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2-Amino-3-(4-pyridylmethyleneamino)butenedinitrile

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Key indicators

Single-crystal X-ray study
T = 571 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.038
wR factor = 0.099
Data-to-parameter ratio = 8.5

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

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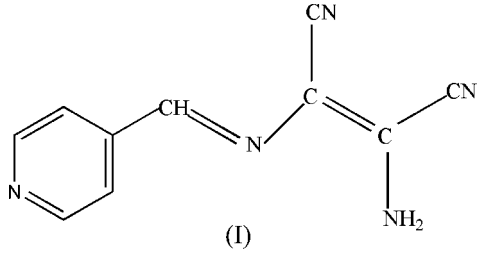
In the title crystal structure, $C_{10}H_7N_5$, intermolecular N—H···$N_{pyridine}$ hydrogen bonds connect molecules to form one-dimensional chains propagating along [031]. In powder form, the compound exhibits second harmonic generation effects.

Comment

We have an interest in molecules containing the diaminomaleonitrile group, which are useful for the synthesis of dyes and pharmaceuticals (Begland *et al.*, 1974; Begland & Del, 1975), and we have already determined the structures of 2-amino-3-(4-methoxybenzylideneamino)butenedinitrile monohydrate (Shi *et al.*, 2006) and 2-amino-3-(4-hydroxybenzylideneamino)butenedinitrile (Wu *et al.*, 2006). We report here the crystal structure of a related compound, (I), Fig. 1.



(I)

All non-H atoms of the molecule are essentially coplanar with a maximum deviation of 0.395 (9) Å for atom N5′ from the molecular plane. Atom N5′ is a disorder component of a nitrile group comprising N5/N5′. An intermolecular N—H···N hydrogen bond involving the amino group and the N atom of the pyridyl ring (Table 1) connects molecules to form

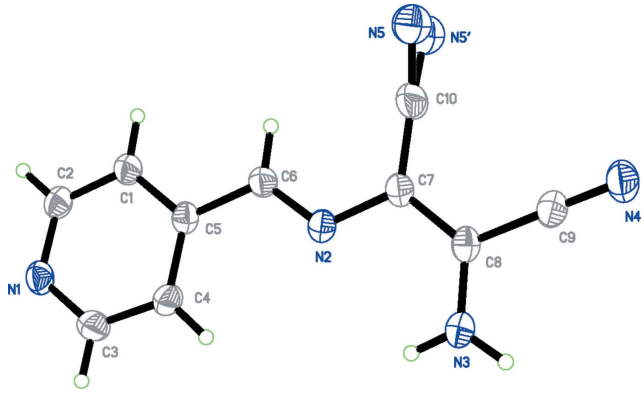


Figure 1
The molecular structure of (I), showing the crystallographic numbering scheme with displacement ellipsoids drawn at the 30% probability level. Atom labels N5/N5′ represent the two components of the disordered atom.

a one-dimensional chain propagating along [031]. As a powder, the title compund displays second harmonic generation with an intensity 0.43 times that of urea.

Experimental

4-Pyridylaldehyde (3.5 g, 0.0327 mol) and diaminomaleonitrile (3.5 g, 0.0324 mol) were dissolved in ethanol (70 ml) and refluxed for half an hour. The crude product was filtered, washed with ethanol and dissolved in acetonitrile. Yellow single crystals were obtained on standing at room temperature for two weeks. Analysis found: C 60.74, H 3.26, N 35.71%; calculated for $C_{10}H_7N_5$: C 60.90, H 3.58, N 35.52%. The IR stretching vibrations of the nitrile groups appeared at 2241 and 2194 cm^{-1}, whereas the vibrations of C═N bond appeared at 1616 and 1573 cm^{-1}.

Crystal data

$C_{10}H_7N_5$
M_r = 197.21
Orthorhombic, $Pna2_1$
a = 15.405 (6) Å
b = 4.8694 (19) Å
c = 13.587 (5) Å
V = 1019.2 (7) Å^3
Z = 4
D_x = 1.285 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 571 (2) K
Prism, yellow
0.27 × 0.17 × 0.15 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.977, T_{max} = 0.987
5568 measured reflections
1153 independent reflections
1021 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.099
S = 1.05
1153 reflections
136 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.104P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3B···N1^{i}	0.86	2.02	2.865 (3)	167

Symmetry code: (i) $-x+\frac{3}{2}, y+\frac{3}{2}, z+\frac{1}{2}$.

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. The H atoms were placed in calculated positions and refined as riding, with C—H = 0.93 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2U_{eq}(N). The N atom of one of the nitrile groups was modelled as disordered over two sites and the components were refined isotropically with final occupancies of 0.51 (2)/0.49 (2) for N5/N5′.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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