

1-(Carboxymethyl)uracil

Jing Xiong,* Miao-Chang Liu and
Ji-Xin YuanSchool of Chemistry and Materials Science,
Wenzhou Normal College, Wenzhou 325027,
People's Republic of ChinaCorrespondence e-mail:
theresa_xiong@yahoo.com.cn

Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.010\text{ \AA}$
 R factor = 0.066
 wR factor = 0.148
Data-to-parameter ratio = 8.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $\text{C}_6\text{H}_6\text{N}_2\text{O}_4$, the planar carboxyl group makes a dihedral angle of $74.61(2)^\circ$ with the uracil plane. In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into a three-dimensional network.

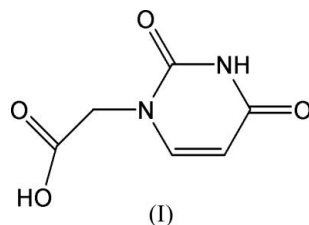
Received 24 June 2005

Accepted 15 July 2005

Online 23 July 2005

Comment

Thymine and uracil are important nucleobases in biology; they contribute to a wide range of biochemical processes, such as complementary base pairing in genetic information storage and transfer, molecular recognition and for some enzyme reactions (Bazzicalupi *et al.*, 2001; Honda *et al.*, 2002; Leiros *et al.*, 2003). As a result, great efforts have been undertaken to investigate thymine, uracil and their derivatives (Jolibois *et al.*, 1998; Koike *et al.*, 1998). In a continuation of our research in this field, we present here the synthesis and crystal structure of the title compound, namely 1-(carboxymethyl)uracil, (I).



In (I) (Fig. 1), the carboxyl group (C6/O3/O4/H4) and uracil ring (C1–C4/N1/N2) are each almost planar, with r.m.s. deviations of 0.0196 and 0.0093 Å, respectively. The dihedral angle between their mean planes is $74.61(2)^\circ$. Moreover, the C–N bond lengths in the uracil ring (Table 1) range from 1.369 (9) to 1.382 (8) Å, which are shorter than a C–N single bond length (*ca* 1.443 Å) (Jin *et al.*, 2004), but longer than a typical C=N bond length (*ca* 1.269 Å), indicating delocalization. On the other hand, the conjugation of O2/C2/C3/C4

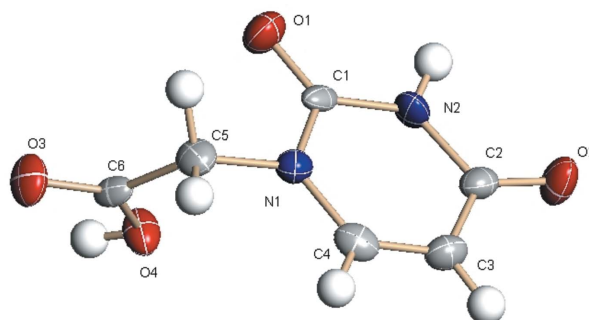


Figure 1
View of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

results in a decrease of the C2—C3 bond length [1.419 (10) Å].

In the crystal structure (Fig. 2), intermolecular N—H···O and O—H···O hydrogen bonds (Table 2) link each molecule with the four adjacent molecules to form a three-dimensional network. This hydrogen-bonding motif can be described in graph-set notation as $R_4^4(20)$ and $R_2^2(8)$ (Hu *et al.*, 2005).

Experimental

Bromoacetic acid (45 mmol, 6.25 g) in water (10 ml) was added to a solution of uracil (30 mmol, 3.33 g), potassium hydroxide (11.5 mmol, 6.41 g) and water (20 ml) at 313 K. The mixture was cooled after 2 h reaction and the pH was adjusted to 5.5 using hydrochloric acid. The precipitate was filtered off and the pH of the filtrate was adjusted to 1, again using hydrochloric acid, and the solution was cooled for 6 h in a refrigerator and then filtered. The precipitate was washed with water and dried to obtain the title compound. Single crystals were obtained by recrystallization from anhydrous ethanol.

Crystal data

$C_6H_6N_2O_4$	$D_x = 1.669 \text{ Mg m}^{-3}$
$M_r = 170.13$	Mo $K\alpha$ radiation
Monoclinic, Pc	Cell parameters from 861 reflections
$a = 5.0999$ (8) Å	$\theta = 2.8\text{--}24.8^\circ$
$b = 4.6273$ (8) Å	$\mu = 0.14 \text{ mm}^{-1}$
$c = 15.0106$ (19) Å	$T = 298$ (2) K
$\beta = 107.093$ (5)°	Rod, colourless
$V = 338.58$ (9) Å ³	$0.21 \times 0.11 \times 0.10 \text{ mm}$
$Z = 2$	

Data collection

Bruker APEX area-detector diffractometer	621 independent reflections
φ and ω scans	607 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2002)	$R_{\text{int}} = 0.035$
$T_{\text{min}} = 0.971$, $T_{\text{max}} = 0.983$	$\theta_{\text{max}} = 25.2^\circ$
3071 measured reflections	$h = -6 \rightarrow 6$
	$k = -5 \rightarrow 5$
	$l = -17 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 0.2892P]$
$R[F^2 > 2\sigma(F^2)] = 0.066$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.148$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.26$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
621 reflections	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$
75 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Selected bond lengths (Å).

