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Bis(2-pyridylmethyl)amine–borane

Noelia Sanchez Ballester,[a] Julia Barreira Fontecha,[a] Jan Wikaira[b]* and Vickie McKee[a]

[a]Chemistry Department, Loughborough University, Loughborough, Leics LE11 3TU, England, and [b]Chemistry Department, Canterbury University, Christchurch, PB 4800, New Zealand

Correspondence e-mail: jan.wikaira@canterbury.ac.nz

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.116
Data-to-parameter ratio = 19.3

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

The title compound, $C_{12}H_{12}N_3{\cdot}BH_3$ or $C_{12}H_{15}BN_3$, contains a BH_3 group and two picolyl groups attached to a central N atom. Both edge-to-face and face-to-face π-stacking interactions are found.

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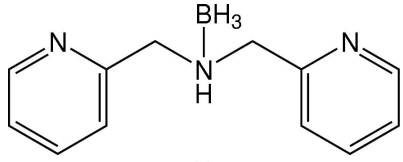
Comment

The asymmetric unit of the title compound, (I), contains one molecule. The two planar pyridyl rings are twisted (Fig. 1) about the central N atom, with an interplanar angle of 110.9°. The amine N atom is not involved in any hydrogen bonding but pyridyl atom N1 interacts with atom C3 in an adjacent ring (Table 1).



(I)

An edge-to-face interaction is found between the H atom on C2 and the plane of the pyridine ring containing atom N3 (Fig. 2). This H atom is 2.806 Å from the mean plane of the pyridine ring at $(\frac{1}{2}+x,\ \frac{1}{2}-y,\ \frac{1}{2}+z)$. The pyridine ring containing atom N3 is π-stacked with its symmetry equivalent by inversion (symmetry code: $2-x,\ -y,\ 1-z$). The interplanar and the centroid-to-centroid distances are 3.496 (2) and 3.971 Å respectively (Fig. 2).

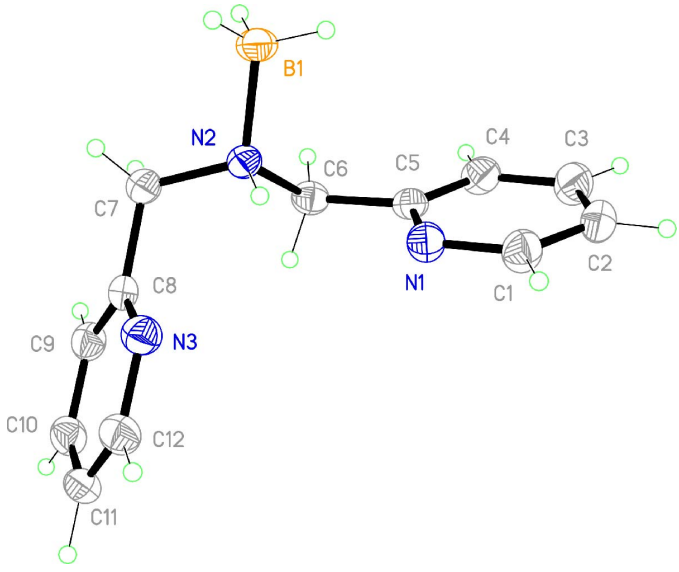


Figure 1
Perspective view of (I), showing 50% probability displacement ellipsoids.

Experimental

2-(Aminomethyl)pyridine (4.95 g, 44.77 mmol) and pyridine-2-carboxaldehyde (4.96 g, 46.31 mmol) were dissolved in methanol (150 ml) (Lambert *et al.*, 1997). The solution was stirred for 2 h at room temperature (yellow–orange solution). After slow addition of an excess of sodium borohydride, stirring was continued for 1 h (pale-yellow solution). The solvent was removed by rotary evaporation to give bis(pyridin-2-ylmethyl)amine (6.43 g, 73%) as an orange oil. Colourless crystals of the borane adduct appeared as a minor product after the oil was stored in a freezer overnight.

Crystal data

$C_{12}H_{15}BN_3$
M_r = 213.09
Monoclinic, $P2_1/n$
a = 5.3172 (4) Å
b = 24.8494 (19) Å
c = 9.3896 (7) Å
β = 102.938 (1)°
V = 1209.14 (16) Å^3
Z = 4
D_x = 1.171 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3633 reflections
θ = 2.4–27.5°
μ = 0.07 mm^{-1}
T = 150 (2) K
Needle, colourless
0.55 × 0.17 × 0.09 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.946, T_{max} = 0.990
10293 measured reflections
2860 independent reflections
2156 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 28.8°
$h = -7 \rightarrow 6$
$k = -32 \rightarrow 32$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.116
S = 1.02
2860 reflections
148 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.3966P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···N1^{i}	0.95	2.66	3.5215 (19)	150

