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A monoclinic polymorph of 1,3-bis(2-pyridylaminomethyl)benzene

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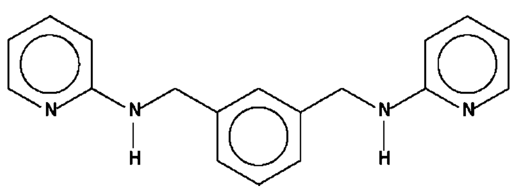
Key indicators: single-crystal X-ray study; *T* = 295 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.047; *wR* factor = 0.150; data-to-parameter ratio = 16.9.

The molecules of the title compound, $C_{18}H_{18}N_4$, are linked by two different N—H···$N_{pyridyl}$ hydrogen bonds into a linear chain.

Related literature

In the orthorhombic polymorph, the molecule lies on a twofold rotation axis; adjacent molecules are hydrogen-bonded into a linear chain, see: Zhu *et al.* (2007).



Experimental

Crystal data

$C_{18}H_{18}N_4$
M_r = 290.36
Monoclinic, $C2/c$
a = 35.647 (2) Å
b = 5.7899 (4) Å
c = 14.9019 (9) Å
β = 94.074 (2)°
V = 3067.9 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 295 (2) K
0.32 × 0.28 × 0.24 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.589, T_{max} = 0.982
13991 measured reflections
3512 independent reflections
1833 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.150
S = 1.02
3512 reflections
208 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2N···N1^{i}	0.86 (1)	2.19 (1)	3.047 (2)	175 (2)
N3—H3N···N4^{ii}	0.86 (1)	2.24 (1)	3.104 (2)	177 (2)

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

Data collection: *RAPID-AUTO* (Rigaku Corporation, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2210).

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supplementary materials

Acta Cryst. (2008). E**64**, o252 [**doi:10.1107/S1600536807063878**]

A monoclinic polymorph of 1,3-bis(2-pyridylaminomethyl)benzene

L.-N. Zhu, S. Gao and S. W. Ng

Experimental

The compound was synthesized as described (Zhu *et al.*, 2007), and crystals were obtained upon recrystallization from methanol.

Refinement

Carbon-bound H atoms were placed in calculated positions [C—H 0.93–0.97 Å and U_{iso}(H) 1.25U_{eq}(C), were included in the refinement in the riding-model approximation. The nitrogen-bound H atoms were located and difference Fourier map and were refined with a distance restraint of N–H 0.86±0.01 Å.

Figures

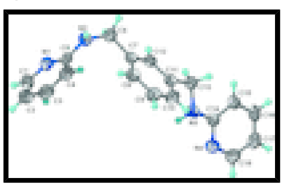


Fig. 1. Thermal ellipsoid plot of a portion of the hydrogen-bonded structure; displacement ellipsoids are drawn at the 50% probability level, and H atoms as spheres of arbitrary radius.

1,3-bis(2-pyridylaminomethyl)benzene

Crystal data

$C_{18}H_{18}N_4$	$F(000) = 1232$
$M_r = 290.36$	$D_x = 1.257$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 7473 reflections
$a = 35.647$ (2) Å	$\theta = 3.0$–27.5°
$b = 5.7899$ (4) Å	$\mu = 0.08$ mm^{-1}
$c = 14.9019$ (9) Å	$T = 295$ K
$\beta = 94.074$ (2)°	Prism, colorless
$V = 3067.9$ (3) Å^3	0.32 × 0.28 × 0.24 mm
$Z = 8$	

Data collection

Rigaku R-AXIS RAPID diffractometer	3512 independent reflections
Radiation source: fine-focus sealed tube	1833 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.047$

Detector resolution: 10.000 pixels mm^{-1}	θ_{max} = 27.5°, θ_{min} = 3.0°
ω–scans	h = −46→46
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	k = −7→7
T_{min} = 0.589, T_{max} = 0.982	l = −19→19
13991 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.048	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.150	$w = 1/[\sigma^2(F_o^2) + (0.0752P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
3512 reflections	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
208 parameters	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
2 restraints	Extinction correction: *SHELXL*, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0013 (5)

