Refinement

Refinement on *F*
$R = 0.045$
$wR = 0.071$
$S = 2.459$
2758 reflections
317 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.02$
$\Delta\rho_{\max} = 0.342$ e Å^{-3}
$\Delta\rho_{\min} = -0.358$ e Å^{-3}
Extinction correction: Becker & Coppens type 1, isotropic
Extinction coefficient: 1.0166×10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	U_{eq}
Cl1	0.27730 (19)	0.02987 (4)	0.06612 (7)	0.0816 (4)
Cl2	0.27354 (19)	0.20683 (4)	0.32066 (9)	0.0862 (4)
Cl3	0.22017 (14)	−0.12438 (4)	0.77274 (7)	0.0671 (3)
Cl4	−0.94626 (16)	−0.17921 (4)	0.02035 (10)	0.0793 (3)
O1	0.2722 (4)	−0.02052 (9)	0.29773 (19)	0.0613 (8)
O2	0.2695 (5)	−0.11181 (11)	0.4347 (2)	0.0828 (12)
O3	0.1964 (3)	−0.17503 (9)	0.5537 (2)	0.0575 (8)
O4	−0.1038 (3)	−0.19167 (10)	0.3518 (2)	0.0644 (8)
O5	−0.5642 (4)	−0.16776 (10)	0.1887 (2)	0.0688 (9)
N1	0.2556 (4)	0.03340 (11)	0.5294 (2)	0.0503 (8)
N2	0.2508 (4)	−0.01559 (10)	0.5260 (2)	0.0476 (8)
C1	0.2648 (5)	0.05756 (13)	0.4170 (3)	0.0468 (9)
C2	0.2705 (5)	0.03084 (12)	0.3071 (3)	0.0468 (9)
C3	0.2774 (5)	0.06122 (13)	0.2034 (3)	0.0532 (10)
C4	0.2799 (5)	0.11463 (13)	0.2072 (3)	0.0547 (10)
C5	0.2742 (5)	0.13962 (13)	0.3167 (3)	0.0562 (10)
C6	0.2645 (5)	0.11164 (13)	0.4210 (3)	0.0538 (10)
C7	0.2414 (5)	−0.03989 (14)	0.6390 (3)	0.0500 (10)
C8	0.2355 (6)	−0.00657 (15)	0.7479 (3)	0.0664 (11)
C9	0.2349 (5)	−0.09207 (14)	0.6383 (3)	0.0496 (10)
C10	0.2361 (5)	−0.12654 (14)	0.5301 (3)	0.0510 (10)
C11	0.1946 (5)	−0.21376 (14)	0.4582 (3)	0.0601 (11)
C12	−0.0026 (5)	−0.23223 (13)	0.4179 (3)	0.0574 (10)
C13	−0.2902 (6)	−0.20558 (15)	0.2979 (4)	0.0691 (12)
C14	−0.3685 (6)	−0.15970 (17)	0.2271 (4)	0.0720 (14)
C15	−0.6513 (6)	−0.12308 (15)	0.1284 (4)	0.0702 (14)
C16	−0.8574 (6)	−0.12765 (16)	0.1198 (4)	0.0747 (14)

Table 2. *Selected geometric parameters* (Å, °)

