Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1*H*-Indole-3-carbaldehyde azine

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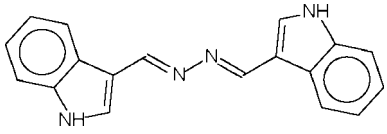
Received 22 January 2008; accepted 28 January 2008

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.122; data-to-parameter ratio = 15.8.

The molecule of the title compound, $C_{18}H_{14}N_4$, lies on a center of inversion such that there is one half-molecule in the asymmetric unit. The N—N single bond adopts a *trans* configuration and the indole fused-ring system is nearly coplanar with the –CH═N—N═CH– fragment [dihedral angle = 9.8 (2)°]. Adjacent molecules are linked by indole–azine N—H···N hydrogen bonds into a layer motif.

Related literature

For the synthesis, see: Alemany *et al.* (1970); Swaminathan & Narasimhan (1964). For the crystal structures of some aromatic azines, for example, benzalazine, see: Burke-Laing & Laing (1976); Mom & de With (1978); Sinha, 1970). For other heterocyclic aldehyde azines, see: Lin *et al.* (2001*a*,*b*); Wu *et al.* (2006).



Experimental

Crystal data

$C_{18}H_{14}N_4$
M_r = 286.33
Monoclinic, $P2_1/c$
a = 5.0849 (2) Å
b = 10.6708 (4) Å
c = 13.4435 (5) Å
β = 94.366 (3)°
V = 727.33 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 295 (2) K
0.33 × 0.27 × 0.17 mm

Data collection

Bruker APEX2 diffractometer
Absorption correction: none
5388 measured reflections
1659 independent reflections
1085 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.121
S = 1.01
1659 reflections
105 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N2^{i}	0.87 (2)	2.21 (2)	3.065 (2)	167 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

We thank the Science Fund (12–02-03–2031) for supporting this study, and the University of Malaya for the purchase of the diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2186).

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supporting information

Acta Cryst. (2008). E**64**, o555 [doi:10.1107/S1600536808003164]

1*H*-Indole-3-carbaldehyde azine

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S1. Comment

Azines are readily synthesized by condensing hydrazine with an aldehyde; the crystal structures of a large number of substituted benzaldehdye azines have been reported. The structure of the parent aromatic compound, benzalazine, has been known for a long time (Burke-Laing & Laing, 1976; Mom & de With, 1978; Sinha, 1970). There are few examples of heterocyclic azines, and their rarity can be attributed to the difficulty of synthesizing the starting aldehyde reactant. Among the few are, for example, unsubstituted and methyl-subsituted thiophene-2-aldehyde azine (Lin *et al.*, 2001*a*, 2001*b*) and a pyrrole derivative has recently been reported (Wu *et al.*, 2006).

3-Indole azine has been known for some time; it was first synthesized from indole-3-carboxaldehyde and hydrazine in order to examine its psychopharmacological activity (Alemany *et al.*, 1970; Swaminathan Narasimhan, 1964). The title compound was the unexpected decomposition product of the Schiff base derived from the condensation of carbohydrazide and indole-3-carboxaldehyde. The molecule (Scheme I, Fig. 1) lies about a center-of-inversion such that there is half a molecule in the asymmetric unit. The N–N single-bond adopts a *trans* configuration and the indolyl fused-ring is nearly coplanar with the –CH=N–N=CH– fragment. Adjacent molecules are linked by an N–$H_{indole}\cdots N_{azine}$ hydrogen bonds into layer motif (Fig. 2).

S2. Experimental

The reaction of carbohydrazide (0.3 g, 3.3 mmol) and indole -3-carboxaldehyde (1 g, 6.6 mmol) in ethanol under reflux for 2 h gave the corresponding Schiff base. This compound (0.2 g, 0.6 mmol), zinc acetate (0.06 g,0.3 mmol) and several drops of triethylamine were dissolved in 10 ml e thanol. The contents were heated in a 25-ml, stainless-steel Paar bomb for for 2 d at 373 K. The bomb was cooled to room temperature over several hours. Well formed crystals were isolated from the cooled bomb.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U(C). The amino H-atom was located in a difference Fourier map, and was freely refined.

