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Polyhedral azaborane chemistry, 6-(C_5H_5N)-*arachno*-4-NB_8H_{11}

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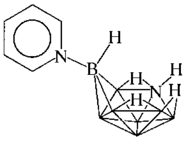
The title compound, 6-pyridyl-4-aza-*arachno*-nonaborane(11), $C_5H_{16}B_8N_2$, has an *arachno* nine-vertex {4-NB_8H_{11}} cluster structure with a pyridine ligand in the *exo*-6-position. The cluster has close geometric similarities to the thiaborane and carbaborane analogues.

Comment

There is current interest in smaller heteroboranes (Davison *et al.*, 2000). Our own interest in the nine-vertex azaboranes stems firstly from the fact that these species are not well investigated in an absolute sense, but clearly have potentially very interesting chemistry (Dörfler *et al.*, 1997), and also in that nine-vertex *arachno* species in general have a propensity to couple upon simple heating to give big-borane 'macropolyhedral' clusters, as in thiaborane, platinaborane and iridaborane chemistry (Bould *et al.*, 1999, and references therein). In main-group heteroborane chemistry in particular, [6-(SMe_2)-*arachno*-4-SB_8H_{10}] gives the interesting species [$S_2B_{17}H_{19}(SMe_2)$] with a hexagonal pyramidal structural feature (Kaur *et al.*, 1996). The nine-vertex thiaboranes [6-*L*-*arachno*-4-SB_8H_{10}] and carbaboranes [6-*L*-*arachno*-4-CB_8H_{12}] are well characterized structurally (Holub, Kennedy & Štíbr, 1994; Holub, Štíbr *et al.*, 1994; Plešek *et al.*, 1994). Several corresponding azaborane analogues [6-*L*-*arachno*-4-NB_8H_{11}], where *L* is a two-electron ligand, are known (Jelínek *et al.*, 1994). However, structural work on these azaboranes is lacking, although the related 'parent' non-ligand species (*arachno*-4-NB_8H_{13}) itself was examined crystallographically some time ago (Baše *et al.*, 1975; Ragatz *et al.*, 1975). Here we report the structure of the pyridine derivative [6-(C_5H_5N)-*arachno*-4-NB_8H_{11}], (I).



(I)

The molecular structure (Fig. 1) closely resembles those of the thiaborane and carbaborane analogues [$SB_8H_{10}(N_4C_6H_6)$] and [$CB_8H_{12}(N_4C_6H_{12})$], where $N_4C_6H_{12}$ is hexamethylene tetramine. Apart from the variation of the nitrogen ligands between NC_5H_5 and $N_4C_6H_6$, which does not affect the cluster interatomic distances significantly, the compounds differ in the isolobal cluster constituents, CH_2, NH and S in the 4-position. There are obvious corresponding differences in the boron-to-heteroatom distances, but the other interatomic distances within the clusters are very similar. For all three classes of compound, there appears to be an asymmetry in the bonding of the bridging H atoms, with the shorter distances being towards B8, although this is not so marked for H78 in the title compound. A similar asymmetry is also noted for the four bridging H atoms in the parent non-ligand analogue NB_8H_{13} (Ragatz *et al.*, 1975).

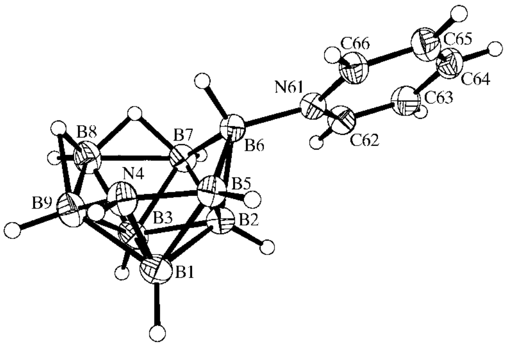


Figure 1
Perspective view of a single molecule of (I) drawn with 40% probability ellipsoids and with H atoms shown as small circles of artificial radii for clarity.

Experimental

The title compound may be prepared by the action of pyridine on [*arachno*-4-NB_8H_{13}], following a previously detailed procedure (Jelínek *et al.*, 1994). The crystallographic sample was obtained from $CHCl_3$/pentane.

