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N-[(3-Methyl-5-oxo-1-phenyl-4,5-dihydro-1*H*-pyrazol-4-ylidene)(phenyl)methyl]glycine ethyl ester

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Key indicators

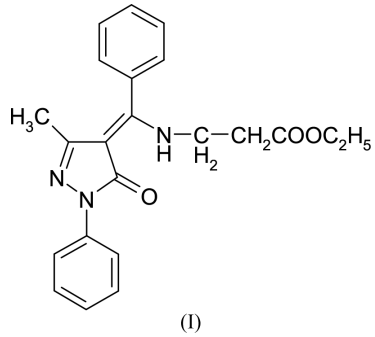
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.131
Data-to-parameter ratio = 12.4

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

In the title compound, $C_{21}H_{21}N_3O_3$, the pyrazolone and the N atom of the glycine ethyl ester group are essentially coplanar. The compound is a neutral tridentate ligand in an enamine–keto form, stabilized by two strong intramolecular N—H···O hydrogen bonds.

Comment

1-Phenyl-3-methyl-4-benzoylpyrazolon-5-one (HPMBP), a β-diketonate, is widely used and well known for its extractive ability. In recent years, both HPMBP and its metal complexes have been found to possess good antibacterial activity. The metal complexes also have analgesic activity (Liu *et al.*, 1980; Li *et al.*, 1997; Zhou *et al.*, 1999). Amino acid esters also possess antibacterial activity (Xiong *et al.*, 1993). Therefore, the study of the reaction of HPMBP with amino acid esters is warranted.



(I)

The title compound, (I), prepared by condensation of 1-phenyl-3-methyl-4-benzoyl-pyrazolon-5-one (HPMBP) and glycine ethyl ester, is a neutral potentially tridentate molecule in which the O atom of the 5-methyl-2-phenylpyrazol-3-one moiety, and the O and N atoms of the glycine ethyl ester group are available for coordination with metals. The torsion angles O1—C1—C3—C4 and N3—C18—C19—O2 are 2.2 (3) and 3.7 (3)°, respectively.

Atom O1 lies 0.0585 (3) Å from the plane defined by atoms C1, C3 and C4 of the PMBP moiety and atom N3 of the glycine ethyl ester; the largest deviation from the plane is 0.0268 (2) Å for atom C3. The dihedral angle between this plane and the pyrazoline ring of PMBP is 2.02 (9)°, so they are practically coplanar, as seen in 4-{[3,4-dihydro-5-methyl-3-oxo-2-phenyl-2*H*-pyrazol-4-ylidene](phenyl)methylamino}-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one, (II) [3.56 (3)°; Wang *et al.*, 2003]. The bond lengths in this part of the molecule lie between classical single- and double-bond values, indicating extensive delocalization.

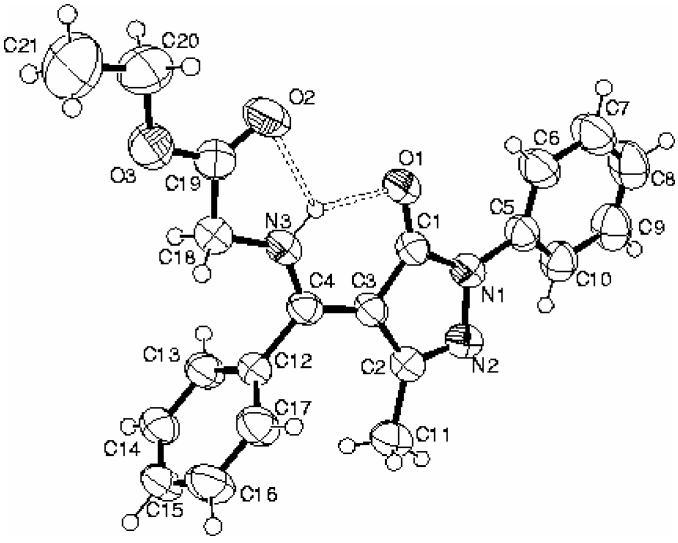


Figure 1
The molecular structure of compound (I), with ellipsoids shown at the 50% probability level. Dashed lines indicate hydrogen bonds.

Atoms N3, C18, C19 and O2 of the glycine ethyl ester are also coplanar, the largest deviation from this plane being 0.0189 (3) Å for atom C19. The dihedral angle between this plane and the plane defined by atoms C1, C3, C4 and N3 is 11.46 (9)°. The bond lengths in this part of the molecule also indicate the delocalization of the glycine amino ethyl ester. Strong intramolecular N3—H3···O1 hydrogen bonds (Table 1) are observed, indicative of the enamine–keto form. This is similar to the situation in (II) [N···O = 2.745 (4) Å and N—H···O = 146 (4)°: Wang *et al.*, 2003]. Two other intramolecular hydrogen bonds (N3—H3···O2 and C6—H6···O1) and an intermolecular hydrogen bond (C18—H18*B*···O1^{i}; symmetry code: (i) $-x$, $-y$, $-z$] are also found, stabilizing the structure.

Experimental

The title compound was synthesized by refluxing a mixture of PMBP (10 mmol) and glycine ethyl ester (10 mmol) in ethane (80 ml) over a steam bath for about 6 h. Excess solvent was removed by evaporation and the solution was cooled to room temperature. After 2 d, pale-yellow blocks were obtained and dried in air. The product was recrystallized from a mixture of ethyl acetate and ethane (1:1), affording pale-yellow crystals suitable for X-ray analysis.

Crystal data

$C_{21}H_{21}N_3O_3$
M_r = 363.41
Triclinic, $P\overline{1}$
a = 9.268 (3) Å
b = 9.985 (3) Å
c = 11.193 (4) Å
α = 100.386 (5)°
β = 92.652 (5)°
γ = 106.543 (5)°
V = 971.4 (6) Å^3
Z = 2
D_x = 1.242 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 905 reflections
θ = 3.1–24.1°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, pale yellow
0.40 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
8043 measured reflections
3957 independent reflections
2551 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.4°
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.131
S = 1.01
3957 reflections
318 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 0.1991P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···O1	0.93 (2)	1.94 (2)	2.704 (2)	137.9 (18)
N3—H3···O2	0.93 (2)	2.26 (2)	2.666 (2)	105.9 (15)
C6—H6···O1	0.96 (2)	2.33 (2)	2.939 (3)	120.3 (17)
C18—H18*B*···O1^{i}	0.95 (2)	2.48 (2)	3.317 (3)	146.5 (18)

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

All H atoms were freely refined, except for the H atoms bound to C21, which were positioned geometrically (C—H = 0.96 Å) and treated as riding, with $U_{iso} = 1.5U_{eq}$(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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