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6-(Trifluoromethyl)pyrimidine-2,4(1*H*,3*H*)-dione monohydrate

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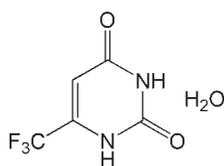
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 Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.036; wR factor = 0.100; data-to-parameter ratio = 13.0.

The title compound, $\text{C}_5\text{H}_3\text{F}_3\text{N}_2\text{O}_2 \cdot \text{H}_2\text{O}$, was prepared by the reaction of ethyl 4,4,4-trifluoro-3-oxobutanoate with urea. In the crystal, the 6-(trifluoromethyl)pyrimidine-2,4(1*H*,3*H*)-dione and water molecules are linked by $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds. A ring dimer structure is formed by additional intermolecular $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds.

Related literature

For applications of pyrimidine derivatives as pesticides and pharmaceutical agents, see: Condon *et al.* (1993); as agrochemicals, see: Maeno *et al.* (1990); as antiviral agents, see: Gilchrist (1997); as herbicides, see: Selby *et al.* (2002).



Experimental

Crystal data

 $\text{C}_5\text{H}_3\text{F}_3\text{N}_2\text{O}_2 \cdot \text{H}_2\text{O}$
 $M_r = 198.11$

 Monoclinic, $P2_1/c$
 $a = 5.0250$ (8) Å
 $b = 7.046$ (1) Å
 $c = 20.769$ (2) Å
 $\beta = 91.300$ (7)°

 $V = 735.16$ (17) Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.19$ mm⁻¹
 $T = 113$ K
 $0.24 \times 0.20 \times 0.18$ mm

Data collection

 Rigaku Saturn724 CCD diffractometer
 Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku/MS, 2009)
 $T_{\min} = 0.956$, $T_{\max} = 0.966$

 6863 measured reflections
 1747 independent reflections
 1382 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.100$
 $S = 1.07$
 1747 reflections
 134 parameters

 H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O3}-\text{H3B} \cdots \text{O1}^i$	0.825 (17)	2.017 (18)	2.7815 (13)	153.9 (17)
$\text{O3}-\text{H3A} \cdots \text{O2}^{ii}$	0.86 (2)	1.95 (2)	2.8066 (13)	176.0 (17)
$\text{N2}-\text{H2} \cdots \text{O3}$	0.896 (17)	1.824 (17)	2.7191 (14)	177.9 (16)
$\text{N1}-\text{H1} \cdots \text{O1}^{iii}$	0.954 (17)	1.896 (18)	2.8490 (14)	176.4 (16)

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

Data collection: *CrystalClear-SM Expert* (Rigaku/MS, 2009); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku/MS, 2009); software used to prepare material for publication: *CrystalStructure*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2309).

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supporting information

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6-(Trifluoromethyl)pyrimidine-2,4(1*H*,3*H*)-dione monohydrate**Gong-Chun Li, Hong-Sheng Wang, Yu-Jiao Niu and Feng-Ling Yang****S1. Comment**

Pyrimidine derivatives are very important molecules in biology and have many application in the areas of pesticide and pharmaceutical agents (Condon *et al.*, 1993). For example, imazosulfuron, ethirmol and mepanipyrim have been commercialized as agrochemicals (Maeno *et al.*, 1990). Pyrimidine derivatives have also been developed as antiviral agents, such as AZT, which is the most widely used anti-AIDS drug (Gilchrist, 1997). Recently, a new series of highly active herbicides of substituted azolopyrimidines were reported (Selby *et al.*, 2002). In order to discover further biologically active pyrimidine compounds, the title compound, (I), was synthesized and its crystal structure determined (Fig. 1).

