Structural studies of organoboron compounds. XLII.[1] 4,6-Bis(1-cyano-1-methylethyl)-2-mesityl-1,3-dioxa-4,6-diaza-2-boracyclohexane

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The condensation of *N,N'*-bis(1-cyano-1-methylethyl)-*N,N'*-dihydroxymethanediamine and mesitylboronic acid gives 4,6-bis(1-cyano-1-methylethyl)-2-mesityl-1,3-dioxa-4,6-diaza-2-boracyclohexane in good yield. Crystals of the latter compound are orthorhombic, a = 15.825(1), b = 17.958(1), c = 14.014(1) Å, Z = 8, space group *Pbca*. The structure was solved by direct methods and was refined by full-matrix least-squares procedures to $R = 0.037$ and $R_w = 0.051$ for 2844 reflections with $I \geq 3\sigma(I)$. The molecule has a six-membered cycloboronate structure featuring the first structurally characterized BONCNO ring. The ring was found to have a *C*-envelope conformation in the solid state. Bond lengths include: O—B(sp^2) = 1.356(2) and 1.370(2), and C(aryl)—B = 1.560(2) Å.

Key words: organoboron compound, boron compound, crystal structure.

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La condensation de la *N,N'*-bis(1-cyano-1-méthyléthyl)-*N,N'*-dihydroxyméthanediamine et de l'acide mésitylboronique fournit le 4,6-bis(1-cyano-1-méthyléthyl)-2-mésityl-1,3-dioxa-4,6-diaza-2-boracyclohexane avec un bon rendement. Les cristaux de ce dernier composé sont orthorhombiques, groupe d'espace *Pbca*, avec a = 15,825(1), b = 17,958(1) et c = 14,014(1) Å et Z = 8. On a résolu la structure par des méthodes directes et on l'a affinée par là méthode des moindres carrés (matrice entière) jusqu'à des valeurs de R = 0,037 et R_w = 0,051 pour 2844 réflexions avec $I \geq 3\sigma(I)$. La molécule possède un cycloboronate à six chaînons comprenant le premier cycle BONCNO à être caractérisé. On a trouvé que, à l'état solide, le cycle existe dans une conformation *C*-enveloppe. Les longueurs des liaisons observées comprennent : O—B(sp^2) = 1,356(2) et 1,370(2) et C(aryl)—B = 1,560(2) Å.

Mot clés : composés organoboriques, composés boriques, structure cristalline.

[Traduit par la revue]

Introduction

N,N'-Dihydroxy-*N,N'*-dialkylaminals **1** react with arylboronic acids to give bicyclic pyroboronates **3** with transannular N—B coordination (1, 2). The bicyclo[3.3.0]octane skeletal structure of these compounds has been established by an X-ray crystallographic analysis of the *N*-methyl/*B*-phenyl-substituted derivative **3***a* (3).

Reaction of **1** and arylboronic acid in various molar ratios (including 1:1) leads to type **3** products, rather than the six-membered "diol boronate" **4**. Cyclic esters derived from 1,3-alkanediols similar to **4** are well-known in the literature (4). The structures of several arylboronates of 1,3-diols have been elucidated by X-ray crystallographic analyses (5–8). To achieve the formation of a type **4** boronate with the "diol" compound **1** we reduced the basicity of the aminal nitrogen atom by incorporation of an α-cyano group into the *N*-alkyl substituent. The decrease in basicity should weaken the dative N—B interaction which is possible in the hypothetical intermediate **2** leading to the bicyclic 2:1 condensate **3**. In addition, steric hindrance of the boronic acid by *ortho* substitution in the aryl residue could impede an extension of the coordination at boron from trigonal planar sp^2 to tetrahedral sp^3. Indeed, the condensation of a *N*-(α-cyanoalkyl)aminal **1** (R = $C(CH_3)_2CN$) and mesitylboronic acid (Ar = 2,4,6-$(CH_3)_3C_6H_2$) gave a crystalline 1:1 condensation product, according to elemental analyses and spectroscopic data. Although mass spectral data were consistent with a monomeric condensate, it was not possible to decide unambiguously between the expected six-membered cycloboronate structure **4** and the less probable *N*-oxide form **5** or its rearrangement product **6**, its dimer **7**, and possible oligomers like **8** (n = 2, 3, or higher). Thus an X-ray crystallographic analysis has been undertaken to determine the molecular structure of the condensate and to provide data on the conformation of the new heterocyclane ring, at least in the solid state.

