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N-(3,4-Dibenzoyl-2,5-dimethylpyrrol-1-yl)-4-methoxybenzamide

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.062
wR factor = 0.167
Data-to-parameter ratio = 15.6

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

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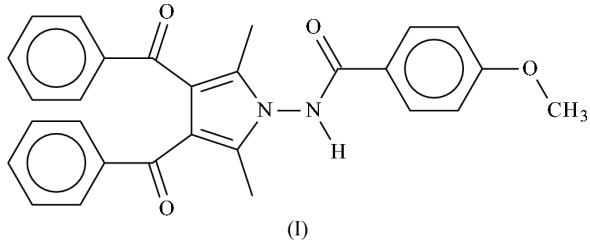
4-Methoxybenzoylhydrazine condenses with benzoylacetone to form the title compound, $C_{28}H_{24}N_2O_4$, a pyrrole that is substituted at all five positions of the five-membered ring. The –N—C(O)—C fragment of the benzoylamide unit is twisted by 80.0 (1)° with respect to the pyrrole ring; two molecules are linked across a centre of inversion by a pair of amide–carbonyl hydrogen bonds [N···O = 2.876 (3) Å].

Comment

Benzoylhydrazine, $C_6H_5C(O)NHNH_2$, condenses with an α,β-diketone, such as acetylacetone, to form the expected Schiff base, which can be used to bind to metal atoms in the *O*,*N*,*O*-chelating mode (Bansse *et al.*, 1994; Ghosh *et al.*, 2004; Liu & Gao, 1998). The reagent reacts with benzoylacetone to furnish the analogous Schiff base, which binds in an identical manner (Chakravarty *et al.*, 1994; Yusupov *et al.*, 1992). In our experiment, the reaction of benzoylacetone with the 4-methoxy-subsituted benzoylhydrazine yielded the anticipated condensation product; however, it reacted with an additional molar equivalent of the diketone to give the title *N*-substituted pyrrole (Fig. 1). A search of the Cambridge Structural Database (Version 5.25; Allen, 2002) found only one example of a pyrrole linked to –NH—C(O)—*R*, *viz.* methyl 1-(3-chlorobenzoylamino)-4-diethylaminocarbonyl-2,5-dimethyl-1*H*-pyrrole-3-carboxylate (Giuseppetti *et al.*, 1985); details of diethyl 1-benzoylamino-2-(4-nitrophenyl)-5-methylpyrrole-3,4-dicarboxylate (Attanasi *et al.*, 1987) were not published.



(I)

The title pyrrole is extremely crowded as all five positions of the ring are substituted. The planar benzoylamide substituent is twisted by 80.0 (1)° with respect to the pyrrole ring; two molecules are linked across a centre of inversion by a pair of amide–carbonyl hydrogens bond [N···O = 2.876 (3) Å; Fig. 1].

Experimental

Glacial acetic acid (3 ml) was added to a methanol soloution (3 ml) of benzoylacetone (21.1 g, 0.13 mol) and 4-methoxybenzoylhydrazine (16.2 g, 0.10 mol) in the condensation of the α,β-diketone with the hydrazine. The mixture was refluxed for 2.5 h. The pale-yellow title compound was isolated as crystals when the filtered solution was set

aside for several days. Analysis calculated for $C_{28}H_{24}N_2O_4$: C 74.32, H 5.35, N 6.19%; found: C 74.58, H 5.29, N 5.09%.

