Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4,6-Dimethylpyrimidin-2(1*H*)-one–urea–water (1/1/1)

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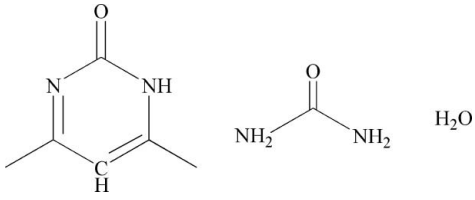
Received 30 May 2008; accepted 7 July 2008

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.171; data-to-parameter ratio = 12.1.

In the crystal structure of the title compound, $C_6H_8N_2O\cdot CH_4N_2O\cdot H_2O$, molecules are linked *via* N—H···O, O—H···N and O—H···O hydrogen bonds, forming a three–dimensional framework.

Related literature

For general background, see: Zhao *et al.*, (2004); Zheng *et al.*, (2005).



Experimental

Crystal data

$C_6H_8N_2O\cdot CH_4N_2O\cdot H_2O$
M_r = 202.22
Triclinic, $P\overline{1}$
a = 8.1246 (5) Å
b = 8.4062 (5) Å
c = 8.9268 (9) Å
α = 105.007 (3)°
β = 103.857 (3)°
γ = 114.379 (2)°
V = 493.05 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
0.22 × 0.16 × 0.11 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.977, T_{max} = 0.998
5424 measured reflections
1728 independent reflections
1439 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.170
S = 1.10
1728 reflections
143 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2	0.92 (3)	1.87 (3)	2.779 (2)	172 (3)
N3—H3A···O1	0.87 (3)	2.09 (3)	2.953 (3)	169 (3)
N3—H3B···O1^{i}	0.83 (3)	2.16 (3)	2.896 (3)	149 (3)
N4—H4A···O2^{ii}	0.93 (3)	2.04 (3)	2.968 (3)	174 (3)
N4—H4B···O1W^{i}	0.83 (3)	2.12 (4)	2.948 (3)	175 (3)
O1W—H1WA···N1	0.850 (11)	2.121 (15)	2.930 (2)	159.1 (13)
O1W—H1WB···O1^{iii}	0.850 (11)	2.209 (11)	3.009 (3)	156.9 (3)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; 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: *WinGX* (Farrugia, 1999).

We acknowledge the support of the National Natural Science Foundation of China (No. 20662003) and the Foundation of the Governor of Guizhou Province, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2094).

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Zhao, Y. J., Xue, S. F., Zhu, Q. J., Tao, Z., Zhang, J. X., Wei, Z. B., Long, L. S., Hu, M. L., Xiao, H. P. & Day, A. I. (2004). *Chin. Sci. Bull.* **49**, 1111–1116.
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supporting information

Acta Cryst. (2008). E**64**, o1488 [doi:10.1107/S1600536808020928]

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Feng Wu, Yun-Qian Zhang, Qian-Jiang Zhu, Sai-Feng Xue and Zhu Tao

S1. Comment

Recent years, we used different alkyl–substituted glycolurils as the building blocks to synthesize the partly alkyl substituted cucurbit[*n*]urils (Zhao *et al.*, 2004; Zheng *et al.*, 2005). In this work, we further report the crystal structure of a pyrimidine–substituted semi–glycoluril.

The crystal structure of the title compound $C_6H_8N_2O{\cdot}CH_4N_2O{\cdot}H_2O$, **I**, consists from 4,6–dimethylpyrimidin molecule, a urea molecule and a lattice water molecule. These molecules are linked *via* N—H···O, O—H···N and O—H···O hydrogen bonds forming a three–dimensional framework (Fig. 2).

S2. Experimental

In a 3–neck flask fitted with water knockout trap and the thermometer, (36 g, 0.6 mol) of urea dissolved in 100 ml of toluene, stirred vigorously, at room temperature. At the same time, (24 g, 0.24 mol) of acetylacetone was added into flask in one portion. The 1.5 ml of trifluoroacetic acid was added too in order to make the value of pH of the solvent is around 4. The reaction mixture was stired and heated and maintained at reflux for 4 h. After cooling to room temperature, the reaction mixture was filtered. The filtrate was concentrated by rotary evaporation at 318–323 K and then maintained overnight at room temperature and crystals of **I** appear (yield: 4.5 g, 0.036 mol, 15%)

S3. Refinement

Water H atoms and H atoms of amino group were located in a difference Fourier map and refined freely with U_{iso}(H) = 1.2U_{eq}(O, N). All H atoms based on C were placed in calculated positions and refined as riding, with C—H = 0.930–0.960Å with U_{iso}(H) = 1.2–1.5 U_{eq}(C).

