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5-Methyl-2-phenyl-4-[(*Z*)-(2-tolylamino)-phenylmethylene]pyrazol-3(2*H*)-one

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.133
Data-to-parameter ratio = 16.8

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

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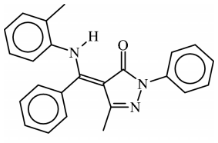
The NH unit on the exocyclic C═C double bond in the title compound, $C_{24}H_{21}N_3O$, lies on the same side of the double bond as the carbonyl unit of the pyrazolone ring, and the two interact through an N—H···O hydrogen bond [2.695 (2) Å].

Comment

As the metal complexes of *N*,*O*-chelates are effective catalysts in the polymerization of α and other polar olefins (Peukert & Keim, 1983; Reuben & Wittcoff, 1988), a number of studies have been directed towards the development of the late transition metal complexes of such ligands (Brookhart & Wagner, 1996; Johnson *et al.*, 1996; Mecking *et al.*, 1998; Rix *et al.*, 1996; Wang *et al.*, 1998). The studies have been complemented by a report that the β-ketoamino unit of the Ni complex is responsible for catalytic activity in the homo- and copolymerization of norbornene and methyl methacrylate (He *et al.*, 2003). The class of pyrazolones, as represented by the title compound, (I), can be conveniently synthesized by condensing commercially available 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone with an amine (Holzer *et al.*, 1999; Wang *et al.*, 2003). In the title compound, the pyrazole (N1/N2/C7–C9) ring is flat, and is coplanar with the C8/C11/C12/N3 fragment [dihedral angle = 3.8 (1)°]. The partial double-bond character of the C8—C11 bond gives rise to a planar configuration of the C8/C11/C12/C18/N3 fragment; the planar feature must be responsible for locking the amino H atom into a hydrogen-bonding position with the carbonyl O atom [N3···O1 = 2.695 (2) Å]. With respect to the C11—N3 bond, the aromatic ring bonded to the C end is twisted by 83.2 (2)° whereas that bonded to the N end is twisted by 60.0 (1)°. The compound resulting from the condensation of 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone and *o*-aminophenol also features an N—H···O interaction [2.750 (3) Å], but it exists as a centrosymmetric hydrogen-bonded dimer owing to the hydroxycarbonyl interaction [O—H···O = 2.724 (3) Å] (Wang *et al.*, 2002). The 1-naphthyl homolog also features a short intramolecular N···O hydrogen bond [2.69 (3) Å] (Wang *et al.*, 2003).



(I)

Experimental

1-Phenyl-3-methyl-4-benzoyl-5-pyrazolone (2.50 g, 9.0 mmol) and 2-toluidine (1.01 g, 9.4 mmol) were dissolved in ethanol (35 ml) and the solution heated under reflux for several hours. The solvent was removed and the pure product obtained upon recrystallization from a

1:1 ethanol/*n*-heptane mixture (35 ml) in about 80% yield. Crystals were grown from ethanol as solvent. CHN elemental analysis, calculated for $C_{24}H_{21}N_3O$: C 78.45, H 5.76, N 111.44%; found: C 78.25, H 5.70, N 11.41%.

Crystal data

$C_{24}H_{21}N_3O$
$M_r = 367.44$
Triclinic, $P\bar{1}$
$a = 9.792$ (1) Å
$b = 9.832$ (1) Å
$c = 11.556$ (2) Å
$\alpha = 101.560$ (2)°
$\beta = 99.974$ (2)°
$\gamma = 108.034$ (2)°
$V = 1002.9$ (2) Å^3
$Z = 2$
$D_x = 1.217$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 848 reflections
$\theta = 2.5$–26.5°
$\mu = 0.08$ mm^{-1}
$T = 298$ (2) K
Block, yellow
0.50 × 0.35 × 0.20 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: none
8614 measured reflections
4346 independent reflections
2832 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.012$
$\theta_{\max} = 27.1°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.133$
$S = 1.00$
4346 reflections
259 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2 + 0.1426P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.15$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.245 (2)	C2—C3	1.381 (2)
N1—C7	1.376 (2)	C3—C4	1.357 (3)
N1—N2	1.402 (2)	C4—C5	1.366 (3)
N1—C1	1.418 (2)	C5—C6	1.382 (3)
N2—C9	1.300 (2)	C7—C8	1.441 (2)
N3—C11	1.329 (2)	C8—C9	1.430 (2)
N3—C18	1.427 (2)	C8—C11	1.390 (2)
C1—C6	1.377 (2)	C9—C10	1.497 (2)
C1—C2	1.380 (2)	C11—C12	1.488 (2)
N2—N1—C7	111.9 (1)	O1—C7—C8	128.5 (1)
N2—N1—C1	118.6 (1)	N1—C7—C8	104.5 (1)
C1—N1—C7	129.5 (1)	C7—C8—C9	105.3 (1)
N1—N2—C9	106.5 (1)	C7—C8—C11	122.4 (1)
C11—N3—C18	128.3 (1)	C9—C8—C11	132.3 (1)
N1—C1—C2	119.5 (2)	N2—C9—C8	111.8 (1)
N1—C1—C6	120.9 (2)	N2—C9—C10	119.0 (2)
C2—C1—C6	119.6 (2)	C8—C9—C10	129.2 (2)
C3—C2—C1	119.5 (2)	N3—C11—C8	118.9 (1)
C4—C3—C2	121.4 (2)	N3—C11—C12	119.5 (1)
C3—C4—C5	118.8 (2)	C8—C11—C12	121.6 (1)
C4—C5—C6	121.5 (2)	N3—C18—C23	118.6 (2)
C1—C6—C5	119.3 (2)	N3—C18—C19	120.1 (2)
O1—C7—N1	127.0 (1)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3N···O1	0.87 (1)	1.94 (1)	2.695 (2)	144 (2)

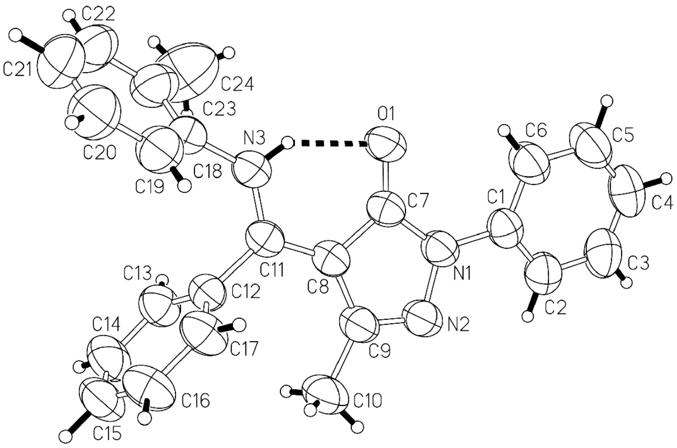


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), with displacement ellipsoids shown at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

H atoms were placed at calculated positions in the riding model approximation (C—H = 0.93 Å for the aromatic H atoms and C—H = 0.96 Å for the aliphatic H atoms), and their displacement parameters were set to $1.2U_{\rm eq}$ of their parent atoms. The amino H atom was located and refined with an N—H distance restraint of 0.85 (1) Å.

Data collection: *SMART* (Bruker, 1999); 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*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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