Experimental

4,6-Bis(1-cyano-1-methylethyl)-2-mesityl-1,3-dioxa-4,6-diaza-2-boracyclohexane, **4**a *(R = $C(CH_3)_2CN$, Ar = 2,4,6-$(CH_3)_3C_6H_2$)*

N,N'-Bis(1-cyano-2-methylethyl)-*N,N'*-dihydroxymethanediamine **1** (R = $C(CH_3)_2CN$) (9) (0.64 g, 3 mmol) and mesitylboronic acid (0.49 g, 3 mmol) are dissolved in 20 mL of benzene and refluxed for 1 h with continuous removal of water. After partial evaporation *in vacuo* colorless crystals formed upon cooling. Yield: 0.83 g (81%). Melting point (decomp.) 158–160°C (from benzene). *Anal.* calcd. for $C_{18}H_{25}BN_4O_2$: C 63.54, H 7.41, B 3.18, N 16.47; found: C 63.55, H 7.59, B 3.20, N 16.47. Infrared (KBr): 2230 (C≡N), 1610 cm^{-1} (aryl-C═C). ^1H nmr (90 MHz, $CDCl_3$/TMS): δ(ppm) = 1.67 (s, 2 $C(CH_3)_2$), 2.27 (s, p-CH_3), 2.43 (s, 2 o-CH_3), 4.35 (s, N—CH_2—N), 6.78 (s, 2 aromatic H). Mass spectrum (70 eV, 60°C): m/z = 340 (56%, M^+), 298 (14%), 272 (8%, M − $C(CH_3)_2CN$), 241 (14%), 227 (36%, M − H_2C═N(OH)$C(CH_3)_2CN$), 161 (15%), 147 (41%, $(CH_3)_3C_6H_2$), 133 (26%), 113 (76%, H_2C═N(OH)$C(CH_3)_2CN$), 97 (10%), 85 (26%), 68 (100%, $C(CH_3)_2CN$), 56 (8%), 45 (64%, CH_2NOH). Crystals suitable for X-ray analysis were obtained by recrystallization from dry benzene.

