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Ethyl 3-(anilinocarbonothioyl)-6-methyl-2,4-diphenyl-1,2,3,4-tetrahydropyrimidine-5-carboxylate

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The title compound, $C_{27}H_{27}N_3O_2S$, was obtained *via* a multicomponent reaction using benzaldehyde, ethyl acetoacetate, ammonium acetate and phenyl isothiocyanate. The pyrimidine ring assumes a half-boat conformation. The crystal packing is stabilized by N—H···O and N—H···S hydrogen bonds. The phenyl group of the anilinocarbonothioyl group displays some rotational disorder.

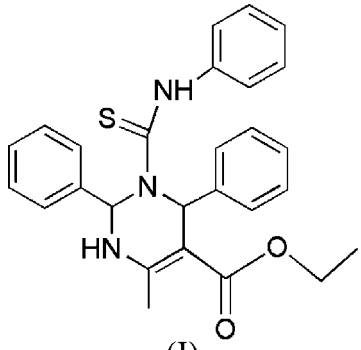
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.047
wR factor = 0.143
Data-to-parameter ratio = 13.1

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

Comment

Reactions in which more than two starting compounds react to form a product in such a way that the majority of the atoms of the starting materials can be found in the product are called multicomponent reactions (MCRs) (Domling & Ugi, 2000). Some important MCRs, such as the Ugi reaction (Ugi *et al.*, 1959), the Biginelli reaction (Biginelli, 1893) and the Hantzsch reaction (Hantzsch, 1882), have attracted wide attention in recent research. Pyrimidine derivatives have shown interesting biological activities, such as antihypertensive (Atwal *et al.*, 1991) and anticancer (Kato, 1984) activities. In this paper, we report the crystal structure of the title compound, (I), a pyrimidine derivative obtained *via* a 4-MCR using benzaldehyde, ethyl acetoacetate, ammonium acetate and phenyl isothiocyanate.



(I)

The tetrahydropyrimidine ring of (I) is not planar but adopts a half-boat conformation, with puckering parameters q_2 = 0.370 (2) Å, q_3 = −0.248 (3) Å, φ_2 = 159.0 (4)° and Q_T = 0.445 (2) Å (Cremer & Pople, 1975). The dihedral angle between the phenyl rings (C15–C20 and C21–C26) attached to the tetrahydropyrimidine ring is 30.63 (12)°. The ester group is *syn*-periplanar to the tetrahydropyrimidine ring double bond, with a C8—C9—C10—O1 torsion angle of −4.8 (5)°.

In the crystal structure, molecules are linked into centrosymmetric dimers through intermolecular N—H···O interactions (Table 1). Additional weak N—H···S hydrogen-

bonding interactions result in the formation of a three-dimensional network stabilizing the structure.

Experimental

A mixture of benzaldehyde (4 mmol), ethyl acetoacetate (2 mmol), ammonium acetate (4 mmol) and phenyl isothiocyanate (2 mmol) in ethanol (10 ml) was refluxed in a 100 ml flask for 2 h at 351 K. The reaction was monitored by thin-layer chromatography. The solid product formed was then filtered off, washed with water and dried to obtain the title compound (yield 64%). Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{27}H_{27}N_3O_2S$
M_r = 457.58
Orthorhombic, *Pbca*
a = 11.668 (4) Å
b = 20.397 (6) Å
c = 20.804 (6) Å
V = 4951 (3) Å^3
Z = 8
D_x = 1.228 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 294 (2) K
Prism, colourless
0.30 × 0.28 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.955, T_{max} = 0.962
23789 measured reflections
4361 independent reflections
2199 reflections with $I > 2\sigma(I)$
R_{int} = 0.080
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.143
S = 1.01
4361 reflections
333 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 4.1532P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.90 (3)	2.01 (3)	2.877 (4)	160 (3)
N3—H3···S1^{ii}	0.82 (3)	2.73 (3)	3.466 (3)	150 (3)

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

H atoms bound to C atoms were placed in calculated positions, with C—H = 0.93–0.98 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). N-bound H atoms were located in a difference Fourier map and refined freely. The phenyl ring C2–C7 was found to be disordered and was refined over two positions, with occupancies of 0.55 (4) and 0.45 (4) for the unprimed and primed atoms, respectively.

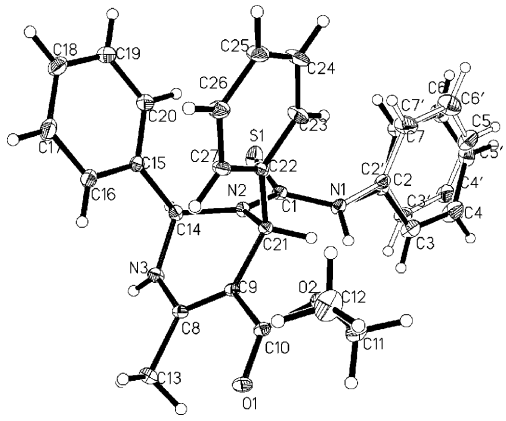


Figure 1
The molecular structure of (I), with 10% probability displacement ellipsoids. The minor component of the disorder (primed atoms) is shown with hollow bonds.

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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