Cl1—C3	1.729 (3)	N2—C7	1.416 (4)
Cl2—C5	1.727 (3)	C1—C2	1.410 (5)
Cl3—C9	1.730 (4)	C1—C6	1.390 (5)
Cl4—C16	1.781 (4)	C2—C3	1.402 (5)
O1—C2	1.324 (4)	C3—C4	1.373 (5)
O2—C10	1.186 (4)	C4—C5	1.385 (5)
O3—C10	1.312 (4)	C5—C6	1.377 (5)
O3—C11	1.456 (4)	C7—C8	1.491 (5)
O4—C12	1.410 (4)	C7—C9	1.341 (5)
O4—C13	1.418 (5)	C9—C10	1.497 (5)
O5—C14	1.410 (5)	C11—C12	1.486 (5)
O5—C15	1.424 (5)	C13—C14	1.480 (6)
N1—N2	1.259 (4)	C15—C16	1.461 (6)
N1—C1	1.409 (4)		
C10—O3—C11	118.7 (3)	C4—C5—C6	120.9 (3)
C12—O4—C13	114.1 (3)	C1—C6—C5	119.6 (3)
C14—O5—C15	111.4 (3)	N2—C7—C8	118.8 (3)
N2—N1—C1	114.7 (2)	N2—C7—C9	116.2 (3)
N1—N2—C7	114.7 (2)	C8—C7—C9	125.0 (3)
N1—C1—C2	124.7 (3)	Cl3—C9—C7	118.7 (3)
N1—C1—C6	114.3 (3)	Cl3—C9—C10	115.0 (3)
C2—C1—C6	121.0 (3)	C7—C9—C10	126.2 (3)
O1—C2—C1	123.7 (3)	O2—C10—O3	124.1 (3)
O1—C2—C3	119.2 (3)	O2—C10—C9	124.0 (3)
C1—C2—C3	117.0 (3)	O3—C10—C9	111.8 (3)
Cl1—C3—C2	118.4 (3)	O3—C11—C12	110.1 (3)
Cl1—C3—C4	119.4 (3)	O4—C12—C11	107.9 (3)
C2—C3—C4	122.2 (3)	O4—C13—C14	106.2 (3)
C3—C4—C5	119.2 (3)	O5—C14—C13	108.7 (3)
Cl2—C5—C4	119.1 (3)	O5—C15—C16	109.2 (3)
Cl2—C5—C6	120.0 (3)	Cl4—C16—C15	111.1 (3)

Data were corrected for Lorentz and polarization effects, and for absorption using *DIFABS* (Walker & Stuart, 1983). H atoms were located by difference Fourier synthesis and refined isotropically.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN PROCESS* (Fair, 1990). Program(s) used to solve structure: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Program(s) used to refine structure: *MolEN LSFM*. Software used to prepare material for publication: *PLATON* (Spek, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, and torsion angles have been deposited with the IUCr (Reference: SE1046). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Azabicyclo[4.4.0]decane Borine Adduct

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Abstract

The title compound, (1-azoniabicyclo[4.4.0]decane)trihydroborate, $C_9H_{20}BN$, adopts a *trans*-decalin-type con-

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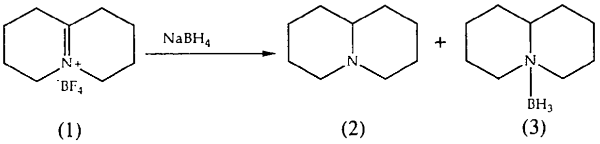
formation and lies on a mirror plane at $y = \frac{1}{4}$. Atoms C(6), H(6), N and B lie on this plane. The C(6)—N—B bond angle of 116.5° is indicative of distortion of the borine substituent to relieve the 1,3-diaxial interactions of the H atoms at positions C(5) and C(5^{i}). The B—N bond length, 1.638 (2) Å, is in agreement with those of other similar tertiary amine borine adducts [Wang & Paul (1977). *Acta Cryst.* B**33**, 2977–2979; Hanic & Šubrtová (1969). *Acta Cryst.* B**25**, 405–408].

Comment

Iminium ions have been shown to be reduced to amines upon treatment with reducing agents such as $NaBH_4$ and $LiAlH_4$ (Cook, 1988). However, reaction of iminium ion (1) using $NaBH_4$ under standard conditions afforded not only the expected azadecalin (2), but also (3) as another major product. The title compound (3), which is slightly air sensitive, was separated from (2) by distillation. Rod-like crystals were obtained by recrystallization from methanol.



