(1R,2R)-N,N'-Dimethylcyclohexane-1,2-diamine

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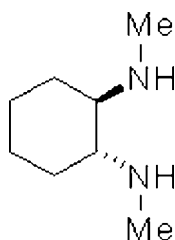Correspondence e-mail: mail@carsten-strohmann.de

The molecule of the title compound, $C_8H_{18}N_2$, possesses C_2 symmetry. Owing to its stereochemistry, it is used in the synthesis of chiral ligands and metal complexes for asymmetric synthesis. The cyclohexane ring shows a chair conformation with the amino groups in equatorial positions. Contrary to the literature, the title compound is not a liquid, but a crystalline solid at room temperature (293 K). The absolute configuration is assigned from the synthesis.

Related literature

The synthesis of the title compound is described by Kizirian *et al.* (2005). For related literature, see: Larrox and Jacobsen (1994); Cole *et al.* (2005); Seebach *et al.* (1977); Strohmann & Gessner (2007); Strohmann *et al.* (2003, 2004); Strohmann, Däschlein & Auer (2006); Strohmann, Dilsky & Strohfeldt (2006); Strohmmann & Gessner (2007a,b).

Experimental

Crystal data

$C_8H_{18}N_2$	$V = 910.0$ (8) Å^3
$M_r = 142.24$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 7.552$ (4) Å	$\mu = 0.06$ mm^{-1}
$b = 8.521$ (5) Å	$T = 173$ (2) K
$c = 14.142$ (8) Å	$0.40 \times 0.10 \times 0.10$ mm

Data collection

Bruker APEX CCD diffractometer	4816 measured reflections
Absorption correction: multi-scan	953 independent reflections
(*SADABS*; Bruker, 1999)	784 reflections with $I > 2\sigma(I)$
$T_{min} = 0.912$, $T_{max} = 0.982$	$R_{int} = 0.050$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$	H atoms treated by a mixture of
$wR(F^2) = 0.111$	independent and constrained
$S = 1.08$	refinement
953 reflections	$\Delta\rho_{max} = 0.12$ e Å^{-3}
101 parameters	$\Delta\rho_{min} = -0.12$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D \cdots A$	D—H$\cdots A$
N1—H1$N \cdots$N2^i	0.91 (4)	2.36 (4)	3.250 (4)	166 (3)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1999); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2055).

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supplementary materials

(1*R*,2*R*)-*N*,*N*'-Dimethylcyclohexane-1,2-diamine

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Comment

Due to their strong coordination ability diamine bases have become powerful agents in various fields of chemistry *e.g.* for the deaggregation of organolithium compounds or the coordination of transition metals. Especially chiral amines have attracted special attention in asymmetric synthesis. Thereby, (1*R*,2*R*)-*N*,*N*'-dimethylcyclohexane-1,2-diamine is an important chiral amine, which serves as a starting material for the synthesis of numerous diamine bases with a cyclohexane framework. The amine crystallizes at room temperature as colourless needles in the orthorhombic crystal system, space group $P2_12_12_1$. The asymmetric unit contains one molecule of the C_2 symmetric amine (see figure 1).

In the unit cell molecules are interconnected *via* hydrogen bonding to give infinite layers (see figure 2). H atoms (H1N) are arranged in direction to the nitrogen atom (N2) of an adjacent molecule (N1—HN1—N2' angle: 166 (3)°). However, the long N1—N2' distance of 3.250 (4) Å and the short N1—HN1 distance of 0.91 (4) Å indicate weak N–H···N hydrogen bonds.

Experimental

Treatment of the enantiomerically pure (*R*,*R*)-1,2-diammoniumcyclohexane mono-(+)-tartrate with two equivalents of ethyl-chloroformate in the presence of a stochiometric amount of NaOH resulted in the formation of diethyl-(1*R*,2*R*)-cyclohex-ane-1,2-diyldicarbamat. Subsequent reduction with an excess of LiAlH$_4$ gave colourless crystals of the title compound during bulb-to-bulb destillation. Contrary to a formerly published synthesis, (1*R*,2*R*)-*N*,*N*'-diemthylcyclohexane-1,2-diamine is not liquid but a highly hygroscopic crystalline solid.

^{1}H-NMR (500.1 MHz, CDCl$_3$): 0.86–0.94 (m, 2H; C*H$_2$*CHN), 1.13–1.19 (m, 2H; C*H$_2$*CH$_2$CHN), 1.61–1.67 (m, 2H; C*H$_2$*CH$_2$CHN), 1.68–1.75 (br, 2H, N*H*), 1.93–2.00 (m, 2H; C*H$_2$*CHN), 2.02–2.06 (m, 2H; C*H*NCHN), 2.33 (s, 6H; NC*H$_3$*).