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

	x	y	z	U_{iso}*/U_{eq}
N1	0.26513 (4)	0.1786 (3)	0.38622 (11)	0.0551 (4)
N2	0.28167 (4)	0.5057 (3)	0.46611 (11)	0.0571 (4)
H2N	0.2672 (5)	0.452 (3)	0.5052 (12)	0.076 (7)*
N3	0.47595 (4)	0.7010 (3)	0.58328 (13)	0.0615 (5)
H3N	0.4725 (6)	0.599 (3)	0.5412 (11)	0.080 (7)*
N4	0.53909 (4)	0.6575 (3)	0.56996 (12)	0.0632 (5)
C1	0.27020 (6)	0.0330 (4)	0.31870 (14)	0.0648 (6)
H1	0.2536	−0.0903	0.3102	0.078*
C2	0.29845 (6)	0.0538 (4)	0.26093 (14)	0.0717 (6)
H2	0.3009	−0.0517	0.2146	0.086*
C3	0.32289 (6)	0.2357 (4)	0.27402 (15)	0.0682 (6)
H3	0.3424	0.2542	0.2364	0.082*
C4	0.31870 (5)	0.3903 (4)	0.34224 (14)	0.0597 (5)
H4	0.3352	0.5141	0.3513	0.072*
C5	0.28904 (5)	0.3579 (3)	0.39817 (12)	0.0491 (4)
C6	0.30779 (5)	0.6811 (3)	0.49945 (14)	0.0566 (5)
H6A	0.2950	0.7808	0.5398	0.068*
H6B	0.3144	0.7747	0.4490	0.068*
C7	0.34377 (5)	0.5957 (3)	0.54853 (12)	0.0469 (4)
C8	0.34573 (6)	0.3867 (3)	0.59374 (13)	0.0566 (5)
H8	0.3249	0.2898	0.5912	0.068*

C9	0.37829 (6)	0.3207 (3)	0.64242 (15)	0.0630 (6)
H9	0.3792	0.1803	0.6728	0.076*
C10	0.40950 (5)	0.4621 (3)	0.64621 (13)	0.0601 (5)
H10	0.4313	0.4171	0.6796	0.072*
C11	0.40859 (5)	0.6714 (3)	0.60048 (12)	0.0496 (5)
C12	0.37567 (5)	0.7345 (3)	0.55229 (12)	0.0480 (4)
H12	0.3748	0.8741	0.5214	0.058*
C13	0.44274 (5)	0.8269 (3)	0.60434 (15)	0.0573 (5)
H13A	0.4384	0.9527	0.5619	0.069*
H13B	0.4466	0.8928	0.6641	0.069*
C14	0.51105 (5)	0.7920 (3)	0.59598 (13)	0.0525 (5)
C15	0.51840 (5)	1.0098 (3)	0.63396 (13)	0.0600 (5)
H15	0.4987	1.1056	0.6482	0.072*
C16	0.55490 (6)	1.0800 (4)	0.64988 (14)	0.0664 (6)
H16	0.5602	1.2243	0.6751	0.080*
C17	0.58374 (6)	0.9363 (4)	0.62849 (15)	0.0667 (6)
H17	0.6088	0.9778	0.6412	0.080*
C18	0.57426 (6)	0.7309 (4)	0.58794 (16)	0.0681 (6)
H18	0.5937	0.6357	0.5718	0.082*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0488 (9)	0.0608 (10)	0.0551 (10)	−0.0034 (7)	0.0003 (7)	−0.0014 (8)
N2	0.0473 (9)	0.0641 (10)	0.0604 (10)	−0.0077 (8)	0.0084 (8)	−0.0103 (9)
N3	0.0474 (9)	0.0637 (11)	0.0737 (12)	−0.0021 (8)	0.0062 (8)	−0.0216 (9)
N4	0.0485 (10)	0.0596 (10)	0.0820 (12)	−0.0003 (7)	0.0071 (9)	−0.0096 (9)
C1	0.0689 (13)	0.0630 (12)	0.0615 (12)	−0.0010 (10)	−0.0031 (11)	−0.0067 (10)
C2	0.0773 (15)	0.0803 (15)	0.0573 (13)	0.0147 (12)	0.0043 (12)	−0.0080 (11)
C3	0.0612 (13)	0.0913 (15)	0.0530 (12)	0.0132 (12)	0.0111 (10)	0.0059 (12)
C4	0.0474 (11)	0.0717 (12)	0.0603 (12)	−0.0037 (9)	0.0058 (9)	0.0065 (10)
C5	0.0391 (9)	0.0574 (10)	0.0503 (10)	0.0008 (8)	−0.0006 (8)	0.0036 (9)
C6	0.0507 (11)	0.0534 (11)	0.0649 (12)	−0.0010 (9)	−0.0013 (9)	−0.0014 (9)
C7	0.0471 (10)	0.0456 (10)	0.0484 (10)	0.0008 (8)	0.0054 (8)	−0.0038 (8)
C8	0.0578 (12)	0.0499 (11)	0.0628 (12)	−0.0049 (9)	0.0086 (10)	0.0036 (9)
C9	0.0671 (13)	0.0556 (12)	0.0666 (13)	0.0046 (10)	0.0052 (11)	0.0162 (10)
C10	0.0543 (12)	0.0654 (12)	0.0600 (12)	0.0093 (10)	−0.0009 (10)	0.0063 (10)
C11	0.0487 (11)	0.0519 (10)	0.0486 (10)	0.0042 (8)	0.0065 (9)	−0.0029 (8)
C12	0.0507 (10)	0.0437 (9)	0.0500 (10)	0.0027 (8)	0.0056 (8)	0.0009 (8)
C13	0.0468 (11)	0.0598 (11)	0.0648 (12)	0.0014 (9)	0.0011 (9)	−0.0083 (10)
C14	0.0478 (11)	0.0531 (11)	0.0563 (11)	−0.0012 (8)	0.0014 (9)	−0.0037 (9)
C15	0.0580 (12)	0.0596 (12)	0.0618 (12)	−0.0011 (9)	0.0008 (10)	−0.0120 (10)
C16	0.0708 (14)	0.0676 (13)	0.0598 (12)	−0.0146 (11)	−0.0022 (11)	−0.0083 (10)
C17	0.0519 (12)	0.0822 (14)	0.0650 (13)	−0.0120 (11)	−0.0034 (10)	0.0022 (11)
C18	0.0488 (12)	0.0708 (13)	0.0848 (16)	0.0002 (10)	0.0050 (11)	−0.0039 (12)

