule of DMF in the asymmetric unit. This solvate exhibits a sheet structure, with the DMF molecules present on both surfaces of the sheet and 5-fluorouracil molecules within the sheath of DMF molecules.

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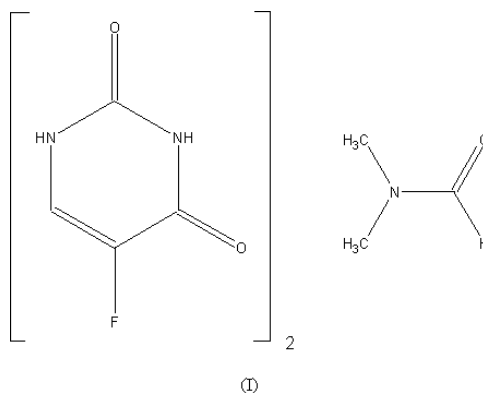
Key indicators

Single-crystal X-ray study
 $T = 150\text{ K}$
 Mean $\sigma(C-C) = 0.003\text{ \AA}$
 R factor = 0.049
 wR factor = 0.108
 Data-to-parameter ratio = 14.0

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. One of the solvate structures, (I), containing two independent molecules of 5-fluorouracil and one molecule of dimethyl formamide (DMF) in the asymmetric unit (Fig. 1), and crystallizing in the space group $P2_1/n$, is reported here.



The two 5-fluorouracil molecules in the asymmetric unit are linked to one another *via* $N11-H11 \cdots O7$ [$N \cdots O =$

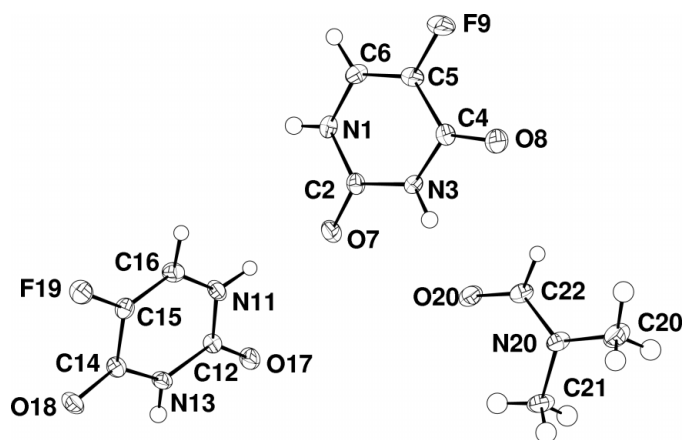


Figure 1

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

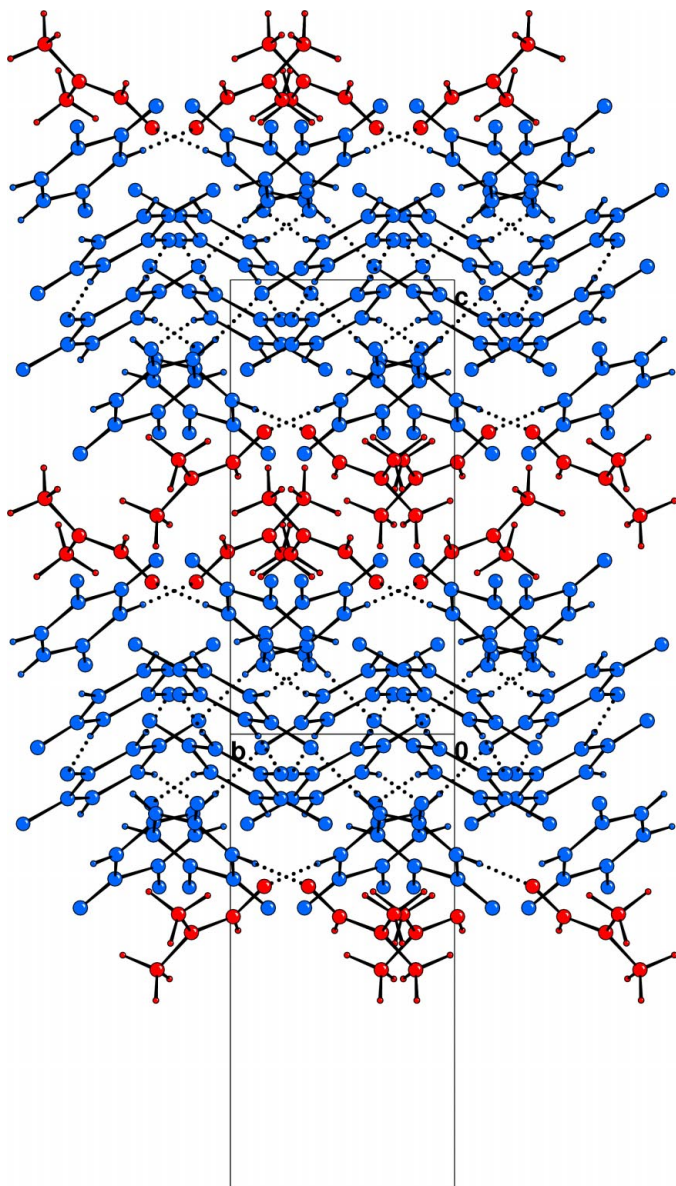


Figure 2

View parallel to the plane of two sheets of the structure. 5-Fluorouracil molecules are coloured blue, while the DMF molecules are red. Dashed lines indicate hydrogen bonds.