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

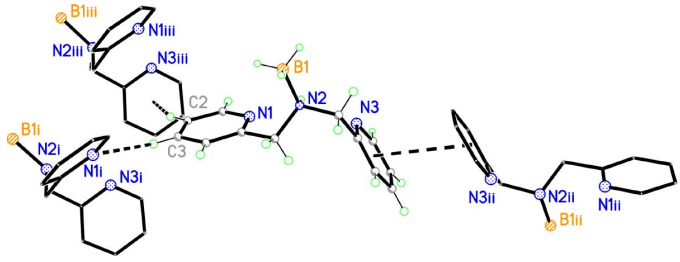


Figure 2
View showing the C—H···N bond (C3 and N1), the interaction between the H atom bonded to C2 and the pyridyl ring, and the π-stacking of the N3-containing pyridine rings [symmetry codes: (i) $\frac{1}{2}+x, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $2-x, -y, 1-z$; (iii) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$.]

H atoms bonded to C and B atoms were placed at calculated positions; the constrained C—H distances were 0.95, 0.98 and 0.99 Å for H atoms bonded to Csp^2, Bsp^3 and methylene C atoms, respectively. They were refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(B,C). The H atom bonded to the amine N atom was located in a difference map and the coordinates freely refined with a fixed U_{iso} value of 0.03 Å.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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supporting information

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Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3633 reflections
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2156 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 28.8°$, $\theta_{min} = 2.4°$
$h = -7\rightarrow6$
$k = -32\rightarrow32$
$l = -12\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.116$
$S = 1.02$
2860 reflections
148 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
$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.3966P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.7734 (2)	0.21154 (5)	0.06397 (12)	0.0294 (3)
C1	0.7038 (3)	0.26208 (6)	0.02464 (16)	0.0330 (3)
H1	0.5762	0.2787	0.0663	0.040*
C2	0.8076 (3)	0.29147 (6)	−0.07349 (16)	0.0364 (3)
H2	0.7520	0.3273	−0.0985	0.044*
C3	0.9934 (3)	0.26772 (6)	−0.13425 (17)	0.0391 (4)
H3	1.0690	0.2869	−0.2016	0.047*
C4	1.0676 (3)	0.21532 (6)	−0.09506 (16)	0.0323 (3)
H4	1.1947	0.1979	−0.1354	0.039*
C5	0.9533 (3)	0.18883 (5)	0.00379 (14)	0.0249 (3)
C6	1.0282 (3)	0.13226 (5)	0.05302 (14)	0.0268 (3)
H6A	1.1212	0.1153	−0.0157	0.032*
H6B	1.1461	0.1331	0.1509	0.032*
N2	0.7962 (2)	0.09966 (4)	0.05927 (12)	0.0247 (3)
C7	0.8683 (3)	0.05115 (5)	0.15252 (14)	0.0315 (3)
H7A	1.0137	0.0326	0.1235	0.038*
H7B	0.7201	0.0260	0.1365	0.038*
C8	0.9453 (3)	0.06549 (5)	0.31264 (14)	0.0261 (3)
C9	1.1746 (3)	0.04780 (5)	0.40128 (15)	0.0303 (3)
H9	1.2931	0.0271	0.3618	0.036*
C10	1.2282 (3)	0.06080 (6)	0.54864 (15)	0.0327 (3)
H10	1.3837	0.0491	0.6121	0.039*
C11	1.0516 (3)	0.09103 (6)	0.60112 (15)	0.0320 (3)
H11	1.0821	0.1003	0.7017	0.038*
C12	0.8290 (3)	0.10767 (6)	0.50462 (15)	0.0320 (3)
H12	0.7091	0.1288	0.5415	0.038*
N3	0.7732 (2)	0.09558 (5)	0.36190 (13)	0.0305 (3)
B1	0.6297 (3)	0.08390 (7)	−0.10096 (17)	0.0302 (3)
H13A	0.5839	0.1167	−0.1591	0.045*
H13B	0.4720	0.0652	−0.0916	0.045*
H13C	0.7321	0.0603	−0.1495	0.045*
H1N	0.700 (3)	0.1212 (6)	0.1050 (17)	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0338 (6)	0.0275 (6)	0.0291 (6)	0.0024 (5)	0.0116 (5)	0.0003 (5)
C1	0.0376 (8)	0.0296 (7)	0.0338 (7)	0.0037 (6)	0.0125 (6)	−0.0006 (6)
C2	0.0500 (9)	0.0249 (7)	0.0345 (8)	−0.0005 (6)	0.0100 (7)	0.0020 (6)
C3	0.0540 (10)	0.0337 (8)	0.0345 (8)	−0.0076 (7)	0.0201 (7)	0.0014 (6)

