

Structure of 2,10-Bis(diisopropylamino)-1,5-dihydro-1,5-epiborano-2H-benzo[c]borepine

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Abstract. $C_{22}H_{36}B_2N_2$, $M_r = 350.2$, monoclinic, $C2/c$, $a = 10.701$ (9), $b = 17.789$ (9), $c = 24.143$ (12) Å, $\beta = 90.84$ (3)°, $V = 4595.4$ Å³, $Z = 8$, $D_x = 1.01$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.05$ mm⁻¹, $F(000) = 1536$, $T = 298$ K, $R = 0.078$ for 1450 observed reflections. The compound was obtained by borylation of naphthalene in such a way that one BN[CH(CH₃)₂]₂ unit has been added across one of the benzo rings, while the other has been inserted into the ring skeleton.

Experimental. Single crystals were obtained (Meller, Maringele, Elter, Bromm, Noltemeyer & Sheldrick, 1987) from the highly viscous pure substance by tempering at 303 K for 1 year. A specimen of size 0.3 × 0.3 × 0.6 mm was investigated on a Stoe AED-2 four-circle diffractometer (revision 6.2), with graphite-monochromated Mo $K\alpha$ radiation and profile-fitted 2θ - ω scans involving variable scan width and speed (Clegg, 1981). Lattice parameters were determined from a least-squares fit of 42 reflections in the range $20 < 2\theta < 25^\circ$. 9076 reflections were measured in the range 2θ to 45° and $-11 \leq h \leq 11$, $-19 \leq k \leq 19$, $-25 \leq l \leq 25$; 2997 were unique ($R_{\text{int}} = 0.04$). The structure was solved by direct methods (Sheldrick, 1985). 1450 reflections with $F > 3\sigma(F)$ were used (Sheldrick, 1976), with 235 refined parameters, $S = 1.4$, $R = 0.078$, $wR = 0.055$. The weighting scheme was $w^{-1} = \sigma^2(|F_o|) + 0.0004|F_o|^2$, residual electron density = ± 0.2 e Å⁻³ and $(\Delta/\sigma)_{\text{max}} = 0.001$. All non-H atoms were refined anisotropically. H atoms were included using a riding model [C—H 0.96 Å, $U(\text{H}) = 0.08$ Å²]. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Atomic coordinates are given in Table 1,† selected bond lengths and

angles in Table 2. The molecule, which corresponds to formula diagram (B) is shown in Fig. 1.

Related literature. In the course of investigations on carbene analogous reactions of the dehalogenation products of dichloro(diisopropylamino)borane with

Table 1. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å² × 10³)

	x	y	z	U_{eq}^*
C(1)	92 (4)	3551 (3)	3530 (2)	45 (2)
B(2)	899 (6)	4013 (4)	3983 (3)	50 (3)
C(3)	994 (5)	3593 (4)	4551 (3)	65 (3)
C(4)	514 (5)	2914 (4)	4622 (2)	68 (3)
C(5)	-208 (5)	2487 (3)	4177 (2)	57 (2)
C(5a)	-1430 (5)	2889 (3)	4068 (2)	49 (2)
C(6)	-2608 (7)	2712 (4)	4251 (2)	67 (3)
C(7)	-3598 (6)	3154 (4)	4086 (3)	80 (3)
C(8)	-3421 (6)	3756 (4)	3744 (3)	77 (3)
C(9)	-2258 (6)	3942 (3)	3550 (2)	65 (3)
C(9a)	-1262 (5)	3498 (3)	3713 (2)	46 (2)
B(10)	576 (6)	2714 (4)	3638 (3)	46 (3)
N(10)	1478 (4)	2316 (3)	3362 (2)	49 (2)
C(11)	816 (6)	988 (3)	3369 (3)	87 (3)
C(12)	1864 (5)	1539 (3)	3490 (3)	62 (3)
C(13)	2362 (6)	1446 (3)	4075 (3)	97 (3)
C(14)	3523 (5)	2826 (4)	3089 (3)	103 (3)
C(15)	2183 (5)	2680 (3)	2913 (3)	59 (3)
C(16)	2115 (5)	2256 (3)	2379 (3)	82 (3)
N(2)	1461 (4)	4702 (3)	3870 (2)	60 (2)
C(21)	684 (6)	5860 (4)	3401 (3)	129 (4)
C(22)	1260 (7)	5103 (4)	3351 (3)	84 (4)
C(23)	2368 (7)	5100 (4)	2976 (3)	113 (4)
C(24)	1683 (6)	5438 (4)	4747 (3)	108 (4)
C(25)	2292 (7)	5107 (4)	4247 (3)	82 (3)
C(26)	3475 (5)	4680 (4)	4387 (3)	115 (4)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. Selected bond lengths (Å) and angles (°)