X-ray crystallographic analysis of **4**a

A crystal *ca.* 0.25 × 0.25 × 0.35 mm in size was mounted on a glass

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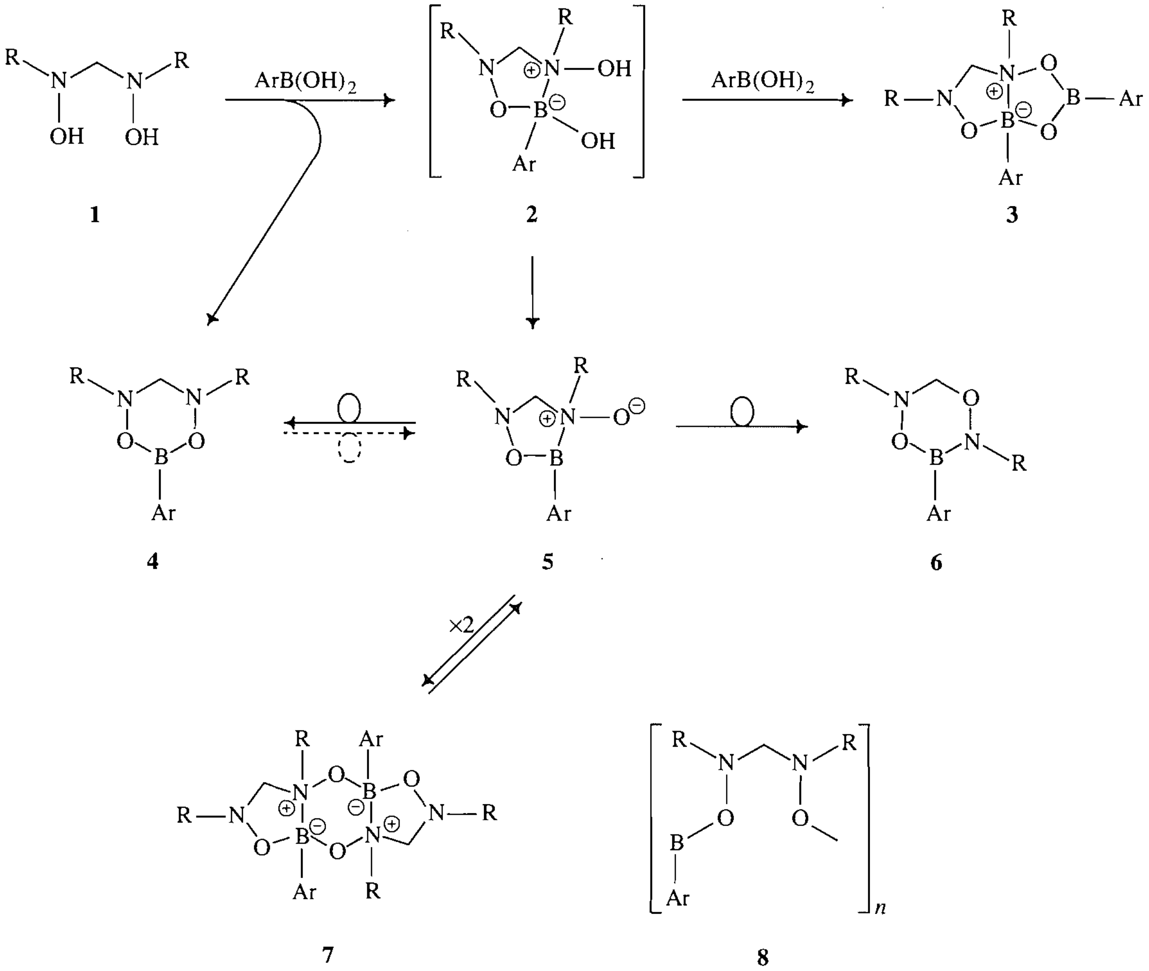
[1] Previous paper in this series: ref. 25.



fiber. Unit-cell parameters were refined by least squares on setting angles for 25 reflections ($2\theta = 92.4$–$118.1°$) measured on a diffractometer with Cu-K_α radiation ($\lambda = 1.54178$ Å). Crystal data at 21°C are as follows

$C_{18}H_{25}BN_4O_2$ f.w. = 340.23

Orthorhombic, $a = 15.825(1)$, $b = 17.958(1)$, $c = 14.014(1)$ Å, $V = 3982.6(5)$ Å^3, $Z = 8$, $\rho_c = 1.135$ Mg m^{-3}, $F(000) = 1456$, μ(Cu-K_α) = 5.66 cm^{-1}. Absent reflections: $0kl$, k odd, $h0l$, l odd, and $hk0$, h odd, uniquely indicate the space group *Pbca* (No. 61).

Intensities were measured with graphite-monochromated Cu-K_α radiation on a Rigaku AFC6S diffractometer. An ω–2θ scan at 32° min^{-1} over a range of $(1.10 + 0.30 \tan \theta)°$ in ω (with up to eight rescans, background/scan time ratio = 0.5) was employed. Data were measured to $2\theta = 155°$. The intensities of three check reflections, measured every 150 reflections throughout the data collection, showed only small random variations. After data reduction,[2] an empirical absorption correction, based on azimuthal scans for three reflections, was applied. Transmission factors range from 0.909 to 1.00. Of the 4294 independent reflections measured, 2844(66.2%) had intensities greater than or equal to $3\sigma(F^2)$ above background where $\sigma^2(F^2) = [S^2(C + 4B) + (0.035F^2)^2]/\mathrm{Lp}^2$ with S = scan speed, C = scan count, B = total background count, and Lp = Lorentz-polarization factor.

The structure was solved by direct methods, the coordinates of all non-hydrogen atoms being determined from an E-map. The non-hydrogen atoms were refined with anisotropic thermal parameters and the hydrogen atoms were refined with isotropic thermal parameters. A correction for secondary extinction was applied, the final value of the extinction coefficient being 8.35×10^{-6}. Scattering factors for all atoms and anomalous dispersion corrections for the non-hydrogen atoms were taken from ref. 10. The weighting scheme $w = 4F_o^2/\sigma^2(F_o^2)$ gave uniform average values of $w(|F_o| - |F_c|)^2$ over ranges of both $|F_o|$ and $\sin \theta/\lambda$ and was employed in the final stages of full-matrix least-squares refinement of 327 variables. Reflections with $I < 3\sigma(I)$ were not included in the refinement. Convergence was reached at $R = 0.037$ and $R_w = 0.051$ for 2844 reflections with $I \geq 3\sigma(I)$. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$, $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $R_w = (\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2)^{1/2}$.

