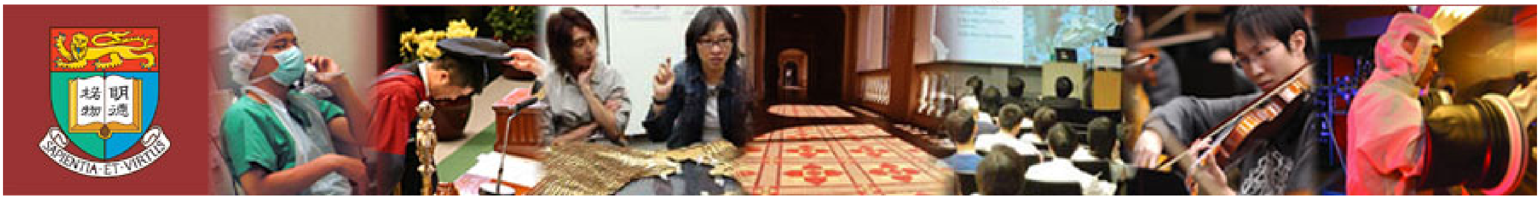

Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2,4-Dioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylic acid monohydrate

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Received 7 May 2004
Accepted 20 May 2004
Online 29 May 2004

The title compound, $C_5H_4N_2O_4{\cdot}H_2O$, a uracil derivative, contains several important functional groups, which give rise to its use in biomedical applications. The crystal structure consists of molecules held together by extensive intermolecular hydrogen bonding between neighbouring pyrimidine rings and between the acid groups and water molecules, resulting in a layered sheet structure. The asymmetric unit contains two molecules of the carboxylic acid and two water molecules.

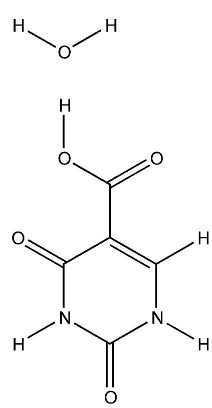
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.047
Data-to-parameter ratio = 7.7

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

Comment

Pyrimidines, especially those incorporating the uracil functional group, are known for their applications in biomedical studies (Iltzsch & Tankersley, 1994; Javaid *et al.*, 1999), for example the use of uracil derivatives as antithyroid drugs as well as enzyme inhibitors. The study of the title compound, (I), may offer useful leads in relation to previously studied uracil compounds, helping to improve and further our understanding of drug design.



(I)

It is known that the 5–6 double bond of uracil and uracil derivatives such as thiouracils is particularly reactive, especially towards nucleophiles; the range of reagents used for selective reduction of the carbonyl functional group at position 5 of the aromatic ring without reducing the double bond is relatively small. It is also possible to reduce the carbonyl ester at position 5 of the ring without affecting the aromaticity (5–6 double bond) of the ring. The choice of reaction medium has been found to be of importance (Gosh *et al.*, 1984; Pal, 1978), as it has been established that the reactivity of a carboxylic acid is greater than that of an olefinic bond towards certain reagents in tetrahydrofuran (THF). Hence, as well as the

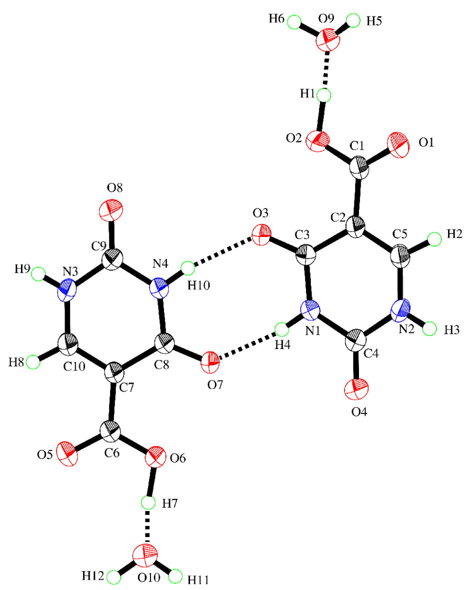


Figure 1
An *ORTEP*-3 (Farrugia, 2001) diagram of (I), showing 50% probability ellipsoids and the atom-labeling scheme. Hydrogen-bonding interactions are shown as dashed lines.

solubility factor influencing the choice of the reaction medium, THF was the reagent of choice for the conversion of the ester functional group to the corresponding acid and alcohol.

