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N-Acetoylphenylhydrazone†

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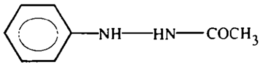
Abstract

The phenyl ring and acetoylhydrazone moiety of the title molecule, $C_8H_{10}N_2O$, are individually planar and these planes are nearly perpendicular to each other. The N atom bonded to the phenyl ring exhibits a pyramidal coordination. Both N atoms are involved in N—H···O hydrogen bonds.

Comment

Acetoylphenylhydrazones are intermediates for dyestuffs and pharmaceuticals. The X-ray structure determination of the title compound, (I), is presented here.



(I)

The mean C—C bond length in the phenyl ring is 1.382 (2) Å. The N1 atom adopts a planar coordination, whereas the coordination of N2 is pyramidal. The C—N bond lengths are normal. The acetoylhydrazone moiety is planar and nearly perpendicular to the plane of the phenyl ring (dihedral angle 86.27°).

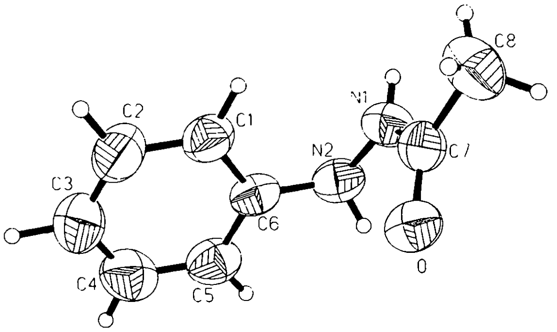


Fig. 1. The structure of title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

In the crystal, the carbonyl O atoms are involved in bifurcated N—H···O hydrogen bonds. Centrosymmetrically related molecules are connected by N2—H1···O hydrogen bonds and pack as linear chains along the **a** direction. These molecular chains are interconnected by N1—H1···O hydrogen bonds.

Experimental

Phenylhydrazine was converted to acetoylphenylhydrazone by treatment with acetic acid in a reaction of the Fischer Indole type (Karmakar, Kar & Ray, 1997). Single crystals were grown by slow evaporation of a chloroform solution of the compound.

Crystal data

$C_8H_{10}N_2O$
$M_r = 150.18$
Orthorhombic
Pbca
$a = 8.836\,(1)$ Å
$b = 10.065\,(1)$ Å
$c = 19.071\,(5)$ Å
$V = 1696.1\,(5)$ Å^3
$Z = 8$
$D_x = 1.176$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 40 reflections
$\theta = 5.49–12.42°$
$\mu = 0.080$ mm^{-1}
$T = 293\,(2)$ K
Thin elongated slab
0.90 × 0.58 × 0.22 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2565 measured reflections
1949 independent reflections
1390 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$

$\theta_{\rm max} = 27.50°$
$h = -1 \rightarrow 11$
$k = -1 \rightarrow 13$
$l = -1 \rightarrow 24$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.141$
$S = 0.987$
1949 reflections
141 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0943P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.015 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O—C7	1.2333 (15)	N1—N2	1.399 (2)
N1—C7	1.330 (2)	N2—C6	1.402 (2)
C7—N1—N2	122.53 (11)	O—C7—N1	121.75 (12)
N1—N2—C6	116.07 (10)	O—C7—C8	122.39 (14)
C5—C6—N2	118.95 (12)	N1—C7—C8	115.86 (14)
C1—C6—N2	122.61 (12)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O^{i}	0.83 (2)	2.00 (2)	2.828 (1)	177 (1)
N2—H1N2···O^{ii}	0.83 (2)	2.15 (2)	2.972 (1)	174 (2)

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

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† Alternative name: *N*′-phenylacetohydrazide.

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The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were located from a difference Fourier map and refined isotropically.

Programs used: data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994); structure solution and molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); structure refinement: *SHELXL93* (Sheldrick, 1993); geometrical calculations *PARST* (Nardelli, 1983).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1336). Services for accessing these data are described at the back of the journal.

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1-(*p*-Chlorophenyl)-5-oxo-3-(2-thienyl)-pyrrolidine-2-carboxylic Acid

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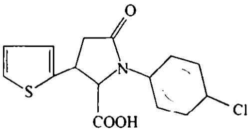
Abstract

The chlorophenyl and thiophene rings of the title molecule, $C_{15}H_{12}ClNO_3S$, are individually planar. The pyrrolidine ring is in a half-chair conformation. The thiophene ring is disordered. The structure is stabilized by a three-dimensional network of C—H···O and O—H···O hydrogen bonds.

Comment

In recent work, we have observed that *N*-phenyl γ-lactam derivatives exhibited Gram-positive and Gram-negative antibacterial activities (Ray, Kar, Roy & Brahma, 1995). The bioactivity of the γ-lactam derivatives depends on the ability of several proteins to inhibit the cross-linking of the bacterial cell wall (Baldwin, Lynch & Pitlick, 1991) and this property is controlled by substituents in the γ-lactam ring (Baldwin, Chan, Gallecher & Otsnka, 1984). In connection with our studies on the synthesis of novel γ-lactam analogues with potential as biological surrogates, we introduced a thiophene ring which can help in activating the γ-lactam system (Roy, Ray & Kar, 1997). The crystal structure determination of the title compound, (I), one of these derivatives, was carried out in order to elucidate the molecular conformation.



(I)

In general, the bond lengths and angles observed in this structure agree with those in related structures (Sivakumar, Fun, Ray, Roy & Nigam, 1995*a*,*b*), but the unusually long C12—C15 [1.596 (4) Å] and C13—C15 [1.522 (6) Å] distances are probably systematically in error because of the disorder of the thiophene ring (as explained below).

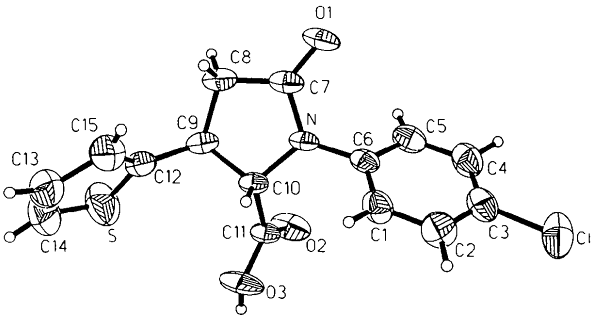


Fig. 1. The structure of title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme. Only the major conformer is shown.

The pyrrolidine ring is in a half-chair conformation, with asymmetry parameter $\Delta C_2(C7) = 0.005\,(2)°$ (Nardelli, 1983*a*). The deviations of atoms C9 and C10 from the plane defined by N, C7 and C8 are −0.204 (4) and 0.260 (3) Å, respectively. The chloro-

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