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The crystal structure of 2-(benzo[*d*][1,3]dioxol-5-yl)-2,3-dihydro-1H-naphtho[1,8-*de*][1,3,2]diazaborinine, C₁₇H₁₃BN₂O₂

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Abstract

C₁₇H₁₃BN₂O₂, orthorhombic, *Pbca* (no. 61), $a = 10.0820(14)$ Å, $b = 13.1135(19)$ Å, $c = 20.644(3)$ Å, $V = 2729.4$ Å³, $Z = 8$, $R_{gt}(F) = 0.0373$, $wR_{ref}(F^2) = 0.1005$, $T = 193$ 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.

Source of material

In air, Bpin{Bis(pinacolato)diboron}-B(dan){naphthalene-1,8-diamino boronamide}; (0.1 mmol, 1.0 eq.), benzo[*d*][1,3]dioxol-5-amine (0.2 mmol, 2.0 eq.), TBAI (tetrabutylammonium iodide; 0.01 eq.), NaOAc (0.15 eq.), and BPO (0.01 eq.)

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Table 1: Data collection and handling.

Crystal:	Pink block
Size:	0.12 × 0.10 × 0.10 mm
Wavelength:	Ga Kα radiation (1.34139 Å)
μ:	0.48 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	60.6°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	20002, 3078, 0.048
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 2833
$N(param)_{refined}$:	200
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

were sequentially weighed and added to a screw-capped Schlenk tube containing a magnetic stir bar. The vessel was evacuated and refilled with nitrogen for three times. The tBuONO (0.2 eq.) and MeCN (0.6 mL) were added in turn under N₂ atmosphere using syringes through a septum which was temporarily used to replace the screw cap. The reaction mixture was then vigorously stirred at 80 °C for the indicated time. The resulting mixture was filtered through a pad of Celite, and the filter cake was washed with ethyl acetate (3 mL × 2). The combined filtrate was evaporated under vacuum to dryness and the residue was purified by column chromatography to yield the desired product as pink solid.

Experimental details

All the H atoms on the benzene rings were placed geometrically and refined without any constraints or restraints.

Comment

Recently, organoboron materials have caught the researchers' eyes because of their use in material discovery, synthetic science and drug chemistry [5]. They can be used in the synthetic strategies to form new C–C bonds or C–X bonds [6, 7]. The two nitrogen atoms in the naphthalene-1,8-diaminato (dan) ligand can interact with boron to form the naphthalene-1,8-diamino boronamide (Bdan). Bdan compounds are relatively stable, and in most organic chemical reactions they can avoid unnecessary side reactions. More importantly, the Bdan group

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.10474 (8)	0.25412 (6)	0.61458 (4)	0.0353 (2)
O2	0.01125 (8)	0.40691 (7)	0.58396 (4)	0.0434 (2)
N1	0.48663 (9)	0.45974 (7)	0.39012 (4)	0.0297 (2)
H1	0.507478	0.394714	0.392771	0.036*
N2	0.34936 (9)	0.60087 (7)	0.42162 (4)	0.0293 (2)
H2	0.282731	0.625577	0.444242	0.035*
C1	0.38514 (11)	0.76835 (8)	0.37447 (5)	0.0331 (3)
H1A	0.316883	0.796379	0.400727	0.040*
C2	0.45463 (12)	0.83120 (9)	0.33104 (6)	0.0374 (3)
H2A	0.431688	0.901281	0.327802	0.045*
C3	0.55460 (12)	0.79335 (9)	0.29337 (5)	0.0357 (3)
H3	0.599634	0.837057	0.263987	0.043*
C4	0.59150 (10)	0.68945 (8)	0.29784 (5)	0.0297 (2)
C5	0.69781 (11)	0.64768 (9)	0.26189 (5)	0.0350 (3)
H5	0.747410	0.690028	0.233473	0.042*
C6	0.72969 (11)	0.54681 (9)	0.26776 (6)	0.0385 (3)
H6	0.800485	0.519766	0.242712	0.046*
C7	0.66001 (11)	0.48241 (9)	0.31001 (5)	0.0347 (3)
H7	0.683893	0.412531	0.313403	0.042*
C8	0.55694 (10)	0.52042 (8)	0.34659 (5)	0.0272 (2)
C9	0.52002 (9)	0.62470 (8)	0.34087 (5)	0.0258 (2)
C10	0.41567 (10)	0.66627 (8)	0.37910 (5)	0.0270 (2)
C11	0.31147 (10)	0.42714 (8)	0.48093 (5)	0.0271 (2)
C12	0.18969 (10)	0.45790 (8)	0.50846 (5)	0.0318 (2)
H12	0.149761	0.520874	0.496755	0.038*
C13	0.13074 (10)	0.39425 (8)	0.55256 (5)	0.0312 (2)
C14	0.18598 (10)	0.30236 (8)	0.57032 (5)	0.0286 (2)
C15	0.30347 (10)	0.26872 (8)	0.54486 (5)	0.0304 (2)
H15	0.341221	0.205130	0.556945	0.037*
C16	0.36486 (10)	0.33351 (8)	0.49995 (5)	0.0286 (2)
H16	0.446858	0.312706	0.481522	0.034*
C17	0.01058 (11)	0.33085 (10)	0.63350 (6)	0.0383 (3)
H17A	−0.079013	0.300699	0.637805	0.046*
H17B	0.036120	0.361119	0.675619	0.046*
B1	0.38360 (11)	0.49682 (9)	0.43031 (5)	0.0273 (2)

can be converted into its corresponding boronic acids in high yield in acidic aqueous solution, and then participate in the following reactions. Because of these characteristics, they were often used in the synthesis of multi-boron compounds [8–12].

There is one molecule in the asymmetric unit (see the figure). All bonds and angles in the crystal structure are

within the normal range. In conclusion, we have developed a facile process for the synthesis of 2-(benzo[*d*][1,3]dioxol-5-yl)-2,3-dihydro-1*H*-naphtho[1,8-*de*][1,3,2]diazaborinine.

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