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4-Amino-5-fluoropyrimidin-2(1H)-one–2-amino-5-fluoropyrimidin-4(3H)-one–water (1/1/1)

Ashley T. Hulme* and Derek A. Tocher

Christopher Ingold Laboratory, Department of Chemistry, 20 Gordon St., London WC1H 0AJ, England

Correspondence e-mail: a.hulme@ucl.ac.uk

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Key indicators

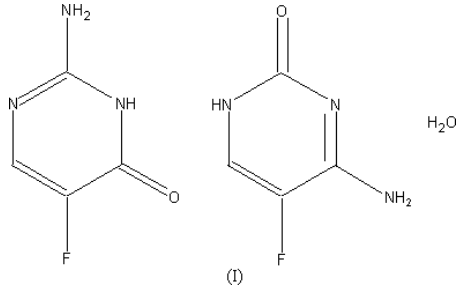
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.123
Data-to-parameter ratio = 11.3

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

The title co-crystal, $C_4H_4FN_3O\cdot C_4H_4FN_3O\cdot H_2O$, has one molecule of 4-amino-5-fluoropyrimidin-2(1*H*)-one, one molecule of its isomer 2-amino-5-fluoropyrimidin-4(3*H*)-one and a molecule of water in the asymmetric unit. 4-Amino-5-fluoropyrimidin-2(1H)-one is commonly known as 5-fluorocytosine.

Comment

The title co-crystal, (I) (Fig. 1), was grown by evaporation of a 50% aqueous solution of ethanol saturated with 5-fluorocytosine. Two different crystal forms were obtained from this solution. The major crystallisation product exhibited a block morphology and was the known monohydrate of 5-fluorocytosine (Louis *et al.*, 1982). A small number of needle-shaped crystals were observed as the minor crystallization product. These crystals proved to be the co-crystal, (I). The isomer of 5-fluorocytosine was assumed to have been present in the commercial sample of 5-fluorocytosine purchased from Fluorochem (98% pure, Old Glossop, UK) that was used to prepare the initial solution.



(I)

The simplest hydrogen-bonded subunit observed is a two-molecule unit, containing one molecule of each isomer. Each molecule of 5-fluorocytosine forms three hydrogen bonds to a

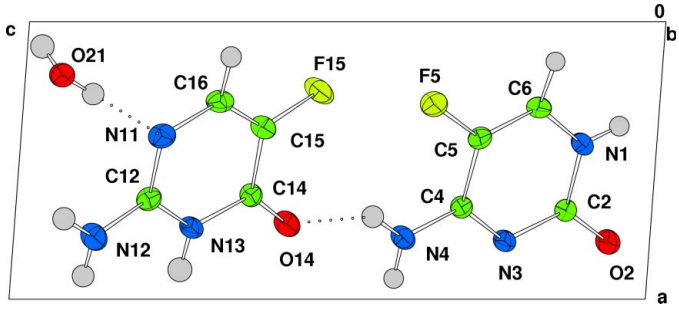


Figure 1
The asymmetric unit of the title co-crystal. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres. Dotted lines indicate hydrogen bonds.

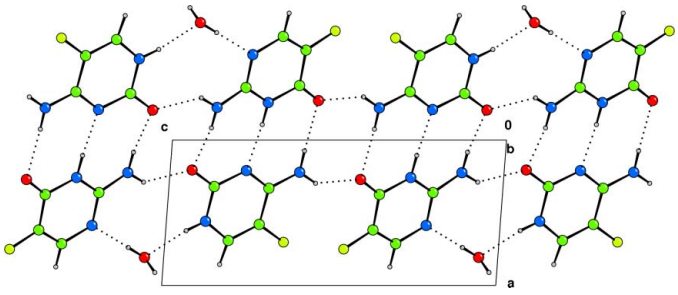


Figure 2
The hydrogen bonded ribbon present in the title structure. Dotted lines indicate hydrogen bonds.

molecule of the isomer (N4—H2···O14, N13—H13···N3 and N12—H12···O2), forming two adjoining $R_2^2(8)$ hydrogen bond rings (Table 1). Two different $R_2^4(8)$ hydrogen-bond rings join these subunits together to form a ribbon (Fig. 2).

The role of the water molecules in the structure is to join together the ribbons into a hydrogen-bonded sheet. The water hydrogen bonds to two molecules from one ribbon, acting both as donor and acceptor, and as a donor to a third molecule, from a different ribbon (Table 1). The ribbons form stepped sheets, parallel to the $01\bar{1}$ planes (Fig. 3).