2.7962 (18) Å] hydrogen bonds. The DMF molecule forms a hydrogen bond to one of the 5-fluorouracil molecules in the asymmetric unit [N3—H3···O20, 2.7518 (19) Å]. A further two N—H···O hydrogen bonds link 5-fluorouracil molecules in the crystal structure; these bonds are N1—H1···O17ⁱ [N···O = 2.8205 (19) Å], and N13—H13···O18ⁱⁱ [N···O = 2.8203 (18) Å]. The N13—H13···O18ⁱⁱ hydrogen bonds link the 5-fluorouracil molecules into a centrosymmetric hydrogen-bonded dimer.

This solvate exhibits a sheet structure, in which the sheet has a discrete thickness of approximately 13.9 Å in the direction perpendicular to the plane of the sheet, and stacks parallel to the (10 $\bar{1}$) planes. The DMF molecules are present on both surfaces of the sheet with 5-fluorouracil molecules within this sheath of DMF molecules. The parallel sheets

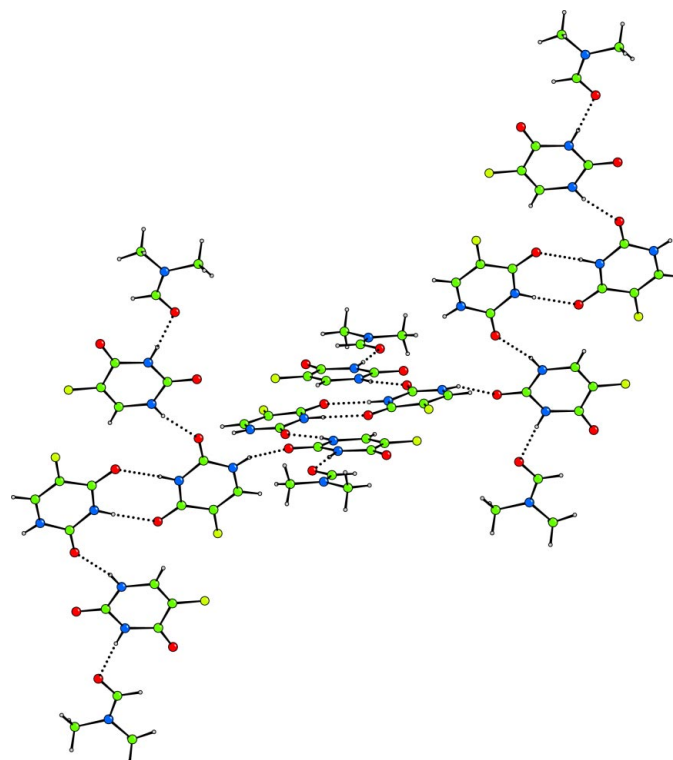


Figure 3

Three adjacent ribbons of a larger sheet, showing an alternating crossed orientation. Other ribbons stack parallel to each of the ribbons in the diagram. Dashed lines indicate hydrogen bonds.

approach each other closely, though there are no short intermolecular contacts between DMF molecules in adjacent sheets. Within the sheets the 5-fluorouracil molecules do not lie parallel to each other, but form a series of smaller blocks of parallel ribbons, as shown in Fig. 2. These ribbons are finite in length and are terminated by surface DMF molecules. Each of these ribbons is *ca* 26.8 Å long (Fig. 3).

Experimental

5-Fluorouracil was obtained from the Aldrich Chemical Company Inc. The crystals were grown by vapour diffusion of diethyl ether into a saturated solution of 5-fluorouracil in dimethylformamide.

Crystal data

$2C_4H_3FN_2O_2 \cdot C_3H_7NO$
 $M_r = 333.26$
 Monoclinic, $P2_1/n$
 $a = 14.7361$ (18) Å
 $b = 5.8693$ (7) Å
 $c = 16.397$ (2) Å
 $\beta = 100.524$ (2)°
 $V = 1394.3$ (3) Å³
 $Z = 4$

$D_x = 1.588$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2752 reflections
 $\theta = 2.5$ – 27.7°
 $\mu = 0.14$ mm⁻¹
 $T = 150$ (2) K
 Block, colourless
 $0.42 \times 0.21 \times 0.11$ mm

Data collection

Bruker SMART APEX diffractometer
 Narrow-frame ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.942$, $T_{max} = 0.984$
 11 701 measured reflections

3331 independent reflections
 2768 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.032$
 $\theta_{max} = 28.3^\circ$
 $h = -19 \rightarrow 19$
 $k = -7 \rightarrow 7$
 $l = -21 \rightarrow 20$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.108$
 $S = 1.10$
 3331 reflections
 238 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.5804P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1 \cdots O17^i$	0.83 (2)	2.01 (2)	2.8205 (19)	167 (2)
$N3-H3 \cdots O20$	0.85 (2)	1.90 (2)	2.7518 (19)	176 (2)
$N11-H11 \cdots O7$	0.86 (2)	1.97 (2)	2.7962 (18)	160 (2)
$N13-H13 \cdots O18^{ii}$	0.86 (2)	1.96 (2)	2.8203 (18)	175 (2)

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

The methyl H atoms were placed in geometrically idealized positions ($C-H = 0.98 \text{ \AA}$) and allowed to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. All other H atoms were located in a difference

map and were refined isotropically; N–H and C–H distances were in the range 0.83 (2)–0.86 (2) and 0.94 (2)–0.97 (2) \AA , respectively.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINTE* (Bruker, 1998); data reduction: *SAINTE*; 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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