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3,3-Pentamethylenediaziridine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.034
wR factor = 0.088
Data-to-parameter ratio = 9.6

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

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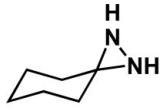
The title compound, $C_6H_{12}N_2$, is the first free *N*,*N*-unsubstituted 3,3-dialkyldiaziridine to be structurally characterized. The crystal structure indicates the presence of intermolecular N—H···N hydrogen bonds, resulting in a three-dimensional network.

Comment

During the course of a project aiming at developing new aminating reagents, we synthesized 3,3-pentamethylenediaziridine, (I), as a precursor to the corresponding diazirine, (II). This compound shows good aminating efficiency towards Grignard reagents (Schmitz & Ohme, 1961). Diazirines can also be used as carbene precursors.



(I)

(II)

Surprisingly, no crystal structure of a free *N*,*N*-unsubstituted 3,3-dialkyldiaziridine has been published so far. In all the reported compounds, either they were *N*-substituted or *N*-coordinated to a metal (Adedapo, Avent *et al.*, 1993; Adedapo, Benyunes *et al.*, 1993). The only two other examples of *N*,*N*-unsubstituted diaziridines are 3-methyldiaziridine (Mashyukov *et al.*, 1974), which was studied by electron diffraction, and a C-alkoxy diaziridine (Blüchel *et al.*, 2001). Crystals suitable for analysis were easily obtained, although it was necessary to perform the analysis using the sealed capillary method (see *Experimental*). A view of (I) is shown in Fig. 1 and selected geometric parameters are presented in Table 1. The H atom on each N atom is disordered over two positions. The observed N—N bond length of 1.5083 (17) Å is greater than the corresponding bond length observed in 3-methyldiaziridine (1.468 Å) but less than that observed in the C-alkoxy diaziridine (1.539 Å). In the crystal structure, the molecules are held together by a cooperative hydrogen-bonding system (Table 2). Every N atom is both a donor and an acceptor of a hydrogen bond. The hydrogen-bond network propagates along the *b* axis (Fig. 2).

Experimental

The title compound was prepared according to the method of Schmitz & Ohme (1965). Crystals suitable for analysis were prepared by slow recrystallization from toluene. However, the title compound showed spontaneous sublimation during the first attempt at structure determination. Accordingly, the analysis was made using a crystal sealed in a capillary and immobilized using silicone grease.

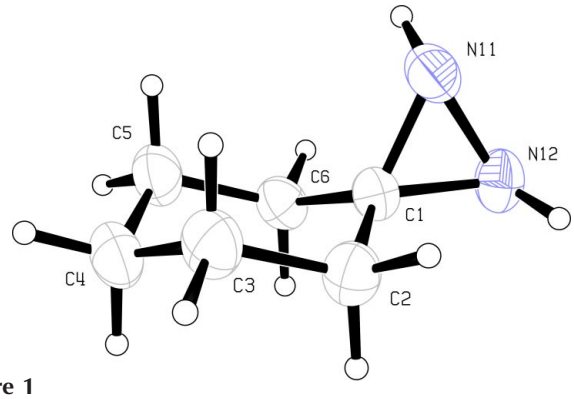


Figure 1
A view of the asymmetric unit of (I), with displacement ellipsoids drawn at the 30% probability level. Only one disorder component is shown.

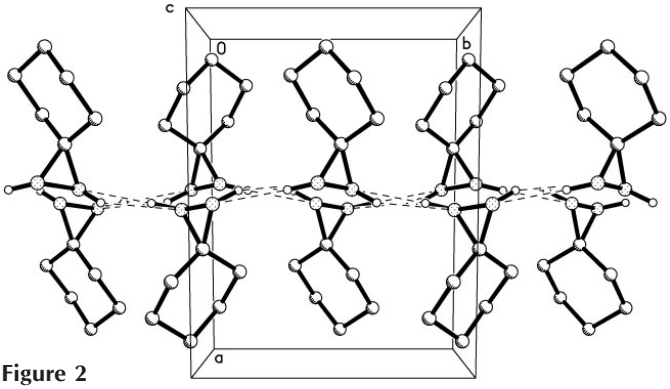


Figure 2
A view of the unit cell, showing the disordered hydrogen-bond network along the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms attached to C atoms have been omitted.

Crystal data

$C_6H_{12}N_2$
M_r = 112.18
Monoclinic, $P2_1/c$
a = 10.373 (3) Å
b = 8.037 (3) Å
c = 8.077 (3) Å
β = 98.86 (3)°
V = 665.3 (4) Å^3
Z = 4
D_x = 1.120 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 20.0–25.0°
μ = 0.54 mm^{-1}
T = 293 (2) K
Block, colourless
0.40 × 0.36 × 0.23 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: by integration (*ABSORP* in *NRCVAX*; Gabe *et al.*, 1989)
T_{min} = 0.819, T_{max} = 0.898
19 833 measured reflections
1253 independent reflections
872 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 69.9°
$h = -12 \rightarrow 12$
$k = -9 \rightarrow 9$
$l = -9 \rightarrow 9$
5 standard reflections
frequency: 60 min
intensity decay: 2.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.088
S = 1.05
1253 reflections
130 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.072 (4)

Table 1
Selected geometric parameters (Å, °).

N11—C1	1.4420 (16)	N12—C1	1.4427 (14)
N11—N12	1.5083 (17)		
C1—N11—N12	58.50 (7)	N12—C1—C6	118.28 (11)
C1—N12—N11	58.45 (7)	N11—C1—C2	117.09 (12)
N11—C1—N12	63.05 (8)	N12—C1—C2	117.45 (10)
N11—C1—C6	116.70 (11)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N11—H11A···N12^{i}	0.91 (5)	2.20 (5)	3.082 (2)	164 (3)
N11—H11B···N11^{ii}	0.87 (3)	2.26 (3)	3.132 (3)	173 (3)
N12—H12A···N11^{iii}	0.94 (3)	2.37 (3)	3.082 (2)	132 (2)
N12—H12B···N12^{iv}	0.95 (2)	2.23 (2)	3.173 (3)	171 (2)

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

Two H atoms were introduced on each N atom to satisfy the short contacts observed.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *NRC*-2 and *NRC*-2*A* (Ahmed *et al.*, 1973); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997) and *PLATON* (Spek, 1995); software used to prepare material for publication: *SHELXL*97.

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