On the final cycle of refinement the maximum parameter shift corresponded to 0.15σ. The mean error in an observation of unit weight was 2.09. The final difference map showed maximum fluctuations of ±0.15 e Å^{-3}. The final positional and (equivalent) isotropic thermal parameters for the non-hydrogen atoms appear in Table 1. Bond lengths and angles are given in Tables 2 and 3 and intra-annular torsion angles in Table 4. Hydrogen atom parameters, anisotropic thermal parameters, torsion angles, intermolecular contacts, least-squares planes, bond lengths and angles involving hydrogen, and structure factors have been deposited.[3]

Results and discussion

The X-ray analysis shows that compound **4***a* has the expected six-membered cycloboronate structure of the type **4** (Fig. 1), and represents (to the best of our knowledge) the first heterocyc-

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[2] TEXSAN/TEXRAY structure analysis package which includes versions of the following: MITHRIL, integrated direct methods, by C.J. Gilmore; DIRDIF, direct methods for difference structures, by P.T. Beurskens; ORFLS, full-matrix least-squares, and ORFFE, function and errors, by W.R. Busing, K.O. Martin, and H.A. Levy; ORTEP II, illustrations, by C.K. Johnson.

[3] Supplementary material mentioned in the text may be purchased from the Depository of Unpublished Data, CISTI, National Research Council of Canada, Ottawa, Ont., Canada K1A 0S2.

TABLE 1. Final atomic coordinates (fractional) and B (eq)

Atom	x	y	z	B (eq)
O(1)	0.27031(7)	0.54983(5)	0.56908(7)	3.71(4)
O(2)	0.21797(6)	0.44590(5)	0.47966(8)	3.85(5)
N(1)	0.32810(8)	0.50019(7)	0.6197(1)	3.45(5)
N(2)	0.27315(8)	0.39828(6)	0.5366(1)	3.46(5)
N(3)	0.4556(1)	0.6237(1)	0.5003(1)	5.59(8)
N(4)	0.3550(1)	0.3639(1)	0.3138(1)	6.2(1)
C(1)	0.3503(1)	0.44081(8)	0.5531(1)	3.51(6)
C(2)	0.3985(1)	0.54942(8)	0.6483(1)	3.66(6)
C(3)	0.2847(1)	0.33028(8)	0.4779(1)	3.68(6)
C(4)	0.4304(1)	0.5917(1)	0.5644(1)	3.99(7)
C(5)	0.4708(1)	0.5037(1)	0.6906(2)	4.83(9)
C(6)	0.3643(1)	0.6039(1)	0.7222(1)	5.0(1)
C(7)	0.3241(1)	0.3489(1)	0.3849(1)	4.27(7)
C(8)	0.3418(1)	0.2764(1)	0.5324(2)	4.75(9)
C(9)	0.1985(1)	0.2946(1)	0.4605(2)	4.93(9)
C(10)	0.1613(1)	0.57343(7)	0.4423(1)	3.39(6)
C(11)	0.0811(1)	0.59247(8)	0.4759(1)	3.81(6)
C(12)	0.0293(1)	0.6377(1)	0.4201(2)	4.68(8)
C(13)	0.0558(1)	0.66651(9)	0.3341(1)	4.76(8)
C(14)	0.1370(1)	0.6500(1)	0.3039(1)	4.46(8)
C(15)	0.1900(1)	0.60324(8)	0.3553(1)	3.78(6)
C(16)	0.0519(1)	0.5669(1)	0.5725(2)	5.5(1)
C(17)	−0.0012(2)	0.7144(2)	0.2735(3)	7.5(2)
C(18)	0.2768(1)	0.5853(2)	0.3178(2)	5.2(1)
B	0.2195(1)	0.52032(9)	0.5010(1)	3.34(7)