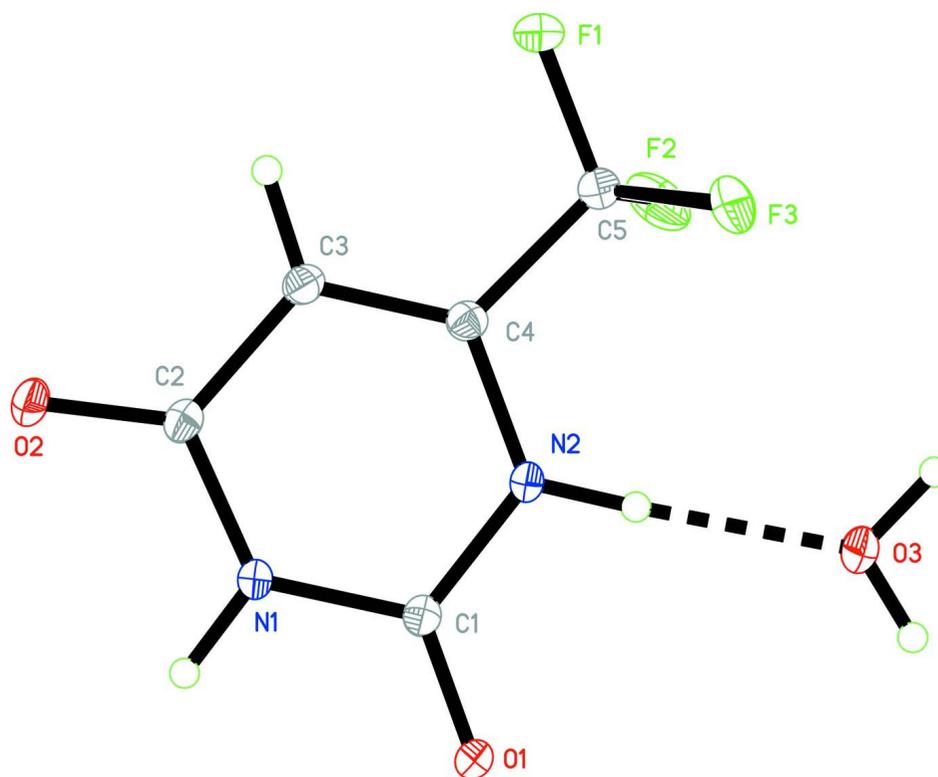
In the crystal structure, The part of 6-(trifluoromethyl)pyrimidine-2,4(1*H*,3*H*)-dione and water molecule are linked by N—H \cdots O and O—H \cdots O hydrogen bonds. The ring dimer structure is formed by addition intermolecular N—H \cdots O hydrogen bonds.

S2. Experimental

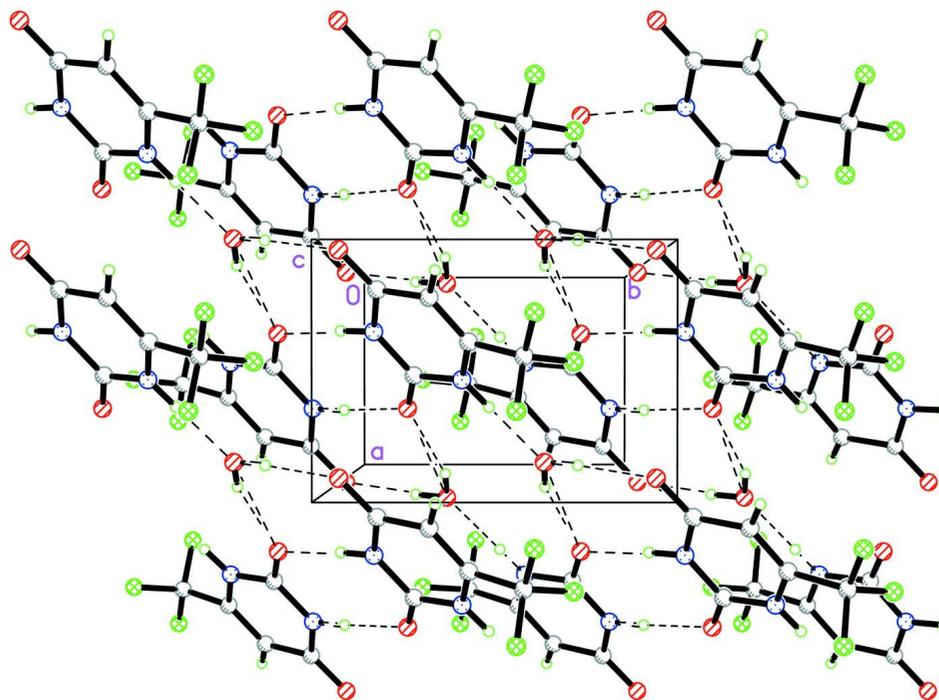
To 35 ml absolute ethanol sodium (1.38 g, 60 mmol) was added. When sodium was disappeared, ethyl 4,4,4-trifluoro-3-oxobutanoate(5.50 g, 30 mmol) and urea (1.80 g, 30 mmol) were added to the solution. The mixture was refluxed for 20 hr., The solvent was evaporated *in vacuo* and the residue was washed with water. The title compound was recrystallized from water and single crystals of (I) were obtained by slow evaporation.

