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The crystal structure of 4–(4,4,5,5–tetramethyl–1,3,2–dioxaborolan–2–yl)morpholine, $C_{10}H_{20}BNO_3$

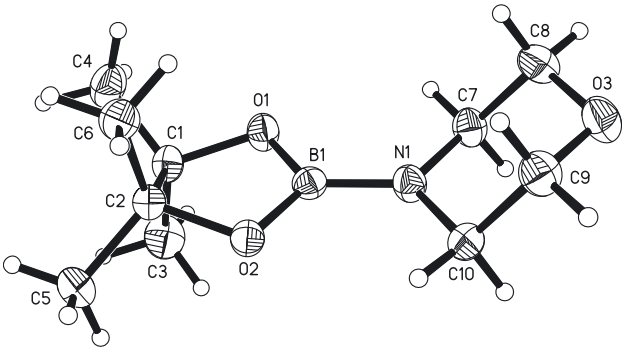

Table 1: Data collection and handling.

Crystal:	Colourless plate
Size:	0.45 × 0.25 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.08 mm^{-1}
Diffractometer, scan mode:	STOE IPDS 2T, Omega scan
θ_{max}, completeness:	27.2°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	14,127, 2663, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2235
$N(param)_{refined}$:	140
Programs:	X-Red [1], Shelx [2, 3], Ortep-III [4]

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Abstract

$C_{10}H_{20}BNO_3$, monoclinic, $P2_1/c$ (no. 14), a = 11.0681(8) Å, b = 6.1883(3) Å, c = 18.1603(13) Å, β = 105.111(6)°, V = 1200.84(14) $Å^3$, Z = 4, $R_{gt}(F)$ = 0.0338, $wR_{ref}(F^2)$ = 0.0861, T = 153 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

In a representative experiment 1.037 g morpholine (11.9 mmol, Sigma–Aldrich, distilled from CaH_2) was placed in a 100-ml round bottom flask. About 20 mL of THF (VWR AnalaR Normapur, dried with MBraun SPS 800) and 1.472 g TMEDA (12.7 mmol, Sigma–Aldrich, distilled from CaH_2) were added. The solution was cooled in an ice bath and CO_2 (Linde, 5.4) was introduced into this solution for 20 min forming a white suspension within 5 min. To this supension 1.874 g HBpin (14.6 mmol, TCI, >97%) was added. A gas evolution took place during the addition and afterwards the solution was heated to about 50 °C for about 1 h. After standing overnight at room temperature, crystals were obtained. All volatiles were removed *in vacuo* and a white solid was yielded used for further analyses.

NMR ($CDCl_3$, ppm): ^{11}B NMR (160 MHz) δ = 23; ^{1}H NMR (500 MHz) δ = 3.55–3.49 (m, 4H, O–CH_2), 3.08–3.02 (m, 4H, N–CH_2), 1.20 (s, 12H, CH_3); **^{13}C NMR** (125 MHz) δ = 81.7 (C1 and C2), 67.9 (O–CH_2), 44.1 (N–CH_2), 24.3 (CH_3).

IR (cm^{-1}): 2991.2 (w), 2970.0 (w), 2929.5 (vw), 1546.7 (w), 1508.1 (m), 1475.3 (m), 1456.1 (m), 1409.8 (w), 1386.6 (w), 1367.4 (m), 1357.7 (w), 1305.6 (w), 1269.0 (s), 1216.9 (vw), 1151.4 (s), 1124.4 (vw), 1101.2 (vs), 1080.0 (m), 1062.6 (m), 1049.1 (w), 1002.9 (vs), 989.4 (vs), 954.6 (vs), 933.4 (s), 918.0 (m), 875.6 (vs), 865.9 (vs), 840.9 (m), 784.9 (w), 756.0 (vw), 730.9 (w), 705.9 (w), 676.9 (s), 642.2 (w), 603.6 (w).

2 Experimental details

The carbon-bound hydrogen atoms were geometrically placed (C—H = 0.98–0.99 Å) and refined as riding atoms with U_{iso} (H) = 1.2–1.5 U_{eq} (C).