The asymmetric unit of the title compound contains two molecules of the carboxylic acid and two water molecules. Bond lengths and angles are typically in accord with values for known uracil compounds. The C—O bond lengths are in the range 1.216 (3)–1.307 (3) Å, while the aromatic C—C and C—N distances are in the range 1.345 (3)–1.452 (3) Å.

Inspection of the structure shows hydrogen-bonding interactions with the surrounding water molecules. These interactions cause stacking of the molecules, giving a layered sheet structure (Figs. 2 and 3). Examination of the structure (Fig. 1) shows that there are intermolecular hydrogen-bond interactions between the acid groups and the water molecules [O2—H1···O9 = 2.537 (3) Å and O6—H7···O10 = 2.502 (4) Å]. The exceptionally long O2—H1 and O6—H7 distances [1.15 (3) Å] indicate that the acid protons are probably delocalized between O2 and O9, and between O6 and O10. There are also intermolecular N—H···O interactions between neighbouring pyrimidine moieties.

It is interesting to note that the hydrogen at the end of the acid group does not form intramolecular hydrogen bond with the oxygen on the pyrimidine ring. Instead it forms intermolecular bonds with the water molecules that are present.

Study of the two-dimensional cross-section in Fig. 3 shows the layering and the sheet-like formation. The shortest out-of-plane distance of the centroid of one pyrimidine ring to the mean plane of the next pyrimidine ring sitting above or below it is 3.104 (5) Å. The undulation of the sheets is caused by the rather flexible and large carbonyl functional groups attached to the aromatic ring, which is otherwise approximately planar. The two acid molecules are found to be significantly different

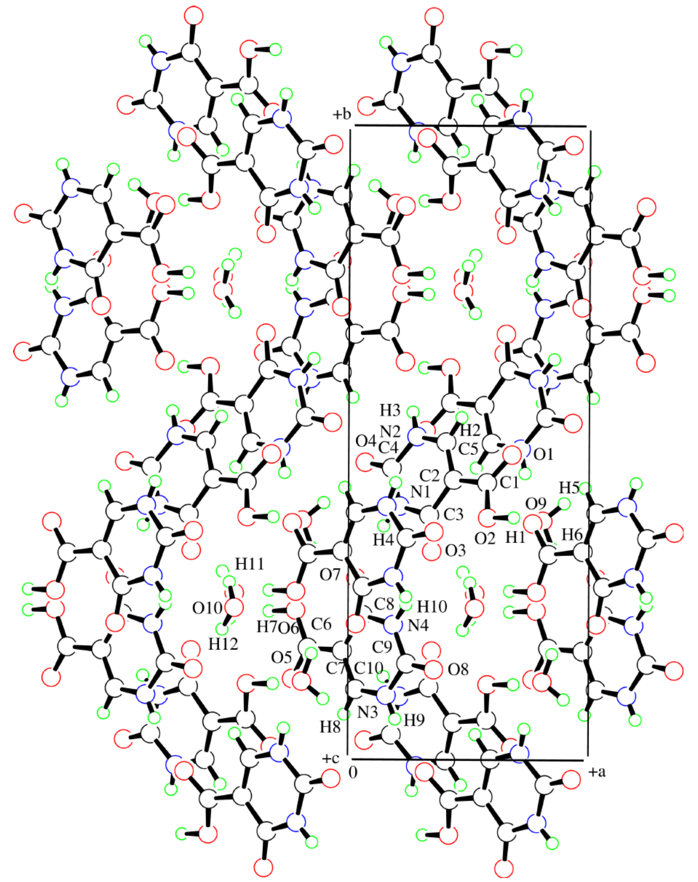


Figure 2
Packing diagram of (I), viewed along the *c* axis.

in conformation. The dihedral angle between the planar pyrimidine ring and the protruding acid group is 12.24 (9)° for one (C2/C3/N1/C4/N2/C5 and C2/C1/O1/O2) and 1.24 (8)° for the other (C7/C8/N4/C9/N3/C10 and C7/C6/O5/O6). Neighbouring symmetry-independent pyrimidine rings are not exactly coplanar, the dihedral angle being 12.28 (7)°; these effects together produce the slight wave in the structure.