Crystal data

$C_5H_{16}B_8N_2$
$M_r = 190.68$
Orthorhombic, $Pna2_1$
$a = 14.36180 (10)$ Å
$b = 10.46000 (10)$ Å
$c = 7.57790 (10)$ Å
$V = 1138.39 (2)$ Å^3
$Z = 4$
$D_x = 1.113$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 26 404 reflections
$\theta = 2.41$–$30.48°$
$\mu = 0.056$ mm^{-1}
$T = 190 (2)$ K
Prism, yellow
0.24 × 0.18 × 0.06 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Area-detector φ and ω scans
Absorption correction: multi-scan (Blessing, 1995)
$T_{min} = 0.987$, $T_{max} = 0.997$
26 404 measured reflections

3204 independent reflections
$R_{int} = 0.042$
$\theta_{max} = 30.48°$
$h = -20 \rightarrow 20$
$k = -14 \rightarrow 14$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.0813P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$(\Delta/\sigma)_{\max} = 0.001$
$wR(F^2) = 0.093$	$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$S = 1.067$	$\Delta\rho_{\min} = -0.16$ e Å^{-3}
3204 reflections	
185 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

B6—N61	1.5671 (13)	B8—B9	1.8497 (17)
B6—B7	1.7798 (14)	N4—B9	1.4989 (14)
B6—B5	1.8287 (15)	N4—B5	1.5180 (14)
B7—B8	1.8616 (18)	N4—B1	1.6064 (15)
N61—B6—B7	121.09 (8)	B9—N4—B5	123.13 (8)
N61—B6—B5	116.90 (7)	B9—N4—B1	73.30 (7)
B7—B6—B5	106.47 (7)	B5—N4—B1	70.20 (7)
B6—B7—B8	112.70 (8)	N4—B5—B6	112.74 (8)
B9—B8—B7	101.84 (8)	N4—B9—B8	121.10 (9)

The title compound crystallizes in the polar orthorhombic space group $Pna2_1$. The direction of the polar axis is ambiguous since the structure does not possess significant anomolous scatterers of Mo $K\alpha$ radiation. An enantiopole parameter is therefore not quoted. Pyridine-H atoms were constrained to calculated positions with isotropic displacement parameters equal to $1.2U_{\rm eq}$ of the parent C atom. Cluster-associated H atoms were located *via* Fourier difference syntheses and all associated parameters were refined freely.

Data collection: *KCCD* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1996); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEX*5 (McArdle, 1995); software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BR1272). Services for accessing these data are described at the back of the journal.

References

Baše, K., Plešek, J., Heřmánek, S., Huffman, J., Ragatz, P. & Schaeffer, R. (1975). *J. Chem. Soc. Chem. Commun*. pp. 934–935.

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.

Bould, J., Clegg, W., Teat, S. J., Barton, L., Rath, N. P., Thornton-Pett, M. & Kennedy, J. D. (1999). *Inorg. Chim. Acta*, **289**, 95–124.

Davison, M. G., Hughes, A. K., Marder, T. B. & Wade, K. (2000). Editors. *Contemporary Boron Chemistry*. Royal Society of Chemistry: Cambridge. In the press.

Dörfler, U., Kennedy, J. D., Barton, L., Collins, C. M. & Rath, N. P. (1997). *J. Chem. Soc. Dalton Trans.* pp. 707–708.

Holub, J., Kennedy, J. D. & Štíbr, B. (1994). *Collect. Czech. Chem. Commun.* **59**, 367–373.

Holub, J., Štíbr, B., Kennedy, J. D., Thornton-Pett, M., Jelínek, T. & Plešek, J. (1994). *Inorg. Chem.* **33**, 4545–4552.

Jelínek, T., Štíbr, B. & Kennedy, J. D. (1994). *Collect. Czech. Chem. Commun.* **59**, 2244–2252.

Kaur, P., Holub, J., Rath, N. P., Bould, J., Barton, L., Štíbr, B. & Kennedy, J. D. (1996). *J. Chem. Soc. Chem. Commun.* pp. 273–275.

McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65–67.

Nonius (1998). *KCCD*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1996). *Methods Enzymol.* **276**, 307–326.

Plešek, J., Štíbr, B., Fontaine, X. L. R., Jelínek, T., Thornton-Pett, M., Heřmánek, S. & Kennedy, J. D. (1994). *Inorg. Chem.* **33**, 2994–3002.

Ragatz, P., Plešek, J., Heřmánek, S., Baše, K. & Huffman, J. C. (1975). Entry 7513, http://www.iumsc.indiana.edu.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.