Within the ribbon structure, there is also a close F···F contact, between F5 and F15, of 2.9003 (15) Å; however, this is likely to have arisen as a consequence of the adjacent $R_2^4(8)$ hydrogen-bond ring.

Experimental

Crystals were grown from a 50% aqueous ethanol solution, by evaporation at room temperature. The crystal form reported was the minor crystallisation product.

Crystal data

$C_4H_4FN_3O\cdot C_4H_4FN_3O\cdot H_2O$
M_r = 276.22
Triclinic, $P\bar{1}$
a = 5.4122 (16) Å
b = 8.447 (2) Å
c = 12.083 (4) Å
α = 89.454 (5)°
β = 85.718 (5)°
γ = 77.096 (4)°
V = 536.9 (3) Å^3
Z = 2
D_x = 1.708 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1511 reflections
θ = 3.0–28.1°
μ = 0.16 mm^{-1}
T = 150 (2) K
Needle, colourless
0.44 × 0.14 × 0.11 mm

Data collection

Bruker SMART APEX diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.934, T_{max} = 0.984
4532 measured reflections
2405 independent reflections
1884 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 28.3°
$h = -6 \rightarrow 6$
$k = -11 \rightarrow 10$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.123
S = 1.05
2405 reflections
212 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 0.0364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

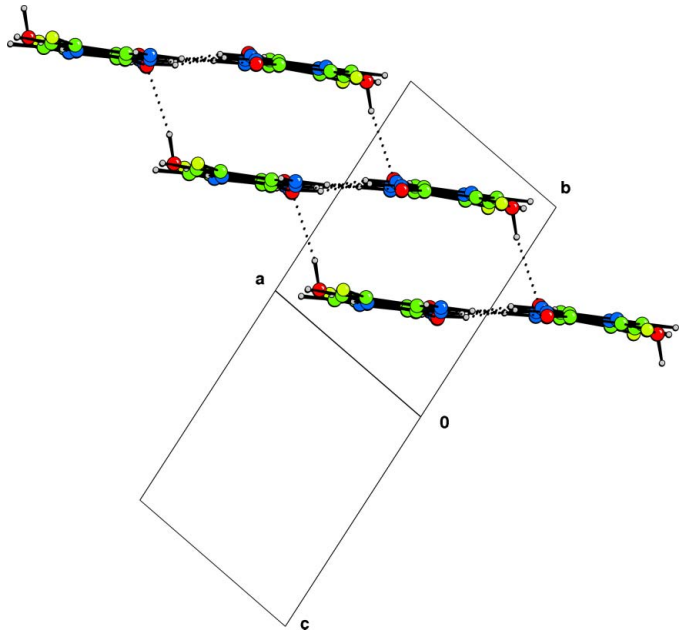


Figure 3
The stepped structure of the sheet, comprising ribbons which are hydrogen bonded (dotted lines) *via* water molecules.

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

D—H···A	D—H	H···A	D···A	D—H···A
N12—H11···O2^{i}	0.86 (3)	2.13 (3)	2.870 (2)	143 (2)
N12—H12···O2^{ii}	0.91 (2)	1.99 (2)	2.889 (2)	172 (2)
N13—H13···N3^{ii}	0.91 (3)	2.01 (3)	2.922 (2)	175 (2)
N1—H1···O21^{iii}	0.83 (2)	1.95 (2)	2.775 (2)	173 (2)
N4—H2···O14^{ii}	0.88 (2)	2.07 (2)	2.9482 (19)	177 (2)
N4—H3···O14	0.91 (3)	2.01 (2)	2.8285 (19)	149 (2)
O21—H21···N11	0.84 (3)	1.94 (3)	2.785 (2)	177 (2)
O21—H22···O2^{iv}	0.81 (3)	2.03 (3)	2.826 (2)	170 (2)

Symmetry codes: (i) $x, y+1, z+1$; (ii) $-x+2, -y, -z+1$; (iii) $x, y-1, z-1$; (iv) $x-1, y+1, z+1$.

All H atoms were located [C—H = 0.95 (2)–0.96 (2), N—H = 0.83 (2)–0.91 (3) and O—H = 0.95 (2)] in a difference map and were refined isotropically.

