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# Dicyano{[(1*S*)-(1-phenylethyl)aziridin-2-yl]methanolato-$\kappa^2N,O$}boron

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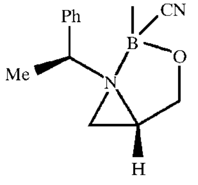
In the title compound, $C_{13}H_{14}BN_3O$, the aziridine ring is an almost equilateral triangle, the C—C distance being slightly shorter than the C—N distances, probably because of the dative B—N bond. The five-membered ring, composed of two C atoms and N, B and O atoms, is fused with the aziridine ring to form a six-membered ring with a chair conformation.

## Comment

During the course of synthetic studies employing the chiral aziridine 1-[(*S*)-1-phenylethyl]-2-(*tert*-butyldimethylsilyloxymethyl)aziridine (Lee & Ha, 2003), we expected that the azidirine ring, known to be labile to Lewis acids (Pearson *et al.*, 1996), would be opened by a nitrile nucleophile with the assistance of boron trifluoride. However, although the spectral data for the product, *viz.* the title compound, (I), do not allow an unambiguous structural assignment, they clearly show that (I) contains two cyano groups, with conservation of the three-membered aziridine ring. Compound (I) is stable at room temperature in air for up to a month, and the structure determination described here was undertaken to establish its exact nature.



(I)

The structure of (I) is shown in Fig. 1. Contrary to expectation, the nitrile nucleophiles have replaced the fluorides at the B atom instead of causing ring opening, leaving the aziridine ring intact. Atoms N1, C1 and C2 form an almost equilateral triangle, the C1—C2 distance [1.468 (3) Å] being slightly shorter than the C—N distances [1.484 (2) and 1.506 (2) Å; Table 1]. Usually, the C—C distance in aziridine rings is longer than the C—N distances (Hargreaves *et al.*, 1997; Willems *et al.*, 1997). The unusual behaviour of (I) is probably due to the electron-donating nature of the N atom towards the B atom in forming the dative B—N bond. The geometry around atom N1 is distorted significantly from ideal tetrahedral, with high strain.

A five-membered ring composed of atoms N1, B, O, C3 and C2 is observed. This ring is fused with the aziridine ring, thus forming a six-membered ring that displays a chair conformation. The interplanar angle between the azidirine ring plane and the main plane of the chair (B/N1/C2/C3) is 68.96 (12)°, which is greater than that formed with the plane composed of atoms O, B and C3 [35.98 (17)°].

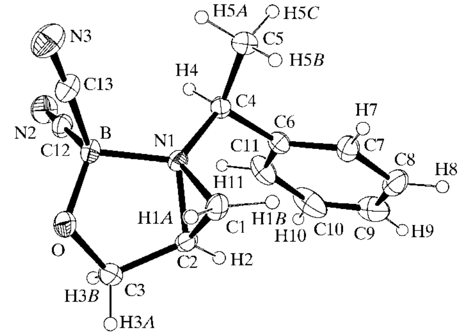


**Figure 1**
The molecular structure of (I), showing 30% probability displacement ellipsoids.

Compound (I) has a characteristic coordinative bond between the N and B atoms, with a bond distance of 1.608 (3) Å, which is significantly longer than typical covalent bond distances (~1.4 Å; Allen *et al.*, 1987) but agrees well with values for other dative B—N bonds (*e.g.* in $NH_3BF_3$; Penner *et al.*, 2002). The geometry around the B atom can be described as being typical of tetrahedral coordination [~104.08 (14)–114.09 (16)°].

There is no strong hydrogen bond in (I) (Spek, 1990). However, there are some short C—H···N contacts that could be regarded as weak hydrogen bonds (Table 2). Additionally, there is an intermolecular contact between atoms C1 and N3(1 + *x*, *y*, *z*) [3.163 (4) Å].