S3. Refinement

All H atoms were placed in calculated positions, with C—H = 0.95 Å, O—H = 0.86 Å or 0.825 Å, and included in the final cycles of refinement using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

**Figure 1**

The asymmetric unit of the title compound, (I), with displacement ellipsoids drawn at the 30% probability level.

**Figure 2**

The packing diagram of the title compound. Intermolecular hydrogen bonds are shown as dashed line.

6-(Trifluoromethyl)pyrimidine-2,4(1*H*,3*H*)-dione monohydrate

Crystal data

C₅H₃F₃N₂O₂·H₂O $M_r = 198.11$ Monoclinic, $P2_1/c$ $a = 5.0250$ (8) Å $b = 7.046$ (1) Å $c = 20.769$ (2) Å $\beta = 91.300$ (7)° $V = 735.16$ (17) Å³ $Z = 4$ $F(000) = 400$ $D_x = 1.790$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å

Cell parameters from 2492 reflections

 $\theta = 2.0$ – 27.9 ° $\mu = 0.19$ mm⁻¹ $T = 113$ K

Prism, colorless

 $0.24 \times 0.20 \times 0.18$ mm

Data collection

Rigaku Saturn724 CCD

diffractometer

Radiation source: rotating anode

Multilayer monochromator

 ω scans

Absorption correction: multi-scan

(CrystalClear-SM Expert; Rigaku/MSO, 2009)

 $T_{\min} = 0.956$, $T_{\max} = 0.966$

6863 measured reflections

1747 independent reflections

1382 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.029$ $\theta_{\max} = 27.9$ °, $\theta_{\min} = 2.0$ ° $h = -6 \rightarrow 6$ $k = -6 \rightarrow 9$ $l = -27 \rightarrow 27$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.036$ $wR(F^2) = 0.100$ $S = 1.07$

1747 reflections

134 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.0166P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.33$ e Å⁻³ $\Delta\rho_{\min} = -0.21$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.74748 (17)	0.37877 (12)	0.26992 (4)	0.0431 (3)
F2	0.33139 (17)	0.43344 (12)	0.28202 (4)	0.0433 (3)
F3	0.54045 (16)	0.25207 (11)	0.34843 (4)	0.0359 (2)
O1	0.32964 (16)	0.78481 (12)	0.48978 (4)	0.0247 (2)

O2	1.03039 (17)	0.99065 (12)	0.37275 (4)	0.0261 (2)
O3	0.09114 (19)	0.33980 (15)	0.43644 (5)	0.0299 (3)
N1	0.67605 (19)	0.88773 (14)	0.42979 (5)	0.0199 (2)
N2	0.46193 (19)	0.60395 (14)	0.40583 (5)	0.0187 (2)
C1	0.4798 (2)	0.76007 (17)	0.44449 (5)	0.0191 (3)
C2	0.8566 (2)	0.87123 (17)	0.38039 (6)	0.0193 (3)
C3	0.8191 (2)	0.70430 (17)	0.34028 (6)	0.0196 (3)
H3	0.9296	0.6828	0.3045	0.023*
C4	0.6262 (2)	0.58089 (16)	0.35448 (5)	0.0183 (3)
C5	0.5641 (2)	0.40964 (18)	0.31352 (6)	0.0237 (3)
H1	0.681 (3)	0.999 (2)	0.4561 (9)	0.048 (5)*
H2	0.340 (3)	0.518 (2)	0.4169 (8)	0.041 (5)*
H3A	0.081 (3)	0.234 (3)	0.4166 (10)	0.049 (5)*
H3B	-0.024 (3)	0.336 (3)	0.4642 (8)	0.043 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0459 (5)	0.0380 (5)	0.0467 (5)	-0.0137 (4)	0.0277 (4)	-0.0219 (4)
F2	0.0400 (5)	0.0422 (6)	0.0467 (5)	0.0051 (4)	-0.0173 (4)	-0.0201 (4)
F3	0.0481 (5)	0.0177 (4)	0.0423 (5)	-0.0059 (4)	0.0095 (4)	-0.0025 (3)
O1	0.0267 (5)	0.0250 (5)	0.0229 (4)	-0.0091 (4)	0.0097 (4)	-0.0050 (3)
O2	0.0237 (5)	0.0225 (5)	0.0325 (5)	-0.0076 (4)	0.0092 (4)	-0.0012 (4)
O3	0.0307 (5)	0.0248 (6)	0.0347 (6)	-0.0110 (4)	0.0140 (4)	-0.0049 (4)
N1	0.0204 (5)	0.0192 (6)	0.0204 (5)	-0.0058 (4)	0.0042 (4)	-0.0025 (4)
N2	0.0191 (5)	0.0165 (5)	0.0208 (5)	-0.0046 (4)	0.0043 (4)	-0.0009 (4)
C1	0.0190 (6)	0.0193 (6)	0.0190 (5)	-0.0028 (4)	0.0014 (4)	0.0000 (5)
C2	0.0176 (5)	0.0187 (6)	0.0215 (6)	-0.0006 (5)	0.0023 (4)	0.0028 (4)
C3	0.0192 (6)	0.0195 (7)	0.0201 (6)	0.0010 (5)	0.0039 (4)	0.0010 (4)
C4	0.0185 (5)	0.0174 (6)	0.0191 (6)	0.0019 (4)	0.0013 (4)	0.0007 (5)
C5	0.0227 (6)	0.0216 (7)	0.0270 (6)	-0.0019 (5)	0.0064 (5)	-0.0038 (5)