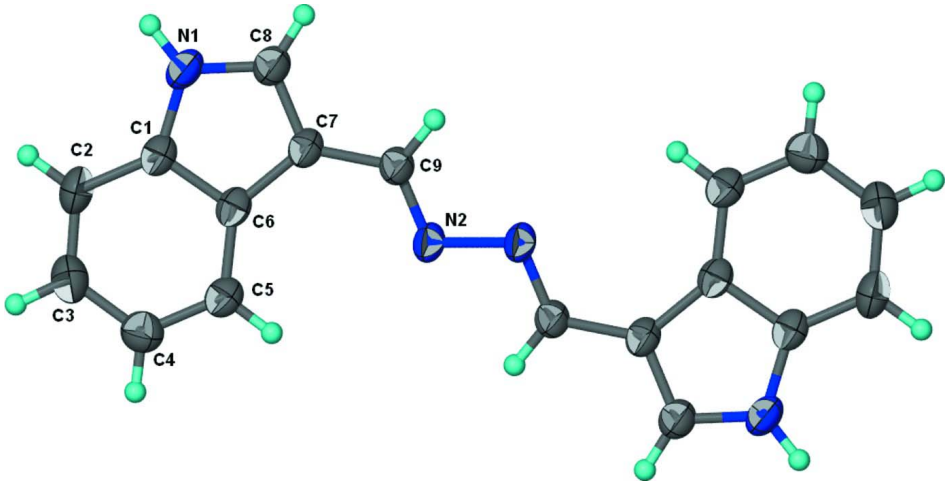

Figure 1

Displacement ellipsoid plot of (I) at the 50% probability level. H atoms are drawn as spheres of arbitrary radiius.

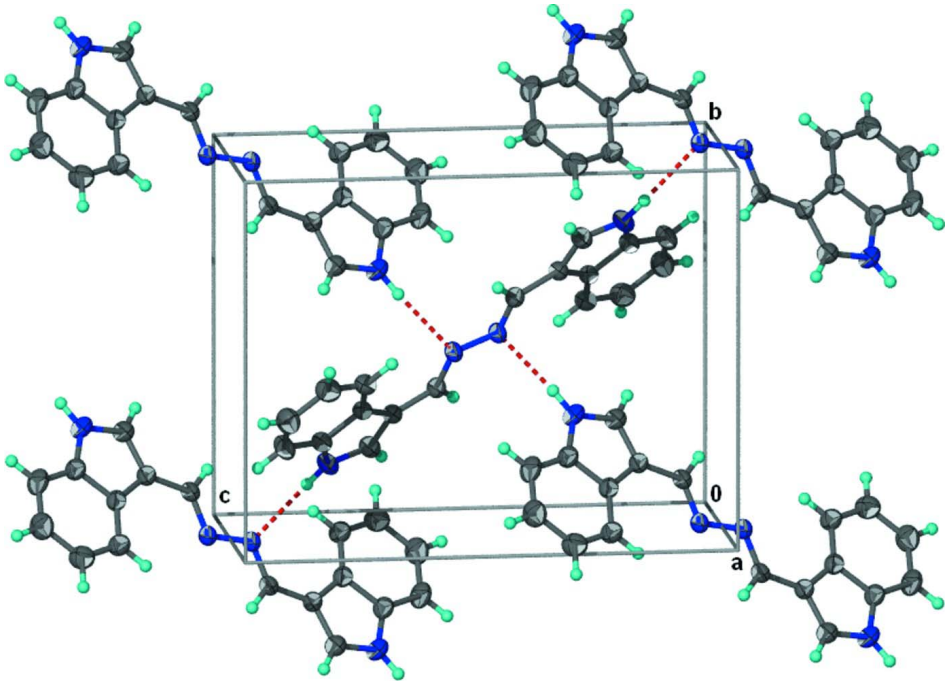

Figure 2

Layer structure of (I).

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Crystal data

$C_{18}H_{14}N_4$
$M_r = 286.33$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.0849\ (2)$ Å
$b = 10.6708\ (4)$ Å
$c = 13.4435\ (5)$ Å
$\beta = 94.366\ (3)°$
$V = 727.33\ (5)$ Å^3
$Z = 2$
$F(000) = 300$
$D_x = 1.307$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1012 reflections

θ = 2.3–23.6°
μ = 0.08 mm^{-1}
T = 295 K

Irregular block, green–yellow
0.33 × 0.27 × 0.17 mm

Data collection

Bruker APEX2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
5388 measured reflections
1659 independent reflections