## Experimental

The title compound was prepared from the reaction of 1-[(*S*)-1-phenylethyl]-2-(*tert*-butyldimethylsilyloxymethyl)aziridine with three molar equivalents of cyanotrimethylsilane and one equivalent of boron trifluroride diethyl etherate at 233 K in $CH_2Cl_2$. The initial product (85% yield), a white solid, was recrystallized from dichloromethane to afford single crystals suitable for X-ray diffraction studies. Analysis found: C 65.2, H 5.77, N 17.5%; calculated for $C_{13}H_{14}BN_3O$: C 65.3, H 5.90, N 17.6%.

### Crystal data

$C_{13}H_{14}BN_3O$
$M_r = 239.08$
Triclinic, $P1$
$a = 6.710\ (3)$ Å
$b = 6.767\ (4)$ Å
$c = 8.059\ (5)$ Å
$\alpha = 71.51\ (4)^\circ$
$\beta = 84.68\ (5)^\circ$
$\gamma = 70.16\ (3)^\circ$
$V = 326.4\ (3)$ Å$^3$

$Z = 1$
$D_x = 1.216$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 20 reflections
$\theta = 10\text{–}14^\circ$
$\mu = 0.08$ mm$^{-1}$
$T = 293\ (2)$ K
Rectangular, colourless
0.5 × 0.4 × 0.3 mm

### Data collection

MacScience MXC3 diffractometer
$\omega\text{–}2\theta$ scans
1595 measured reflections
1483 independent reflections
1435 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\rm max} = 27.5^\circ$

$h = -8 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = 0 \rightarrow 10$
2 standard reflections every 100 reflections intensity decay: none

### Refinement

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.090$
$S = 1.08$
1483 reflections
165 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.0238P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.17$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.15$ e Å$^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.09 (2)

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| C1—C2 | 1.468 (3) | C12—B | 1.618 (3) |
| C1—N1 | 1.484 (2) | C13—N3 | 1.128 (3) |
| C2—N1 | 1.506 (2) | C13—B | 1.604 (3) |
| C2—C3 | 1.513 (3) | B—O | 1.425 (3) |
| C3—O | 1.427 (3) | B—N1 | 1.608 (3) |
| C12—N2 | 1.140 (3) | | |
| | | | |
| C2—C1—N1 | 61.34 (12) | N1—B—C12 | 105.71 (15) |
| C1—C2—N1 | 59.85 (12) | C1—N1—C2 | 58.81 (13) |
| N2—C12—B | 179.8 (2) | C1—N1—C4 | 118.49 (14) |
| N3—C13—B | 178.1 (2) | C2—N1—C4 | 123.43 (14) |
| O—B—C13 | 110.71 (16) | C1—N1—B | 113.35 (15) |
| O—B—N1 | 104.08 (14) | C2—N1—B | 102.86 (14) |
| C13—B—N1 | 112.53 (15) | C4—N1—B | 122.79 (14) |
| O—B—C12 | 114.09 (16) | B—O—C3 | 107.80 (15) |
| C13—B—C12 | 109.59 (15) | | |
| | | | |
| C1—C2—C3—O | −40.6 (2) | O—B—N1—C2 | −18.52 (16) |
| N1—C2—C3—O | 23.63 (19) | O—B—N1—C4 | −163.54 (14) |
| O—B—N1—C1 | 42.79 (19) | | |

**Table 2**
Hydrogen-bonding and short contact geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C2—H2···N2$^{\rm i}$ | 0.98 | 2.67 | 3.465 (3) | 138 |
| C1—H1$B$···N2$^{\rm ii}$ | 0.97 | 2.64 | 3.404 (3) | 136 |
| C9—H9···N3$^{\rm iii}$ | 0.93 | 2.70 | 3.553 (4) | 152 |

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

In the absence of significant anomalous scattering, the absolute configuration could not be determined and was therefore assigned by reference to the starting materials. Friedel reflections were merged. H atoms were positioned geometrically and treated as riding (*SHELXL*97; Sheldrick, 1997).

Data collection: *MXC*3 *Diffractometer Control Software* (MacScience, 1994); cell refinement: *MXC*3 *Diffractometer Control Software*; data reduction: *MXC*3 *Diffractometer Control Software*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1576). Services for accessing these data are described at the back of the journal.

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