3 Comment

Aminoboranes $R_2N–BR_2$ are utilized in a variety of transformations like Strecker type aminative cyanation, Mannich-

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
B1	0.26364 (11)	0.28816 (19)	0.48106 (7)	0.0234 (2)
O1	0.35529 (7)	0.23210 (13)	0.54606 (4)	0.02751 (19)
O2	0.18477 (7)	0.44944 (12)	0.49341 (4)	0.02599 (18)
C1	0.34902 (10)	0.39470 (18)	0.60321 (6)	0.0268 (2)
C2	0.21010 (10)	0.47318 (17)	0.57596 (6)	0.0248 (2)
C3	0.44204 (12)	0.5722 (2)	0.59845 (8)	0.0391 (3)
H3A	0.525489	0.509092	0.605470	0.059*
H3B	0.444228	0.679558	0.638443	0.059*
H3C	0.416247	0.642113	0.548371	0.059*
C4	0.38405 (12)	0.2889 (2)	0.68126 (7)	0.0375 (3)
H4A	0.332664	0.159548	0.680496	0.056*
H4B	0.369452	0.390741	0.719365	0.056*
H4C	0.472648	0.248115	0.694328	0.056*
C5	0.18759 (13)	0.70723 (19)	0.59385 (7)	0.0354 (3)
H5A	0.240123	0.801645	0.571840	0.053*
H5B	0.208805	0.727580	0.649260	0.053*
H5C	0.099303	0.743467	0.571987	0.053*
C6	0.11912 (11)	0.3267 (2)	0.60222 (7)	0.0315 (3)
H6A	0.033124	0.365720	0.575342	0.047*
H6B	0.130744	0.343934	0.657269	0.047*
H6C	0.134889	0.175989	0.590961	0.047*
N1	0.25101 (8)	0.19277 (15)	0.40961 (5)	0.0260 (2)
C7	0.32938 (10)	0.01700 (18)	0.39482 (6)	0.0287 (2)
H7A	0.388500	−0.028772	0.443184	0.034*
H7B	0.378696	0.067386	0.359821	0.034*
C8	0.24809 (12)	−0.17242 (19)	0.35943 (7)	0.0344 (3)
H8A	0.301403	−0.287892	0.346932	0.041*
H8B	0.205397	−0.231453	0.396646	0.041*
O3	0.15666 (8)	−0.10884 (14)	0.29171 (5)	0.0361 (2)
C9	0.07691 (11)	0.0550 (2)	0.30894 (7)	0.0324 (3)
H9A	0.032906	−0.002381	0.345761	0.039*
H9B	0.013130	0.095937	0.261815	0.039*
C10	0.15182 (10)	0.25192 (18)	0.34232 (6)	0.0271 (2)
H10A	0.189093	0.317892	0.303686	0.033*
H10B	0.095932	0.359964	0.356573	0.033*

type reaction, and reductive amination [5, 65,6]. Herein we describe the crystal structure of a simple aminoborane formed out of pinacolborane ("HBpin") and morpholine.

The asymmetric unit contains one molecule of 4–(4,4,5,5–tetramethyl–1,3,2–dioxaborolan–2–yl)morpholine. The bond lengths are in the expected ranges. Bond angles in the five membered 1,3,2–dioxaborolane ring are smaller than expected due to the five membered ring, for instance B1—O1—C1 105.85(8)° and B1—O2—C2 106.20(8)°. The planes O1—B1—O2 and C7—N1—C10 are nearly coplanar, as can be seen from the values of the torsion angles O1—B1—N1—C7 and O2—B1—N1—C10 with 2.38(17)° and −1.39(17)°, respectively. The five membered 1,3,2–dioxaborolane ring is twisted on C1 and C2. These two atoms are 0.1848(6) Å (C1) below and 0.1876(6) Å above the least squares plane of the ring atoms C1—C2—O2—B1—O1. The six membered morpholine ring adopts a chair conformation.

About 70 related crystal structures of aminoboranes containing the 1,3,2–dioxaborolane ring bound to secondary amines are listed in the Cambridge Structural Database [7].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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