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Methyl 4-benzoyl-1,5-diphenyl-1*H*-pyrazole-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.026
wR factor = 0.045
Data-to-parameter ratio = 10.1

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

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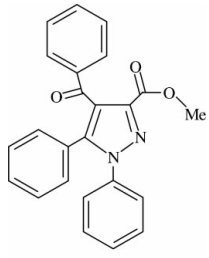
The title compound, $C_{24}H_{18}N_2O_3$, is a derivative of 1*H*-pyrazole-3-carboxylic acid. The pyrazole substituted N atom deviates by 0.0037 (11) Å from the pyrazole ring. The molecules are connected by C—H···O, π—π and C—H···π(phenyl) interactions. In the C—H···π interaction, the C···*Cg* distance is 3.6514 (19) Å (*Cg* is the ring centroid), with a C—H···π angle of 147.6 (11)°.

Comment

Pyrazole derivatives in general are well known nitrogen-containing heterocyclic compounds and these derivatives have been the subject of much research due to their importance in various applications and their widespread potential biological and pharmacological activities such as 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), antidepressant activities (Bailley *et al.*, 1985). The reaction of 4-benzoyl-5-phenyl-2,3-dihydrofuran-2,3-dione with various phenylhydrazones and phenylhydrazine leads to pyrazolecarboxylic acid and pyridazinones (Akçamur *et al.*, 1986, 1997; Şener *et al.*, 2002). 4-Aroyl-5-aryl-2,3-dihydrofuran-2,3-diones represent easily accessible building blocks for the synthesis of heterocyclic systems (Kollenz *et al.*, 1991; Yıldırım & İlhan, 1997; Hökelek *et al.*, 2002). In view of these important properties,, we have undertaken the X-ray diffraction study of the title compound, (I).



(I)

The structure of (I) (Fig. 1) consists of one pyrazole ring (ring *A*: N1/N2/C1–C3) with a carboxylate group (C4/C5/O1/O2) substituted at C1 and three phenyl rings (ring *B*: C7–C12; ring *C*: C13–C18; and ring *D*: C19–C24) substituted at C2, C3 and N1, respectively. Ring *B* is linked to the pyrazole ring by a keto group. As expected, rings *A*, *B*, *C* and *D* are planar. The maximum deviation of the pyrazole ring from planarity is 0.0037 (11) Å for atom N1. The N1—N2 bond length is

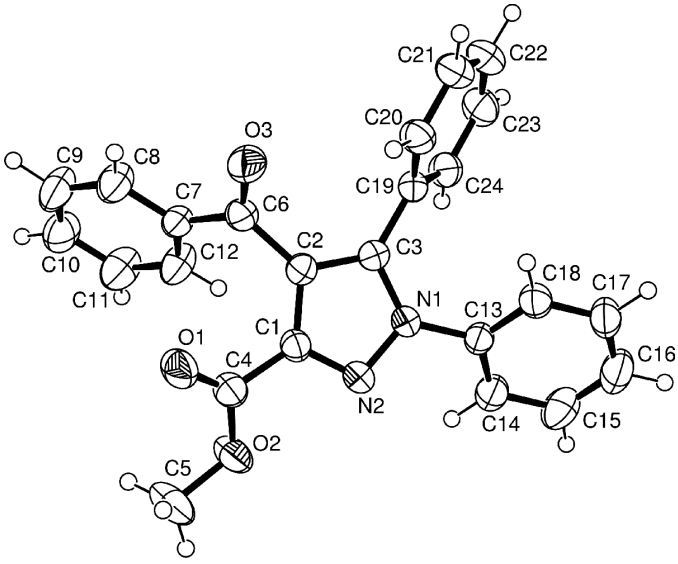


Figure 1
An *ORTEP*-3 (Farrugia, 1997) drawing of the title compound, (I), showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

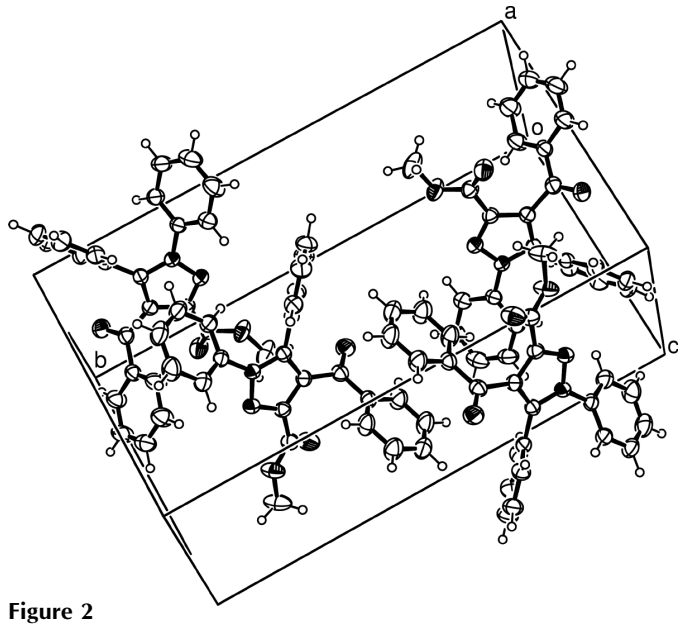


Figure 2
A view of the packing structure of (I), illustrating the π–π and C—H$\cdots\pi$ interactions.

