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2,2′-Bis(2-nitrobenzylidene)pyridine-2,6-dicarbohydrazide

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Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.056
wR factor = 0.132
Data-to-parameter ratio = 13.9

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

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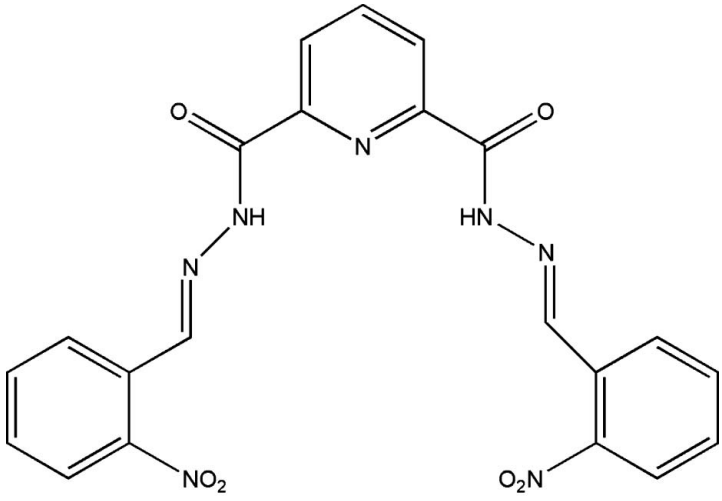
The title molecule, $C_{21}H_{15}N_7O_6$, has crystallographic twofold symmetry. In the crystal structure, intermolecular N—H···O hydrogen bonds [H···O = 2.125 (17) Å] link the molecules, forming one-dimensional chains along [001].

Comment

Tridentate ligands containing the 2,6-dipicolinoylhydrazone group have been intensively studied due to their interesting coordination modes (Chen *et al.*, 1997; Thompson, 2002; Zhao *et al.*, 2004). We report here the synthesis and crystal structure of a new tridentate ligand, (I).



(I)

The molecule of (I) (Fig. 1) has crystallographic twofold symmetry. The dihedral angle between the planes formed by the pyridine and nitrobenzene rings is 10.88 (1)°.

In the crystal structure, intermolecular N—H···O hydrogen bonds link the molecules, forming one-dimensional chains running parallel to the *c* axis. These chains are further reinforced by weak intermolecular C—H···O hydrogen bonds (Fig. 2 and Table 1).

Experimental

To a solution of 2-nitrobenzaldehyde (1.66 g, 11 mmol) in absolute ethanol (40 ml), a suspension of 2,6-dipicolinoylhydrazone in the same solvent (50 ml) was added at 323 K. The mixture was refluxed for 6 h. The product, as yellow needles, was then filtered off, washed with hot ethanol (20 ml portion) three times, and dried *in vacuo*. Crystals suitable for X-ray diffraction were obtained from a solution of (I) in dimethylformamide:methanol (3:1 *v*/*v*) over a period of about three weeks.

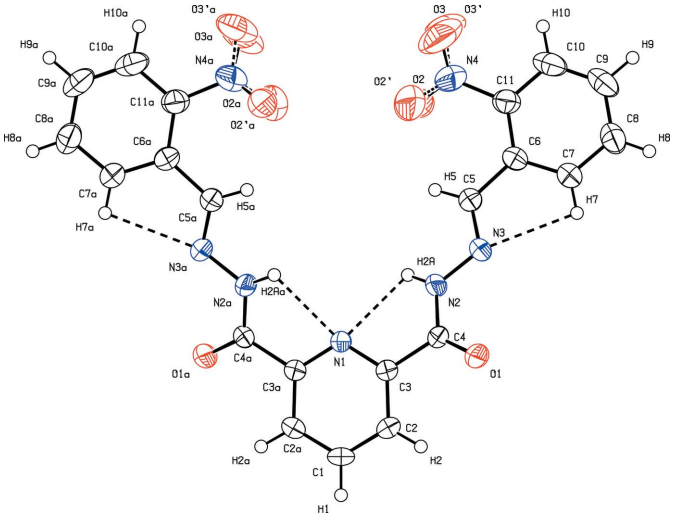


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids. The dashed lines indicate intramolecular hydrogen bonds or, with respect to the N—O bonds in the nitro groups, they indicate the minor component of disorder. Atoms labelled with suffix 'a' are related by the symmetry operator $(-x, y, -z-\frac{1}{2})$.

Crystal data

$C_{21}H_{15}N_7O_6$
M_r = 461.40
Monoclinic, $C2/c$
a = 19.889 (3) Å
b = 12.5106 (17) Å
c = 8.3010 (11) Å
β = 92.861 (3)°
V = 2062.9 (5) Å^3
Z = 4
D_x = 1.486 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 292 (2) K
Needle, yellow
0.20 × 0.10 × 0.06 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: none
9263 measured reflections
2454 independent reflections
1578 reflections with $I > 2\sigma(I)$
R_{int} = 0.129
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.133
S = 0.92
2454 reflections
177 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O2	0.93	2.26	2.839 (3)	120
C5—H5···O1^{i}	0.93	2.49	3.199 (2)	134
N2—H2A···O1^{i}	0.893 (17)	2.125 (17)	2.9612 (19)	155.8 (15)
N2—H2A···N1	0.893 (17)	2.322 (17)	2.6690 (15)	103.0 (12)
C7—H7···N3	0.93	2.49	2.801 (2)	100

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

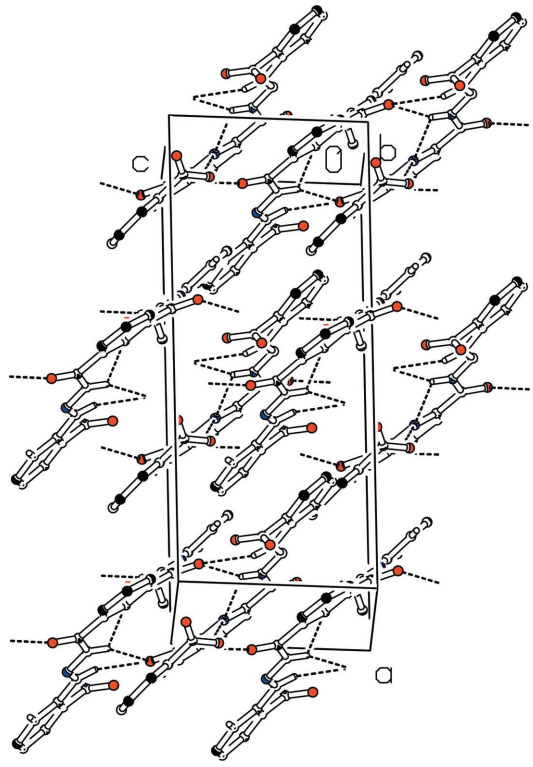


Figure 2
Partial packing plot of (I), with dashed lines indicating hydrogen bonds.

Initially all H atoms were visible in the difference map, but H atoms bonded to C atoms were subsequently placed in calculated positions with C—H = 0.93 Å and included in the riding-model aproximation, with U_{iso}(H)= 1.2U_{eq}(C). The positional parameters of the H atom bonded to N2 were refined freely, with U_{iso}=1.2U_{eq}(N). The O atoms of the nitro group were refined freely with the sum of the occupancies restrained to be 1. The occupancy of the major component is 0.67 and the minor 0.33.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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