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N,*N*′-Dimethyl-*N*,*N*′-bis(pyridin-2-yl)-methanediamine

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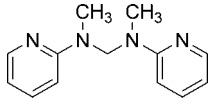
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.125; data-to-parameter ratio = 11.5.

The title compound, $C_{13}H_{16}N_4$, consists of two pyridine rings which are linked by an *N*,*N*′-dimethylmethaneamine chain. The pyridine rings adopt a twist conformation and the dihedral angle between them is 60.85 (5)°. The crystal packing is stabilized by weak C—H$\cdots\pi$ interactions.

Related literature

For the synthesis of the title compound, see: Kahn *et al.* (1945). For applications of heteroaromatic amines, see: Mehrkhodavandi & Schrock (2001); Hall *et al.* (1998); Lee (2003).



Experimental

Crystal data

$C_{13}H_{16}N_4$
M_r = 228.30
Monoclinic, $P2_1/n$
a = 11.6652 (7) Å
b = 8.3921 (5) Å
c = 12.8966 (7) Å
β = 106.634 (2)°
V = 1209.69 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 100 K
0.12 × 0.08 × 0.08 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.963, T_{max} = 0.986
27844 measured reflections
2516 independent reflections
2105 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.125
S = 0.96
2516 reflections
218 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the N1/C1–C5 and N4/C9–C15 rings, respectively.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C1—H1$\cdots Cg2^{i}$	0.99 (2)	2.64 (2)	3.484 (1)	143 (1)
C6—H7$\cdots Cg2^{ii}$	1.00 (2)	2.75 (2)	3.545 (2)	137 (1)
C10—H13$\cdots Cg1^{iii}$	0.95 (2)	2.90 (1)	3.637 (1)	135 (1)

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

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: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2378).

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supplementary materials

Acta Cryst. (2011). E**67**, o3226 [**doi:10.1107/S1600536811046320**]

N,*N*'-Dimethyl-*N*,*N*'-bis(pyridin-2-yl)methanediamine

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Comment

Heteroaromatic amines based metal complexes have been extensively studied due to their numerous potential applications as catalysts, drugs, biomimetic chemistry, and so on. (Mehrkhodavandi, *et al.*, (2001) , Hall, *et al.*,(1998), Lee, (2003). We are interested in the use of chelates containing pyridylamine. We report here the crystal structure of the title compound, Fig. 1, which consists of two 2-pyridyl rings which are linked together by a *N*,*N*'-dimethylmethaneamine chain .The pyridine rings adopt a twist conformation and the dihedral angle between them is 60.85 (5)°. The crystal packing is stabilized by weak C—H···π interactions, Fig. 2, Table 1.

Experimental

N,*N*'-dimethyl-*N*,*N*'-di(pyridin-2-yl)methanediamine was prepared by a reported method, Kahn, *et al.*, (1945). A solution of 2-(methylamino)pyridine (5.00 g, 4.62 × 10 $^{-2}$ mol) in water (50 ml) was added dropwise to 37% formaldehyde solution (1.83 ml, 2.31 × 10^{-2} mol) at 0°C. The reation mixture was stirred at room temperature for overnight and the white precipitate formed was filtered,washed with water and dried. It was dissolved in acetone, dried over $MgSO_4$ and concentrated. It was recrystallized in acetonitrile. Yield: 92% (4.88 g)

Refinement

All H atoms were geometrically positioned and refined using a riding model, with C—H = 0.95 Å for the aryl, 0.99 Å for the methylene, and 0.00 Å for the methyl H atoms, respectively, and with U_{iso}(H) = 1.2Ueq(C) for the aryl and methylene H atoms, and 1.5Ueq(C) for the methyl H atoms.

Figures

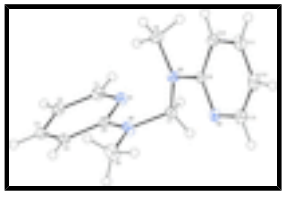

Fig. 1. A view of the title compound showing the labelling of the atoms. Displacement ellipsoids are shown at the 50% probability level.

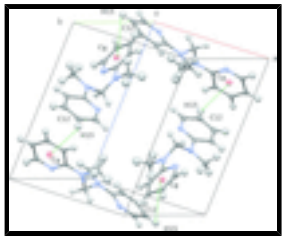

Fig. 2. Part of the crystal structure of (I) showing the formation of a C—H···π interactions.