C(1)—B(2)	1.609 (9)	C(1)—B(10)	1.596 (9)
B(2)—C(3)	1.562 (10)	B(2)—N(2)	1.394 (9)
C(3)—C(4)	1.326 (9)	C(4)—C(5)	1.518 (8)
C(5)—B(10)	1.610 (9)	B(10)—N(10)	1.377 (8)
C(1)—B(2)—C(3)	112.3 (5)	C(1)—B(2)—N(2)	123.0 (6)
C(3)—B(2)—N(2)	124.8 (6)	C(1)—B(10)—C(5)	101.2 (5)
C(1)—B(10)—N(10)	129.0 (5)	C(5)—B(10)—N(10)	129.7 (5)

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† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52998 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

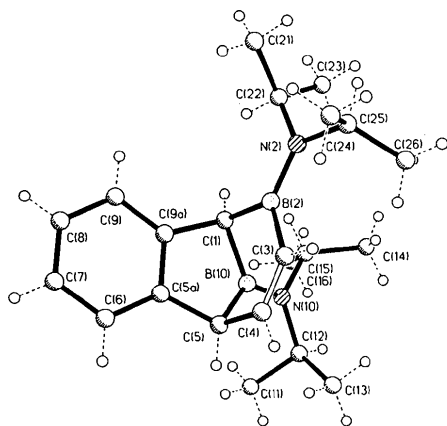
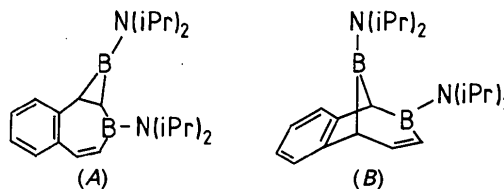


Fig. 1. The molecular structure of $C_{22}H_{36}B_2N_2$.

ano-2*H*-benzo[*c*]borepine (*B*) (Meller, Bromm, Maringgele, Böhler & Elter, 1988).



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aromatic compounds a product, $C_{10}H_8 \cdot 2BN(C_3H_7)_2$, was obtained with naphthalene. It was not possible to determine by NMR (H, B, C, N) and mass spectrometry whether the structure of this product was that of 1,2-bis(diisopropylamino)-1,1a,2,8b-tetrahydroborireno[2,3-*b*][3]benzoborepine (*A*) or 2,10-bis(diisopropylamino)-1,5-dihydro-1,5-epibor-

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Structures of Solvated Hexaphenyldisiloxanes

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Abstract. $C_{36}H_{30}OSi_2 \cdot 2C_6H_6$ (1); $M_r = 691.03$, trigonal, space group $R\bar{3}$, $a_H = 11.325$ (4), $c_H = 26.738$ (6) Å [$a_R = 11.054$ (2) Å, $\alpha_R = 61.63$ (2)°], $V = 2969.86$ Å³, $Z = 3$, $D_x = 1.159$ g cm⁻³, $\mu = 0.93$ cm⁻¹, $\lambda(Mo K\alpha) = 0.71069$ Å, $F(000) = 1098$, $T = 293$ K, final $R = 0.044$ for 1293 observed reflections with $I > 3\sigma(I)$. $C_{36}H_{30}OSi_2 \cdot 2C_7H_8$ (2); $M_r = 719.08$, trigonal, space group $R\bar{3}$, $a_H = 11.542$ (4), $c_H = 26.735$ (6) Å [$a_R = 11.129$ (2) Å, $\alpha_R = 62.47$ (2)°], $V = 3084.41$ Å³, $Z = 3$, $D_x = 1.161$ g cm⁻³, $\mu = 0.91$ cm⁻¹, $\lambda(Mo K\alpha) = 0.71069$ Å, $F(000) = 1146$, $T = 293$ K, final $R = 0.066$ for 727 observed reflections with $I > 3\sigma(I)$. (1) and (2) are isotypic. The molecule has 3 symmetry with the phenyl groups in staggered

conformation. It consists of a central fragment Si—O—Si along the crystallographic *c* axis being linear by an inversion center. The principal interatomic distances are $d(Si—O) = 1.622$ (1) and $d(Si—C) = 1.899$ (2) Å in (1), and $d(Si—O) = 1.620$ (2) and $d(Si—C) = 1.866$ (6) Å in (2). In (1) the anisotropic thermal displacement parameters for the O atom, however, indicate a deviation from the linearity of the Si—O—Si fragment caused by static or dynamic disorder. This deviation would yield $d(Si—O) = 1.64$ Å and $\beta(Si—O—Si) = 163^\circ$.

Experimental. $[(C_6H_5)_3Si]_2O \cdot 2C_6H_6$ (1) and $[(C_6H_5)_3Si]_2O \cdot 2C_7H_8$ (2): colourless crystals of dimensions $0.1 \times 0.1 \times 0.2$ mm and $0.1 \times 0.1 \times 0.15$ mm. Diffraction maxima with $2\theta \leq 55^\circ$ were collected on a computer-controlled four-circle Syntex $P\bar{I}$ diffrac-

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