Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

6-Ethyl-5-fluoro-2-methoxypyrimidin-4(3*H*)-one

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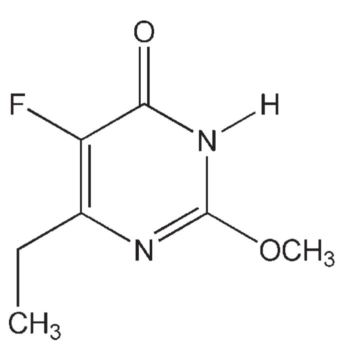
Received 23 August 2009; accepted 2 September 2009

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.106; data-to-parameter ratio = 16.6.

In the title compound, $C_7H_9FN_2O_2$, the methoxy and ethyl groups form dihedral angles of 1.4 (2) and 73.5 (3)°, respectively, with the mean plane of the pyrimidine ring. In the crystal structure, two molecules are linked by a pair of N—H···O hydrogen bonds, forming a centrosymmetric dimer.

Related literature

For fluoro-containing pyrimidines as intermediates for the synthesis of some anticancer and antifungal drugs, see: Bergmann *et al.* (1959); Butters *et al.* (2001).



Experimental

Crystal data

$C_7H_9FN_2O_2$
M_r = 172.16
Triclinic, $P\overline{1}$
a = 4.5711 (4) Å
b = 8.4985 (8) Å
c = 10.8546 (11) Å
α = 88.043 (2)°
β = 79.737 (3)°
γ = 79.616 (2)°
V = 408.13 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 296 K
0.40 × 0.28 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995) T_{min} = 0.948, T_{max} = 0.979
4010 measured reflections
1842 independent reflections
945 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.106
S = 1.00
1842 reflections
111 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.86	1.91	2.763 (2)	174

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *PROCESS-AUTO* (Rigaku/MSC, 2004); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2004).

The authors acknowledge support from the Educational Commission of Zhejiang Province (200803289).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2454).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2582 [doi:10.1107/S1600536809035430]

6-Ethyl-5-fluoro-2-methoxypyrimidin-4(3*H*)-one

Yu-Yuan Ye and Kai Yang

S1. Comment

The fluoro-containing pyrimidines have been used as a kind of important intermediates for the synthesis of some anticancer drugs and antifungal drugs (Bergmann *et al.*, 1959; Butters *et al.*, 2001). In the synthesis of the novel antifungal drug-Voriconazole, we have prepared the title compound 6-ethyl-5-fluoro-2-methoxypyrimidin-4(3*H*)-one as an intermediate, which was synthesized by reacting methyl 2-fluoro-3-oxopentanoate with *o*-methylisourea sulfate in a solution of sodium methylate in methanol.

The molecular structure of the title compound, (I), is illustrated in Fig. 1. The bond lenghth of C4—O2 and C1—O1 are 1.238 (3) and 1.321 (2) Å, respectively, corresponding to a double C=O bond and a Csp^2—O single bond. In the six-membered pyrimidine ring, the even bond lengths of C—N and C—C are 1.361 (3) and 1.380 (3) Å, respectively, indicating these bond forming a conjugating system. The atoms in the pyrimidine ring (C1–C4/N1/N2) form a good plane with a mean deviation of 0.006 Å. An intermolecular N—H···O hydrogen bond was found to link two molecules as a pair (Fig. 2 and Table 1).

S2. Experimental

To a 250 ml flask was added a 80 ml solution of 25% sodium methylate in methanol. The solution was cooled to 278 K, and then 40 g *o*-methylisourea sulfate and 20 g methyl 2-fluoro-3-oxopentanoate were added. After the addition, the mixture were stirred at 298 K for half an hour and refluxed for three hours. The mixture was concentrated under reduced pressure, and the residue was dissolved with 200 ml water. The aqueous solution was treated with 6*M* hydrochloric acid to pH3 and cooled in refrigerator for three hours. The resulted precipitate was filtered, to give 12.5 g product as white powder (yield 53.8%; m.p. 447–449 K). Since the product was not found to be suitable for X-ray diffraction studies, a few samples were dissolved in absolute ethanol, which was allowed to evaporate slowly to give colourless crystals of (I) suitable for X-ray diffraction studies.