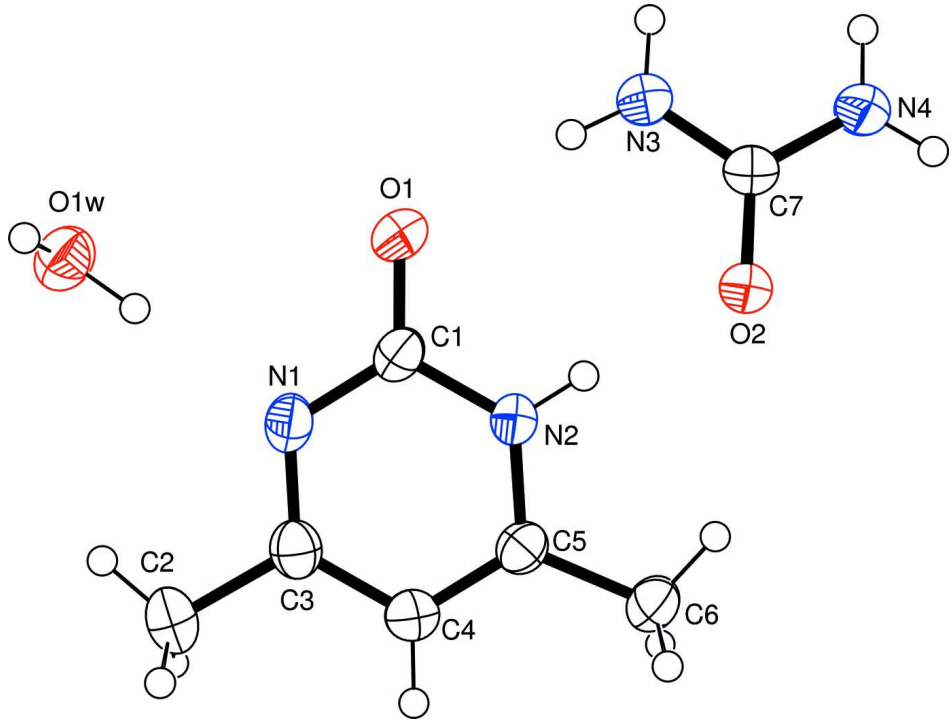

Figure 1

The molecular structure of **I** showing the atom–labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

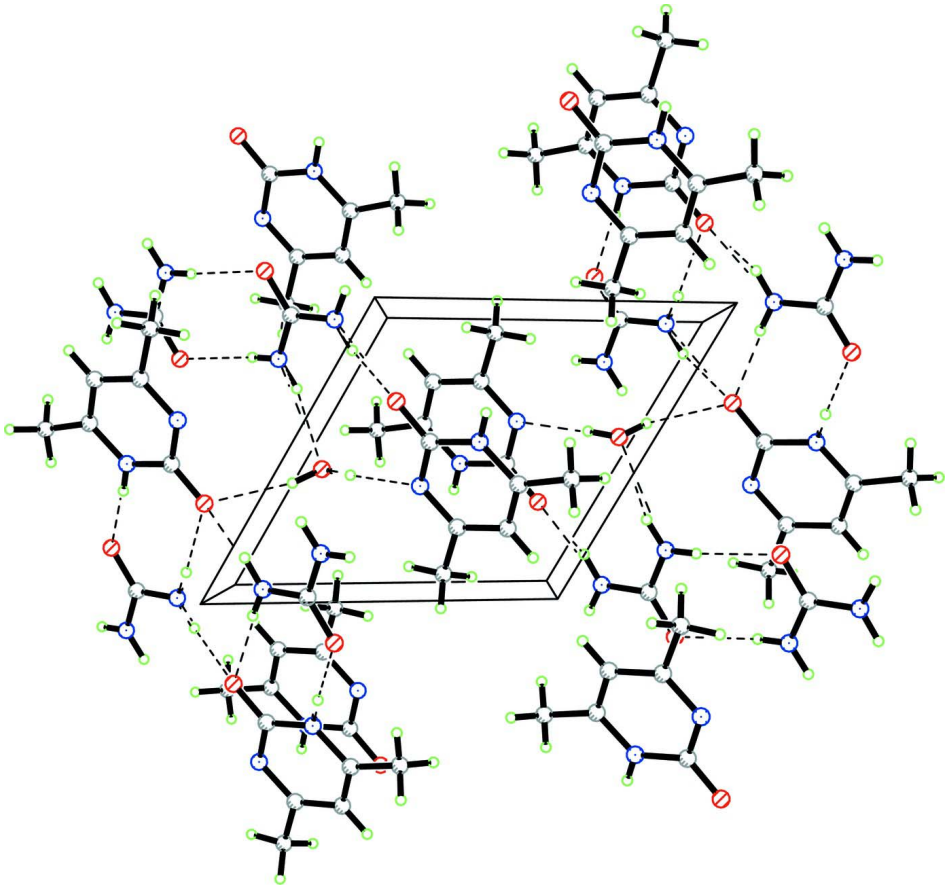

Figure 2

Packing diagram of **I**. H–bonds are shown as dashed lines.