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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Crystal data

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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.4122\ (16)$ Å
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$\alpha = 89.454\ (5)°$
$\beta = 85.718\ (5)°$
$\gamma = 77.096\ (4)°$
$V = 536.9\ (3)$ Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.708$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1511 reflections
$\theta = 3.0–28.1°$
$\mu = 0.16$ mm^{-1}
$T = 150$ K
Lathe, colourless
0.44 × 0.14 × 0.11 mm

Data collection

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Radiation source: fine-focus sealed tube
Graphite monochromator
ω rotation with narrow frames scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
$T_{min} = 0.934$, $T_{max} = 0.984$
4532 measured reflections
2405 independent reflections
1884 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.7°$
$h = -6\rightarrow6$
$k = -11\rightarrow10$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.123$
$S = 1.05$
2405 reflections
212 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0762P)^2 + 0.0364P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.36$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
F15	0.24930 (19)	0.40288 (12)	0.53690 (9)	0.0329 (3)
O14	0.7285 (2)	0.21415 (14)	0.57184 (10)	0.0266 (3)
N11	0.4124 (3)	0.59592 (17)	0.77839 (12)	0.0252 (3)
N12	0.7754 (3)	0.5260 (2)	0.87373 (13)	0.0299 (4)
H11	0.710 (4)	0.600 (3)	0.923 (2)	0.045 (6)*
H12	0.917 (4)	0.449 (3)	0.8860 (19)	0.044 (6)*
N13	0.7418 (3)	0.37337 (17)	0.72161 (11)	0.0216 (3)
H13	0.894 (5)	0.306 (3)	0.735 (2)	0.053 (7)*
C12	0.6385 (3)	0.50003 (19)	0.79159 (14)	0.0217 (3)
C14	0.6254 (3)	0.33173 (19)	0.63165 (13)	0.0202 (3)
H14	0.127 (4)	0.631 (3)	0.6791 (18)	0.038 (6)*
C15	0.3812 (3)	0.4364 (2)	0.62024 (14)	0.0230 (4)
C16	0.2874 (3)	0.5612 (2)	0.69146 (15)	0.0246 (4)
F5	0.29725 (18)	0.17388 (12)	0.35499 (8)	0.0297 (3)
O2	0.7937 (2)	−0.28047 (14)	0.06559 (9)	0.0270 (3)
N1	0.4410 (3)	−0.08912 (17)	0.11725 (12)	0.0222 (3)
H1	0.376 (4)	−0.113 (3)	0.0611 (19)	0.036 (6)*
N3	0.7899 (2)	−0.14513 (16)	0.22807 (11)	0.0205 (3)
N4	0.7783 (3)	0.00182 (18)	0.38789 (12)	0.0250 (3)
H2	0.925 (4)	−0.065 (3)	0.3978 (17)	0.035 (6)*
H3	0.703 (4)	0.082 (3)	0.438 (2)	0.044 (6)*
C2	0.6802 (3)	−0.17508 (19)	0.13548 (13)	0.0201 (3)
C4	0.6684 (3)	−0.02817 (19)	0.29956 (13)	0.0195 (3)
C5	0.4193 (3)	0.06105 (19)	0.27865 (13)	0.0214 (4)
C6	0.3091 (3)	0.0281 (2)	0.18955 (14)	0.0229 (4)
H4	0.144 (4)	0.079 (2)	0.1689 (17)	0.033 (5)*
O21	0.1925 (2)	0.82377 (16)	0.94260 (11)	0.0270 (3)
H21	0.261 (4)	0.757 (3)	0.892 (2)	0.043 (6)*
H22	0.089 (5)	0.783 (3)	0.977 (2)	0.051 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F15	0.0266 (5)	0.0369 (6)	0.0339 (6)	−0.0006 (4)	−0.0139 (4)	−0.0056 (5)
O14	0.0257 (6)	0.0249 (6)	0.0269 (7)	0.0010 (5)	−0.0069 (5)	−0.0079 (5)
N11	0.0226 (7)	0.0216 (7)	0.0285 (8)	0.0006 (6)	−0.0002 (6)	−0.0039 (6)

