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Ethyl 4-(4-hydroxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate

Ming Li,* Wei-Si Guo, Li-Rong Wen and Wen-Ying Qi

College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, Peoples' Republic of China

Correspondence e-mail: nick8110@163.com

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Key indicators

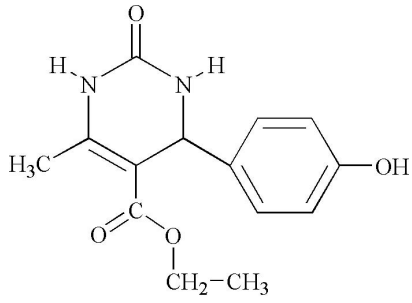
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.043
wR factor = 0.134
Data-to-parameter ratio = 12.4

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

In the title compound, $C_{14}H_{16}N_2O_4$, the ethyl chain of the ethoxycarbonyl group displays rotational disorder. In the crystal structure, the molecules are connected in a three-dimensional network through intermolecular N—H···O and O—H···O hydrogen-bond interactions.

Comment

5-Ethoxycarbonyl-4-substituted phenyl-6-methyl-3,4-dihydropyrimidin-2(1*H*)-ones have attracted considerable interest because of their versatile activities as calcium channel blockers, antihypertensive agents and α-1a-antagonists (Kappe, 1993). The biological activity of some isolated alkaloids has also been attributed to the presence of the dihydropyrimidinone moiety in the molecules (Overman *et al.*, 1995). Most important among them are batzelladine alkaloids, which have been found to be potent HIVgp-120-CD4 inhibitors (Snider *et al.*, 1996). In order to develop new biological activities, we synthesized the title compound, (I), the structure of which is reported here.



(I)

The pyrimidine moiety is non-planar, as indicated by the displacement of atom C7 from the least-squares plane [0.212 (2) Å] and by the C8-N1-C7-C10 torsion angle [33.2 (2)°]. The benzene ring is planar, the largest displacement observed being 0.005 (2) Å for atom C2. The dihedral angle between the pyrimidine and benzene rings is 82.84 (6)°, close to the value of 91.57° found in 4-(2-chlorophenyl)-6-methyl-5-ethoxycarbonyl-3,4-dihydropyrimidin-2(*H*)-one.

In the crystal structure, centrosymmetric molecules are connected as dimers through intermolecular N—H···O interactions (Table 2), giving rise to eight-membered rings. Additional weak hydrogen-bond interactions involving atoms N1, O1 and O2 result in a three-dimensional network, stabilizing the structure.

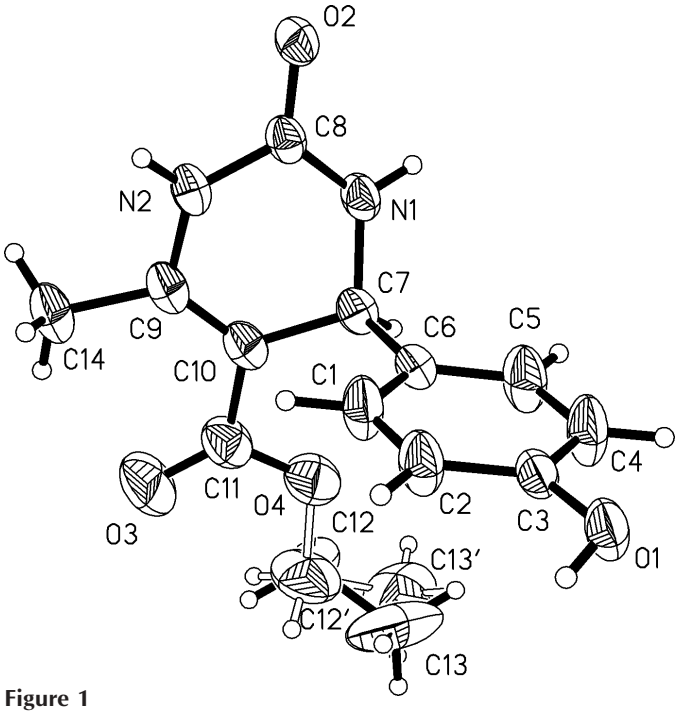


Figure 1
View of the title compound, with 35% probability displacement ellipsoids. Both disordered components are shown.