S3. Refinement

H atoms were placed in calculated positions (C—H = 0.96–0.97 Å and N—H = 0.86 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C, N) or 1.5U_{eq}(methyl C).

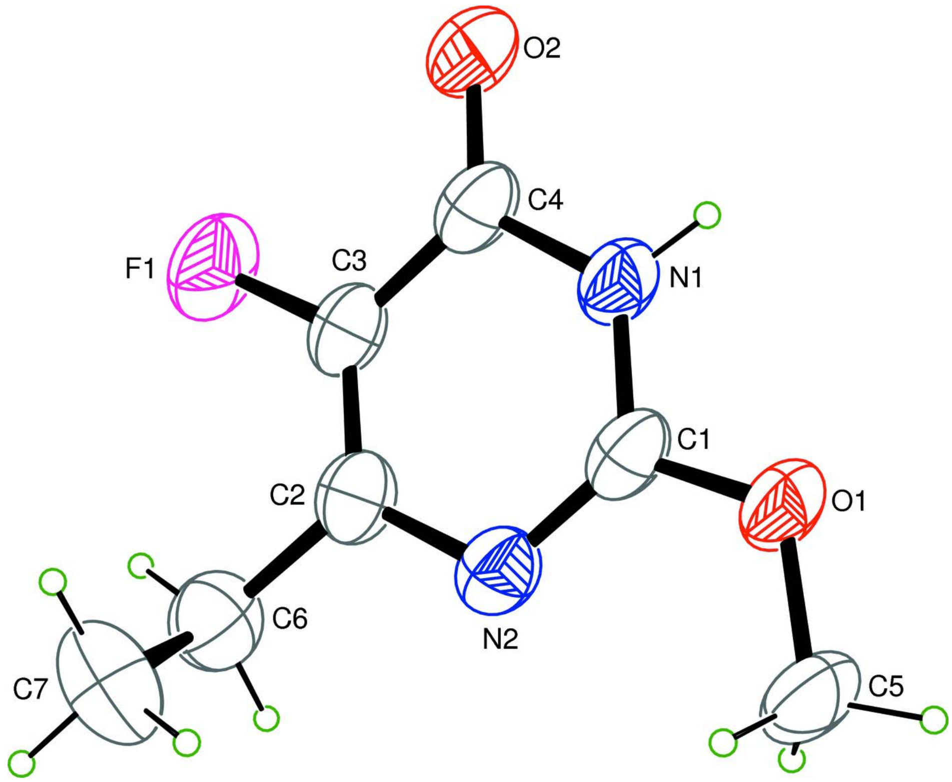

Figure 1

The molecular structure of (I) with 30% probability displacement ellipsoids.