(1) (2) (3)

^{13}C NMR spectroscopy indicated that (3) was a symmetrical structure similar to (2). Furthermore, (3) gave rise to a 70 eV electron-impact mass spectrometry fragmentation pattern which was essentially identical to that of (2). It is worthwhile noting that reduction of (1) using $LiAlH_4$ in THF yielded a white solid as the sole product which showed an identical ^{13}C NMR spectrum to that of (3); the product this was not analysed further, but was presumed to be the corresponding AlH_3 adduct.

The crystals of (3) proved to be unstable under exposure to X-ray radiation at room temperature; thus, data were collected at 180 K using an Oxford Cryostream cooling device and showed no significant decomposition. Structure solution was by direct methods and refinement of the data resulted in the structure shown as the *ORTEP* (Johnson, 1965) diagram in Fig. 1.

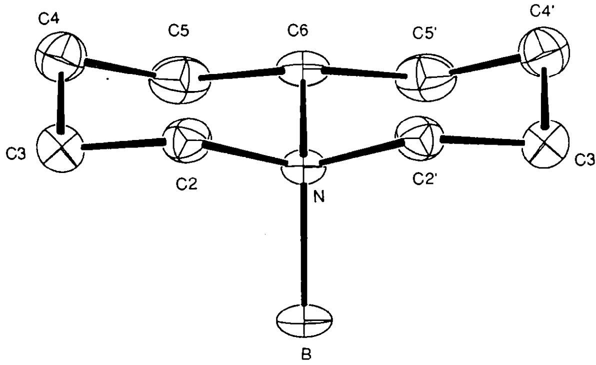


Fig. 1. *ORTEP* (Johnson, 1965) diagram of the title compound.

Experimental

Crystal data

$C_9H_{20}BN$
$M_r = 153.08$
Orthorhombic
Pnma
$a = 13.554$ (2) Å
$b = 11.766$ (2) Å
$c = 6.1480$ (8) Å
$V = 980.4$ (2) Å^3
$Z = 4$
$D_x = 1.037$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13$–18°
$\mu = 0.06$ mm^{-1}
$T = 180$ K
Block
0.5 × 0.4 × 0.4 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1663 measured reflections
903 independent reflections
806 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.016$
$\theta_{\max} = 25°$
$h = -2 \rightarrow 16$
$k = -13 \rightarrow 2$
$l = -1 \rightarrow 7$
3 standard reflections
frequency: 150 min
intensity variation: <2%

Refinement

Refinement on F^2
$R(F) = 0.036$
$wR(F^2) = 0.100$
$S = 1.04$
903 reflections
98 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.25P]$
where $P = [\max(F_o^2,0) + 2F_c^2]/3$

$(\Delta/\sigma)_{\max} = -0.037$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.13$ e Å^{-3}
Extinction correction: empirical
Extinction coefficient: 0.025 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
B	0.1355 (1)	1/4	0.3306 (3)	0.030 (1)
N	0.1668 (1)	1/4	0.5880 (2)	0.023 (1)
C(2)	0.2297 (1)	0.3525 (1)	0.6347 (2)	0.029 (1)
C(3)	0.1737 (1)	0.4637 (1)	0.6228 (2)	0.036 (1)
C(4)	0.0853 (1)	0.4629 (1)	0.7755 (2)	0.038 (1)
C(5)	0.0224 (1)	0.3582 (1)	0.7361 (2)	0.035 (1)
C(6)	0.0825 (1)	1/4	0.7524 (2)	0.026 (1)

Table 2. *Selected geometric parameters* (Å, °)

B—N	1.638 (2)	N—C(2)	1.5048 (12)
N—C(6)	1.525 (2)	C(2)—C(3)	1.514 (2)
C(3)—C(4)	1.523 (2)	C(4)—C(5)	1.517 (2)
C(5)—C(6)	1.5151 (13)		
C(2)—N—C(2^{i})	106.56 (11)	C(2)—N—C(6)	107.33 (7)
C(2)—N—B	109.33 (7)	C(2^{i})—N—B	109.33 (7)
C(6)—N—B	116.50 (11)	N—C(2)—C(3)	113.53 (9)
C(2)—C(3)—C(4)	111.08 (9)	C(3)—C(4)—C(5)	110.41 (9)
C(4)—C(5)—C(6)	111.70 (9)	C(5^{i})—C(6)—C(5)	114.37 (12)
C(5)—C(6)—N	111.03 (7)		