N,*N*'-Dimethyl-*N*,*N*'-bis(pyridin-2-yl)methanediamine

Crystal data

$C_{13}H_{16}N_4$	$F(000) = 488$
$M_r = 228.30$	$D_x = 1.254$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P2yn	Cell parameters from 2516 reflections
$a = 11.6652$ (7) Å	$\theta = 2.1$–$26.5°$
$b = 8.3921$ (5) Å	$\mu = 0.08$ mm^{-1}
$c = 12.8966$ (7) Å	$T = 100$ K
$\beta = 106.634$ (2)°	Rod, colourless
$V = 1209.69$ (12) Å^3	0.12 × 0.08 × 0.08 mm
$Z = 4$	

Data collection

Bruker APEXII diffractometer	2105 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.050$
graphite	$\theta_{max} = 26.5°$, $\theta_{min} = 2.1°$
$\theta/2\varphi$ scans	$h = -14 \rightarrow 14$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$k = -10 \rightarrow 10$
$T_{min} = 0.963$, $T_{max} = 0.986$	$l = -16 \rightarrow 14$
27844 measured reflections	4 standard reflections every 30 min
2516 independent reflections	intensity decay: 0.0%

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.125$	All H-atom parameters refined
$S = 0.96$	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
2516 reflections	$(\Delta/\sigma)_{max} < 0.001$
218 parameters	$\Delta\rho_{max} = 0.23$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.21$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.43474 (8)	0.22148 (11)	0.82931 (7)	0.0194 (3)
N2	0.61745 (8)	0.11262 (11)	0.82658 (8)	0.0203 (3)
N3	0.59838 (8)	−0.15394 (11)	0.89105 (8)	0.0189 (3)
N4	0.79220 (8)	−0.22454 (12)	0.98754 (7)	0.0182 (3)
C1	0.34526 (10)	0.32394 (15)	0.78856 (9)	0.0211 (3)
C2	0.33876 (11)	0.42500 (14)	0.70242 (9)	0.0214 (3)
C3	0.43186 (10)	0.41792 (14)	0.65514 (9)	0.0200 (3)
C4	0.52555 (10)	0.31431 (14)	0.69472 (9)	0.0173 (3)
C5	0.52473 (10)	0.21664 (13)	0.78366 (9)	0.0160 (3)
C6	0.71691 (12)	0.09942 (17)	0.78056 (12)	0.0272 (3)
C7	0.62117 (10)	0.01339 (13)	0.91939 (9)	0.0173 (3)
C8	0.47475 (11)	−0.19594 (16)	0.83643 (11)	0.0240 (3)
C9	0.67862 (10)	−0.27049 (13)	0.94043 (8)	0.0163 (3)
C10	0.64315 (11)	−0.43154 (14)	0.93928 (9)	0.0205 (3)
C11	0.72811 (12)	−0.54307 (15)	0.98755 (9)	0.0248 (3)
C12	0.84533 (12)	−0.49686 (15)	1.03581 (9)	0.0255 (3)
C13	0.87199 (11)	−0.33721 (14)	1.03368 (9)	0.0221 (3)
H1	0.2816 (13)	0.3259 (16)	0.8256 (12)	0.031 (4)*
H2	0.2701 (12)	0.4955 (17)	0.6790 (10)	0.029 (4)*
H3	0.4313 (12)	0.4841 (17)	0.5946 (11)	0.028 (4)*
H4	0.5883 (13)	0.3065 (16)	0.6629 (11)	0.025 (3)*
H5	0.7601 (15)	0.201 (2)	0.7856 (14)	0.048 (5)*
H6	0.7696 (15)	0.022 (2)	0.8232 (13)	0.047 (4)*
H7	0.6898 (15)	0.059 (2)	0.7046 (14)	0.045 (4)*
H8	0.5576 (11)	0.0536 (14)	0.9520 (10)	0.018 (3)*
H9	0.6997 (13)	0.0194 (15)	0.9712 (11)	0.023 (3)*
H10	0.4350 (13)	−0.1019 (18)	0.8023 (12)	0.032 (4)*
H11	0.4723 (12)	−0.2806 (19)	0.7818 (13)	0.035 (4)*
H12	0.4316 (13)	−0.2327 (17)	0.8900 (12)	0.033 (4)*
H13	0.5616 (13)	−0.4628 (16)	0.9100 (11)	0.028 (4)*
H14	0.7042 (13)	−0.6532 (18)	0.9862 (12)	0.032 (4)*
H15	0.9091 (13)	−0.5706 (18)	1.0696 (11)	0.033 (4)*
H16	0.9557 (12)	−0.3009 (15)	1.0693 (11)	0.021 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

