Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 2-{[(1*Z*)-(3-methyl-5-oxo-1-phenyl-1,5-dihydropyrazol-4-ylidene)(phenyl)methyl]amino}-3-phenylpropanoate

Xin Zhang,[a]* Hua-Ling Zhu,[b] Hai-Zhen Xu[a] and Mei Dong[a]

[a]College of Chemistry and Life Science, Tianjin Normal University, Tianjin, Weijin Road No. 241, Tianjin, People's Republic of China, and [b]Tianjin Agricultural College, Tianjin, Jinjing Road, Tianjin, People's Republic of China

Correspondence e-mail: zxin_tj@eyou.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.051
wR factor = 0.130
Data-to-parameter ratio = 13.0

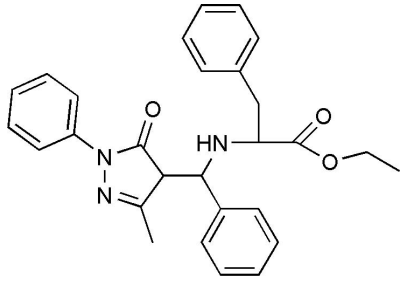
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 16 March 2005
Accepted 3 May 2005
Online 14 May 2005

In the title compound, $C_{28}H_{27}N_3O_3$, the pyrazolone ring and the N atom of the 2-amino-3-phenylpropanoate group are essentially coplanar. The compound is in an enamine–keto form and its structure is stabilized by one strong intramolecular N—H···O hydrogen bond.

Comment

In recent years, the Schiff bases derived from 4-acyl-5-pyrazolones and their metal complexes have been studied widely for their high antibacterial activation (Li *et al.*, 1997, 2004). Since amino acid esters also possess good antibacterial and biological activations (Xiong *et al.*, 1993), we synthesized the title compound, (I) (Fig. 1).



(I)

Atoms O1, C1, C2, C5 and N3 form a plane, the largest deviation being 0.0303 (16) Å for atom C5. The dihedral angle between this mean plane and the pyrazoline ring is 2.39 (8)°, indicating that they are essentially coplanar, as seen in 4-{[3,4-dihydro-5-methyl-3-oxo-2-phenyl-2*H*-pyrazol-4-ylidene]-(phenyl)methyl]amino}-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one [3.56 (3)°; Wang *et al.*, 2003]. The bond lengths within this part of the molecule (Table 1) lie between classical single- and double-bond lengths, indicating extensive conjugation. Atoms N3, C6, C14 and O2 are not coplanar, the torsion angle being 41.1 (4)°, different from that in some other 4-acylpyrazolone Schiff bases (Zhang *et al.*, 2004; Wang *et al.*, 2003). The bond lengths in this part of the molecule (Table 1) indicate that only C14═O2 is a classical double bond and the other bonds are classical single bonds. The dihedral angle between the benzene ring of ethyl 2-amino-3-phenylpropanoate and the pyrazolone ring is 78.28 (8)°, to avoid steric hindrance. A strong intramolecular hydrogen bond N3—H3···O1 (Table 2) is indicative of the enamine–keto form. This is similar to the situation in a related compound [N···O = 2.745 (4) Å and N—H···O 146 (4)°; Wang *et al.*, 2003].

Experimental

The title compound, (I), was synthesized by refluxing 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone and ethyl 2-amino-3-phenylpropanoate in ethanol for about 6 h. The product was recrystallized from a mixture of ethanol and ethyl acetate (1:1) to afford pale yellow crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{28}H_{27}N_3O_3$
M_r = 453.53
Monoclinic, $P2_1/c$
a = 10.521 (3) Å
b = 16.629 (5) Å
c = 14.080 (4) Å
β = 105.315 (5)°
V = 2375.9 (12) Å^3
Z = 4
D_x = 1.268 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4184 reflections
θ = 1.9–26.4°
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, yellow
0.32 × 0.24 × 0.22 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
12 125 measured reflections
4184 independent reflections
2661 reflections with $I > 2\sigma(I)$
R_{int} = 0.039
θ_{max} = 25.0°
$h = -12 \rightarrow 12$
$k = -19 \rightarrow 19$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.130
S = 1.01
4184 reflections
321 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0536P)^2 + 0.8249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.015
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N3—C5	1.330 (3)	C1—C2	1.442 (3)
N3—C6	1.466 (3)	C2—C5	1.389 (3)
O1—C1	1.250 (3)	C6—C14	1.524 (3)
O2—C14	1.197 (3)		
N3—C6—C14—O2	41.1 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O1	0.87	2.06	2.770 (2)	139

All H atoms were positioned geometrically (C—H = 0.93–1.06 Å and N—H = 0.87 Å) and treated as riding, with U_{iso}(H) = 1.2U_{eq}(C); the U_{iso} value for the H atoms bonded to N atoms was refined freely. The ethyl group was found to be disordered and was refined as two components with equal occupancy, with the aid of restraints on geometry and displacement parameters.

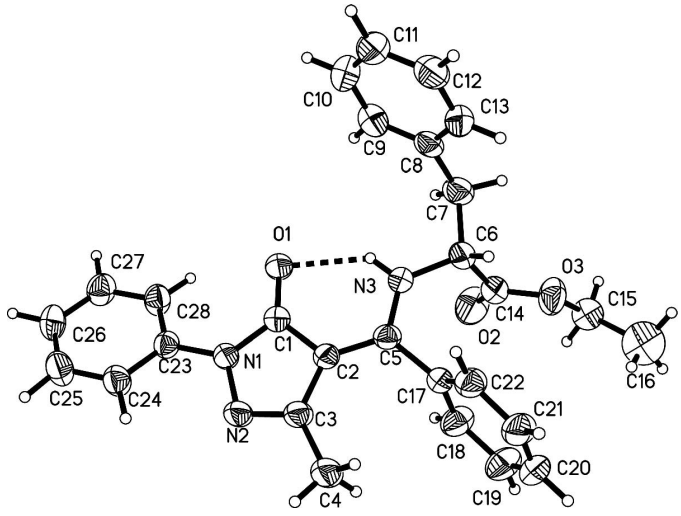


Figure 1
A view of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The intramolecular hydrogen bond is indicated by a dashed line.

Data collection: *SMART* (Bruker, 1999); 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*.

This work was supported by the Foundation of Tianjin Science Committee (research grant No. 023606511).

References

Bruker (1999). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, J.-Z., Jiang, L. & An, Y.-M. (2004). *Chin. J. Appl. Chem.* **21**, 150–153.
Li, J.-Z., Yu, W.-J. & Du, X.-Y. (1997). *Chin. J. Appl. Chem.* **14**, 98–100.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wang, J.-L., Yang, Y., Zhang, X. & Miao, F.-M. (2003). *Acta Cryst.* E**59**, o430–o432.
Xiong, G.-H., Yang, Z.-M. & Guo, A.-L (1993). *Fine Chemicals*, **6**, 1–3.
Zhang, X.,Zhu, H.-L., Xu, H.-Z. & Dong, M. (2004). *Acta Cryst.* E**60**, o1157–o1158.