TABLE 2. Bond distances (Å) with estimated standard deviations in parentheses

Atom	Atom	Distance	Atom	Atom	Distance
O(1)	B	1.356(2)	C(3)	C(7)	1.484(2)
O(1)	N(1)	1.461(2)	C(3)	C(9)	1.527(2)
O(2)	B	1.370(2)	C(3)	C(8)	1.529(2)
O(2)	N(2)	1.460(2)	C(10)	C(11)	1.396(2)
N(1)	C(1)	1.460(2)	C(10)	C(15)	1.406(2)
N(1)	C(2)	1.478(2)	C(10)	B	1.560(2)
N(2)	C(1)	1.458(2)	C(11)	C(12)	1.394(2)
N(2)	C(3)	1.484(2)	C(11)	C(16)	1.502(3)
N(3)	C(4)	1.138(2)	C(12)	C(13)	1.378(3)
N(4)	C(7)	1.142(2)	C(13)	C(14)	1.385(3)
C(2)	C(4)	1.488(2)	C(13)	C(17)	1.507(3)
C(2)	C(6)	1.525(2)	C(14)	C(15)	1.388(2)
C(2)	C(5)	1.527(3)	C(15)	C(18)	1.506(3)

lane with a BONCNO ring skeleton to be structurally characterized. The structure is comparable to that of a simple 1,3-diol boronate and possesses a "semi-planar" (C(1)—sofa) conformation for the six-membered heterocyclic ring, similar to those postulated for and established by X-ray analyses of several crystalline phenylboronates of 1,3-diols **9** (5–7) and **10** (8) as well as related ethylboronates (11, 12) and a simple boric acid ester of a 1,3-diol (13).

The essential planarity (to within 0.026 Å) of the N—O—B—O—N moiety in the boronate portion of **4***a* results, as does that of corresponding ring fragments in compounds **9**, **10**, and other known six-membered cyclic esters of ethylboronic or boric aid, from the partial double bond character of the O—B bonds caused by O—B $pp(\pi)$ back donation. This is supported by the relatively short O—B distances in **4***a* (1.356(2) and 1.370(2) Å). Corresponding O—B distances in the related compounds mentioned above range from 1.354 to 1.398 (average 1.373 Å) (5–8,11–13). Correlation of these O—B bond lengths with O—B π-bond order (14) yields a value of around 50%. Thus, a mean bond order of 1.5 is characteristic of the O—B bonds in the cycloboronate **4***a*.

In contrast to phenylboronates like **9** and **10**, in which the phenyl rings lie approximately coplanar with the C—O—B—O—C moieties of the boronate rings (suggesting at least some π-interaction between the aromatic system and the unoccupied boron p_z-orbital), the mesityl group of **4***a* is oriented perpendicular (dihedral angle between normals to the aromatic ring and boron coordination mean planes = 87.2°) to the boronate ring. This is apparently due to the steric hindrance of the ortho methyl substituents. The B—C(aryl) distance in **4***a* (1.560(2) Å) is nearly equal to those in **9***b* (6) (1.564 Å) and **9***c* (7) (1.561 Å), marginally longer than that in **10** (8) (1.554 Å), and distinctly longer than the reported (5) value for **9***a* (1.506 Å). The similarity of the above-mentioned distances suggests that for the B(sp^2)-C(aryl) bonds, in contrast to the O—B(sp^2) bonds, $p\pi$–$p\pi$ interactions appear to play a relatively minor role.

