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

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.148
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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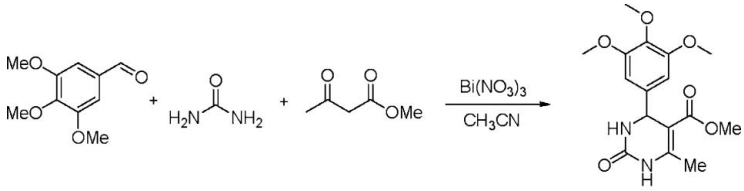
The title compound, $C_{16}H_{20}N_2O_6$, was synthesized from 3,4,5-trimethoxybenzaldehyde, methyl 3-oxobutanoate and urea in acetonitrile catalysed by $Bi(NO_3)_3$. The tetrahydropyrimidin-2-one ring is twisted and two molecules are connected by N—H···O hydrogen bonds.

Comment

Dihydropyrimidinones (DHPMs) and their derivatives exhibit a wide range of biological activities such as antibacterial, antiviral, antitumor and anti-inflamatory actions (Kappe, 1993). These compounds also exhibit pharmacological activities as calcium channel blockers, antihypertensive agents, and neuropeptide Y(NPY) antagonists (Atwal *et al.*, 1989, 1991; Rovnyak *et al.*, 1992; Kappe & Fabian, 1997). The structure of the title compound, (I), is shown in Fig. 1.



(I)

The tetrahydropyrimidin-2-one ring is twisted [C11—N1—C10—C14 = 30.5 (4)°]; the phenyl ring is almost perpendicular

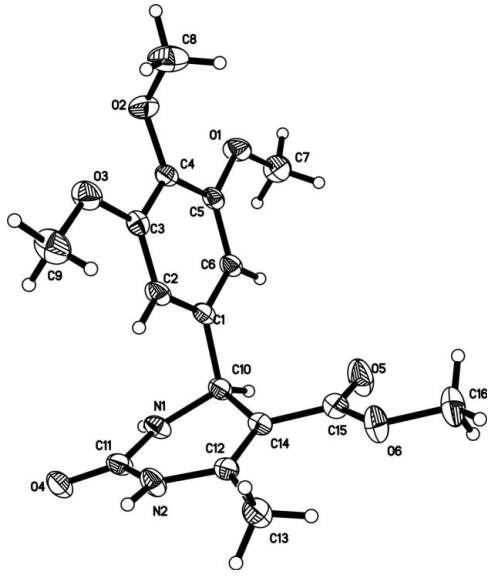


Figure 1
The molecular structure of (I), shown with 30% probability ellipsoids. H atoms have been omitted for clarity.

to the tetrahydropyrimidin-2-one ring [C11—N1—C10—C1 = −94.0 (3)° and C1—C10—C14—C15 = −78.4 (3)]. In the crystal structure, molecules are connected by N—H···O hydrogen bonds (Table 1).

Experimental

A solution of methyl 3-oxobutanoate (1.74 g, 15 mmol), 3,4,5-trimethoxybenzaldehyde (1.96 g, 10.0 mmol) and urea (0.76 g, 10 mmol) in acetonitrile (5 ml) was heated under reflux in the presence of a catalytic amount of $Bi(NO_3)_3$ for 3 h. The reaction mixture was washed thoroughly with water, filtered and recrystallized from methanol to afford the pure product. The title product was dissolved in 100 ml absolute methanol and crystals suitable for X-ray analysis were grown by slow evaporation of the absolute methanol solution at room temperature over a period of 15 d.

Crystal data

$C_{16}H_{20}N_2O_6$
M_r = 336.34
Triclinic, $P\overline{1}$
a = 7.949 (3) Å
b = 10.176 (3) Å
c = 11.802 (4) Å
α = 111.237 (6)°
β = 101.026 (6)°
γ = 103.892 (6)°
V = 821.9 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.977, T_{max} = 0.988
4232 measured reflections
2885 independent reflections
1432 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.148
S = 1.00
2885 reflections
228 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O4^{i}	0.84 (3)	1.99 (3)	2.836 (4)	176 (3)
N1—H1···O1^{ii}	0.89 (3)	2.40 (3)	3.068 (3)	132 (3)
N1—H1···O2^{ii}	0.89 (3)	2.34 (3)	3.171 (4)	155 (3)

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

Carbon-bound H atoms were positioned geometrically, with C—H = 0.93–0.96 Å, and refined in a riding model, with U_{iso}(H) = 1.2U_{eq}(carrier). The positional parameters of the nitrogen-bound H atoms were refined freely, with U_{iso}(H) = 1.2U_{eq}(N).

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

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