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1-(Carboxymethyl)thymine

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In the title compound, 5-methyl-2,6-dioxo-1,2,3,6-tetrahydropyrimidine-3-acetic acid, $C_7H_8N_2O_4$, the planar carboxymethyl moiety makes a dihedral angle of 83.89 (6)° with the thymine heterocycle plane. O—H···O, N—H···O, C—H···O and C—H···π interactions are observed in the structure, leading to the formation of a three-dimensional network.

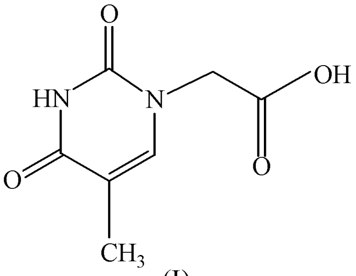
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.107
Data-to-parameter ratio = 11.7

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

Comment

Thymine and uracil are important nucleobases in biology, being responsible for a wide range of biochemical processes, such as complementary base pairing in genetic information storage and transfer, molecular recognition and some enzymatic reactions (Bazzicalupi *et al.*, 2001; Honda *et al.*, 2002; Leiros *et al.*, 2003). Therefore, considerable efforts have been made to investigate thymine, uracil and their derivatives (Jolibois *et al.*, 1998; Koike *et al.*, 1998). In earlier studies, we reported the synthesis and crystal structures of 1-benzylthymine (Ding *et al.*, 2002), 1,3-dibenzylthymine (Ding *et al.*, 2003) and 1-benzyluracil (Ding *et al.*, 2004). Since then, a new thymine derivative, 1-carboxymethylthymine, (I), has been obtained and its crystal structure is reported here.



(I)

In (I), the carboxymethyl group is essentially planar, with an average deviation from planarity of 0.0008 Å. The atoms of the thymine ring (C3–C6/N1/N2) are coplanar, with an average deviation of 0.0146 Å; the value in 1,3-dibenzylthymine (Ding *et al.*, 2003) is very similar, *viz*. 0.013 Å. The carboxymethyl plane is almost perpendicular to the thymine ring; the dihedral angle is 83.89 (6)°.

The N1—C3 [1.376 (2) Å] and N1—C6 [1.375 (2) Å] bond distances are significantly shorter than the corresponding lengths of 1.404 (3) and 1.400 (3) Å, respectively, in acetylthymine (Tari & Secco, 1992). This difference indicates that the exocyclic carboxyl group is electron withdrawing with respect to atom N1 and makes the N1—C bond lengths longer. However, in the title compound, the presence of a methylene group decreases this effect.

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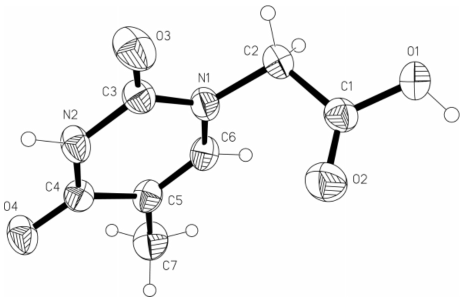

Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

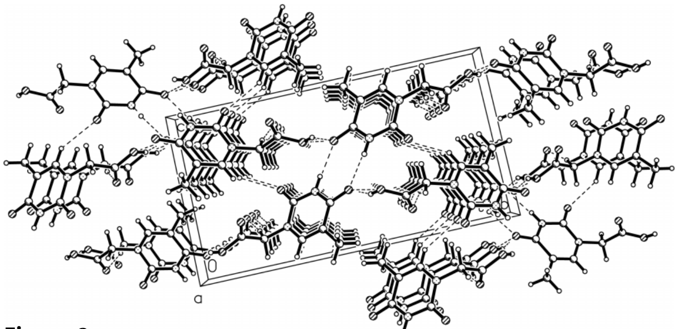

Figure 2
The crystal packing of (I), viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

In the crystal structure, intermolecular O1—H1···O4^{i} and C2—H2*A*···O2^{iii} hydrogen bonds (symmetry codes as in Table 1) link the molecules into layers parallel to the *ab* plane. The adjacent layers are interlinked by N2—H2···O4^{ii} and C6—H6···O3^{iv} hydrogen bonds, resulting in the formation of a three-dimensional network (Fig. 2). A weak C—H···π interaction involving the thymine ring is also observed in the structure (Table 1).

Experimental

Chloroacetic acid (1.63 g, 15 mmol) was added to a solution of thymine (1.22 g, 10 mmol), KOH (2.8 g, 5 mmol) and H_2O (7 ml, 389 mmol) at 313 K. The mixture was stirred for 2 h at the same temperature, and then it was cooled. Hydrochloric acid was added to the mixture to adjust the pH to 5.5. The precipitate was filtered off and the pH of the filtrate was adjusted to 2 by addition of hydrochloric acid. The solution was cooled for 6 h in a refrigerator and then filtered. The filtrate was washed with water and dried to obtain the title compound. Single crystals were obtained by recrystallization from anhydrous ethanol.

Crystal data

$C_7H_8N_2O_4$
M_r = 184.15
Monoclinic, $P2_1/n$
a = 5.0372 (2) Å
b = 17.1314 (7) Å
c = 9.2598 (4) Å
β = 99.476 (2)°
V = 788.16 (6) Å^3
Z = 4

D_x = 1.552 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2012 reflections
θ = 2.4–25.2°
μ = 0.13 mm^{-1}
T = 298 (2) K
Block, colorless
0.31 × 0.25 × 0.14 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min}$ = 0.951, $T_{\max}$ = 0.972
4127 measured reflections

1409 independent reflections
1291 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.015
$\theta_{\max}$ = 25.2°
$h = -4 \rightarrow 6$
$k = -20 \rightarrow 18$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.107
S = 1.11
1409 reflections
120 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.263P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.15 e Å^{-3}
$\Delta\rho_{\min}$ = −0.25 e Å^{-3}

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

Cg is the thymine ring centroid.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4^{i}	0.82	1.89	2.673 (2)	158
N2—H2···O4^{ii}	0.86	2.05	2.904 (2)	176
C2—H2*A*···O2^{iii}	0.97	2.47	3.425 (2)	170
C6—H6···O3^{iv}	0.93	2.44	3.265 (2)	148
C7—H7*C*···*Cg*^{v}	0.96	2.79	3.675 (2)	154

Symmetry codes: (i) $\frac{5}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $2-x, -y, 2-z$; (iii) $x-1, y, z$; (iv) $\frac{1}{2}+x, \frac{1}{2}-y, z-\frac{1}{2}$; (v) $1+x, y, z$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of 0.82 Å for O—H, 0.86 Å for N—H, 0.93 Å for Csp^2—H, 0.96 Å for $C_{\rm methyl}$—H and 0.97 Å for $C_{\rm methylene}$—H. Isotropic displacement parameters were set to $1.5U_{\rm eq}$(parent atom) for carboxyl and methyl H atoms and $1.2U_{\rm eq}$(parent atom) for other H atoms.

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

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