C4	0.0362 (8)	0.0322 (7)	0.0319 (7)	−0.0047 (6)	0.0148 (6)	−0.0039 (6)
C5	0.0260 (7)	0.0266 (7)	0.0217 (6)	−0.0024 (5)	0.0044 (5)	−0.0030 (5)
C6	0.0249 (6)	0.0301 (7)	0.0251 (6)	0.0018 (5)	0.0048 (5)	−0.0018 (5)
N2	0.0294 (6)	0.0220 (5)	0.0227 (5)	0.0020 (4)	0.0057 (4)	−0.0023 (4)
C7	0.0448 (8)	0.0217 (6)	0.0259 (7)	0.0020 (6)	0.0035 (6)	−0.0013 (5)
C8	0.0339 (7)	0.0192 (6)	0.0249 (6)	−0.0031 (5)	0.0059 (5)	0.0021 (5)
C9	0.0334 (7)	0.0252 (7)	0.0325 (7)	0.0010 (6)	0.0077 (6)	0.0011 (5)
C10	0.0324 (8)	0.0325 (8)	0.0302 (7)	−0.0014 (6)	0.0005 (6)	0.0054 (6)
C11	0.0399 (8)	0.0337 (7)	0.0222 (6)	−0.0078 (6)	0.0066 (6)	0.0008 (5)
C12	0.0346 (8)	0.0340 (8)	0.0291 (7)	0.0000 (6)	0.0108 (6)	0.0001 (6)
N3	0.0315 (6)	0.0308 (6)	0.0283 (6)	0.0009 (5)	0.0051 (5)	−0.0001 (5)
B1	0.0334 (8)	0.0297 (8)	0.0248 (7)	0.0011 (6)	0.0012 (6)	−0.0048 (6)

Geometric parameters (Å, º)

N1—C1	1.3375 (18)	C7—C8	1.5097 (18)
N1—C5	1.3396 (17)	C7—H7A	0.9900
C1—C2	1.384 (2)	C7—H7B	0.9900
C1—H1	0.9500	C8—N3	1.3417 (17)
C2—C3	1.379 (2)	C8—C9	1.385 (2)
C2—H2	0.9500	C9—C10	1.387 (2)
C3—C4	1.386 (2)	C9—H9	0.9500
C3—H3	0.9500	C10—C11	1.377 (2)
C4—C5	1.3845 (19)	C10—H10	0.9500
C4—H4	0.9500	C11—C12	1.383 (2)
C5—C6	1.5054 (18)	C11—H11	0.9500
C6—N2	1.4881 (17)	C12—N3	1.3402 (18)
C6—H6A	0.9900	C12—H12	0.9500
C6—H6B	0.9900	B1—H13A	0.9800
N2—C7	1.4887 (17)	B1—H13B	0.9800
N2—B1	1.6138 (18)	B1—H13C	0.9800
N2—H1N	0.910 (16)		
C1—N1—C5	117.31 (12)	N2—C7—C8	111.80 (11)
N1—C1—C2	123.53 (14)	N2—C7—H7A	109.3
N1—C1—H1	118.2	C8—C7—H7A	109.3
C2—C1—H1	118.2	N2—C7—H7B	109.3
C3—C2—C1	118.61 (14)	C8—C7—H7B	109.3
C3—C2—H2	120.7	H7A—C7—H7B	107.9
C1—C2—H2	120.7	N3—C8—C9	123.11 (12)
C2—C3—C4	118.67 (13)	N3—C8—C7	114.89 (12)
C2—C3—H3	120.7	C9—C8—C7	121.98 (13)
C4—C3—H3	120.7	C8—C9—C10	118.83 (13)
C5—C4—C3	118.89 (13)	C8—C9—H9	120.6
C5—C4—H4	120.6	C10—C9—H9	120.6
C3—C4—H4	120.6	C11—C10—C9	118.67 (13)
N1—C5—C4	122.98 (13)	C11—C10—H10	120.7
N1—C5—C6	115.40 (11)	C9—C10—H10	120.7

C4—C5—C6	121.61 (12)	C10—C11—C12	118.74 (13)
N2—C6—C5	110.83 (11)	C10—C11—H11	120.6
N2—C6—H6A	109.5	C12—C11—H11	120.6
C5—C6—H6A	109.5	N3—C12—C11	123.60 (14)
N2—C6—H6B	109.5	N3—C12—H12	118.2
C5—C6—H6B	109.5	C11—C12—H12	118.2
H6A—C6—H6B	108.1	C12—N3—C8	117.02 (12)
C6—N2—C7	110.95 (11)	N2—B1—H13A	109.5
C6—N2—B1	112.45 (10)	N2—B1—H13B	109.5
C7—N2—B1	111.54 (10)	H13A—B1—H13B	109.5
C6—N2—H1N	104.8 (10)	N2—B1—H13C	109.5
C7—N2—H1N	107.2 (10)	H13A—B1—H13C	109.5
B1—N2—H1N	109.5 (10)	H13B—B1—H13C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···N1^{i}	0.95	2.66	3.5215 (19)	150

Symmetry code: (i) $x+1/2, -y+1/2, z-1/2$.