Geometric parameters (Å, °)

N1—C1	1.335 (2)	C7—C8	1.384 (2)

N1—C5	1.347 (2)	C7—C12	1.390 (2)
N2—C5	1.365 (2)	C8—C9	1.378 (3)
N2—C6	1.442 (2)	C8—H8	0.9300
N2—H2N	0.86 (1)	C9—C10	1.379 (3)
N3—C14	1.358 (2)	C9—H9	0.9300
N3—C13	1.444 (2)	C10—C11	1.389 (2)
N3—H3N	0.86 (1)	C10—H10	0.9300
N4—C18	1.333 (2)	C11—C12	1.381 (3)
N4—C14	1.346 (2)	C11—C13	1.512 (2)
C1—C2	1.376 (3)	C12—H12	0.9300
C1—H1	0.9300	C13—H13A	0.9700
C2—C3	1.372 (3)	C13—H13B	0.9700
C2—H2	0.9300	C14—C15	1.399 (3)
C3—C4	1.371 (3)	C15—C16	1.368 (3)
C3—H3	0.9300	C15—H15	0.9300
C4—C5	1.405 (2)	C16—C17	1.377 (3)
C4—H4	0.9300	C16—H16	0.9300
C6—C7	1.514 (3)	C17—C18	1.366 (3)
C6—H6A	0.9700	C17—H17	0.9300
C6—H6B	0.9700	C18—H18	0.9300

C1—N1—C5	117.91 (15)	C7—C8—H8	119.7
C5—N2—C6	122.91 (15)	C8—C9—C10	120.23 (18)
C5—N2—H2N	115.5 (15)	C8—C9—H9	119.9
C6—N2—H2N	115.1 (15)	C10—C9—H9	119.9
C14—N3—C13	122.39 (16)	C9—C10—C11	120.47 (19)
C14—N3—H3N	116.7 (14)	C9—C10—H10	119.8
C13—N3—H3N	115.2 (15)	C11—C10—H10	119.8
C18—N4—C14	117.70 (17)	C12—C11—C10	118.35 (17)
N1—C1—C2	124.1 (2)	C12—C11—C13	120.98 (16)
N1—C1—H1	118.0	C10—C11—C13	120.67 (17)
C2—C1—H1	118.0	C11—C12—C7	122.05 (16)
C3—C2—C1	117.6 (2)	C11—C12—H12	119.0
C3—C2—H2	121.2	C7—C12—H12	119.0
C1—C2—H2	121.2	N3—C13—C11	111.21 (15)
C4—C3—C2	120.34 (18)	N3—C13—H13A	109.4
C4—C3—H3	119.8	C11—C13—H13A	109.4
C2—C3—H3	119.8	N3—C13—H13B	109.4
C3—C4—C5	118.65 (19)	C11—C13—H13B	109.4
C3—C4—H4	120.7	H13A—C13—H13B	108.0
C5—C4—H4	120.7	N4—C14—N3	115.54 (16)
N1—C5—N2	115.33 (14)	N4—C14—C15	121.11 (17)
N1—C5—C4	121.35 (17)	N3—C14—C15	123.36 (16)
N2—C5—C4	123.30 (17)	C16—C15—C14	119.09 (18)
N2—C6—C7	116.16 (15)	C16—C15—H15	120.5
N2—C6—H6A	108.2	C14—C15—H15	120.5
C7—C6—H6A	108.2	C15—C16—C17	119.81 (19)
N2—C6—H6B	108.2	C15—C16—H16	120.1
C7—C6—H6B	108.2	C17—C16—H16	120.1
H6A—C6—H6B	107.4	C18—C17—C16	117.59 (19)

