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Ethyl 4-(4-methoxyphenyl)-2-methyl-1,4-dihydrobenzo[4,5]imidazo[1,2-*a*]pyrimidine-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.137
Data-to-parameter ratio = 13.0

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

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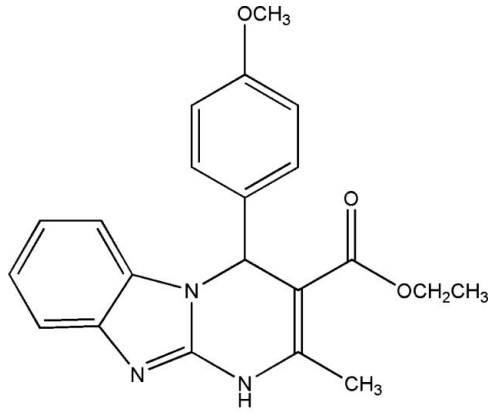
In the molecule of the title compound, $C_{21}H_{21}N_3O_3$, the pyrimidine ring adopts a boat conformation. Intermolecular N—H···N hydrogen bonds link the independent molecules into dimers, which may be effective in stabilizing the crystal structure.

Comment

It has been suggested that the substituent effect may be attributable to intramolecular hydrogen bonding between the alkoxy oxygen and the proton of the pyrimidine ring NH group (Broughton *et al.*, 1975). Recently, much attention has been devoted to dihydropyrimidine derivatives because of their significant therapeutic and medicinal properties (Kappe, 1993; Kappe *et al.*, 1997). Several marine alkaloids having the dihydropyrimidine core unit have been found to show interesting biological activities, such as antiviral, antibacterial and anti-inflammatory (Snider & Shi, 1993; Overman *et al.*, 1995). Many functionalized derivatives are used as calcium channel blockers and antihypertensive agents (Atwal *et al.*, 1990; Atwal *et al.*, 1991). We report here the crystal structure of the title compound, (I).



(I)

In the molecule of the title compound, (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Pyrimidine ring *A* (atoms N1/N2/C1–C4) is not planar, having a total puckering amplitude, Q_T of 0.179 (3) Å, and a boat conformation [φ = 26.74 (5)° and θ = 82.02 (3)°] (Cremer & Pople, 1975). Rings *B* (N2/N3/C1/C5/C6), *C* (C5–C10) and *D* (C14–C19) are, of course, planar and the dihedral angles between them are *B*/*C* = 2.55 (4), *B*/*D* = 85.84 (3) and *C*/*D* = 87.90 (5)°.

As can be seen from the packing diagram (Fig. 2), intermolecular N—H···N hydrogen bonds (Table 1) link molecules

into dimers, which may be effective in stabilizing the crystal structure. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.

Experimental

The title compound, (I), was prepared by the reaction of 4-methoxybenzaldehyde (0.27 g, 2 mmol) and ethyl 3-oxobutanoate (0.26 g, 2 mmol) in the presence of 2-aminobenzimidazole (0.27 g, 2 mmol) in water (5 ml) under microwave irradiation for 5 min. Upon completion of the reation, monitored by thin-layer chromatography, the reaction mixture was cooled to room temperature and then poured into cold water. The solid product was filtered off, washed with water and EtOH (95%), and subsequently dried and recrystallized from EtOH (95%) to give the pure product. Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a 95% aqueous ethanol solution (yield 0.64 g, 88%; m.p. 545–546 K).

Crystal data

$C_{21}H_{21}N_3O_3$
$M_r = 363.41$
Triclinic, $P\overline{1}$
$a = 6.3173\ (13)$ Å
$b = 10.970\ (2)$ Å
$c = 13.956\ (3)$ Å
$\alpha = 73.277\ (3)^\circ$
$\beta = 80.566\ (3)^\circ$
$\gamma = 89.565\ (3)^\circ$
$V = 912.9\ (3)$ Å^3
$Z = 2$
$D_x = 1.322$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 298\ (2)$ K
Block, colorless
0.20 × 0.15 × 0.09 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.982$, $T_{max} = 0.992$
4860 measured reflections
3171 independent reflections
2069 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.0^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.137$
$S = 1.01$
3171 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.3234P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3^{i}	0.86	2.07	2.917 (3)	169

Symmetry code: (i) $-x, -y+1, -z+1$.

H atoms were positioned geometrically, with N—H = 0.86 Å, C—H = 0.93, 0.98, 0.97 and 0.96 Å for aromatic, methine, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,N)$, where $x = 1.5$ for methyl and $x = 1.2$ for all other H 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*.

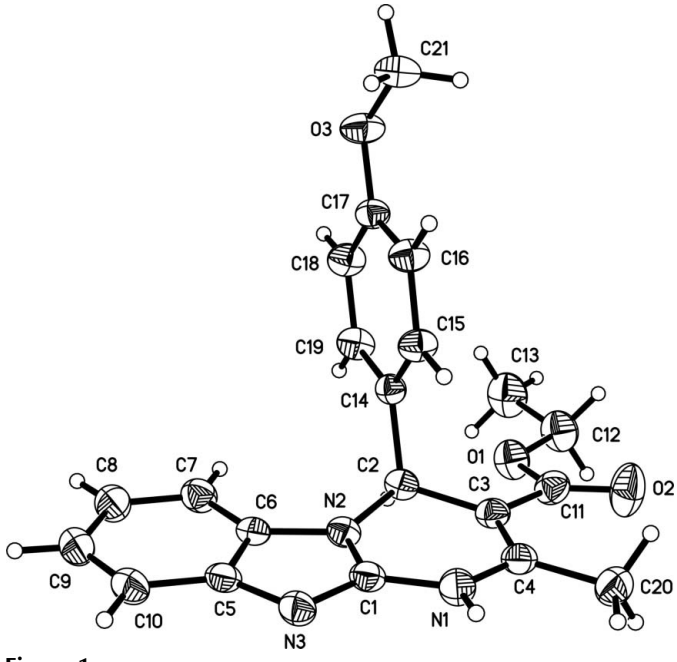


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

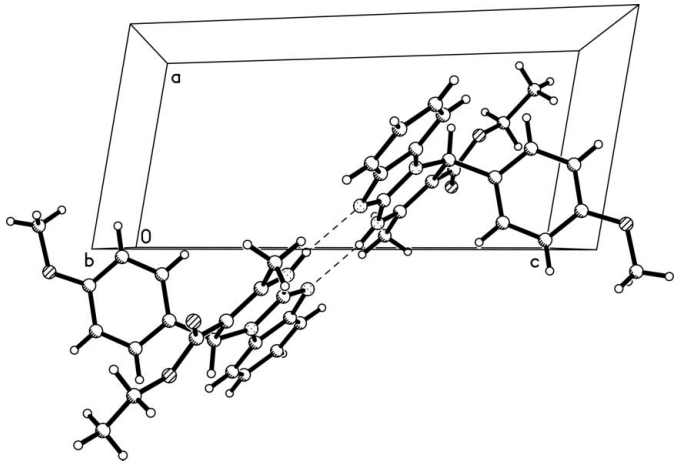


Figure 2
The dimer formation in (I). Hydrogen bonds are shown as dashed lines.

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