

A monoclinic modification of *N*-(3,4-dibenzoyl-2,5-dimethylpyrrol-1-yl)-4-methoxybenzamideShan Gao,^a Li-Hua Huo^a and
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Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

R factor = 0.051

wR factor = 0.139

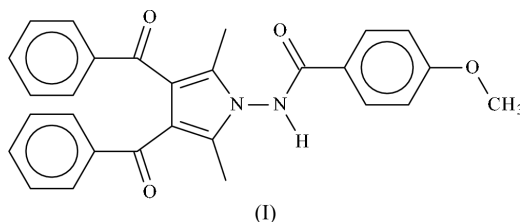
Data-to-parameter ratio = 17.2

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

The molecule of the title compound, $\text{C}_{28}\text{H}_{24}\text{N}_2\text{O}_4$, in the monoclinic modification shows no significant differences in bond dimensions from those in the reported orthorhombic modification [Gao *et al.* (2004). *Acta Cryst. E* **60**, o1733–o1735]. Two molecules are linked across a center of inversion by a pair of amide–carbonyl hydrogen bonds [2.867 (2) \AA].

Comment

N-(Dibenzoyldimethylpyrrol-1-yl)-4-methoxybenzamide represents a pyrrole that is severely crowded owing to substituents on all atoms of the five-membered ring. A previous study had documented the compound belonging to the orthorhombic crystal system (Gao *et al.*, 2004). The synthesis, albeit in a slightly different solvent, afforded the monoclinic modification, (I) (Fig. 1). It is not clear whether the two modifications were obtained in the same reaction, or whether the solvent itself governed the crystallization. The principal bond dimensions are not significantly different in the two modifications; the monoclinic modification is marginally denser ($D_x = 1.263 \text{ Mg m}^{-3}$) than the orthorhombic modification ($D_x = 1.250 \text{ Mg m}^{-3}$).



Experimental

Glacial acetic acid (3 ml) was added to an ethanol solution (3 ml) of benzoylacetone (21.1 g, 0.13 mol) and 4-methoxybenzoylhydrazine (16.2 g, 0.10 mol) in the condensation for the α,β -diketone with the hydrazine. The mixture was refluxed for 3 h. The pale yellow title compound was isolated as crystals when the filtered solution was set aside for several days. Analysis calculated for $\text{C}_{28}\text{H}_{24}\text{N}_2\text{O}_4$: C 74.32, H 5.35, N 6.19%; found: C 74.60, H 5.30, N 6.11%. In the previously reported synthesis (Gao *et al.*, 2004), methanol was used as solvent.

Crystal data

 $\text{C}_{28}\text{H}_{24}\text{N}_2\text{O}_4$ $M_r = 452.49$ Monoclinic, $P2_1/c$ $a = 15.643 (3) \text{ \AA}$ $b = 10.258 (2) \text{ \AA}$ $c = 16.563 (3) \text{ \AA}$ $\beta = 116.49 (3)^\circ$ $V = 2378.7 (8) \text{ \AA}^3$

Z = 4

 $D_x = 1.263 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation

Cell parameters from 20917

reflections

 $\theta = 3.1\text{--}27.5^\circ$ $\mu = 0.09 \text{ mm}^{-1}$

T = 298 (2) K

Prism, yellow

 $0.40 \times 0.27 \times 0.21 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID diffractometer
 ω scans
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.660$, $T_{\max} = 0.982$
 22118 measured reflections

5414 independent reflections
 4210 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -20 \rightarrow 20$
 $k = -13 \rightarrow 13$
 $l = -20 \rightarrow 21$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.139$
 $S = 1.03$
 5414 reflections
 314 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2 + 0.5812P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.32 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.19 \text{ e } \text{\AA}^{-3}$

Table 1

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

| | | | |
|----------|-----------|-----------|-----------|
| N1–N2 | 1.386 (2) | C2–C7 | 1.468 (2) |
| N1–C1 | 1.374 (2) | C3–C4 | 1.376 (2) |
| N1–C4 | 1.376 (2) | C3–C14 | 1.478 (2) |
| C2–C3 | 1.441 (2) | C4–C6 | 1.486 (2) |
| C1–N1–C4 | 112.2 (1) | C3–C2–C7 | 124.6 (1) |
| C1–N1–N2 | 124.7 (1) | C4–C3–C2 | 107.6 (1) |
| C4–N1–N2 | 123.1 (1) | C4–C3–C14 | 123.9 (1) |
| N1–C1–C2 | 106.0 (1) | C2–C3–C14 | 128.5 (1) |
| N1–C1–C5 | 120.2 (1) | C3–C4–N1 | 106.3 (1) |
| C2–C1–C5 | 133.3 (1) | C3–C4–C6 | 132.6 (1) |
| C1–C2–C3 | 107.9 (1) | N1–C4–C6 | 121.0 (1) |
| C1–C2–C7 | 125.9 (1) | | |

H atoms bonded to C atoms were placed in calculated positions [$\text{C–H} = 0.93 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms, and $\text{C–H} = 0.96 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms, which were rotated to fit the electron density] and included in the refinement in the riding-model approximation. The amide H atom was refined isotropically.

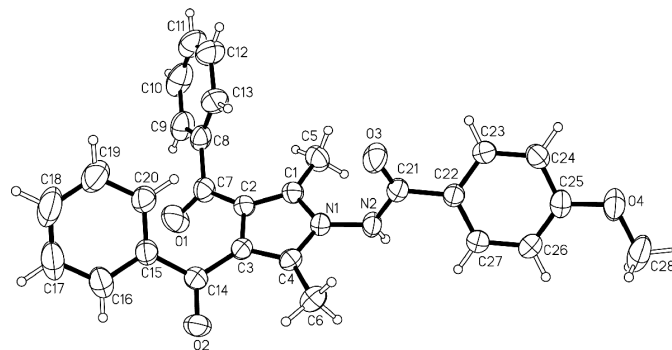


Figure 1 ORTEPII (Johnson, 1976) plot of (I), with ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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