Data collection

Siemens SMART CCD area-detector diffractometer	2460 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.041$
Absorption correction: empirical using *SADABS* (Sheldrick, 1996)	$\theta_{max} = 27.49°$
	$h = 0 \rightarrow 13$
	$k = 0 \rightarrow 16$
$T_{min} = 0.557$, $T_{max} = 0.777$	$l = 0 \rightarrow 20$
18 432 measured reflections	Intensity decay: negligible
3180 independent reflections	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.057$	$\Delta\rho_{max} = 0.94$ e Å^{-3}
$wR(F^2) = 0.182$	$\Delta\rho_{min} = -0.35$ e Å^{-3}
$S = 1.077$	Extinction correction: none
3180 reflections	Scattering factors from
190 parameters	*International Tables for*
H atoms constrained	*Crystallography* (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.1009P)^2 + 2.4231P]$ where $P = (F_o^2 + 2F_c^2)/3$	

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

S1A—C2	1.702 (3)	O1—C3	1.436 (4)
O1—C4	1.366 (4)		
C11—N1—C10	114.6 (3)	O2—C12—O3	126.9 (4)
C11—N1—C13	112.3 (2)	O2—C12—C11	122.0 (3)
C10—N1—C13	111.5 (2)	O3—C12—C11	111.1 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N1···O2	0.90	2.22	2.753 (4)	118
C10—H10A···O1	0.97	2.31	2.758 (4)	107
C11—H11B···Br	0.97	2.87	3.734 (3)	149
O3—H3C···O3^i	1.22	1.22	2.423 (4)	163
N1—H1N1···O2^{ii}	0.90	2.10	2.874 (4)	143
C8—H8A···O2^{ii}	0.93	2.59	3.392 (5)	145
C11—H11B···Br	0.97	2.87	3.734 (3)	149

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

The thiophene ring is highly disordered over two orientations: atoms S1 and C1 were refined with occupancies of 72 and 28%, respectively.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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N,N'-Bis(picolinoyl)hydrazine

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Abstract

The X-ray structure determination of the title compound, N,N'-bis(2-pyridinecarbonyl)hydrazine, $C_{12}H_{10}N_4O_2$,

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indicated that it is non-planar and twisted in the form of butterfly wings. The two carbonyl bonds are located in an s-syn conformation relative to the N—N bond, through which there is a crystallographic twofold axis. The molecules are packed in chains running along the c axis.

Comment

N,N'-Bis(picolinoyl)hydrazine is the basic structural component of some heterocyclic ring systems (Daniel & Peet, 1978). It acts as an inhibitor of the human immunodeficiency virus (HIV) integrase (Zhao et al., 1997) and is a potential antitumour agent (Rutner et al., 1974). Recently, in the synthesis of 4-(p-methoxy-phenyl)-3,5-bis(2-pyridyl)-4H-1,2,4-triazole (Fun et al., 1999), we unexpectedly obtained single crystals of the title compound, (I), and its crystal structure is reported herein.

(I)
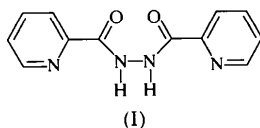
The X-ray analysis of (I) indicated that it is non-planar and twisted in the form of butterfly wings. There is a half molecule in the asymmetric unit and a crystallographic twofold axis through the N2—N2^i bond relates the symmetry-related moieties [symmetry code: (i) $1 - x$, y, $\frac{1}{2} - z$]. The two carbonyl groups are located in an s-syn conformation relative to the N2—N2^i bond. The bond lengths and angles in the structure are in the usual ranges. The conformation of the two planar systems about the N2—N2^i bond is given by the C6—N2—N2^i—C6^i torsion angle of $-70.9(2)°$. The twisting is probably caused mainly by the hydrogen bond involving the O atom, as the O1···O1^i distance of 3.388 (2) Å is much longer than the sum of the van der Waals radii (2.8 Å), indicating that there is no steric hindrance between them.

The presence of intramolecular N—H···N and inter-molecular N—H···O hydrogen bonds enhances the packing of the molecules. The molecules pack as a one-dimensional infinite chain along the c axis because of the intermolecular N—H···O hydrogen bonds between the central hydrazine moiety and the carbonyl-O atom.
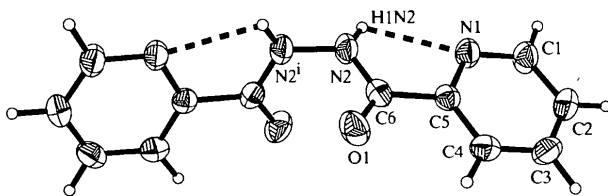

Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry code: (i) $1 - x$, y, $\frac{1}{2} - z$.]
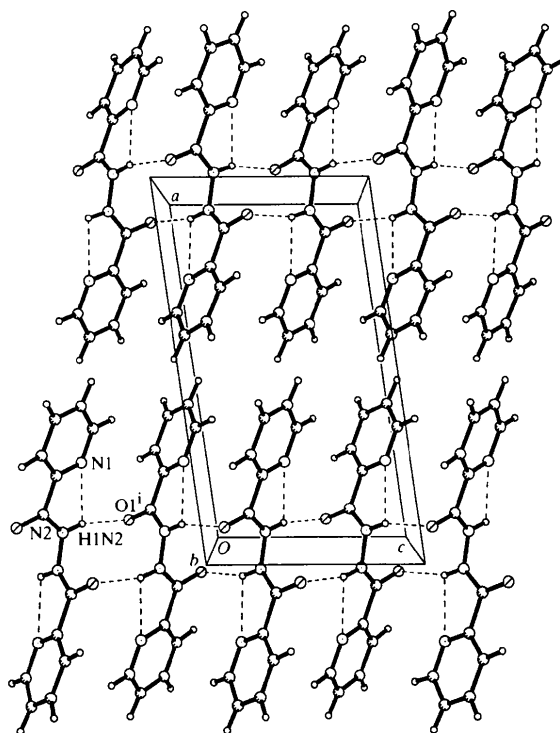
Fig. 2. The packing of molecules of (I) viewed down the b axis showing the infinite chain.

Experimental

The title compound was synthesized by the reaction of equivalent amounts of n-amyl nitrite and 2-pyridoylhydrazine in absolute ethanol for 18 h at 403–413 K (Graf, 1933). Single crystals suitable for X-ray analysis were obtained from acetone.

Crystal data

C₁₂H₁₀N₄O₂	Mo $K\alpha$ radiation
$M_r = 242.24$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 2109
$C2/c$	reflections
$a = 13.4224(1)$ Å	$\theta = 2.39–28.32°$
$b = 11.1181(1)$ Å	$\mu = 0.103$ mm^{-1}
$c = 7.5341(6)$ Å	$T = 293(2)$ K
$\beta = 97.761(3)°$	Block
$V = 1114.0(2)$ Å^3	$0.36 \times 0.18 \times 0.16$ mm
$Z = 4$	Colourless
$D_x = 1.444$ Mg m^{-3}	
D_m not measured	

Data collection

Siemens SMART CCD area-detector diffractometer	$R_{int} = 0.051$
	$\theta_{max} = 28.32°$
ω scans	$h = -16 \rightarrow 17$
Absorption correction: none	$k = -9 \rightarrow 14$
3782 measured reflections	$l = -9 \rightarrow 10$
1375 independent reflections	
999 reflections with	
$I > 2\sigma(I)$	

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.146$
$S = 1.042$
1375 reflections
102 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0845P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: none
Scattering factors from
International Tables for
Crystallography (Vol. C)

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

O1—C6	1.219 (2)	N1—C1	1.339 (2)
N1—C5	1.333 (2)	N2—C6	1.356 (2)
O1—C6—N2	122.7 (1)	N2—C6—C5	114.7 (1)
O1—C6—C5	122.6 (2)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H1N2⋯N1	0.89 (2)	2.36 (2)	2.678 (2)	101 (2)
N2—H1N2⋯O1^i	0.89 (2)	2.28 (2)	3.124 (2)	160 (2)
C2—H2⋯O1^{ii}	0.97 (2)	2.58 (2)	3.220 (2)	124 (2)

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

All the H atoms were refined. C—H distances are in the range 0.97 (2)–0.99 (2) Å.

Data collection: *SMART* (Siemens, 1996a). Cell refinement: *SAINT* (Siemens, 1996b). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *XP* in *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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4-Chloro-2-(morpholinomethyl)phenol

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Abstract

The title compound, $C_{11}H_{14}ClNO_2$, crystallizes in the triclinic system with two independent molecules in the asymmetric unit. The molecular packing is governed by C—H⋯O hydrogen bonds leading to the formation of dimeric pairs.

Comment

Aminomethylphenols have been known for their uses as pigments (Fechner et al., 1982; Fechner, Kranz & Polster, 1983; Fechner, Polster & Kranz, 1983), as insecticides (Nisato & Boveri, 1984) and as intermediates in medicine. Recently, a lot of attention has been paid to aminomethylphenols for their use as excellent mimics for the active site of enzymes. In this paper, we report the structure of a new aminomethylphenol, 4-chloro-2-(morpholinomethyl)phenol, (I), derived from morpholine and *p*-chlorophenol.

(I)
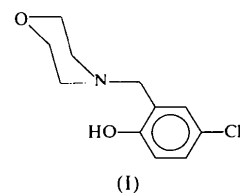
The asymmetric unit of (I) contains two independent molecules (Fig. 1). The two molecules are structurally similar in that the values of the corresponding bond lengths and angles are very similar, and are also comparable with reported values (Shanmuga Sundara Raj et al., 1993; Shanmuga Sundara Raj, Ponnuswamy et al., 1994; Shanmuga Sundara Raj, Velmurugan et al., 1994).

The morpholine rings of the two molecules adopt chair conformations. The best planes of the rings pass

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