Crystal data

$C_{28}H_{24}N_2O_4$
M_r = 452.49
Orthorhombic, *Pbca*
a = 16.603 (3) Å
b = 16.565 (3) Å
c = 17.483 (4) Å
V = 4808 (2) Å^3
Z = 8
D_x = 1.250 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 28 613 reflections
θ = 3.4–27.5°
μ = 0.08 mm^{-1}
T = 295 (2) K
Prism, yellow
0.35 × 0.22 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.405, T_{max} = 0.983
29 843 measured reflections
4844 independent reflections
2726 reflections with $I > 2\sigma(I)$
R_{int} = 0.097
θ_{max} = 26.3°
$h = -20 \rightarrow 20$
$k = -20 \rightarrow 19$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.167
S = 1.01
4844 reflections
310 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0684P)^2 + 1.5206P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C6	1.232 (3)	C1—C5	1.496 (4)
O2—C7	1.233 (3)	C2—C3	1.443 (4)
O3—C21	1.220 (3)	C2—C6	1.479 (4)
O4—C25	1.364 (3)	C3—C4	1.374 (3)
O4—C28	1.414 (4)	C3—C7	1.472 (4)
N1—N2	1.384 (3)	C4—C8	1.487 (4)
N1—C1	1.375 (3)	C6—C9	1.482 (4)
N1—C4	1.380 (3)	C7—C15	1.486 (3)
N2—C21	1.385 (3)	C21—C22	1.477 (4)
C1—C2	1.375 (4)	C22—C23	1.382 (4)
C25—O4—C28	117.5 (2)	O1—C6—C2	119.5 (3)
N2—N1—C1	123.7 (2)	O1—C6—C9	120.4 (2)
N2—N1—C4	124.4 (2)	C2—C6—C9	120.1 (2)
C1—N1—C4	111.8 (2)	O2—C7—C3	119.8 (2)
N1—N2—C21	118.6 (2)	O2—C7—C15	119.4 (2)
N1—C1—C2	106.6 (2)	C3—C7—C15	120.8 (2)
N1—C1—C5	121.4 (2)	C6—C9—C10	119.3 (3)
C2—C1—C5	131.6 (3)	C6—C9—C14	121.9 (2)
C1—C2—C3	107.3 (2)	C7—C15—C16	121.8 (3)
C1—C2—C6	123.5 (2)	C7—C15—C20	118.3 (3)
C3—C2—C6	129.1 (2)	O3—C21—N2	120.6 (3)
C2—C3—C4	108.2 (2)	O3—C21—C22	123.8 (3)
C2—C3—C7	129.4 (2)	N2—C21—C22	115.6 (2)
C4—C3—C7	122.4 (2)	C21—C22—C23	124.3 (3)
N1—C4—C3	106.0 (2)	C21—C22—C27	117.2 (2)
N1—C4—C8	121.3 (2)	O4—C25—C26	115.7 (3)
C3—C4—C8	132.5 (2)	O4—C25—C24	124.0 (3)

H atoms were placed in calculated positions [C—$H_{aromatic}$ = 0.93 Å, N—$H_{aliphatic}$ = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(C,N)], and the methyl groups were rotated so as to fit the electron density [C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C)]. All H atoms were included in the refinement in the riding model approximation.

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku Corporation, 2002); 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.

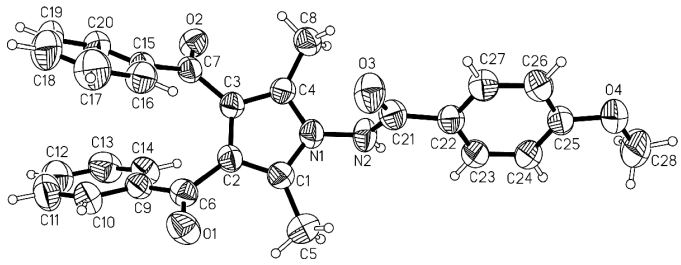


Figure 1
*ORTEP*II (Johnson, 1976) plot of the molecule of (I). Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

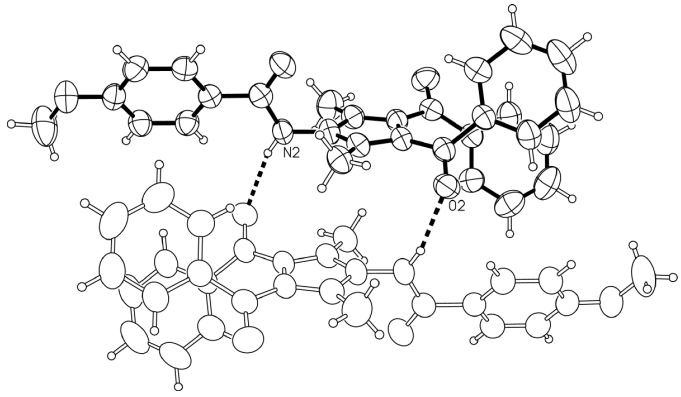


Figure 2
*ORTEP*II (Johnson, 1976) plot of the dimer of (I). Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as spheres of arbitrary radii. Dashed lines indicate hydrogen bonds.

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