C(2^i)—N—C(2)—C(3)	−172.84 (6)	C(6)—N—C(2)—C(3)	−58.11 (11)
B—N—C(2)—C(3)	69.11 (12)	N—C(2)—C(3)—C(4)	56.12 (13)
C(2)—C(3)—C(4)—C(5)	−52.07 (13)	C(3)—C(4)—C(5)—C(6)	54.44 (13)
C(4)—C(5)—C(6)—C(5^i)	174.46 (8)	C(4)—C(5)—C(6)—N	−58.93 (13)
C(2)—N—C(6)—C(5^i)	−172.89 (9)	C(2^i)—N—C(6)—C(5^i)	−58.68 (12)
B—N—C(6)—C(5^i)	64.21 (9)	C(2)—N—C(6)—C(5)	58.68 (12)
C(2^i)—N—C(6)—C(5)	172.89 (9)	B—N—C(6)—C(5)	−64.21 (9)

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

An empirical extinction parameter to cover both primary and secondary extinction was refined (Sheldrick, 1993; Larson, 1970). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1111). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). **C50**, 2076–2077

6-Methyl-2-oxabicyclo[4.3.0]non-4-en-3,7-dione, $C_9H_{10}O_3$

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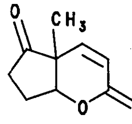
Abstract

The pyrone ring adopts a C(1)-sofa conformation and the *cis*-fused five-membered ring is in a C(9)-envelope conformation. The Me group is in a pseudo-axial position. The molecular packing involves C—H···O intermolecular contacts.

Comment

The title compound, (I), was synthesized as a model compound for natural products isolated from *Otoba parvifolia* (Boscaini, 1991). In order to establish unambiguously its stereochemistry, a crystal structure determination was undertaken.



(I)

The pyrone ring is in a sofa conformation with C(1) 0.470 (2) Å out of the plane formed by atoms O(1) and C(3)–C(6); the carbonyl atom O(2) lies 0.101 (1) Å out of the plane. As in other pyrones (Selladurai & Subramanian, 1992, and references therein) the angle O(2)—C(3)—O(1) is smaller than the angle O(2)—C(3)—C(4). The five-membered ring is in an envelope conformation with C(1), C(6), C(7) and C(8) coplanar to within experimental error and C(9) lying 0.514 (2) Å out of the plane. The dihedral angle between the best least-squares planes through the rings is 66.19 (7)°. The molecular packing involves intermolecular C—H···O interactions as listed in Table 3.

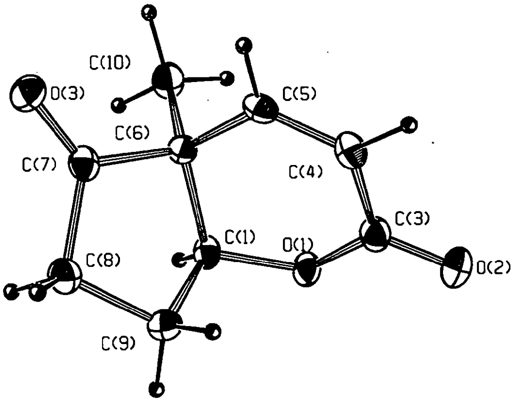


Fig. 1. The molecular structure of $C_9H_{10}O_3$ showing the atom labelling; 50% probability displacement ellipsoids are shown for non-H atoms.

Experimental

Crystal data

$C_9H_{10}O_3$
$M_r = 166.18$
Orthorhombic
Pbca
$a = 8.151$ (1) Å
$b = 10.648$ (1) Å
$c = 18.330$ (2) Å
$V = 1590.9$ (5) Å^3

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–20°
$\mu = 0.97$ mm^{-1}
$T = 83$ K
Irregular