Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Redetermination of *trans*-2,5,7,10-tetraaza-bicyclo[4.4.0]decane

Inge Sänger, Hans-Wolfram Lerner and Michael Bolte*

Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail:
bolte@chemie.uni-frankfurt.de

The structure of the title compound, $C_6H_{14}N_4$, has previously been determined at room temperature by Böttcher, Buchkremer-Hermanns, Hönle & von Schnering [*Z. Kristallogr.* (1987), **181**, 223–226], who found only one position for the amine H atom. We report here the low-temperature structure of the title compound, showing that the amine H atom is disordered over two positions. This H atom must be located either in the equatorial or in the axial position so that N—H···N hydrogen bonds can be formed. A mirror plane contains the bond that the two rings have in common and a twofold rotation axis runs through the mid-points of all three C—C bonds.

Received 8 September 2004
Accepted 13 September 2004
Online 25 September 2004

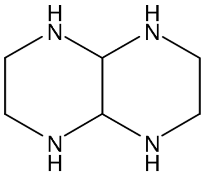
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.050
wR factor = 0.144
Data-to-parameter ratio = 10.0

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

Comment

A perspective view of the title compound, (I), is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, *CONQUEST* Version 1.6 plus three updates; *MOGUL* Version 1.0; Allen, 2002). The molecule possesses crystallographic C_{2h} symmetry. A mirror plane contains the bond that the two rings have in common and a twofold rotation axis runs through the mid-points of all three C—C bonds. As a result, the asymmetric unit comprises a quarter of the molecule.



(I)

The structure of (I) has already been determined at room temperature by Böttcher *et al.* (1987). These authors have

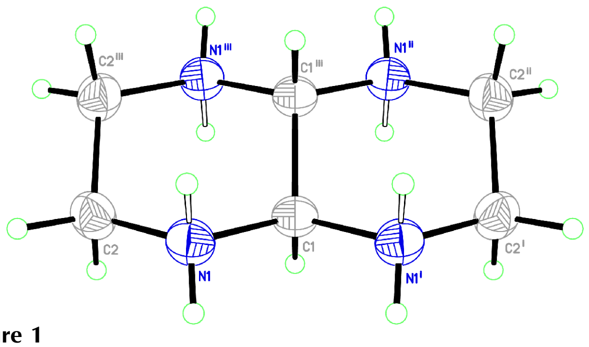


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level. Both positions are shown for each of the disordered H atoms. [Symmetry codes: (i) x, $1 - y$, z; (ii) $-x$, $1 - y$, $1 - z$; (iii) $-x$, y, $1 - z$.]

DOI: 10.1107/S1600536804022688

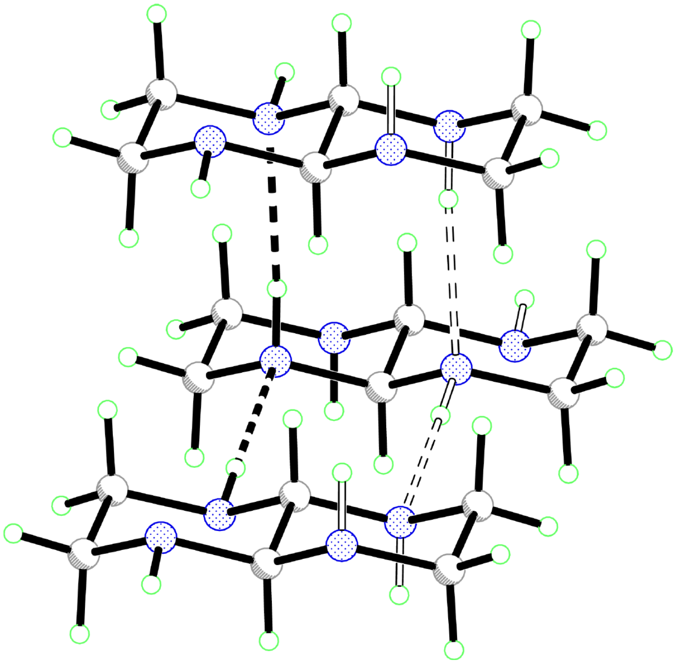


Figure 2
Hydrogen-bond (dashed lines) pattern of the title compound.

located the positions of the H atoms in a ΔF synthesis and refined the H atoms isotropically. They located the amine H atom in an equatorial position. However, they have not reported existence and nature of hydrogen bonds.

We have redetermined the structure at low temperature and have found that the amine H atom is disordered over two positions, either equatorial or axial. If this H atom were located in all molecules at the same position, a short H···H contact to a neighbouring N—H group would arise and no hydrogen bond would be possible. As a result, this H atom must occupy different positions in hydrogen-bonded molecules (Fig. 2).

Experimental

In an attempt to crystallize ^{t}Bu$_3$SiBr (Wiberg *et al.*, 1997) from CH$_3$CN, single crystals of the title compound suitable for X-ray diffraction were obtained.

Crystal data

$C_6H_{14}N_4$
$M_r = 142.21$
Monoclinic, $C2/m$
$a = 5.283$ (2) Å
$b = 15.937$ (4) Å
$c = 4.6205$ (18) Å
$\beta = 108.78$ (3)°
$V = 368.3$ (2) Å^3
$Z = 2$

$D_x = 1.282$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2510 reflections
$\theta = 4.2$–27.7°
$\mu = 0.09$ mm^{-1}
$T = 173$ (2) K
Rod, colourless
0.32 × 0.14 × 0.13 mm

Data collection

Stoe *IPDS*-II two-circle diffractometer
ω scans
Absorption correction: none
2406 measured reflections
449 independent reflections

362 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.069$
$\theta_{\rm max} = 27.8°$
$h = -6 \rightarrow 6$
$k = -18 \rightarrow 20$
$l = -6 \rightarrow 5$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.144$
$S = 0.99$
449 reflections
45 parameters

All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0894P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.26$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.4591 (13)	N1—C2	1.4669 (14)
C1—N1—C2	109.91 (10)	N1—C1—C1$^{\rm ii}$	110.25 (10)
N1$^{\rm i}$—C1—N1	108.77 (13)	N1—C2—C2$^{\rm iii}$	111.18 (9)

Symmetry codes: (i) $x, 1-y, z$; (ii) $-x, 1-y, 1-z$; (iii) $-x, y, 1-z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1′···N1$^{\rm iv}$	0.80 (4)	2.36 (4)	3.146 (3)	167 (3)
N1—H1″···N1$^{\rm v}$	0.93 (3)	2.27 (3)	3.182 (3)	168 (2)

Symmetry codes: (iv) $1-x, y, 2-z$; (v) $-x, y, 2-z$.

All H atoms were located in a difference map and refined isotropically. The two sites for the disordered H atoms bonded to N1 refined to relative occupancies of 0.54 (4) and 0.46 (4) for the equatorial and axial H atoms, respectively.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Böttcher, P., Buchkremer-Hermanns, H., Hönle, W. & von Schnering, H. G. (1987). *Z. Kristallogr.* **181**, 223–226.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus.* Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie, (2001. *X-AREA*.. Stoe & Cie, Darmstadt, Germany.
Wiberg, N., Amelunxen, K., Lerner, H.-W., Schuster, H., Nöth, H., Krossing, I., Schmidt-Amelunxen, M. & Seifert, T. J. (1997). *J. Organomet. Chem.* **542**, 1–18.