

5-Hydroxy-4-(4-methylbenzoyl)-1,3-diphenyl-1H-pyrazole

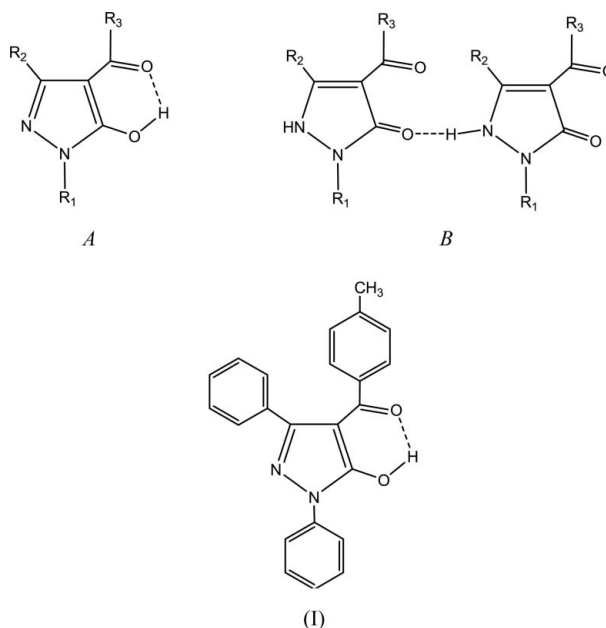
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Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
Disorder in main residue
 R factor = 0.036
 wR factor = 0.096
Data-to-parameter ratio = 13.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The crystal structure of the title compound, $\text{C}_{23}\text{H}_{18}\text{N}_2\text{O}_2$, contains an intramolecular hydrogen bond that links the ketone and hydroxyl groups.Received 11 May 2005
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Comment

Pyrazol-5-one and its derivatives are widely used as biologically active compounds, metal extractants, dyes *etc.* (Ono *et al.*, 1997). The tautomeric structures of these compounds have been extensively studied (Kataeva *et al.*, 2002; Akama *et al.*, 1996; Katritzky *et al.*, 1964). For 4-acyl-substituted pyrazol-5-ones, two forms are stable in the solid state. These are the OH-form (*A*), stabilized by an intramolecular hydrogen bond, and the NH-form (*B*), stabilized by an intermolecular hydrogen bond (see scheme) (Kataeva *et al.*, 2002). The X-ray crystal structure analysis of the title compound, (*I*), was undertaken in order to study its stereochemistry and crystal packing.The present X-ray single-crystal study of compound (*I*) shows (Fig. 1) that it exists in the OH-tautomeric form.

Experimental

The title compound was synthesized according to the method proposed by Jensen (1959) (yield 86%, m.p. 465–466 K). Analysis, required for $\text{C}_{23}\text{H}_{18}\text{N}_2\text{O}_2$: C 77.95, H 5.12, N 7.90%; found: C 77.62, H 5.34, N 7.92%. Block-like single crystals of (*I*) were grown from a mixed solution of methanol and dichloromethane (1:1) by slow evaporation.

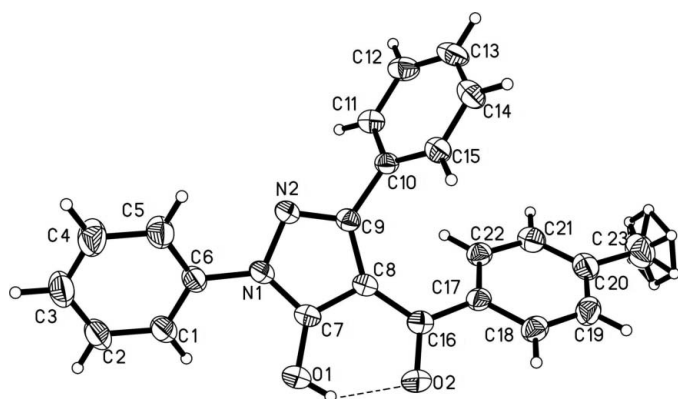


Figure 1
A view of the molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The dashed line indicates a hydrogen bond. Both disorder components are shown.