Compared to the bicyclic pyroboronate **3***a*, the monocyclic boronate **4***a* requires a different configuration at one of the aminal nitrogen atoms. Whereas **3***a* has the same configuration at both amine centres (*R*,*R* or *S*,*S*),[4] compound **4***a* has opposite configurations (*R*,*S*) at the two amine centres. Both of the bulky *N*-cyanoalkyl substituents are found in equatorial positions, resembling the similar influence of *tert*-butyl substituents on the geometry of cyclohexane ring systems (15). Considering the bond lengths and the conformational arrangement of both cyanoalkylamine groups in **4***a*, an anomeric effect (16) seems to contribute to the stabilization of the molecular geometry: the antiperiplanar orientation of both non-bonding electron pairs with respect to the adjoining geminal cyano groups allows the overlap of the nitrogen *n*-orbital with the antibonding σ^* orbital of the C—C(cyano) bond. This is supported by relatively short C—N(amine) and long C—C(cyano) bonds within the cyanoalkylamine moiety of **4***a*. The mean C—N distance of 1.481 Å is somewhat shorter than comparable C—N bonds in *N*-*t*-butyl hydroxylamine derivatives: 1.497 Å (17), 1.496 Å (18). Correspondingly, the mean C—C(cyano) distance of 1.486 Å in **4***a* is longer than the comparable C—C(cyano) distance of 1.460 Å in Me_3C—CN (19) and the average value of 1.469 Å reported for C(sp^3)—C(cyano) bonds (20). An even longer C—C(cyano) bond (1.51 Å) has been reported for the *N*-cyanoalkylpiperidine **11** (21) which has the same *N*-substituent as **4***a* and an analogous conformation of the cyanoalkylamine moiety. Stereoelectronic effects on the geometry of the α-cyanoalkylamine group, similar to those deduced from bond lengths and conformations of both the hydroxylamine derivative **4***a* and the amine derivative **11** (21), have been postulated for α-cyanohydroxylamine derivatives of the general formula **12** (with a preferred axial orientation of the cyano group) on the basis of ^1H nmr data (22, 23).

The mean C≡N bond length of 1.140 Å in **4***a* is in good agreement with the average value 1.137 Å reported for cyano groups (20), and is slightly shorter than the values of 1.15 Å in **11** (21) and 1.158 Å in Me_3C—CN (19). The mean C—N and N—O distances in the aminal group (O—N— CH_2—N—O) of 1.460 and 1.461 Å, respectively, are equal within experimental error to the corresponding values of 1.461 and 1.458 Å reported

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[4] Equal numbers of enantiomers are found in the crystal (3); formula **3***a* depicts the (1*R*,5*S*,7*R*)-enantiomer.

TABLE 3. Bond angles (deg) with estimated standard devitions in parentheses

Atom	Atom	Atom	Angle	Atom	Atom	Atom	Angle
B	O(1)	N(1)	118.3(1)	C(9)	C(3)	C(8)	110.1(2)
B	O(2)	N(2)	116.2(1)	N(3)	C(4)	C(2)	179.3(2)
C(1)	N(1)	O(1)	106.6(1)	N(4)	C(7)	C(3)	179.1(2)
C(1)	N(1)	C(2)	115.4(1)	C(11)	C(10)	C(15)	119.5(1)
O(1)	N(1)	C(2)	103.8(1)	C(11)	C(10)	B	120.5(1)
C(1)	N(2)	O(2)	106.3(1)	C(15)	C(10)	B	120.0(1)
C(1)	N(2)	C(3)	114.6(1)	C(12)	C(11)	C(10)	119.2(2)
O(2)	N(2)	C(3)	104.6(1)	C(12)	C(11)	C(16)	120.2(2)
N(2)	C(1)	N(1)	106.4(1)	C(10)	C(11)	C(16)	120.6(2)
N(1)	C(2)	C(4)	110.3(1)	C(13)	C(12)	C(11)	122.1(2)
N(1)	C(2)	C(6)	107.5(1)	C(12)	C(13)	C(14)	118.0(2)
N(1)	C(2)	C(5)	110.4(1)	C(12)	C(13)	C(17)	121.7(2)
C(4)	C(2)	C(6)	109.3(1)	C(14)	C(13)	C(17)	120.4(2)
C(4)	C(2)	C(5)	109.1(1)	C(13)	C(14)	C(15)	122.1(2)
C(6)	C(2)	C(5)	110.3(2)	C(14)	C(15)	C(10)	119.1(2)
N(2)	C(3)	C(7)	110.7(1)	C(14)	C(15)	C(18)	119.9(2)
N(2)	C(3)	C(9)	108.9(1)	C(10)	C(15)	C(18)	121.0(2)
N(2)	C(3)	C(8)	108.5(1)	O(1)	B	O(2)	123.1(1)
C(7)	C(3)	C(9)	109.2(2)	O(1)	B	C(10)	118.8(1)
C(7)	C(3)	C(8)	109.4(1)	O(2)	B	C(10)	118.1(1)