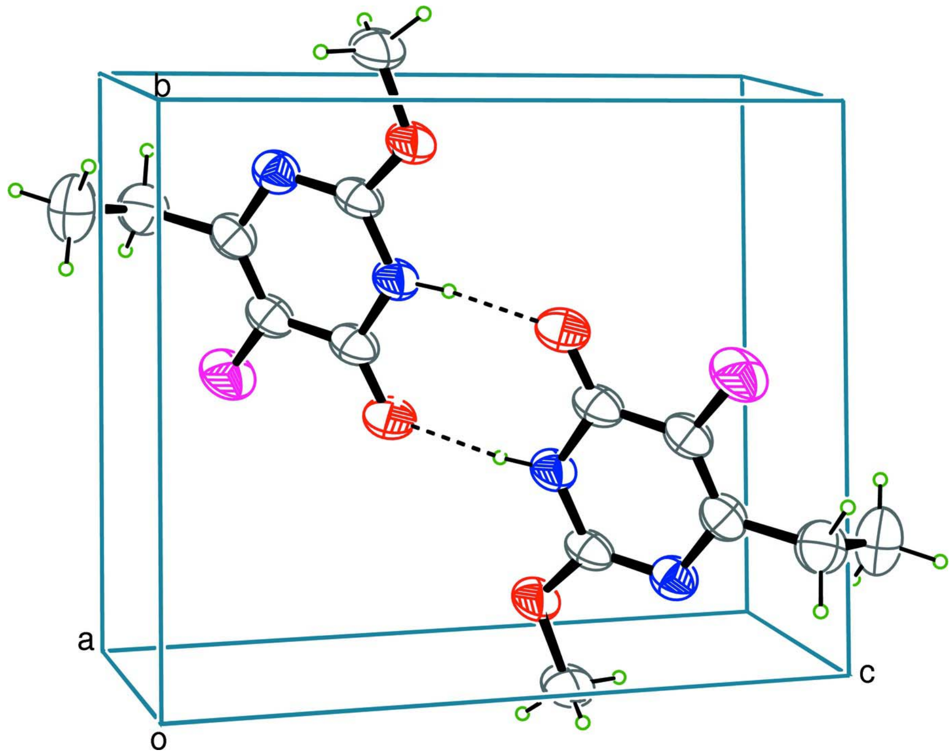

Figure 2

Packing diagram of (I), showing hydrogen bonds as dashed lines.

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Triclinic, $P\overline{1}$
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$a = 4.5711$ (4) Å
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$c = 10.8546$ (11) Å
$\alpha = 88.043$ (2)°
$\beta = 79.737$ (3)°
$\gamma = 79.616$ (2)°
$V = 408.13$ (7) Å^3
$Z = 2$
$F(000) = 180.00$
$D_x = 1.401$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 2411 reflections
$\theta = 3.1$–$27.4°$
$\mu = 0.12$ mm^{-1}
$T = 296$ K
Chunk, colorless
0.40 × 0.28 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.00 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.948$, $T_{max} = 0.979$
4010 measured reflections
1842 independent reflections
945 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 27.4°$
$h = -5 \rightarrow 5$
$k = -10 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.106$
$S = 1.00$
1842 reflections
111 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.P)^2 + 0.345P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.025 (2)

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 using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	−0.2750 (3)	0.5521 (2)	0.78974 (17)	0.0840 (5)
O1	0.6346 (4)	0.0891 (2)	0.60826 (17)	0.0674 (5)
O2	0.1625 (4)	0.6002 (2)	0.58950 (19)	0.0754 (6)
N1	0.3901 (4)	0.3399 (2)	0.6042 (2)	0.0577 (5)
N2	0.2058 (4)	0.1623 (2)	0.7615 (2)	0.0587 (5)
C1	0.4039 (5)	0.1970 (2)	0.6603 (2)	0.0558 (6)
C2	−0.0212 (5)	0.2877 (3)	0.8040 (2)	0.0579 (6)
C3	−0.0446 (5)	0.4310 (3)	0.7475 (2)	0.0590 (7)
C4	0.1656 (6)	0.4689 (3)	0.6427 (2)	0.0603 (7)
C5	0.6683 (7)	−0.0711 (2)	0.6608 (2)	0.0788 (9)
C6	−0.2321 (6)	0.2513 (3)	0.9191 (2)	0.0753 (8)
C7	−0.0903 (8)	0.2402 (4)	1.0339 (2)	0.0997 (11)
H1	0.5276	0.3520	0.5412	0.069*
H51	0.7001	−0.0670	0.7457	0.095*
H52	0.8384	−0.1380	0.6122	0.095*
H53	0.4886	−0.1142	0.6595	0.095*
H61	−0.2930	0.1500	0.9075	0.090*
H62	−0.4085	0.3355	0.9311	0.090*
H71	0.0851	0.1573	1.0231	0.120*
H72	−0.2328	0.2156	1.1050	0.120*
H73	−0.0324	0.3405	1.0473	0.120*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0660 (10)	0.0684 (10)	0.1026 (13)	0.0173 (8)	−0.0007 (9)	−0.0156 (9)
O1	0.0774 (13)	0.0441 (9)	0.0690 (12)	0.0153 (9)	−0.0074 (10)	−0.0028 (8)
O2	0.0822 (14)	0.0447 (10)	0.0864 (14)	0.0146 (9)	−0.0062 (11)	0.0003 (9)
N1	0.0625 (13)	0.0437 (11)	0.0595 (13)	0.0091 (10)	−0.0095 (10)	−0.0023 (10)