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Crystal data

$C_6H_8N_2O·CH_4N_2O·H_2O$
$M_r = 202.22$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 8.1246$ (5) Å
$b = 8.4062$ (5) Å
$c = 8.9268$ (9) Å
$\alpha = 105.007$ (3)°
$\beta = 103.857$ (3)°
$\gamma = 114.379$ (2)°
$V = 493.05$ (7) Å^3
$Z = 2$
$F(000) = 216$
$D_x = 1.362$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1728 reflections
$\theta = 2.6$–25.1°
$\mu = 0.11$ mm^{-1}
$T = 293$ K
Prism, colourless
0.22 × 0.16 × 0.11 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: Fine–focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.977$, $T_{max} = 0.998$
5424 measured reflections
1728 independent reflections
1439 reflections with $I > 2\sigma(I)$

R_{int} = 0.019
θ_{max} = 25.1°, θ_{min} = 2.6°
h = −9→9
k = −8→10
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: Full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.170
S = 1.10
1728 reflections
143 parameters
1 restraint
Primary atom site location: Direct
Secondary atom site location: Difmap
Hydrogen site location: Geom
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0859P)^2 + 0.3229P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted < i>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}
C1	0.4613 (3)	0.3670 (3)	0.6702 (3)	0.0323 (5)
C2	0.9295 (4)	0.6340 (4)	0.6375 (4)	0.0479 (7)
H2A	0.9052	0.5182	0.5560	0.072*
H2B	1.0477	0.6850	0.7341	0.072*
H2C	0.9443	0.7254	0.5883	0.072*
C3	0.7605 (3)	0.5927 (3)	0.6906 (3)	0.0343 (5)
C4	0.7602 (3)	0.7390 (3)	0.8094 (3)	0.0342 (5)
H4	0.8631	0.8644	0.8538	0.041*
C5	0.6073 (3)	0.6940 (3)	0.8582 (3)	0.0304 (5)
C6	0.5869 (4)	0.8321 (3)	0.9851 (3)	0.0393 (6)
H6A	0.4664	0.7658	0.9983	0.059*
H6B	0.5853	0.9287	0.9473	0.059*
H6C	0.6958	0.8907	1.0913	0.059*
C7	−0.0065 (3)	0.2768 (3)	0.8185 (3)	0.0326 (5)
N1	0.6150 (3)	0.4123 (3)	0.6221 (2)	0.0345 (5)
N2	0.4613 (3)	0.5103 (3)	0.7881 (2)	0.0305 (5)
H2	0.361 (5)	0.477 (4)	0.825 (4)	0.037*
N3	−0.0268 (4)	0.1313 (3)	0.6941 (3)	0.0456 (6)
H3A	0.067 (5)	0.150 (4)	0.657 (4)	0.055*
H3B	−0.135 (5)	0.035 (5)	0.631 (4)	0.055*
N4	−0.1593 (3)	0.2454 (3)	0.8633 (3)	0.0411 (5)
H4A	−0.151 (4)	0.352 (5)	0.938 (4)	0.049*

