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Methyl 4-(3-methoxyphenyl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.139
Data-to-parameter ratio = 12.7

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

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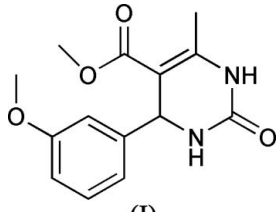
The solid-state structure of the title compound, $C_{14}H_{16}N_2O_4$, is characterized by hydrogen-bonding interactions, linking the molecules into chains. Hydrogen-bonding interactions between neighbouring chains form a two-dimensional network.

Comment

Dihydropyrimidinones are an important class of compounds which are of increasing interest owing to their therapeutic and pharmacological activities (Kappe, 1993). Some of them are integral backbones of several calcium channel blockers, antihypertensive agents and neuropeptide Y (NPY) antagonists (Atwal *et al.*, 1990; Rovnyak *et al.*, 1995). The synthesis of this kind of heterocyclic compound has therefore gained great importance in organic chemistry (Hu *et al.*, 1998). A simple, efficient and practical procedure for dihydropyrimidinones has also been reported by our laboratory (Su *et al.*, 2005).



(I)

The six-membered dihydropyrimidinone ring in the title compound, (I), is almost planar, with an r.m.s. deviation from the mean plane of 0.082 (1) Å. The mean planes through the benzene ring dihydropyrimidinone rings form a dihedral angle of 88.1 (1)°.

The solid-state structure of (I) is characterized by hydrogen-bonding interactions (Table 2). Intermolecular N–H···O hydrogen-bonding interactions link neighbouring molecules into chains (Fig. 2). These chains are connected by hydrogen-bonding interactions, forming a two-dimensional network.

Experimental

A mixture of 3-methoxybenzaldehyde (5 mmol), methyl 3-oxobutanonate (5 mmol), urea (7.5 mmol) and $Sr(OTf)_2$ (OTf is trifluoromethanesulfonate) (0.05 mmol) was heated at 343 K with stirring for 4 h. After cooling, the reaction mixture was poured into cold water and stirred for 5 min. The solid was suction-filtered, washed with cold water, filtered and recrystallized from ethanol to afford the pure product (m.p. 486–487 K). Single crystals suitable for X-ray data collection were obtained from ethanol.

Crystal data

$C_{14}H_{16}N_2O_4$
$M_r = 276.29$
Triclinic, $P\overline{1}$
$a = 7.5344$ (8) Å
$b = 7.5833$ (8) Å
$c = 12.8025$ (14) Å
$\alpha = 78.355$ (2)°
$\beta = 81.757$ (2)°
$\gamma = 68.040$ (2)°
$V = 662.58$ (12) Å^3
$Z = 2$
$D_x = 1.385$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1648 reflections
$\theta = 2.5$–25.0°
$\mu = 0.10$ mm^{-1}
$T = 298$ (2) K
Block, colourless
0.33 × 0.25 × 0.18 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.957$, $T_{max} = 0.972$
3528 measured reflections
2336 independent reflections
2127 reflections with $I > 2\sigma(I)$
$R_{int} = 0.010$
$\theta_{max} = 25.2°$
$h = -8 \rightarrow 9$
$k = -8 \rightarrow 9$
$l = -15 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.139$
$S = 1.07$
2336 reflections
184 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2 + 0.2894P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.011$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.367 (3)	N1—C8	1.466 (2)
O1—C1	1.427 (3)	N2—C9	1.374 (2)
O2—C13	1.340 (2)	N2—C10	1.379 (2)
O2—C14	1.441 (2)	C8—C11	1.521 (2)
O3—C13	1.209 (2)	C10—C11	1.347 (3)
O4—C9	1.235 (2)	C10—C12	1.493 (3)
N1—C9	1.334 (2)	C11—C13	1.466 (3)
C2—O1—C1	117.3 (2)	C9—N1—C8	126.55 (15)
C13—O2—C14	116.35 (17)	C9—N2—C10	123.86 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	2.41	3.146 (2)	144
N2—H2···O4^{ii}	0.86	2.00	2.839 (2)	165

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms (C—H = 0.93, 0.96, 0.97 Å for aromatic, methylene and methyl H atoms, respectively, and N—H = 0.8 Å), with U_{iso}(H) values equal to $1.2U_{eq}$(C,N) or $1.5U_{eq}$(methyl C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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, 2002); software used to prepare material for publication: *SHELXL97*.

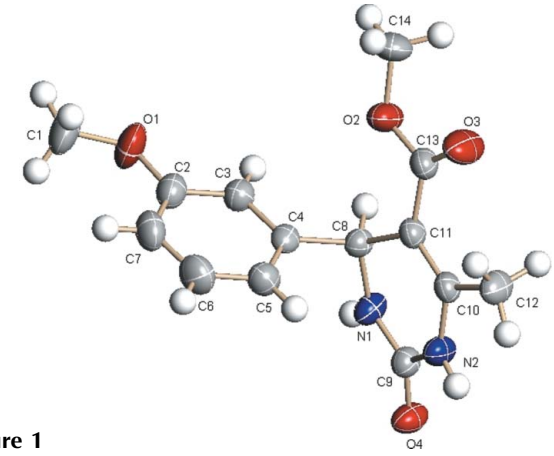


Figure 1
Perspective view of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level.

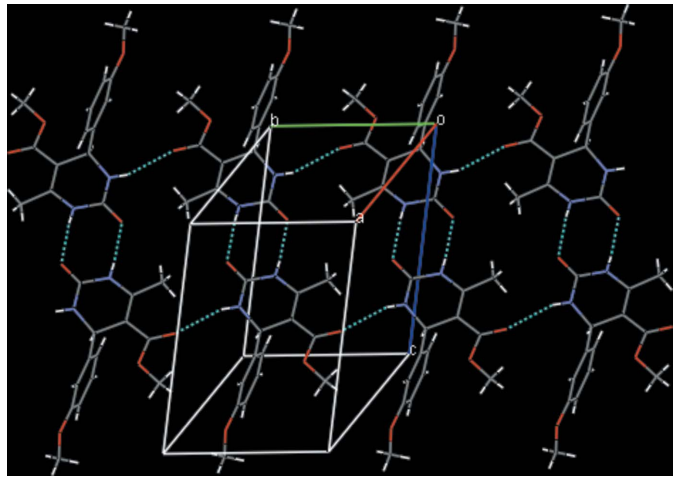


Figure 2
The two-dimensional network formed by intermolecular hydrogen-bonding interactions (dashed lines).

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