N12	0.0289 (8)	0.0285 (8)	0.0279 (8)	0.0050 (7)	−0.0069 (6)	−0.0128 (7)
N13	0.0181 (7)	0.0224 (7)	0.0222 (7)	0.0010 (6)	−0.0048 (5)	−0.0033 (6)
C12	0.0215 (8)	0.0187 (8)	0.0234 (8)	−0.0020 (6)	0.0009 (6)	−0.0031 (6)
C14	0.0209 (8)	0.0192 (8)	0.0198 (8)	−0.0031 (6)	−0.0019 (6)	−0.0012 (6)
C15	0.0205 (8)	0.0238 (8)	0.0247 (8)	−0.0040 (6)	−0.0055 (6)	0.0007 (7)
C16	0.0188 (8)	0.0225 (8)	0.0300 (9)	0.0007 (6)	−0.0019 (7)	0.0025 (7)
F5	0.0241 (5)	0.0311 (6)	0.0284 (5)	0.0057 (4)	−0.0014 (4)	−0.0101 (4)
O2	0.0236 (6)	0.0320 (7)	0.0237 (6)	−0.0013 (5)	−0.0037 (5)	−0.0104 (5)
N1	0.0191 (7)	0.0273 (7)	0.0196 (7)	−0.0027 (6)	−0.0055 (5)	−0.0009 (6)
N3	0.0180 (6)	0.0220 (7)	0.0199 (7)	−0.0002 (5)	−0.0035 (5)	−0.0028 (5)
N4	0.0216 (7)	0.0265 (7)	0.0230 (7)	0.0046 (6)	−0.0064 (6)	−0.0092 (6)
C2	0.0190 (7)	0.0223 (8)	0.0186 (8)	−0.0038 (6)	−0.0012 (6)	−0.0002 (6)
C4	0.0191 (7)	0.0194 (7)	0.0189 (8)	−0.0021 (6)	−0.0014 (6)	−0.0012 (6)
C5	0.0188 (8)	0.0216 (8)	0.0211 (8)	0.0006 (6)	0.0008 (6)	−0.0025 (6)
C6	0.0168 (7)	0.0262 (8)	0.0241 (8)	−0.0010 (6)	−0.0025 (6)	0.0013 (7)
O21	0.0218 (6)	0.0319 (7)	0.0247 (7)	−0.0004 (5)	−0.0020 (5)	−0.0064 (6)

Geometric parameters (Å, º)

F15—C15	1.3441 (19)	O2—C2	1.2552 (19)
O14—C14	1.235 (2)	N1—C6	1.364 (2)
N11—C12	1.328 (2)	N1—C2	1.368 (2)
N11—C16	1.359 (2)	N1—H1	0.83 (2)
N12—C12	1.330 (2)	N3—C4	1.340 (2)
N12—H11	0.86 (3)	N3—C2	1.356 (2)
N12—H12	0.91 (2)	N4—C4	1.313 (2)
N13—C12	1.361 (2)	N4—H2	0.88 (2)
N13—C14	1.383 (2)	N4—H3	0.91 (3)
N13—H13	0.91 (3)	C4—C5	1.429 (2)
C14—C15	1.432 (2)	C5—C6	1.330 (2)
C15—C16	1.350 (2)	C6—H4	0.95 (2)
C16—H14	0.96 (2)	O21—H21	0.84 (3)
F5—C5	1.3566 (18)	O21—H22	0.81 (3)
C12—N11—C16	116.74 (14)	C6—N1—H1	120.8 (15)
C12—N12—H11	119.5 (16)	C2—N1—H1	117.5 (15)
C12—N12—H12	117.7 (15)	C4—N3—C2	120.27 (13)
H11—N12—H12	121 (2)	C4—N4—H2	115.2 (14)
C12—N13—C14	124.20 (14)	C4—N4—H3	122.4 (14)
C12—N13—H13	120.2 (16)	H2—N4—H3	122 (2)
C14—N13—H13	115.5 (16)	O2—C2—N3	121.40 (14)
N11—C12—N12	120.83 (15)	O2—C2—N1	118.93 (15)
N11—C12—N13	122.00 (15)	N3—C2—N1	119.67 (14)
N12—C12—N13	117.17 (14)	N4—C4—N3	119.75 (14)
O14—C14—N13	121.03 (14)	N4—C4—C5	121.07 (15)
O14—C14—C15	126.50 (15)	N3—C4—C5	119.18 (15)
N13—C14—C15	112.47 (14)	C6—C5—F5	121.80 (14)
F15—C15—C16	121.88 (14)	C6—C5—C4	120.43 (15)

F15—C15—C14	116.98 (14)	F5—C5—C4	117.71 (14)
C16—C15—C14	121.13 (15)	C5—C6—N1	118.72 (15)
C15—C16—N11	123.46 (15)	C5—C6—H4	126.9 (13)
C15—C16—H14	118.6 (14)	N1—C6—H4	114.3 (13)
N11—C16—H14	117.9 (14)	H21—O21—H22	106 (2)
C6—N1—C2	121.68 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N12—H11···O2^{i}	0.86 (3)	2.13 (3)	2.870 (2)	143 (2)
N12—H12···O2^{ii}	0.91 (2)	1.99 (2)	2.889 (2)	172 (2)
N13—H13···N3^{ii}	0.91 (3)	2.01 (3)	2.922 (2)	175 (2)
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