H4B	−0.269 (5)	0.148 (5)	0.801 (4)	0.049*
O1	0.3211 (2)	0.2007 (2)	0.6121 (2)	0.0431 (5)
O2	0.1517 (2)	0.4327 (2)	0.8930 (2)	0.0383 (5)
O1W	0.5570 (3)	0.0847 (2)	0.3512 (2)	0.0459 (5)
H1WA	0.5959 (10)	0.175 (2)	0.445 (2)	0.055*
H1WB	0.6047 (12)	0.0249 (14)	0.3918 (11)	0.055*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0355 (12)	0.0298 (12)	0.0289 (11)	0.0177 (10)	0.0113 (9)	0.0074 (9)
C2	0.0432 (14)	0.0535 (16)	0.0554 (16)	0.0269 (13)	0.0298 (13)	0.0205 (13)
C3	0.0341 (12)	0.0401 (13)	0.0342 (12)	0.0221 (11)	0.0143 (10)	0.0162 (10)
C4	0.0327 (12)	0.0300 (12)	0.0364 (12)	0.0140 (10)	0.0135 (10)	0.0117 (10)
C5	0.0341 (11)	0.0292 (11)	0.0289 (11)	0.0173 (10)	0.0116 (9)	0.0117 (9)
C6	0.0460 (14)	0.0297 (12)	0.0414 (13)	0.0186 (11)	0.0216 (11)	0.0103 (10)
C7	0.0318 (11)	0.0287 (12)	0.0343 (12)	0.0133 (10)	0.0123 (10)	0.0128 (9)
N1	0.0366 (10)	0.0378 (11)	0.0332 (10)	0.0227 (9)	0.0160 (8)	0.0113 (9)
N2	0.0304 (10)	0.0294 (10)	0.0310 (10)	0.0156 (9)	0.0135 (8)	0.0093 (8)
N3	0.0370 (12)	0.0340 (12)	0.0463 (13)	0.0084 (10)	0.0188 (10)	0.0023 (10)
N4	0.0302 (10)	0.0337 (11)	0.0488 (13)	0.0114 (9)	0.0167 (10)	0.0085 (10)
O1	0.0404 (10)	0.0296 (9)	0.0449 (10)	0.0129 (8)	0.0169 (8)	0.0025 (7)
O2	0.0328 (9)	0.0305 (9)	0.0433 (10)	0.0113 (7)	0.0184 (7)	0.0078 (7)
O1W	0.0477 (10)	0.0437 (10)	0.0396 (10)	0.0248 (9)	0.0124 (8)	0.0091 (8)

Geometric parameters (Å, º)

C1—O1	1.244 (3)	C6—H6A	0.9600
C1—N1	1.356 (3)	C6—H6B	0.9600
C1—N2	1.379 (3)	C6—H6C	0.9600
C2—C3	1.495 (3)	C7—O2	1.249 (3)
C2—H2A	0.9600	C7—N4	1.339 (3)
C2—H2B	0.9600	C7—N3	1.341 (3)
C2—H2C	0.9600	N2—H2	0.92 (3)
C3—N1	1.329 (3)	N3—H3A	0.87 (3)
C3—C4	1.401 (3)	N3—H3B	0.83 (3)
C4—C5	1.354 (3)	N4—H4A	0.93 (3)
C4—H4	0.9300	N4—H4B	0.83 (3)
C5—N2	1.347 (3)	O1W—H1WA	0.850 (11)
C5—C6	1.492 (3)	O1W—H1WB	0.850 (11)
O1—C1—N1	122.19 (19)	C5—C6—H6B	109.5
O1—C1—N2	119.09 (19)	H6A—C6—H6B	109.5
N1—C1—N2	118.7 (2)	C5—C6—H6C	109.5
C3—C2—H2A	109.5	H6A—C6—H6C	109.5
C3—C2—H2B	109.5	H6B—C6—H6C	109.5
H2A—C2—H2B	109.5	O2—C7—N4	121.7 (2)
C3—C2—H2C	109.5	O2—C7—N3	120.9 (2)

H2A—C2—H2C	109.5	N4—C7—N3	117.3 (2)
H2B—C2—H2C	109.5	C3—N1—C1	118.88 (19)
N1—C3—C4	122.5 (2)	C5—N2—C1	123.10 (19)
N1—C3—C2	116.8 (2)	C5—N2—H2	118.8 (19)
C4—C3—C2	120.7 (2)	C1—N2—H2	117.9 (19)
C5—C4—C3	118.7 (2)	C7—N3—H3A	119 (2)
C5—C4—H4	120.6	C7—N3—H3B	122 (2)
C3—C4—H4	120.6	H3A—N3—H3B	116 (3)
N2—C5—C4	118.0 (2)	C7—N4—H4A	116.4 (19)
N2—C5—C6	116.75 (19)	C7—N4—H4B	119 (2)
C4—C5—C6	125.2 (2)	H4A—N4—H4B	120 (3)
C5—C6—H6A	109.5	H1WA—O1W—H1WB	96.3 (12)
N1—C3—C4—C5	1.1 (4)	O1—C1—N1—C3	−179.4 (2)
C2—C3—C4—C5	−177.7 (2)	N2—C1—N1—C3	0.3 (3)
C3—C4—C5—N2	−0.8 (3)	C4—C5—N2—C1	0.3 (3)
C3—C4—C5—C6	178.9 (2)	C6—C5—N2—C1	−179.4 (2)
C4—C3—N1—C1	−0.8 (3)	O1—C1—N2—C5	179.7 (2)
C2—C3—N1—C1	178.0 (2)	N1—C1—N2—C5	−0.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2	0.92 (3)	1.87 (3)	2.779 (2)	172 (3)
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