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# Benzil 4-nitrophenylhydrazone

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**Key indicators**

Single-crystal X-ray study
*T* = 295 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.049
*wR* factor = 0.127
Data-to-parameter ratio = 16.6

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

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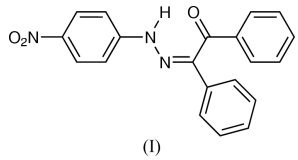
Crystals of the title compound, $C_{20}H_{15}N_3O_3$, were obtained from a condensation reaction of benzil and 4-nitrophenylhydrazine. The phenylhydrazone moiety assumes a planar configuration, and the nitro group and phenyl rings of benzil are inclined to this plane. The overlapped arrangement and the small separation of 3.402 (14) Å between parallel nitrophenyl rings of neighboring molecules suggest the existence of $\pi$–$\pi$ stacking.

## Comment

Some phenylhydrazone derivatives have shown potential application in the field of biochemistry (Okabe *et al.*, 1993). A series of phenylhydrazone derivatives has been prepared in our laboratory (Shan *et al.*, 2003).



(I)

The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles are normal (Table 1). The phenylhydrazone moiety has a planar configuration with a maximum atomic deviation of 0.0124 (12) Å (N2). The two phenyl rings of the benzil moiety are inclined to the phenylhydrazone mean plane, with dihedral angles of 44.07 (5) (C11 ring) and 55.16 (6)° (C21 ring). The nitro group is inclined to the phenylhydrazone plane, with a dihedral angle of 10.28 (13)°. The C1—N2 distance of 1.3916 (18) Å, the N2—N3 distance of

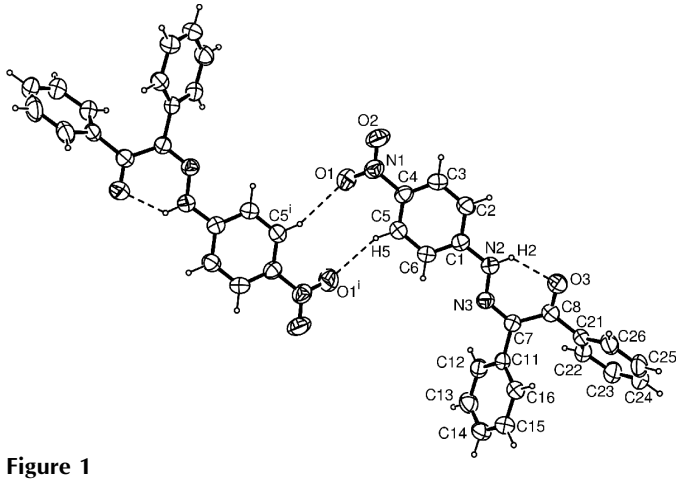



**Figure 1**
Dimer of (I), shown with 40% probability displacement ellipsoids. Dashed lines indicates the hydrogen bonding. [Symmetry code: (i) $1-x$, $y$, $\frac{3}{2}-z$.]

1.3355 (16) Å and the N3—C7 distance of 1.3069 (18) Å suggest electron delocalization within the phenylhydrazone moiety.

The imino H2 atom is intramolecularly hydrogen bonded to the adjacent carbonyl group, with an N2···O3 distance of 2.6172 (17) Å and an N2—H2···O3 angle of 131°. Weak intermolecular C—H···O hydrogen bonding forms dimers of (I), as shown in Fig. 1, the C5···O1$^{i}$ separation and C5—H5···O1$^{i}$ angle being 3.304 (2) Å and 157°, respectively.

The molecular packing is illustrated in Fig. 2. An overlapped arrangement of parallel C4-phenyl and C4$^{ii}$-phenyl rings [symmetry code: (ii) $1-x, 1-y, 1-z$] is observed in the crystal structure. The separation of 3.402 (14) Å suggests the existence of $\pi$–$\pi$ stacking interactions between neighboring molecules.

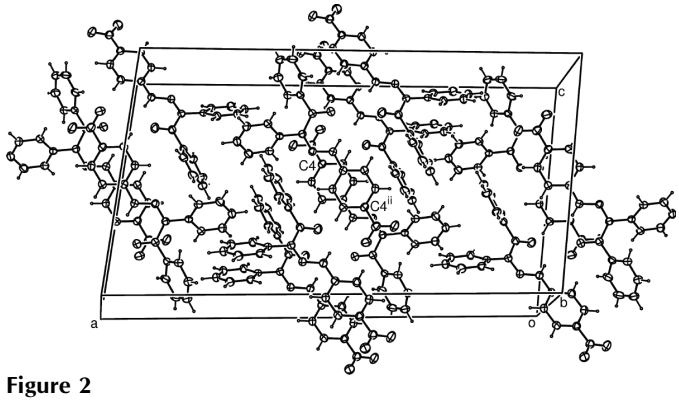



**Figure 2**
A molecular packing diagram showing $\pi$–$\pi$ stacking between neighboring rings. [Symmetry code: (ii) $1-x, 1-y, 1-z$.]

## Experimental

4-Nitrophenylhydrazine (0.31 g, 2 mmol) was dissolved in ethanol (10 ml), then acetic acid (0.2 ml) was slowly added to the ethanol solution with stirring. The solution was heated at about 333 K for several minutes until the solution cleared. An ethanol solution (10 ml) containing benzil (0.42 g, 2 mmol) was slowly dropped into the above solution with continuous stirring. The resulting solution was refluxed for 30 min. When the solution had cooled to room temperature, orange microcrystals appeared. The microcrystals were separated from the solution and washed with cold water three times. Recrystallization was performed twice with benzene, to obtain well-shaped single crystals.

*Crystal data*

$C_{20}H_{15}N_3O_3$
$M_r$ = 345.35
Monoclinic, $C2/c$
$a$ = 29.2648 (15) Å
$b$ = 7.1628 (10) Å
$c$ = 16.4396 (13) Å
$\beta$ = 98.601 (10)°
$V$ = 3407.3 (6) Å$^3$
$Z$ = 8
$D_x$ = 1.346 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 8550 reflections
$\theta$ = 2.0–24.0°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 295 (2) K
Prism, orange
0.37 × 0.20 × 0.20 mm

*Data collection*

Rigaku R-AXIS RAPID diffractometer
$\omega$ scans
Absorption correction: none
15870 measured reflections
3909 independent reflections
3009 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.024
$\theta_{max}$ = 27.5°
$h = -37 \rightarrow 38$
$k = -9 \rightarrow 9$
$l = -19 \rightarrow 21$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.127
$S$ = 1.05
3909 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0588P)^2 + 1.4041P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.14 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.22 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å).

| | | | |
|---|---|---|---|
| O3—C8 | 1.2314 (18) | N3—C7 | 1.3069 (18) |
| N1—C4 | 1.4587 (19) | C7—C8 | 1.487 (2) |
| N2—N3 | 1.3355 (16) | C7—C11 | 1.4920 (19) |
| N2—C1 | 1.3916 (18) | C8—C21 | 1.496 (2) |

H atoms were placed in calculated positions, with C—H = 0.93 Å and N—H = 0.86 Å, and included in the final cycles of refinement as riding atoms, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atom.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); 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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## References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Okabe, N., Nakamura, T. & Fukuda, H. (1993). *Acta Cryst.* C**49**, 1678–1680.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Version 3.00. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381–5209, USA.
Shan, S., Xu, D., Hung, C., Wu, J. & Chiang, M. Y. (2003). *Acta Cryst.* C**59**, o135–o136.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.