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4-Benzoyl-*N*-methyl-1,5-diphenyl-1*H*-pyrazole-3-carboxamide

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The title compound, $C_{24}H_{19}N_3O_2$, is a derivative of 1*H*-pyrazole-3-carboxylic acid. The four rings in the structure are not coplanar. The dihedral angle between the mean planes of the pyrazole ring and the carboxamide group is 26.79 (3)°. Molecules are linked by C—H$\cdots$O, π–π and C—H$\cdots\pi$ interactions. In the latter interactions, C$\cdots$centroid distances are 3.659 (4) and 3.816 (4) Å.

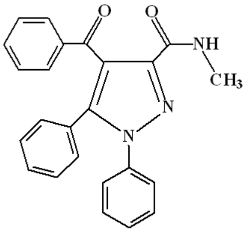
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.058
wR factor = 0.133
Data-to-parameter ratio = 13.5

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

Comment

Pyrazole derivatives have been the subject of much research due to their importance in various applications and their widespread potential biological and pharmacological activities. These include antimicrobial (Mahajan *et al.*, 1991), antiviral (Baraldi *et al.*, 1998), antitumor (Hatheway *et al.*, 1978; Katayama & Oshiyama, 1997), antifungal (Chen & Li, 1998), pesticidal (Londershausen, 1996), anticonvulsant (Lepage & Hublot, 1992), antihistaminic (Mishra *et al.*, 1998) and antidepressant activities (Bailey *et al.*, 1985). The reaction of 4-benzoyl-5-phenyl-2,3-dihydro-2,3-furandione with various phenylhydrazones and phenylhydrazine leads to 4-benzoyl-1,5-diphenyl-1*H*-pyrazole-3-carboxylic acid (Akçamur *et al.*, 1986) and pyridazinones (Akçamur *et al.*, 1997; Verirşen & Erturan, 1998; Şener *et al.*, 2002; Dinçer *et al.*, 2004). 4-Aroyl-5-aryl-2,3-dihydro-2,3-furandiones represent easily accessible building blocks for the synthesis of heterocyclic systems (Terpetschnig *et al.*, 1991; Yıldırım & İlhan, 1997; Hökelek *et al.*, 2002). We describe here the crystal structure of the title compound, (I), as determined by an X-ray crystallographic analysis.



(I)

The structure of (I) (Fig. 1) contains one pyrazole ring (ring *A*: N1/N2/C1/C2/C3) with a carboxamide group (O1/C4/N3/C5) substituted at C3 and three phenyl rings (ring *B*: C7–C12; ring *C*: C13–C18; ring *D*: C19–C24) substituted at C2, C1 and N2, respectively. Ring *B* is attached to the pyrazole ring by a keto group. All four rings are essentially planar. The maximum deviation of the pyrazole ring from planarity is 0.003 (3) Å for atom N1. The four rings are not coplanar. The plane of the pyrazole ring makes dihedral angles of 59.4 (2),

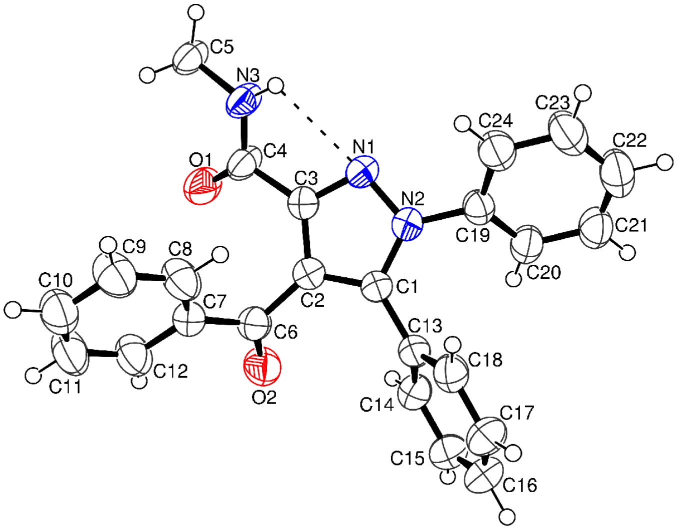


Figure 1
An *ORTEP*-3 (Farrugia, 1997) drawing of the title compound, (I), showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. The intramolecular N—H···N hydrogen bond is represented by a dashed line.

60.8 (2) and 39.5 (2)° with the planes of rings *B*, *C* and *D*, respectively.

The carboxamide group is also planar within 0.008 (3) Å and twisted by 26.79 (3)° out of the mean plane of the pyrazole ring, corresponding to an axial position with respect to the pyrazole ring. The C5—N3—C4—C3 torsion angle is 178.0 (3)° and corresponds to a (+)antiperiplanar configuration. The N1—N2 bond length is 1.370 (4) Å, close to the value found in the literature (Dinçer *et al.*, 2004).

Fig. 1 shows the molecular structure of (I) with the atomic numbering scheme. The packing of (I) is determined by a combination of C—H···O, π–π and C—H···π(phenyl) interactions (Table 2). An intramolecular N—H···N hydrogen bond results in a five-membered ring (N1/C3/C4/N3/H3) fused to the pyrazole ring. The crystal structure has one intermolecular interaction in which atom C5 acts as donor to the symmetry-related atom O2 at $(x, \frac{1}{2} - y, z - \frac{1}{2})$, with a C···O distance of 3.506 (5) Å. Weak intermolecular π–π stacking interactions between ring *B* and its symmetry partner at $(2 - x, 1 - y, -z)$ are observed, with a distance of 3.953 (2) Å between the ring centroids and a perpendicular distance of 3.775 (2) Å. In addition to these interactions, the crystal structure is also stabilized by two C—H···π(phenyl) interactions. In the first of these, atom C10 in the molecule at (x, y, z) forms a C—H···π contact with the centroid (*Cg*3) of ring *C* of the molecule at $(2 - x, 1 - y, -z)$. In the second, atom C8 in the molecule at (x, y, z) forms a C—H···π contact with the centroid (*Cg*4) of ring *D* of the molecule at $(x, \frac{1}{2} - y, z - \frac{1}{2})$.