O1—C1	1.217 (7)	N2—C1	1.369 (9)
O2—C2	1.227 (8)	N2—C2	1.382 (8)
N1—C4	1.371 (8)	C2—C3	1.419 (10)
N1—C1	1.374 (8)		

Table 2

Hydrogen-bond geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O4—H4···O2 ⁱ	0.82 (2)	1.76 (3)	2.553 (7)	160 (7)
N2—H2···O3 ⁱⁱ	0.84 (2)	2.00 (3)	2.828 (7)	169 (7)

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

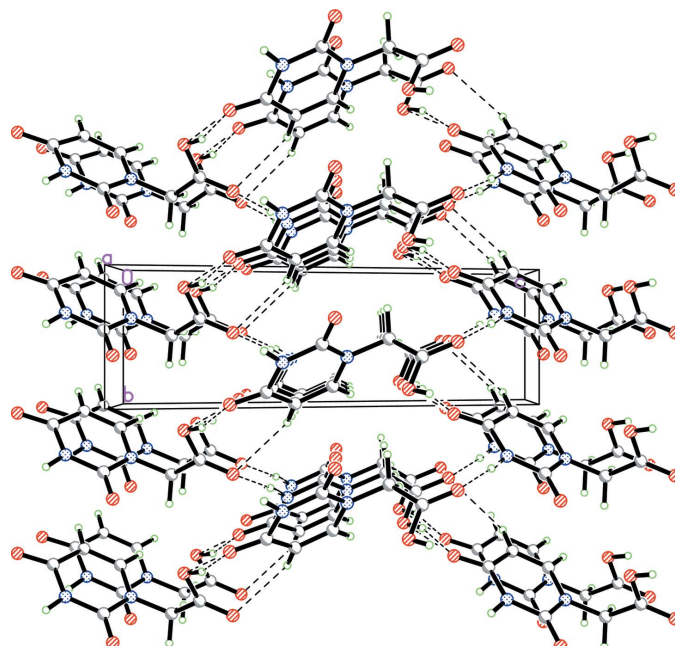


Figure 2

The crystal packing viewed approximately along the a axis. Dashed lines indicate the intermolecular N—H···O and O—H···O hydrogen bonds.

H atoms attached to N and O atoms were located in a difference Fourier map, and refined with O—H and N—H distances restrained to 0.82 (2) and 0.85 (2) Å, respectively, and with $U_{\text{iso}} = 1.2U_{\text{eq}}$ (parent atom). The other H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of $Csp^2\text{—}H = 0.93$ Å, with $U_{\text{iso}} = 1.2U_{\text{eq}}$ (parent atom), and $Csp^3\text{—}H = 0.97$ Å, with $U_{\text{iso}} = 1.5U_{\text{eq}}$ (parent atom). Due to the absence of any significant anomalous scatterers in the molecule, the Friedel pairs were merged before the final refinement.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

We are grateful for funding to Wenzhou Technology Project Foundation of China (No. S2004A004), Zhejiang Provincial Natural Science Foundation of China (No. Y404118) and National Natural Science Foundation of China (No. 20471043).

References

- Bazzicalupi, C., Bencini, A., Berni, E., Ciattini, S., Bianchi, A., Giorgi, C., Paoletti, P. & Valtancoli, B. (2001). *Inorg. Chim. Acta*, **317**, 259–267.
- Bruker (2002). *SADABS* (Version 2.03), *SAINTE* (Version 6.02), *SMART* (Version 5.62) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.
- Honda, T., Inagawa, H., Fukushima, M., Moriyama, A. & Soma, G. I. (2002). *Clin. Chim. Acta*, **322**, 59–66.
- Hu, M.-L., Cheng, Y.-Q., Wang, S., Yuan, J.-X. & Cai, X.-Q. (2005). *Acta Cryst. E61*, o1196–o1197.
- Jin, Z.-M., Li, L., Li, M.-C., Hu, M.-L. & Shen, L. (2004). *Acta Cryst. C60*, o642–o643.

Jolibois, F., D'Ham, C., Grand, A., Subra, R. & Cadet, J. (1998). *J. Mol. Struct. (Theochem)*, **427**, 143–155.
Koike, T., Gotoh, T., Aoki, S., Kimura, E. & Shiro, M. (1998). *Inorg. Chim. Acta*, **270**, 424–432.

Leiros, I., Moe, E., Lanes, O., Smalas, A. O. & Willassen, N. P. (2003). *Acta Cryst. D***59**, 1357–1365.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.