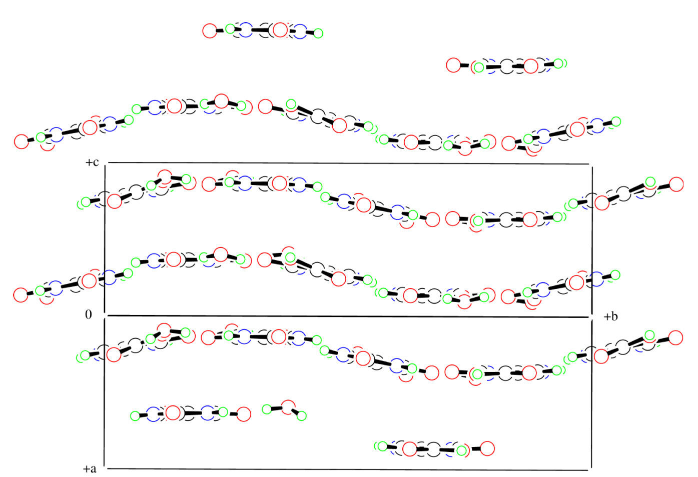


Figure 3
Packing diagram of (I), viewed perpendicular to *b* in the (101) plane.

Experimental

In an attempt to produce a lanthanide complex of ethyl 2,4-dioxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate, (II), the title compound was produced. Compound (II) (0.50 g, 2.71 mmol) was dissolved in THF, terbium nitrate hexahydrate (0.62 g, 1.35 mmol) was added and the resulting solution stirred for 24 h at ambient temperature. The title compound was obtained by slow evaporation of the reaction mixture. Analysis found: C 38.43, H 2.58, N 17.91%; calc for $C_5H_4N_2O_4$: C 38.46, H 2.56, N 17.95%.

Crystal data

$C_5H_4N_2O_4 \cdot H_2O$
M_r = 174.11
Monoclinic, $P2_1/c$
a = 7.876 (2) Å
b = 20.352 (4) Å
c = 8.664 (2) Å
β = 101.87 (1)°
V = 1359.1 (5) Å^3
Z = 8
D_x = 1.702 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16 902 reflections
θ = 1.0–27.5°
μ = 0.16 mm^{-1}
T = 298 (1) K
Block, pale yellow
0.16 × 0.16 × 0.08 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: none
16 902 measured reflections
3154 independent reflections
2050 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.032
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -26 \rightarrow 23$
$l = -11 \rightarrow 10$

Refinement

Refinement on F
R = 0.045
wR = 0.047
S = 1.09
3154 reflections
265 parameters
All H-atom parameters refined
$w = 1/[0.0002F_o^2 + \sigma(F_o^2) + 0.015]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O9	1.15 (3)	1.39 (3)	2.537 (3)	175 (3)
N2—H3···O5^{i}	0.87 (4)	1.91 (4)	2.775 (3)	174 (3)
N1—H4···O7	0.88 (2)	1.98 (2)	2.852 (3)	170 (2)
O9—H5···O4^{ii}	0.90 (5)	1.94 (4)	2.826 (3)	171 (4)
O9—H6···O6^{ii}	0.95 (4)	2.16 (4)	2.932 (3)	137 (3)
O9—H6···O7^{ii}	0.95 (4)	2.00 (4)	2.795 (3)	140 (3)
O6—H7···O10	1.15 (3)	1.36 (3)	2.502 (4)	170 (3)
N3—H9···O1^{iii}	0.83 (2)	1.96 (2)	2.768 (3)	168 (3)
N4—H10···O3	0.87 (4)	2.03 (4)	2.886 (3)	169 (3)
O10—H11···O2^{iv}	0.92 (4)	2.26 (4)	2.947 (3)	131 (3)
O10—H11···O3^{iv}	0.92 (4)	1.97 (4)	2.776 (3)	145 (4)
O10—H12···O8^{iv}	0.95 (4)	1.85 (4)	2.795 (3)	174 (4)

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

All H atoms were located in difference Fourier syntheses and were refined isotropically.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT* and *CrystalStructure* (Rigaku/MSC, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1996); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 2001); software used to prepare material for publication: *CrystalStructure*.

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