1.3596 (13) Å, shorter than the value found in the literature [1.383 (2) Å; Glidewell *et al.*, 2002], probably due to the steric effects of the substituent groups. The dihedral angles between the rings *A*/*B*, *A*/*C* and *A*/*D* are 59.35 (7), 39.35 (7) and 60.90 (7)°, respectively. The plane of ring *B* forms a small dihedral angle of 22.27 (8)° with the plane of ring *C*, indicating that they are almost parallel to each other. The carboxylate group is also planar and twisted by 26.43 (8)° out of the plane of the pyrazole ring. The O1═C4 and O2—C4 bond lengths are 1.1986 (15) and 1.3248 (15) Å, respectively, in good agreement with those previously found for the carboxylate group (Cannon *et al.*, 2001).

Fig. 1 shows the molecular structure of (I) with the atomic numbering scheme. There is only one molecule in the asymmetric unit. The packing structure of (I) is determined by a combination of C—H$\cdots$O, π–π and C—H$\cdots\pi$(phenyl) interactions (Table 1). Atom H5*A* of the carboxylate group (C5) and atom H21 of ring *D* (C21) interact with the benzoyl group O atom (O3), acting as a single acceptor for both hydrogen bonds [C5$\cdots$O3^{i} = 3.513 (2) Å and C21$\cdots$O3^{ii} = 3.3355 (18) Å; symmetry codes: (i) x, $\frac{3}{2}-y$, $\frac{1}{2}+z$; (ii) $-x$, $1-y$, $1-z$]. In (I), the weak π–π stacking involves the phenyl ring (centroid *Cg*2) of the 4-benzoyl group. The ring in the molecule at (x, y, z) stacks above the ring at ($2-x$, $1-y$, $-z$), with a distance of 3.941 (10) Å between the ring centroids, and a perpendicular distance between the rings of 3.762 (10) Å. In the C—H$\cdots\pi$ interaction, occurring between atom H10 of ring *B* and ring *D* (centroid *Cg*4), the H$\cdots$*Cg* distance is 2.783 (15) Å.

Experimental

Appropriate amounts of 4-benzoyl-1,5-diphenyl-1*H*-pyrazole-3-carboxylic acid (0.50 g, 1.30 mmol), easily obtained from 4-benzoyl-5-phenyl-2,3-dihydrofuran-2,3-dione and phenylhydrazine, as given in Akçamur *et al.* (1986), and a large excess of methanol (50–60 ml) were heated, stirring under reflux, together with catalytic amounts of sulfuric acid for 1–2 h. After cooling to 278 K in a refrigerator, the resulting precipitate was filtered off and recrystallized from methanol and dried on P_2O_5 to give white crystals of methyl 4-benzoyl-1,5-diphenyl-1*H*-pyrazole-3-carboxylate, (I) [yield: 0.39 g (75%), m.p. 452–453 K]. Solvents were dried by refluxing with the appropriate drying agents and distilled before use. All other reagents were purchased from Merck, Fluka, Aldrich and Acros Chemical Co., and used without further purification. The melting point was determined on an Electrothermal 9200 apparatus and is uncorrected.

Crystal data

$C_{24}H_{18}N_2O_3$	D_x = 1.319 Mg m^{-3}
M_r = 382.40	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 20375 reflections
a = 8.6467 (6) Å	θ = 2.0–29.5°
b = 20.5855 (13) Å	μ = 0.09 mm^{-1}
c = 10.9985 (8) Å	T = 293 (2) K
β = 100.297 (6)°	Prism, colourless
V = 1926.2 (2) Å^3	0.50 × 0.36 × 0.19 mm
Z = 4	

Data collection

Stoe IPDS 2 diffractometer	3385 independent reflections
ω rotation scans	2148 reflections with $I > 2\sigma(I)$
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)	R_{int} = 0.134
	θ_{max} = 25.0°
T_{min} = 0.948, T_{max} = 0.982	$h = -10 \rightarrow 10$
37508 measured reflections	$k = -24 \rightarrow 24$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0121P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.026	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.045	$\Delta\rho_{max}$ = 0.13 e Å^{-3}
S = 1.01	$\Delta\rho_{min}$ = −0.11 e Å^{-3}
3385 reflections	Extinction correction: *SHELXL*97
335 parameters	Extinction coefficient: 0.0090 (4)
All H-atom parameters refined	

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5A···O3^{i}	0.981 (18)	2.556 (19)	3.513 (2)	165.1 (13)
C21—H21···O3^{ii}	0.992 (14)	2.569 (14)	3.3355 (18)	134.0 (11)
C10—H10···$Cg4^{iii}$	0.982 (14)	2.783 (15)	3.6514 (19)	147.6 (11)

Symmetry codes: (i) $x, \frac{3}{2}-y, \frac{1}{2}+z$; (ii) $-x, 1-y, 1-z$; (iii) $2-x, 1-y, -z$.

The H atoms were located in a difference map and refined isotropically [C—H = 0.918 (16)–0.993 (18) Å].

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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