N1	0.0190 (5)	0.0203 (5)	0.0189 (5)	0.0021 (4)	0.0054 (4)	0.0009 (4)
N2	0.0208 (5)	0.0187 (5)	0.0239 (5)	0.0052 (4)	0.0102 (4)	0.0059 (4)
N3	0.0177 (5)	0.0145 (5)	0.0211 (5)	−0.0003 (4)	0.0003 (4)	0.0002 (4)
N4	0.0185 (5)	0.0185 (5)	0.0166 (5)	0.0017 (4)	0.0035 (4)	0.0008 (4)
C1	0.0178 (6)	0.0235 (6)	0.0218 (6)	0.0025 (5)	0.0051 (5)	−0.0018 (5)
C2	0.0200 (6)	0.0186 (6)	0.0218 (6)	0.0038 (5)	−0.0002 (5)	−0.0010 (5)
C3	0.0260 (6)	0.0146 (6)	0.0170 (6)	−0.0035 (5)	0.0021 (5)	0.0005 (4)
C4	0.0186 (6)	0.0159 (6)	0.0177 (6)	−0.0029 (4)	0.0055 (5)	−0.0019 (4)
C5	0.0178 (6)	0.0133 (6)	0.0162 (5)	−0.0015 (4)	0.0037 (4)	−0.0029 (4)
C6	0.0236 (7)	0.0254 (7)	0.0363 (8)	0.0077 (6)	0.0145 (6)	0.0075 (6)
C7	0.0197 (6)	0.0149 (6)	0.0159 (6)	0.0004 (5)	0.0030 (5)	0.0000 (4)
C8	0.0196 (6)	0.0204 (7)	0.0274 (7)	−0.0006 (5)	−0.0007 (5)	−0.0002 (5)
C9	0.0208 (6)	0.0172 (6)	0.0120 (5)	0.0008 (5)	0.0063 (4)	0.0005 (4)
C10	0.0254 (7)	0.0185 (6)	0.0178 (6)	−0.0024 (5)	0.0067 (5)	−0.0015 (4)
C11	0.0400 (8)	0.0151 (6)	0.0208 (6)	0.0022 (5)	0.0111 (6)	0.0001 (5)
C12	0.0342 (7)	0.0219 (6)	0.0202 (6)	0.0119 (5)	0.0072 (5)	0.0039 (5)
C13	0.0224 (6)	0.0258 (7)	0.0173 (6)	0.0063 (5)	0.0044 (5)	0.0011 (5)

Geometric parameters (Å, °)

N1—C1	1.3382 (15)	C4—H4	0.938 (15)
N1—C5	1.3431 (15)	C6—H5	0.983 (17)
N2—C5	1.3769 (15)	C6—H6	0.951 (18)
N2—C7	1.4485 (14)	C6—H7	0.998 (17)
N2—C6	1.4508 (15)	C7—H8	1.010 (13)
N3—C9	1.3773 (15)	C7—H9	0.968 (14)
N3—C8	1.4556 (15)	C8—H10	0.957 (16)
N3—C7	1.4563 (15)	C8—H11	0.996 (16)
N4—C13	1.3406 (15)	C8—H12	1.012 (16)
N4—C9	1.3463 (15)	C9—C10	1.4123 (16)
C1—C2	1.3823 (17)	C10—C11	1.3756 (17)
C1—H1	0.992 (15)	C10—H13	0.954 (14)
C2—C3	1.3907 (17)	C11—C12	1.3858 (19)
C2—H2	0.972 (14)	C11—H14	0.964 (15)
C3—C4	1.3753 (17)	C12—C13	1.3775 (18)
C3—H3	0.957 (15)	C12—H15	0.969 (15)
C4—C5	1.4121 (16)	C13—H16	1.001 (14)
C1—N1—C5	117.83 (10)	H6—C6—H7	108.0 (13)
C5—N2—C7	122.07 (9)	N2—C7—N3	112.76 (9)
C5—N2—C6	120.81 (10)	N2—C7—H8	107.5 (7)
C7—N2—C6	117.12 (9)	N3—C7—H8	108.9 (7)
C9—N3—C8	119.99 (10)	N2—C7—H9	109.8 (8)
C9—N3—C7	121.18 (9)	N3—C7—H9	107.0 (8)
C8—N3—C7	116.06 (10)	H8—C7—H9	111.0 (10)
C13—N4—C9	117.83 (10)	N3—C8—H10	107.8 (9)
N1—C1—C2	124.54 (11)	N3—C8—H11	109.9 (8)
N1—C1—H1	115.4 (8)	H10—C8—H11	110.5 (12)
C2—C1—H1	120.0 (8)	N3—C8—H12	111.2 (8)
C1—C2—C3	117.13 (11)	H10—C8—H12	107.2 (12)