C8—C7—C12	118.21 (17)	C18—C17—H17	121.2
C8—C7—C6	122.17 (16)	C16—C17—H17	121.2
C12—C7—C6	119.55 (15)	N4—C18—C17	124.49 (18)
C9—C8—C7	120.67 (17)	N4—C18—H18	117.8
C9—C8—H8	119.7	C17—C18—H18	117.8

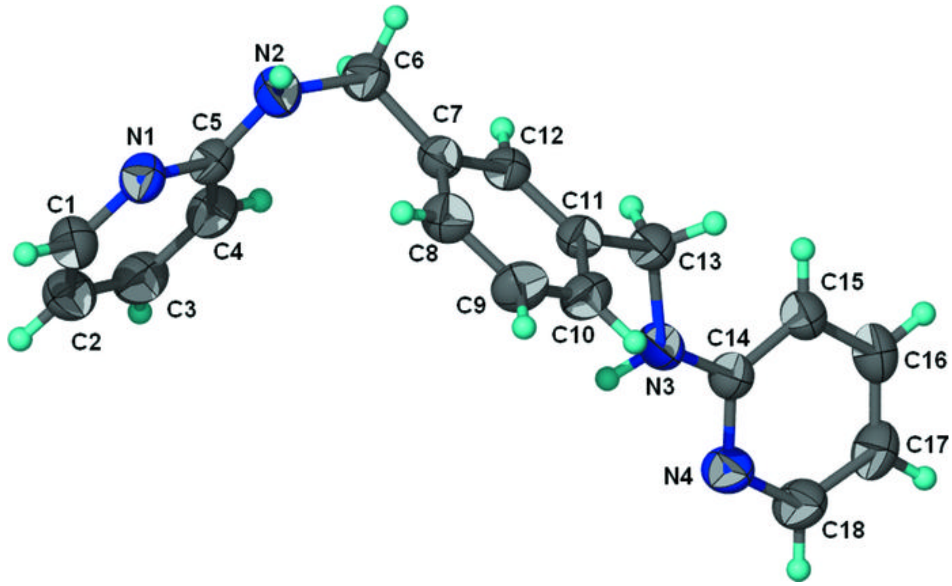
C5—N1—C1—C2	0.4 (3)	C9—C10—C11—C13	−179.93 (17)
N1—C1—C2—C3	0.2 (3)	C10—C11—C12—C7	0.1 (3)
C1—C2—C3—C4	−0.4 (3)	C13—C11—C12—C7	−179.27 (16)
C2—C3—C4—C5	0.1 (3)	C8—C7—C12—C11	−1.0 (3)
C1—N1—C5—N2	177.71 (17)	C6—C7—C12—C11	176.15 (16)
C1—N1—C5—C4	−0.7 (3)	C14—N3—C13—C11	−169.32 (18)
C6—N2—C5—N1	168.25 (17)	C12—C11—C13—N3	−130.03 (18)
C6—N2—C5—C4	−13.4 (3)	C10—C11—C13—N3	50.6 (2)
C3—C4—C5—N1	0.4 (3)	C18—N4—C14—N3	−174.51 (19)
C3—C4—C5—N2	−177.85 (19)	C18—N4—C14—C15	5.1 (3)
C5—N2—C6—C7	−67.6 (2)	C13—N3—C14—N4	−177.51 (17)
N2—C6—C7—C8	−27.6 (2)	C13—N3—C14—C15	2.9 (3)
N2—C6—C7—C12	155.37 (16)	N4—C14—C15—C16	−4.1 (3)
C12—C7—C8—C9	1.2 (3)	N3—C14—C15—C16	175.4 (2)
C6—C7—C8—C9	−175.93 (18)	C14—C15—C16—C17	0.0 (3)
C7—C8—C9—C10	−0.4 (3)	C15—C16—C17—C18	2.9 (3)
C8—C9—C10—C11	−0.6 (3)	C14—N4—C18—C17	−2.1 (3)
C9—C10—C11—C12	0.7 (3)	C16—C17—C18—N4	−1.9 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2N···N1^{i}	0.86 (1)	2.19 (1)	3.047 (2)	175 (2)
N3—H3N···N4^{ii}	0.86 (1)	2.24 (1)	3.104 (2)	177 (2)

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

Fig. 1


N2
C6
C7
C12
N1
C5
C1
C4
C11
C8
C13
C15
C3
C9
C10
C14
C16
C2
N3
C17
N4
C18