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Cyclopentanone 4-nitrophenylhydrazone

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.197
Data-to-parameter ratio = 15.3

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

The title compound, $C_{11}H_{13}N_3O_2$, includes a planar phenylhydrazone moiety and a five-membered carbon ring in an envelope conformation. A short C—C bond distance of 1.435 (6) Å is observed in the five-membered ring. This may be due to the larger displacement parameters of relevant atoms. Neighboring molecules are linked to each other *via* hydrogen bonding, forming a zigzag supramolecular chain along the *b* axis.

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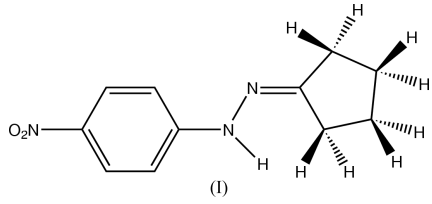
Comment

The molecular structure of (I) is shown in Fig. 1. The phenylhydrazone moiety is approximately planar. The five-membered ring of the compound assumes an envelope conformation, with atom C10 at the flap position. The C9—C10 bond distance of 1.435 (6) Å is much shorter than the expected value for a normal $Csp^3—Csp^3$ bond, and is also



(I)

shorter than the average distance of 1.498 (5) Å for other C—C bonds in the same ring. The shortening of the C9—C10 bond may be due to the larger displacement parameters of both atoms C9 and C10. A similar short C—C bond of 1.448 Å was found in a cyclopentanone derivative, in which the relevant C atoms also had larger displacement parameters (Macias *et al.*, 1989). Neighboring molecules are linked to each other *via* both classical N—H···O hydrogen bonding and weak C—H···O hydrogen bonding, forming a zigzag supramolecular chain along the crystallographic *b* axis, as shown in Fig. 2.

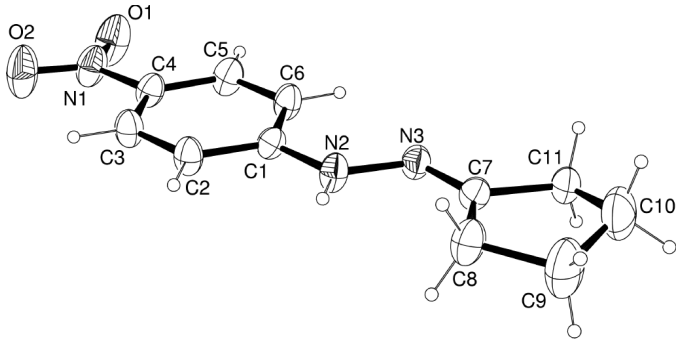


Figure 1
The structure of (I), shown with 30% probability displacement ellipsoids.

Experimental

Cyclopentanone (0.17 g, 0.2 mmol) and acetic acid (2 ml) were added in turn to an ethanol solution (20 ml) of 4-nitrophenylhydrazine (0.30 g, 0.2 mmol). After refluxing the solution for 40 min, it was filtered and the filtrate allowed to stand at room temperature. Yellow powder crystals appeared after 1 d. Well-shaped single crystals were obtained by recrystallization from chloroform.

Crystal data

$C_{11}H_{13}N_3O_2$
$M_r = 219.24$
Orthorhombic, *Pbca*
$a = 21.372\ (3)$ Å
$b = 14.705\ (4)$ Å
$c = 7.211\ (2)$ Å
$V = 2266.2\ (9)$ Å^3
$Z = 8$
$D_x = 1.285$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 4.4$–$12.1°$
$\mu = 0.09$ mm^{-1}
$T = 298\ (2)$ K
Prism, orange
0.58 × 0.46 × 0.28 mm

Data collection

Rigaku AFC-7*S* diffractometer
$\omega/2\theta$ scans
2360 measured reflections
2224 independent reflections
789 reflections with $I > 2\sigma(I)$
$R_{int} = 0.057$
$\theta_{max} = 26.0°$
$h = -10 \rightarrow 26$
$k = -7 \rightarrow 18$
$l = -8 \rightarrow 3$
3 standard reflections
every 150 reflections
intensity decay: 0.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.197$
$S = 1.00$
2224 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0862P)^2 + 0.1882P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C4	1.436 (4)	C7—C11	1.505 (5)
N2—C1	1.364 (4)	C8—C9	1.513 (5)
N2—N3	1.390 (3)	C9—C10	1.435 (6)
N3—C7	1.280 (4)	C10—C11	1.492 (6)
C7—C8	1.483 (5)		
C1—N2—N3	119.7 (2)	C7—N3—N2	115.6 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.86	2.20	3.004 (4)	155
C2—H2···O1^{i}	0.93	2.53	3.287 (4)	139

Symmetry code: (i) $\frac{1}{2} - x, y - \frac{1}{2}, z$.

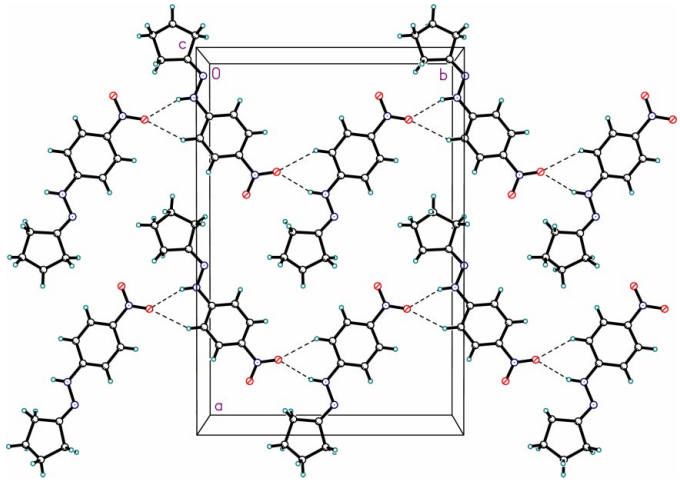


Figure 2
A packing diagram for (I), showing the intermolecular hydrogen bonding.

H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å and N—H = 0.86 Å. All H atoms were included in the final cycles of refinement in the riding mode, with isotropic displacement parameters of $1.2U_{eq}$ of the carrier non-H atoms.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1985); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994).

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