Geometric parameters (Å, °)

F1—C5	1.3245 (14)	N1—H1	0.954 (17)
F2—C5	1.3374 (15)	N2—C1	1.3636 (15)
F3—C5	1.3328 (15)	N2—C4	1.3729 (14)
O1—C1	1.2317 (14)	N2—H2	0.896 (17)
O2—C2	1.2255 (14)	C2—C3	1.4512 (17)
O3—H3A	0.86 (2)	C3—C4	1.3400 (17)
O3—H3B	0.825 (17)	C3—H3	0.9500
N1—C1	1.3742 (15)	C4—C5	1.5050 (17)
N1—C2	1.3898 (15)		
H3A—O3—H3B	105.8 (17)	C4—C3—C2	119.01 (11)
C1—N1—C2	126.37 (10)	C4—C3—H3	120.5
C1—N1—H1	114.8 (11)	C2—C3—H3	120.5
C2—N1—H1	118.8 (11)	C3—C4—N2	123.00 (11)

C1—N2—C4	121.34 (10)	C3—C4—C5	122.52 (11)
C1—N2—H2	115.5 (11)	N2—C4—C5	114.42 (10)
C4—N2—H2	123.2 (11)	F1—C5—F3	107.89 (10)
O1—C1—N2	122.04 (10)	F1—C5—F2	107.48 (10)
O1—C1—N1	122.15 (11)	F3—C5—F2	106.44 (10)
N2—C1—N1	115.80 (10)	F1—C5—C4	112.30 (10)
O2—C2—N1	121.15 (11)	F3—C5—C4	112.34 (10)
O2—C2—C3	124.46 (11)	F2—C5—C4	110.10 (10)
N1—C2—C3	114.39 (10)		
C4—N2—C1—O1	178.24 (10)	C2—C3—C4—C5	176.73 (10)
C4—N2—C1—N1	-1.87 (16)	C1—N2—C4—C3	2.53 (18)
C2—N1—C1—O1	179.13 (11)	C1—N2—C4—C5	-174.89 (10)
C2—N1—C1—N2	-0.76 (17)	C3—C4—C5—F1	10.94 (17)
C1—N1—C2—O2	-177.49 (11)	N2—C4—C5—F1	-171.62 (10)
C1—N1—C2—C3	2.59 (16)	C3—C4—C5—F3	132.78 (12)
O2—C2—C3—C4	178.20 (11)	N2—C4—C5—F3	-49.78 (14)
N1—C2—C3—C4	-1.88 (16)	C3—C4—C5—F2	-108.79 (13)
C2—C3—C4—N2	-0.49 (18)	N2—C4—C5—F2	68.65 (13)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O3—H3 <i>B</i> ...O1 ⁱ	0.825 (17)	2.017 (18)	2.7815 (13)	153.9 (17)
O3—H3 <i>A</i> ...O2 ⁱⁱ	0.86 (2)	1.95 (2)	2.8066 (13)	176.0 (17)
N2—H2...O3	0.896 (17)	1.824 (17)	2.7191 (14)	177.9 (16)
N1—H1...O1 ⁱⁱⁱ	0.954 (17)	1.896 (18)	2.8490 (14)	176.4 (16)

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