^{13}C-NMR (100.6 MHz, CDCl$_3$): 25.0 (*C*H$_2$CH$_2$CHN), 30.8 (*C*H$_2$CHN), 33.7 (*C*H$_3$), 63.2 (*C*HN).

Refinement

Refinement was accomplished by full-matrix least-squares methods (based on $F_o{}^2$, *SHELXL97*); anisotropic thermal parameters for all non-H atoms in the final cycles; the H atoms were refined on a riding model in their ideal geometric positions, except for H(1 N) and H(2 N), which were refined independently.
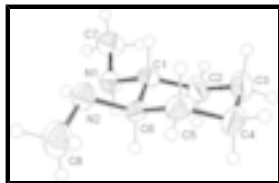
supplementary materials

Figures

Fig. 1. *ORTEP* plot of the molecular structure of (1*R*,2*R*)-*N*,*N'*-diemthylcyclohexane-1,2-diamine. Thermal ellipsoids are drawn at the 50% probability level.
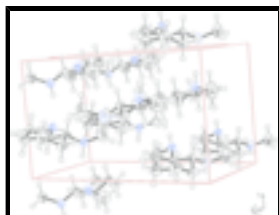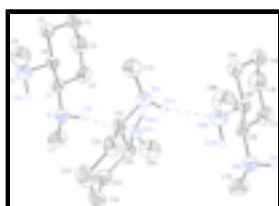

Fig. 2. *ORTEP* plot of the unit cell.

Fig. 3. Display of the hydrogen bonding.

(1*R*,2*R*)-*N*,*N'*-Dimethylcyclohexane-1,2-diamine

Crystal data

$C_8H_{18}N_2$	$F_{000} = 320$
$M_r = 142.24$	$D_x = 1.038$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Melting point: 313 K
Hall symbol: P 2ac 2ab	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.552$ (4) Å	$\theta = 2.8–25.0º$
$b = 8.521$ (5) Å	$\mu = 0.06$ mm^{-1}
$c = 14.142$ (8) Å	$T = 173$ (2) K
$V = 910.0$ (8) Å^3	Needle, colourless
$Z = 4$	$0.40 \times 0.10 \times 0.10$ mm

Data collection

Bruker APEXCCD diffractometer	953 independent reflections
Radiation source: fine-focus sealed tube	784 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.050$
$T = 173(2)$ K	$\theta_{max} = 25.0º$
ω scans	$\theta_{min} = 2.8º$
Absorption correction: multi-scan (SADABS; Bruker, 1999)	$h = -8 \rightarrow 8$
$T_{min} = 0.912$, $T_{max} = 0.982$	$k = -10 \rightarrow 9$

4816 measured reflections

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.111$	$w = 1/[\sigma^2(F_o{}^2) + (0.0405P)^2 + 0.258P]$ where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
953 reflections	$\Delta\rho_{max} = 0.12$ e Å^{-3}
101 parameters	$\Delta\rho_{min} = -0.11$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 > 2sigma(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	$U_{iso}*/U_{eq}$
C1	0.8581 (4)	0.3857 (3)	0.28989 (19)	0.0346 (7)
H1	0.8702	0.5000	0.3046	0.042*
C2	0.6814 (4)	0.3316 (4)	0.3294 (2)	0.0477 (9)
H2A	0.6785	0.3525	0.3982	0.057*
H2B	0.6703	0.2168	0.3202	0.057*
C3	0.5242 (4)	0.4129 (5)	0.2827 (3)	0.0608 (11)
H3A	0.4128	0.3663	0.3067	0.073*
H3B	0.5247	0.5256	0.2999	0.073*
C4	0.5304 (4)	0.3970 (4)	0.1764 (3)	0.0528 (10)
H4A	0.5131	0.2857	0.1586	0.063*
H4B	0.4333	0.4590	0.1479	0.063*
C5	0.7059 (4)	0.4540 (4)	0.1385 (2)	0.0458 (9)
H5A	0.7174	0.5679	0.1509	0.055*
H5B	0.7091	0.4381	0.0691	0.055*
C6	0.8605 (4)	0.3685 (3)	0.18321 (18)	0.0331 (7)
H6	0.8514	0.2545	0.1671	0.040*