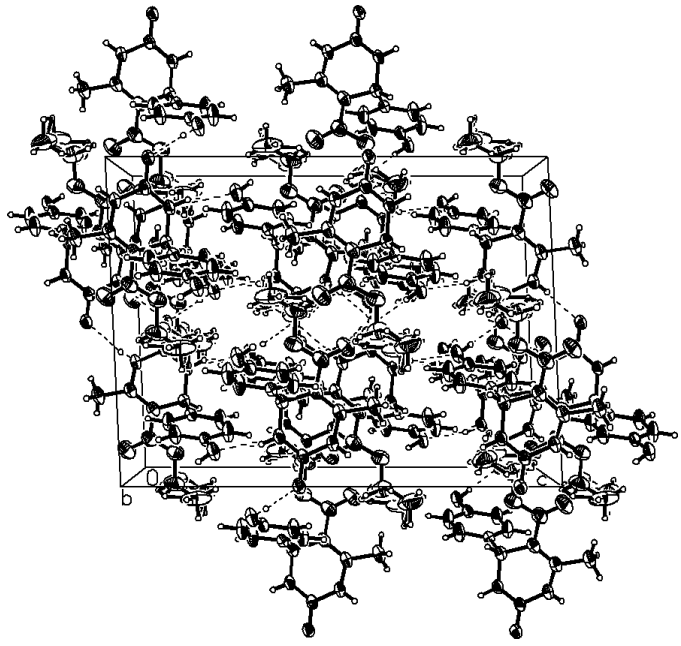


Figure 2
The molecular packing of the title compound, viewed along the *b* axis. Dashed lines represent hydrogen bonds.

Experimental

A mixture of 4-hydroxyaldehyde (20 mmol), ethyl acetoacetate (20 mmol), urea (30 mmol) and 1-butyl-3-methylimidazolium saccharinate (0.1 mmol) was heated in a 100 ml flask at 373 K for 1.5 h (monitored by thin-layer chromatography). The solid product formed was then filtered, washed with water and dried to obtain the primary products. The pure product was isolated by recrystallization from ethanol (m.p. 474 K).

Crystal data

$C_{14}H_{16}N_2O_4$
M_r = 276.29
Monoclinic, $C2/c$
a = 13.669 (7) Å
b = 11.392 (6) Å
c = 18.315 (9) Å
β = 92.697 (7)°
V = 2849 (3) Å^3
Z = 8
D_x = 1.288 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2274 reflections
θ = 2.6–24.5°
μ = 0.10 mm^{-1}
T = 293 (2) K
Prism, colourless
0.58 × 0.35 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
7602 measured reflections
2519 independent reflections
1894 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 25.0°
$h = -14 \rightarrow 16$
$k = -13 \rightarrow 13$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.134
S = 1.06
2519 reflections
203 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0718P)^2 + 1.0565P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0021 (6)

Table 1
Selected geometric parameters (Å, °).

O4—C12	1.436 (9)	N2—C9	1.390 (2)
O4—C12′	1.454 (5)	C7—C10	1.511 (3)
N1—C8	1.317 (2)	C9—C10	1.332 (3)
N1—C7	1.465 (2)	C12—C13	1.491 (10)
N2—C8	1.359 (2)	C12′—C13′	1.465 (7)
C11—O4—C12	121.3 (9)	N1—C8—N2	116.44 (17)
C11—O4—C12′	116.5 (4)	C10—C9—N2	119.11 (17)
C8—N1—C7	123.61 (14)	C9—C10—C7	119.30 (17)
C8—N2—C9	123.07 (16)	O4—C12—C13	102.0 (10)
N1—C7—C10	109.03 (15)	O4—C12′—C13′	106.8 (5)
C8—N1—C7—C10	33.2 (2)	C9—N2—C8—N1	−12.2 (2)
C8—N1—C7—C6	−91.57 (19)	C8—N2—C9—C10	16.9 (3)
C7—N1—C8—N2	−15.1 (2)	C8—N2—C9—C14	−163.79 (17)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.87	2.6873 (19)	176
N1—H1A···O1^{ii}	0.86	2.08	2.911 (2)	164
N2—H2A···O2^{iii}	0.86	2.02	2.847 (2)	162

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

All H atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, O—H = 0.82 Å and N—H = 0.86 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(parent atom). The ethyl group was found to be disordered; atoms C12 and C13, and related H atoms, were refined over two positions [occupancies 0.754 (16) for the primed and 0.246 (16) for the unprimed atoms].

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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