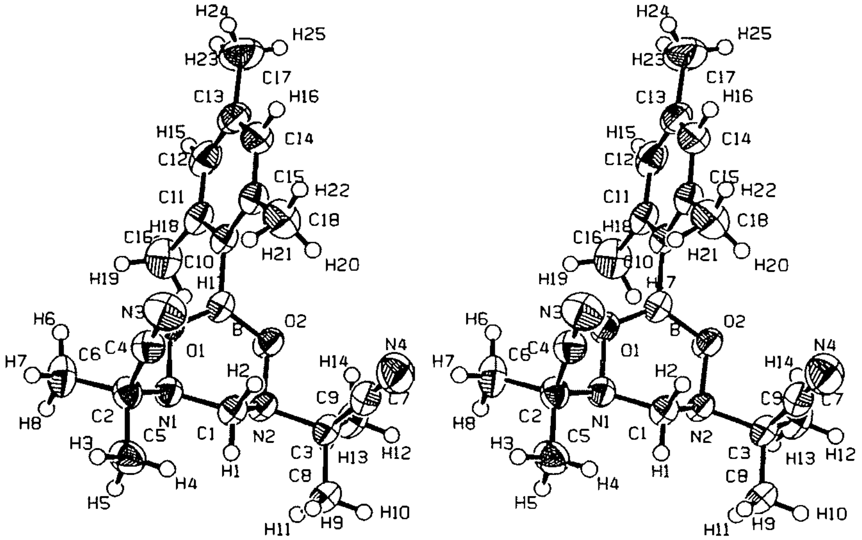
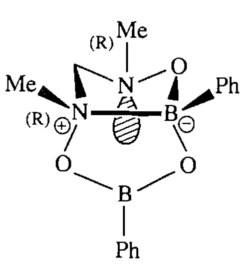
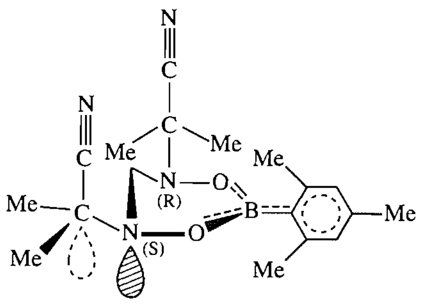


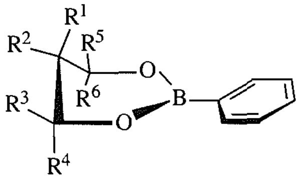
FIG. 1. Stereoview of the 4,6-bis(1-cyano-1-methylethyl)-2-mesityl-1,3-dioxa-4,6-diaza-2-boracyclohexane molecule; 50% probability thermal ellipsoids are shown for the non-hydrogen atoms.



3*a*



4*a*



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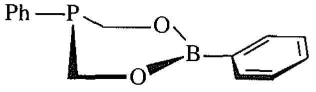

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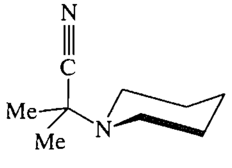
9a: $R^1 = OH, R^2 = R^3 = R^5 = H, R^4/R^6 = CH_2OCH(NHC_6H_4—4\text{-}Br)$
9b: $R^1 = 4\text{-}O_2N—C_6H_4CH{=}N(O)—, R^2 = Me, R^{3-6} = H$
9c: $R^1 = NO_2, R^2 = Me, R^{3-6} = H$



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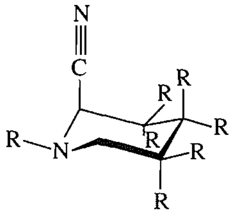

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TABLE 4. Intra-annular torsion angles

Atoms	Torsion angle
B —O(1)—N(1)—C(1)	30.3(2)
O(1)—N(1)—C(1)—N(2)	−66.8(1)
N(1)—C(1)—N(2)—O(2)	70.9(1)
C(1)—N(2)—O(2)—B	−37.4(2)
N(2)—O(2)—B —O(1)	1.7(2)
O(2)—B —O(1)—N(1)	2.1(2)

for the simple aminal **1** (R = CH_3) (24). The crystal structure of **4**_a_ consists of molecules separated by normal van deer Waals distances, the shortest intermolecular distance between non-hydrogen atoms being N(3)···C(1) = 3.367(2) Å.

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