1085 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −6→6
k = −9→13
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.121
S = 1.01
1659 reflections
105 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 +]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.016 (6)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.4730 (3)	0.8059 (1)	0.1978 (1)	0.0484 (4)
N2	0.4464 (2)	0.5165 (1)	0.4519 (1)	0.0422 (4)
C1	0.2718 (3)	0.7222 (1)	0.1726 (1)	0.0425 (4)
C2	0.1064 (3)	0.7130 (2)	0.0860 (1)	0.0538 (5)
C3	−0.0787 (3)	0.6201 (2)	0.0817 (1)	0.0579 (5)
C4	−0.1034 (4)	0.5384 (2)	0.1612 (1)	0.0546 (4)
C5	0.0606 (3)	0.5473 (1)	0.2473 (1)	0.0454 (4)
C6	0.2548 (3)	0.6396 (1)	0.2540 (1)	0.0388 (4)
C7	0.4578 (3)	0.6773 (1)	0.3285 (1)	0.0400 (4)
C8	0.5819 (3)	0.7783 (1)	0.2901 (1)	0.0466 (4)
C9	0.5376 (3)	0.6213 (1)	0.4228 (1)	0.0411 (4)
H1	0.524 (3)	0.865 (2)	0.159 (1)	0.062 (5)*
H2	0.1210	0.7680	0.0330	0.065*
H3	−0.1911	0.6111	0.0243	0.070*
H4	−0.2329	0.4767	0.1561	0.065*
H5	0.0419	0.4926	0.3002	0.054*
H8	0.7215	0.8217	0.3228	0.056*
H9	0.6619	0.6629	0.4650	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.061 (1)	0.041 (1)	0.043 (1)	−0.002 (1)	0.005 (1)	0.012 (1)
N2	0.063 (1)	0.036 (1)	0.027 (1)	−0.001 (1)	−0.003 (1)	0.001 (1)
C1	0.049 (1)	0.039 (1)	0.039 (1)	0.006 (1)	0.006 (1)	0.005 (1)
C2	0.062 (1)	0.058 (1)	0.040 (1)	0.007 (1)	−0.001 (1)	0.014 (1)
C3	0.060 (1)	0.065 (1)	0.047 (1)	0.005 (1)	−0.008 (1)	0.003 (1)
C4	0.054 (1)	0.050 (1)	0.059 (1)	−0.003 (1)	−0.001 (1)	0.000 (1)
C5	0.052 (1)	0.040 (1)	0.045 (1)	0.003 (1)	0.006 (1)	0.004 (1)
C6	0.046 (1)	0.035 (1)	0.036 (1)	0.007 (1)	0.007 (1)	0.003 (1)
C7	0.052 (1)	0.034 (1)	0.034 (1)	0.004 (1)	0.004 (1)	0.001 (1)
C8	0.059 (1)	0.040 (1)	0.041 (1)	−0.002 (1)	0.001 (1)	0.003 (1)
C9	0.056 (1)	0.036 (1)	0.032 (1)	−0.003 (1)	−0.001 (1)	−0.003 (1)

Geometric parameters (Å, º)

N1—C8	1.351 (2)	C6—C7	1.440 (2)
N1—C1	1.381 (2)	C7—C8	1.370 (2)
N2—C9	1.283 (2)	C7—C9	1.432 (2)
N2—N2^{i}	1.409 (2)	N1—H1	0.87 (2)
C1—C2	1.387 (2)	C2—H2	0.9300
C1—C6	1.412 (2)	C3—H3	0.9300
C2—C3	1.365 (2)	C4—H4	0.9300
C3—C4	1.393 (2)	C5—H5	0.9300
C4—C5	1.377 (2)	C8—H8	0.9300
C5—C6	1.393 (2)	C9—H9	0.9300
C8—N1—C1	109.2 (1)	N2—C9—C7	123.4 (1)
C9—N2—N2^{i}	112.0 (1)	C8—N1—H1	126 (1)
N1—C1—C2	129.9 (1)	C1—N1—H1	125 (1)
N1—C1—C6	107.6 (1)	C3—C2—H2	121.4
C2—C1—C6	122.5 (2)	C1—C2—H2	121.4
C3—C2—C1	117.3 (2)	C2—C3—H3	119.2
C2—C3—C4	121.7 (2)	C4—C3—H3	119.2
C5—C4—C3	121.2 (2)	C5—C4—H4	119.4
C4—C5—C6	118.9 (1)	C3—C4—H4	119.4
C5—C6—C1	118.5 (1)	C4—C5—H5	120.5
C5—C6—C7	135.2 (1)	C6—C5—H5	120.5
C1—C6—C7	106.3 (1)	N1—C8—H8	124.8
C8—C7—C9	123.7 (1)	C7—C8—H8	124.8
C8—C7—C6	106.5 (1)	N2—C9—H9	118.3
C9—C7—C6	129.7 (1)	C7—C9—H9	118.3
N1—C8—C7	110.5 (1)		
C8—N1—C1—C2	−179.5 (2)	C2—C1—C6—C7	179.4 (1)
C8—N1—C1—C6	0.5 (2)	C5—C6—C7—C8	−178.6 (2)
N1—C1—C2—C3	−179.6 (2)	C1—C6—C7—C8	0.5 (2)

C6—C1—C2—C3	0.4 (2)	C5—C6—C7—C9	5.1 (3)
C1—C2—C3—C4	0.7 (3)	C1—C6—C7—C9	−175.9 (2)
C2—C3—C4—C5	−0.7 (3)	C1—N1—C8—C7	−0.2 (2)
C3—C4—C5—C6	−0.3 (2)	C9—C7—C8—N1	176.5 (1)
C4—C5—C6—C1	1.3 (2)	C6—C7—C8—N1	−0.2 (2)
C4—C5—C6—C7	−179.7 (2)	N2^{i}—N2—C9—C7	−178.9 (1)
N1—C1—C6—C5	178.6 (1)	C8—C7—C9—N2	−169.5 (1)
C2—C1—C6—C5	−1.4 (2)	C6—C7—C9—N2	6.3 (3)
N1—C1—C6—C7	−0.6 (2)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N2^{ii}	0.87 (2)	2.21 (2)	3.065 (2)	167 (2)

Symmetry code: (ii) −*x*+1, *y*+1/2, −*z*+1/2.