C7	1.0546 (5)	0.3573 (4)	0.4252 (2)	0.0581 (10)
H7A	1.0807	0.4699	0.4250	0.087*
H7B	1.1583	0.2994	0.4480	0.087*
H7C	0.9537	0.3367	0.4670	0.087*
C8	1.0664 (5)	0.3767 (5)	0.0508 (2)	0.0644 (11)
H8A	1.0492	0.2629	0.0465	0.097*
H8B	1.1894	0.4025	0.0350	0.097*
H8C	0.9866	0.4296	0.0065	0.097*
N1	1.0117 (3)	0.3064 (4)	0.32977 (18)	0.0389 (7)
H1N	0.985 (4)	0.203 (4)	0.329 (2)	0.056 (10)*
N2	1.0282 (4)	0.4287 (3)	0.14644 (19)	0.0413 (7)
H2N	1.109 (4)	0.391 (4)	0.193 (2)	0.045 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0385 (17)	0.0216 (15)	0.0438 (17)	0.0043 (16)	0.0006 (14)	0.0013 (13)
C2	0.044 (2)	0.0420 (19)	0.057 (2)	0.0050 (17)	0.0110 (17)	0.0051 (17)
C3	0.039 (2)	0.053 (2)	0.090 (3)	0.0029 (19)	0.010 (2)	0.007 (2)
C4	0.0335 (19)	0.0403 (19)	0.085 (3)	−0.0023 (17)	−0.0126 (19)	0.0081 (19)
C5	0.046 (2)	0.0355 (19)	0.056 (2)	−0.0032 (17)	−0.0131 (16)	0.0054 (16)
C6	0.0341 (16)	0.0251 (16)	0.0400 (17)	−0.0025 (15)	−0.0054 (14)	−0.0008 (13)
C7	0.068 (2)	0.055 (2)	0.052 (2)	0.010 (2)	−0.0109 (18)	−0.0046 (18)
C8	0.057 (2)	0.085 (3)	0.052 (2)	−0.008 (2)	0.0120 (18)	0.005 (2)
N1	0.0389 (15)	0.0388 (16)	0.0390 (15)	0.0029 (14)	−0.0033 (13)	0.0002 (13)
N2	0.0370 (16)	0.0514 (18)	0.0355 (15)	−0.0048 (14)	0.0004 (13)	0.0048 (13)

Geometric parameters (Å, °)

C1—N1	1.455 (4)	C5—H5A	0.9900
C1—C6	1.516 (4)	C5—H5B	0.9900
C1—C2	1.519 (4)	C6—N2	1.462 (4)
C1—H1	1.0000	C6—H6	1.0000
C2—C3	1.525 (4)	C7—N1	1.454 (4)
C2—H2A	0.9900	C7—H7A	0.9800
C2—H2B	0.9900	C7—H7B	0.9800
C3—C4	1.511 (5)	C7—H7C	0.9800
C3—H3A	0.9900	C8—N2	1.452 (4)
C3—H3B	0.9900	C8—H8A	0.9800
C4—C5	1.510 (4)	C8—H8B	0.9800
C4—H4A	0.9900	C8—H8C	0.9800
C4—H4B	0.9900	N1—H1N	0.91 (4)
C5—C6	1.515 (4)	N2—H2N	0.96 (3)
N1—C1—C6	109.4 (2)	C4—C5—H5B	109.2
N1—C1—C2	114.6 (2)	C6—C5—H5B	109.2
C6—C1—C2	110.3 (3)	H5A—C5—H5B	107.9
N1—C1—H1	107.4	N2—C6—C5	110.5 (2)
C6—C1—H1	107.4	N2—C6—C1	109.3 (2)

C2—C1—H1	107.4	C5—C6—C1	111.1 (3)
C1—C2—C3	112.8 (3)	N2—C6—H6	108.6
C1—C2—H2A	109.0	C5—C6—H6	108.6
C3—C2—H2A	109.0	C1—C6—H6	108.6
C1—C2—H2B	109.0	N1—C7—H7A	109.5
C3—C2—H2B	109.0	N1—C7—H7B	109.5
H2A—C2—H2B	107.8	H7A—C7—H7B	109.5
C4—C3—C2	111.4 (3)	N1—C7—H7C	109.5
C4—C3—H3A	109.3	H7A—C7—H7C	109.5
C2—C3—H3A	109.3	H7B—C7—H7C	109.5
C4—C3—H3B	109.3	N2—C8—H8A	109.5
C2—C3—H3B	109.3	N2—C8—H8B	109.5
H3A—C3—H3B	108.0	H8A—C8—H8B	109.5
C5—C4—C3	110.6 (3)	N2—C8—H8C	109.5
C5—C4—H4A	109.5	H8A—C8—H8C	109.5
C3—C4—H4A	109.5	H8B—C8—H8C	109.5
C5—C4—H4B	109.5	C7—N1—C1	113.5 (2)
C3—C4—H4B	109.5	C7—N1—H1N	111 (2)
H4A—C4—H4B	108.1	C1—N1—H1N	106 (2)
C4—C5—C6	111.9 (3)	C8—N2—C6	113.4 (3)
C4—C5—H5A	109.2	C8—N2—H2N	114.6 (19)
C6—C5—H5A	109.2	C6—N2—H2N	100.9 (19)

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N⋯N2[i]	0.91 (4)	2.36 (4)	3.250 (4)	166 (3)

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

Fig. 1

Fig. 2

Fig. 3