organic papers

1-Amino-5-benzoyl-4-phenylpyrimidin-2(1*H*)-one

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In the title compound, $C_{17}H_{13}N_3O_2$, the two phenyl rings are approximately orthogonal. Intermolecular N—H···O hydrogen bonds generate dimers. The crystal structure is stabilized by N—H···O hydrogen bonds, and C—H···π and N—H···π interactions.

Comment

α,β-Unsaturated ketones and their derivatives have been obtained by Claisen–Schmidt reactions of furfural and *p*-substituted acetophenones. These substances were screened for their *in vitro* antimicrobial activity against some bacteria, employing the disc-diffusion technique (Çetin *et al.*, 2003). Transition metal complexes with pyrimidine derivatives are also of special interest (Sönmez *et al.*, 2004; Reinert *et al.*, 1969). The title compound, (V), was prepared as described previously (Altural *et al.*, 1989). In the present study, (V) was synthesized by the reaction of 4-benzoyl-5-phenylfuran-2,3-dione, (IV), and (1*Z*)-1-phenylethan-1-one semicarbazone in moderate yield (45–60%). The 2,3-dione derivative was synthesized from the reaction of dibenzal, (III), with α,β-unsaturated ketone (I). Compound (I) was obtained by Claisen–Schmidt reaction of benzaldehyde and acetophenone. The reaction sequences depicted in the scheme were followed to obtain (V). Initially, the atomic connectivity in (V) was elucidated from IR and ^{1}H NMR spectra.
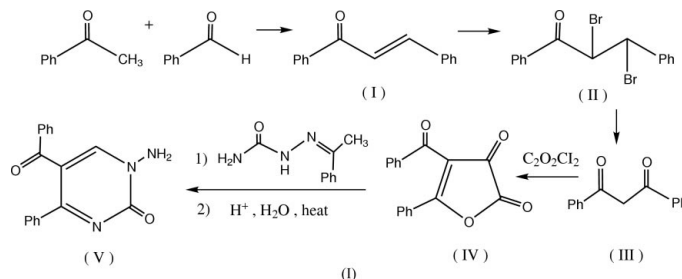
The pyrimidine ring in (V) (Fig. 1) is essentially planar, with a maximum deviation of 0.053 (1) Å for atom C8. The phenyl rings *A* (C1–C6) and *B* (C12–C17) form dihedral angles of 41.05 (5) and 74.84 (3)°, respectively, with the pyrimidine ring. There are two types of intermolecular hydrogen bonds. In the first of these intermolecular interactions, atom N3 acts as a hydrogen-bond donor to O2^i [symmetry code: (i) $1 - x$, $-y$, $-z$]. The N3—H3*A*···O2^i hydrogen bond links inversion-related molecules into dimers. In the second type, atom N3 acts as a donor to O1 at (x, $\frac{1}{2} - y$, $\frac{1}{2} + z$) (Fig. 3 and Table 2). The crystal structure also contains N3—H3*A*···π and C3—H3*C*···π interactions with the centroid, *CgP*, of ring *B* (atoms C12–C17; Fig. 2 and Table 2).
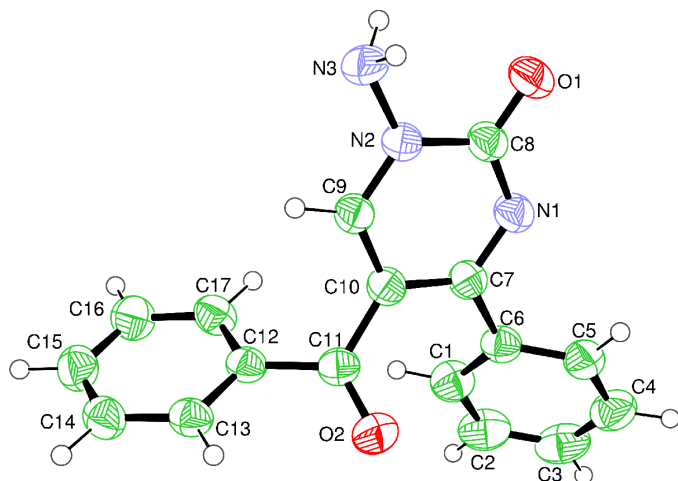
Figure 1
An *ORTEP*-3 (Farrugia, 1997) plot of (V), showing 50% probability displacement ellipsoids and the atomic numbering. H atoms are drawn as spheres of arbitrary radii.

Experimental

A mixture of 4-benzoyl-5-phenylfuran-2,3-dione (1 g), (IV), and (1Z)-1-phenylethan-1-one semicarbazone (0.56 g) (molar ratio 1:1) was refluxed in toluene for 45 min. After cooling, the solid was washed and dried. Water (15 ml) was added to a solution of Schiff base (1 g) in acetic acid (5 ml) and the mixture was then heated under reflux for 30 min. The resulting precipitate was filtered off and then crystallized from a mixture of ethanol–chloroform (3:1) (yield 50%; m.p. 477 K). IR (cm^{-1}, ν): 3297–3176, 1639 (NH$_2$), 3041 (Ar CH), 1683, 1657 (C=O), 1581 (Ar C=C); ^{1}H NMR: δ 5.37 (*br*, 2H, NH$_2$), 7.02–7.08 (*m*, 10H, Ar H), 8.29 (*s*, 1H, C=CH—N); Calculated for C$_{17}$H$_{13}$N$_3$O$_2$ (291): C 70.09, H 4.50, N 14.42%; found: C 70.05, H 4.56, N 14.32%.