Experimental

A solution of methylamine hydrochloride (0.10 g, 1.50 mmol) in 20 ml of triethylamine was added slowly, dropwise, to a solution of 4-benzoyl-1,5-diphenyl-1*H*-pyrazole-3-carboxylic acid chloride (0.50 g, 1.30 mmol), obtained easily from 4-benzoyl-5-phenyl-2,3-dihydro-2,3-furandione and phenylhydrazine. Thionyl chloride (Akçamur *et al.*, 1997) in 30 ml of carbon tetrachloride was then added and stirred, with cooling in ice, for 12 h. After evaporation, the oily residue was treated with cyclohexane and the resulting white crude product was filtered off and recrystallized from 2-propanol and dried over P_2O_5 (yield 0.13 g, 27%; m.p. 415 K). Analysis calculated for $C_{24}H_{19}N_3O_2$: C 75.57, H 5.02, N 11.02%; found: C 75.48, H 5.09, N 10.97%.

Crystal data

$C_{24}H_{19}N_3O_2$
$M_r = 381.42$
Monoclinic, $P2_1/c$
$a = 8.6587$ (10) Å
$b = 20.5881$ (19) Å
$c = 11.0082$ (13) Å
$\beta = 100.187$ (9)°
$V = 1931.5$ (4) Å^3
$Z = 4$

$D_x = 1.312$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6124 reflections
$\theta = 2.0$–26.3°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.29 × 0.17 × 0.09 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{\min} = 0.983$, $T_{\max} = 0.992$
20 198 measured reflections

3400 independent reflections
1440 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.107$
$\theta_{\max} = 25.0°$
$h = -10 \rightarrow 10$
$k = -24 \rightarrow 24$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.133$
$S = 1.01$
3400 reflections
251 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.67$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0091 (11)

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.204 (4)	C1—C2	1.380 (4)
O2—C6	1.224 (4)	C1—C13	1.473 (5)
N1—C3	1.332 (4)	C2—C3	1.402 (5)
N2—C1	1.372 (4)	C2—C6	1.488 (5)
N2—C19	1.439 (4)	C3—C4	1.480 (5)
N3—C4	1.304 (4)	C6—C7	1.487 (5)
N3—C5	1.434 (4)		
C3—N1—N2	103.9 (3)	N1—C3—C4	119.3 (3)
N1—N2—C1	112.3 (3)	C2—C3—C4	127.6 (3)
C4—N3—C5	118.0 (3)	O1—C4—N3	124.4 (3)
N2—C1—C2	106.1 (3)	O1—C4—C3	121.9 (4)
C1—C2—C3	105.1 (3)	N3—C4—C3	113.6 (3)
C1—C2—C6	127.3 (3)	O2—C6—C7	121.0 (4)
C3—C2—C6	127.5 (3)	O2—C6—C2	119.7 (4)
N1—C3—C2	112.6 (3)	C7—C6—C2	119.3 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···N1	0.86	2.41	2.723 (4)	102
C5—H5*A*···O2$^{\rm i}$	0.96	2.56	3.506 (5)	170
C10—H10···*Cg*3$^{\rm ii}$	0.93	2.83	3.659 (4)	149
C8—H8···*Cg*4$^{\rm iii}$	0.93	3.06	3.816 (4)	139

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $2 - x, 1 - y, -z$; (iii) $x, -\frac{1}{2} - y, z - \frac{3}{2}$. *Cg*3 and *Cg*4 are the centroids of rings *C* and *D*, respectively.

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H atoms were positioned geometrically and treated using a riding model, with an N—H distance of 0.86 Å, and C—H distances of 0.93 Å for phenyl H atoms and 0.96 Å for methyl H atoms. The displacement parameters of the H atoms were included as $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(carrier atom) [$1.5U_{\rm eq}$(C) for methyl H atoms].

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); 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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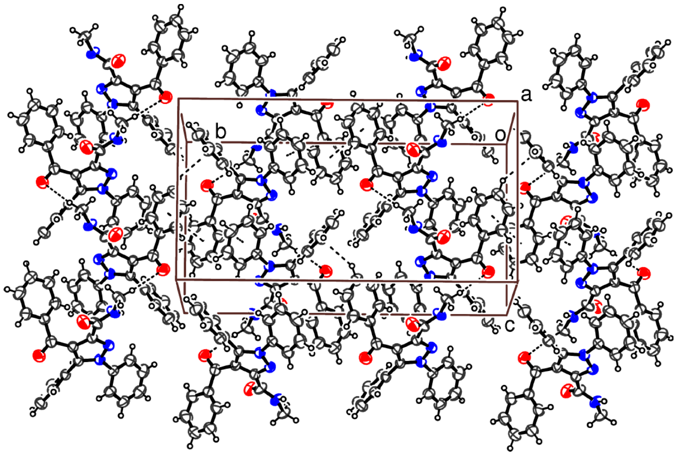

Figure 2

Fig. 2. An *ORTEP*-3 (Farrugia, 1997) drawing of the crystal structure of (I), showing C—H⋯O, π–π and C—H⋯π intermolecular interactions as dashed lines.

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