C1—C2—H2	118.3 (8)	H11—C8—H12	110.2 (13)
C3—C2—H2	124.6 (8)	N4—C9—N3	117.13 (10)
C4—C3—C2	120.11 (11)	N4—C9—C10	121.76 (10)
C4—C3—H3	119.2 (9)	N3—C9—C10	121.10 (10)
C2—C3—H3	120.7 (9)	C11—C10—C9	118.39 (11)
C3—C4—C5	118.62 (11)	C11—C10—H13	119.9 (9)
C3—C4—H4	121.2 (9)	C9—C10—H13	121.6 (8)
C5—C4—H4	120.1 (9)	C10—C11—C12	120.22 (12)
N1—C5—N2	117.75 (10)	C10—C11—H14	118.5 (9)
N1—C5—C4	121.76 (10)	C12—C11—H14	121.3 (9)
N2—C5—C4	120.49 (10)	C13—C12—C11	117.55 (11)
N2—C6—H5	111.2 (10)	C13—C12—H15	118.8 (9)
N2—C6—H6	106.0 (10)	C11—C12—H15	123.7 (9)
H5—C6—H6	108.4 (14)	N4—C13—C12	124.25 (12)
N2—C6—H7	111.2 (10)	N4—C13—H16	116.8 (7)
H5—C6—H7	111.7 (14)	C12—C13—H16	118.9 (7)

Hydrogen-bond geometry (Å, °)

*Cg*1 and *Cg*2 are the centroids of the N1/C1–C5 and N4/C9–C15 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···Cg2^{i}	0.99 (2)	2.64 (2)	3.484 (1)	143 (1)
C6—H7···Cg2^{ii}	1.00 (2)	2.75 (2)	3.545 (2)	137 (1)
C10—H13···Cg1^{iii}	0.95 (2)	2.90 (1)	3.637 (1)	135 (1)

Symmetry codes: (i) −*x*+1, −*y*, −*z*+2; (ii) −*x*+3/2, *y*+1/2, −*z*+3/2; (iii) *x*, *y*−1, *z*.

Fig. 1

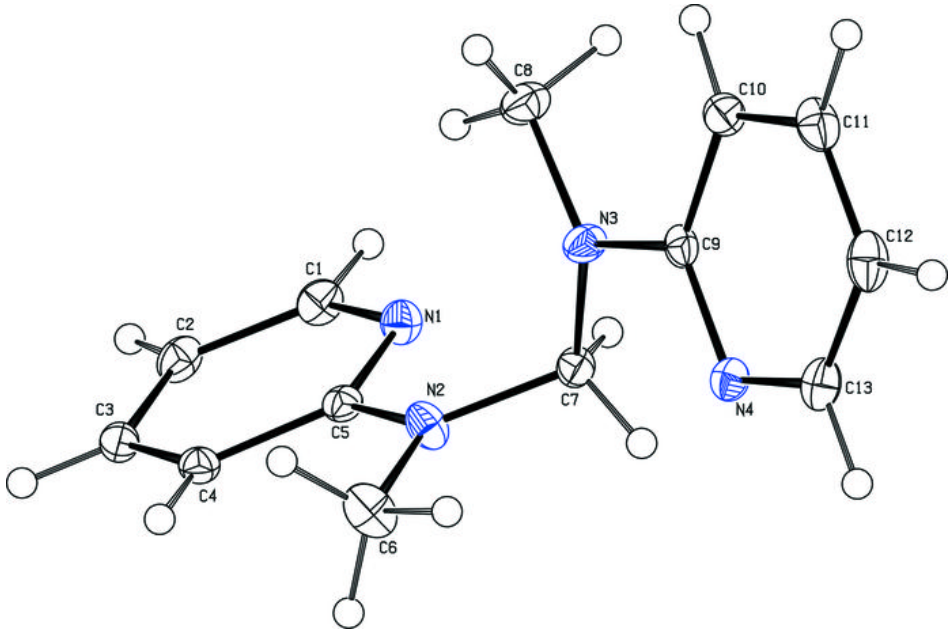

Fig. 2