N2	0.0594 (13)	0.0517 (12)	0.0629 (14)	−0.0013 (10)	−0.0131 (11)	−0.0034 (10)
C1	0.0606 (16)	0.0431 (13)	0.0625 (16)	0.0074 (11)	−0.0230 (13)	−0.0094 (12)
C2	0.0502 (15)	0.0603 (16)	0.0623 (16)	−0.0031 (12)	−0.0122 (12)	−0.0101 (13)
C3	0.0499 (15)	0.0520 (15)	0.0694 (17)	0.0079 (12)	−0.0097 (13)	−0.0117 (13)
C4	0.0620 (17)	0.0456 (14)	0.0698 (17)	0.0082 (12)	−0.0187 (14)	−0.0083 (13)
C5	0.102 (2)	0.0428 (14)	0.082 (2)	0.0155 (15)	−0.0193 (18)	−0.0009 (14)
C6	0.0602 (18)	0.077 (2)	0.085 (2)	−0.0112 (15)	−0.0033 (16)	−0.0051 (17)
C7	0.091 (2)	0.136 (3)	0.069 (2)	−0.029 (2)	0.0017 (18)	0.004 (2)

Geometric parameters (Å, º)

F1—C3	1.359 (2)	C6—C7	1.496 (4)
O1—C1	1.321 (2)	N1—H1	0.860
O1—C5	1.451 (3)	C5—H51	0.960
O2—C4	1.238 (3)	C5—H52	0.960
N1—C1	1.336 (3)	C5—H53	0.960
N1—C4	1.379 (3)	C6—H61	0.970
N2—C1	1.354 (3)	C6—H62	0.970
N2—C2	1.375 (3)	C7—H71	0.960
C2—C3	1.340 (3)	C7—H72	0.960
C2—C6	1.496 (3)	C7—H73	0.960
C3—C4	1.420 (3)		
C1—O1—C5	118.15 (19)	O1—C5—H51	109.5
C1—N1—C4	123.1 (2)	O1—C5—H52	109.5
C1—N2—C2	114.5 (2)	O1—C5—H53	109.5
O1—C1—N1	113.63 (19)	H51—C5—H52	109.5
O1—C1—N2	121.8 (2)	H51—C5—H53	109.5
N1—C1—N2	124.6 (2)	H52—C5—H53	109.5
N2—C2—C3	122.1 (2)	C2—C6—H61	108.7
N2—C2—C6	114.4 (2)	C2—C6—H62	108.7
C3—C2—C6	123.5 (2)	C7—C6—H61	108.7
F1—C3—C2	121.0 (2)	C7—C6—H62	108.7
F1—C3—C4	115.4 (2)	H61—C6—H62	109.5
C2—C3—C4	123.6 (2)	C6—C7—H71	109.5
O2—C4—N1	121.3 (2)	C6—C7—H72	109.5
O2—C4—C3	126.6 (2)	C6—C7—H73	109.5
N1—C4—C3	112.1 (2)	H71—C7—H72	109.5
C2—C6—C7	112.5 (2)	H71—C7—H73	109.5
C1—N1—H1	118.5	H72—C7—H73	109.5
C4—N1—H1	118.5		
C5—O1—C1—N1	−179.1 (2)	N2—C2—C3—F1	178.6 (2)
C5—O1—C1—N2	1.4 (3)	N2—C2—C3—C4	−2.4 (4)
C1—N1—C4—O2	179.0 (2)	N2—C2—C6—C7	72.7 (3)
C1—N1—C4—C3	−0.0 (3)	C3—C2—C6—C7	−105.8 (3)
C4—N1—C1—O1	179.3 (2)	C6—C2—C3—F1	−3.0 (4)
C4—N1—C1—N2	−1.2 (4)	C6—C2—C3—C4	176.0 (3)

C1—N2—C2—C3	1.1 (4)	F1—C3—C4—O2	1.9 (4)
C1—N2—C2—C6	−177.5 (2)	F1—C3—C4—N1	−179.2 (2)
C2—N2—C1—O1	−179.9 (2)	C2—C3—C4—O2	−177.2 (3)
C2—N2—C1—N1	0.6 (4)	C2—C3—C4—N1	1.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2^{i}	0.86	1.91	2.763 (2)	174

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.