Crystal data

C$_{17}$H$_{13}$N$_3$O$_2$	$D_x = 1.383$ Mg m^{-3}
$M_r = 291.30$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 8786
$a = 11.7547$ (11) Å	reflections
$b = 16.6566$ (11) Å	$\theta = 1.8$–28.9°
$c = 7.2986$ (6) Å	$\mu = 0.09$ mm^{-1}
$\beta = 101.722$ (7)°	$T = 296$ K
$V = 1399.2$ (2) Å^3	Prism, light yellow
$Z = 4$	$0.61 \times 0.46 \times 0.23$ mm

Data collection

Stoe IPDS-II diffractometer	$R_{int} = 0.029$
ω scans	$\theta_{max} = 28.8°$
Absorption correction: none	$h = -15 \rightarrow 15$
9982 measured reflections	$k = -22 \rightarrow 22$
3605 independent reflections	$l = -9 \rightarrow 7$
2612 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.037$	$+ 0.0428P]$
$wR(F^2) = 0.105$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
3605 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
252 parameters	$\Delta\rho_{min} = -0.13$ e Å^{-3}
All H-atom parameters refined	Extinction correction: *SHELXL*97
	Extinction coefficient: 0.033 (4)

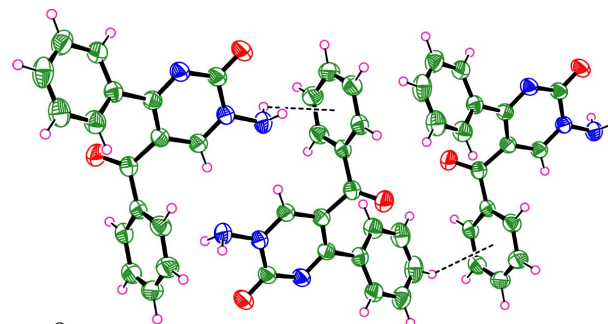**Figure 2**
Part of the crystal structure of (V). Dashed lines show N—H$\cdots\pi$ and C—H$\cdots\pi$ interactions.
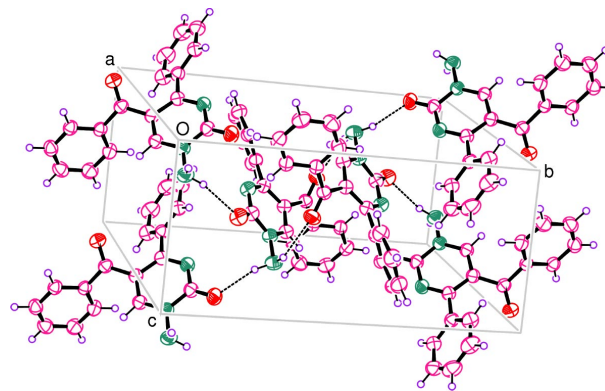

Figure 3
The crystal packing of (V), showing the hydrogen-bonded (dashed lines) three-dimensional network.

Table 1
Selected geometric parameters (Å, °).

N1—C7	1.3118 (14)	O1—C8	1.2157 (13)
N1—C8	1.3699 (15)	O2—C11	1.2191 (14)
N2—C9	1.3401 (14)	C6—C7	1.4855 (15)
N2—C8	1.4112 (15)	C10—C11	1.4863 (15)
N2—N3	1.4204 (13)	C11—C12	1.4835 (16)
C9—N2—C8	121.83 (10)	C9—C10—C7	116.27 (10)
C9—N2—N3	117.73 (10)	C7—C10—C11	124.27 (10)
O1—C8—N1	123.68 (11)	O2—C11—C10	120.68 (11)
O1—C8—N2	119.44 (11)	C12—C11—C10	118.46 (9)
C1—C6—C7—N1	134.50 (12)	C7—C10—C11—O2	−36.96 (17)
C5—C6—C7—C10	145.71 (12)	O2—C11—C12—C17	140.80 (12)
C9—C10—C11—O2	135.53 (12)	C10—C11—C12—C17	−40.29 (15)

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

CgP is the centroid of ring *B* (C12–C17).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N3—H3$A\cdots$O2^i	0.91 (2)	2.37 (2)	3.110 (2)	140 (1)
N3—H3$B\cdots$O1^{ii}	0.92 (2)	2.22 (2)	3.134 (2)	166 (2)
N3—H3$A\cdots CgP^{iii}$	0.91 (2)	2.90 (2)	3.341 (1)	112 (1)
C3—H3$C\cdots CgP^{iv}$	0.95 (2)	2.90 (2)	3.692 (2)	142 (2)

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

All H atoms were located in a difference Fourier map and were refined isotropically. N—H distances are 0.905 (18) and 0.936 (19) Å, and the C—H distances range from 0.947 (14) to 0.993 (13) 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) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Altural, B., Akçamur, Y., Sarıpınar, E., Yıldırım, I. & Kollenz, G. (1989). *Monatsh. Chem.* **20**, 1015–1020.

Çetin, A., Cansız, A. & Digrak, M. (2003). *Heteroatom. Chem.* **14**, 345–347.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Reinert, H., Weiss, R. & Seylers, H. (1969). *Z. Physiol. Chem.* **350**, 1310–1320.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Sönmez, M., Levent, A. & Şekerci, M. (2004). *Russ. J. Coord. Chem.* **30**, 695–699.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED*32 (Version 1.04). Stoe & Cie, Darmstadt, Germany.