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4-{[1-(Methoxycarbonyl)-3-(methylsulfanyl)-propylamino](phenyl)methylidene}-3-methyl-1-phenylpyrazol-5(4*H*)-one

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The title compound, $C_{24}H_{27}N_3O_3S$, is composed of two benzene rings, a pyrazolone ring and an alkyl substituent. The molecule has one intramolecular hydrogen bond.

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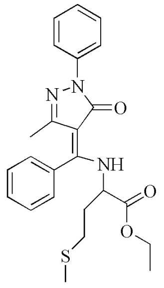
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.063
wR factor = 0.199
Data-to-parameter ratio = 14.5

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

Comment

1-Phenyl-3-methyl-4-benzoylpyrazolon-5-one (HPMBP), an effective β-diketonate, is widely used and well known for its extractive ability. In recent years, it and its metal complexes have also been found to have good antibacterial and biological properties. Its metal complexes have analgesic activity (Liu *et al.*, 1980; Li *et al.*, 1997; Zhou *et al.*, 1999). Amino acid esters also exhibit antibacterial and biological activity. Therefore, the study of the reaction of HPMBP with amino acid esters is very valuable. In order to develop new medicines, we have synthesized the title compound, (I), and its structure is reported here.



(I)

The structure of (I) is shown in Fig. 1. Atoms O1, C9, C8 and C11 of the PMBP moiety and atom N3 of the glycine ethyl ester are coplanar, the largest deviation being 0.0462 (4) Å for atom C11. The dihedral angle between this mean plane and the pyrazoline ring of PMBP is 1.8 (4)°, close to the value of 3.56 (3)° found 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 (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. A strong intramolecular N3—H3*A*···O1 hydrogen bond (Table 2) is observed, leading to an enamine–keto form. This case is similar to that in (II) [N···O = 2.745 (4) Å and N—H···O = 146 (4)°]. Although the torsion angle for O1—C9—C8—C11 [4.8 (6)°] is small, the torsion angle for N3—C18—C22—O2 [18.8 (6)°] is relatively large. This shows that atoms O1, N3 and O2 could not form a ligand donor set, and this may be the reason we have not been able to obtain complexes of this compound with many metal ions.

Experimental

The title compound, (I), was synthesized by refluxing a mixture of PMBP (5 mmol) and methionine ethyl ester (5 mmol) in ethanol (20 ml) with stirring for 6 h at 348–353 K. Some of the ethanol was removed by evaporation and the remaining solution was cooled to room temperature, filtered and the product dried in air. Pale-yellow single crystals were obtained by recrystallization of the yellow solid from ethanol.

Crystal data

$C_{24}H_{27}N_3O_3S$
$M_r = 437.55$
Monoclinic, $P2_1/c$
$a = 13.355\ (7)$ Å
$b = 10.261\ (5)$ Å
$c = 18.159\ (10)$ Å
$\beta = 107.946\ (9)^\circ$
$V = 2367\ (2)$ Å^3
$Z = 4$
$D_x = 1.228$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 685 reflections
$\theta = 3.0$–20.7°
$\mu = 0.17$ mm^{-1}
$T = 293\ (2)$ K
Prism, pale yellow
0.20 × 0.18 × 0.14 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.963$, $T_{\max} = 0.977$
11933 measured reflections
4160 independent reflections
2098 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.053$
$\theta_{\max} = 25.0^\circ$
$h = -15 \rightarrow 15$
$k = -12 \rightarrow 7$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.199$
$S = 1.03$
4160 reflections
287 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0988P)^2 + 0.0751P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.239 (4)	N2—C7	1.295 (4)
N1—C9	1.366 (4)	N3—C11	1.322 (4)
N1—N2	1.402 (4)	C11—C12	1.484 (5)
N1—C6	1.412 (4)		
C9—N1—N2	111.5 (3)	C18—N3—H3*A*	117 (3)
C9—N1—C6	128.7 (3)	O1—C9—N1	126.4 (3)
N2—N1—C6	119.5 (3)	O1—C9—C8	128.8 (3)
C7—N2—N1	107.0 (3)	N1—C9—C8	104.8 (3)
C11—N3—C18	126.3 (3)	C7—C10—H10*A*	109.5
C11—N3—H3*A*	114 (3)		
C9—N1—N2—C7	0.6 (4)	C18—N3—C11—C8	166.2 (4)
C6—N1—N2—C7	−174.5 (3)	C18—N3—C11—C12	−12.3 (6)
C6—N1—C9—O1	−6.1 (6)	C9—C8—C11—N3	−9.0 (5)
N2—N1—C9—C8	−1.2 (4)	N3—C11—C12—C17	−79.4 (4)
C6—N1—C9—C8	173.4 (3)	N3—C11—C12—C13	98.6 (4)
C11—C8—C9—O1	4.8 (6)	N3—C18—C19—C20	−65.9 (4)
C7—C8—C9—O1	−179.3 (4)	N3—C18—C22—O2	−18.8 (6)
C11—C8—C9—N1	−174.6 (3)	N3—C18—C22—O3	161.1 (3)

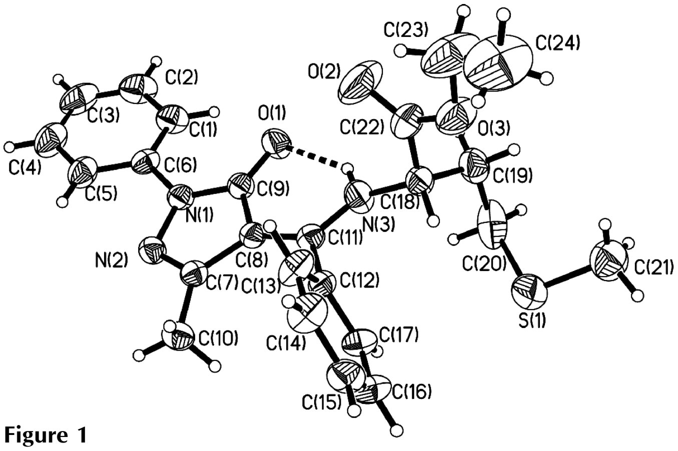


Figure 1
View of the title compound, shown with 40% probability displacement ellipsoids. The intramolecular hydrogen bond is shown dashed.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···O1	0.88 (4)	1.95 (4)	2.705 (4)	143 (4)

The H atom bonded to N3 was located in a difference map and refined freely. All other H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å, and included in the final cycles of refinement using a riding model, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

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: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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