Crystal data

$C_{23}H_{18}N_2O_2$ $Z = 2$
 $M_r = 354.39$ $D_x = 1.278 \text{ Mg m}^{-3}$
 Triclinic, $P\bar{1}$ Mo $K\alpha$ radiation
 Cell parameters from 28 reflections
 $a = 9.705 (1) \text{ \AA}$ $\theta = 3.9\text{--}15.0^\circ$
 $b = 10.0821 (9) \text{ \AA}$ $\mu = 0.08 \text{ mm}^{-1}$
 $c = 10.746 (1) \text{ \AA}$ $T = 294 (2) \text{ K}$
 $\alpha = 115.284 (8)^\circ$ Block, colourless
 $\beta = 102.13 (1)^\circ$ $0.30 \times 0.28 \times 0.26 \text{ mm}$
 $\gamma = 91.61 (1)^\circ$
 $V = 921.2 (2) \text{ \AA}^3$

Data collection

Siemens P4 diffractometer $\theta_{\text{max}} = 25.3^\circ$
 ω scans $h = 0 \rightarrow 11$
 Absorption correction: none $k = -11 \rightarrow 11$
 4049 measured reflections $l = -12 \rightarrow 12$
 3307 independent reflections 3 standard reflections
 2233 reflections with $I > 2\sigma(I)$ every 97 reflections
 $R_{\text{int}} = 0.009$ intensity decay: 4.8%

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0522P)^2]$
 $R[F^2 > 2\sigma(F^2)] = 0.037$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.096$ $(\Delta/\sigma)_{\text{max}} < 0.001$
 $S = 0.97$ $\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$
 3307 reflections $\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$
 249 parameters Extinction correction: *SHELXL97*
 H atoms treated by a mixture of (Sheldrick, 1997)
 independent and constrained refinement Extinction coefficient: 0.045 (4)

Table 1
Selected geometric parameters (\AA , $^\circ$).

O1—C7	1.3193 (16)	N2—C9	1.3142 (18)
O2—C16	1.2655 (17)	C7—C8	1.3962 (19)
N1—C7	1.3369 (19)	C8—C9	1.4366 (19)
N1—N2	1.3915 (16)		
C7—N1—N2	110.47 (11)	C7—C8—C9	103.31 (13)
C9—N2—N1	106.01 (12)	C7—C8—C16	118.56 (13)
O1—C7—N1	123.18 (13)	N2—C9—C8	111.39 (12)
O1—C7—C8	128.05 (14)	O2—C16—C8	117.03 (13)
N1—C7—C8	108.77 (12)		

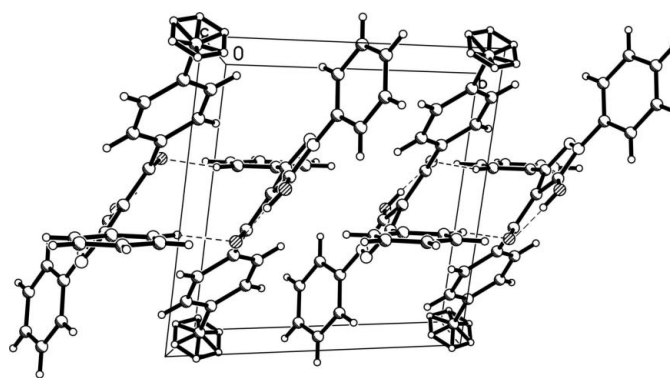


Figure 2
A packing diagram for (I), showing the dimers. Dashed lines indicate hydrogen bonds. Both disorder components are shown.

Table 2
Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1—H1O \cdots O2	0.90 (2)	1.79 (1)	2.5394 (16)	139 (1)
C15—H15 \cdots O2 ⁱ	0.93	2.46	3.378 (2)	170

Symmetry codes: (i) $-x + 1, -y + 2, -z + 1$.

The H atom on atom O1 was located in a difference Fourier map. The O1—H1O distance was restrained to 0.82 (1) \AA and the distance between atoms C7 and H1O was restrained to 1.90 (1) \AA . The H atoms attached to C23 are disordered over two positions, each with 0.5 occupancy. The other H atoms were positioned geometrically, with C—H bond distances of 0.93 (aromatic) or 0.96 \AA (for the CH_3 group), and all H atoms were refined isotropically [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$], except for atom H1O which was refined isotropically.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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