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The crystal structure of 2-(3,4-dimethoxyphenyl)-2,3-dihydro-1*H*-naphtho [1,8-*de*][1,3,2] diazaborinine, C₁₈H₁₇BN₂O₂

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.12 × 0.10 × 0.10 mm
Wavelength:	Ga K α radiation (1.34139 Å)
μ :	0.46 mm ⁻¹
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω
$\theta_{\max}$, completeness:	60.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11,444, 3320, 0.054
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2616
$N(\text{param})_{\text{refined}}$:	210
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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Abstract

C₁₈H₁₇BN₂O₂, monoclinic, $P2_1/c$ (no. 14), $a = 12.5782(14)$ Å, $b = 13.6724(14)$ Å, $c = 8.7113(9)$ Å, $\beta = 97.819(6)^\circ$, $V = 1484.2(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0482$, $wR_{\text{ref}}(F^2) = 0.1357$, $T = 193$ 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.), 3,4-dimethoxyaniline (0.2 mmol, 2.0 eq.), TBAI (tetra-*n*-butylammonium

iodide; 0.01 eq.), NaOAc (0.15 eq.), and BPO (Benzoyl peroxide; 0.01 eq.) 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. 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 yellow solid.

Experimental details

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

Comment

In recent years, organoboron reagents are of interest, because of their widely use in synthetic chemistry, medicinal chemistry and material chemistry [5]. Some boron reagents can be used to construct new C–C or C–X bonds [6, 7]. The most common boron-containing group Bpin has relatively high activity, and its activity cannot be easily controlled during the reaction. Therefore, chemists developed the Bdan group (naphthalene-1,8-diamino

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
O1	0.49195 (10)	0.35408 (9)	0.84556 (13)	0.0338 (3)
O2	0.58801 (9)	0.36300 (9)	0.60273 (13)	0.0308 (3)
N1	0.15891 (12)	0.36022 (10)	0.17069 (17)	0.0309 (3)
H1	0.137261	0.307679	0.215873	0.037*
N2	0.27408 (11)	0.50104 (10)	0.17512 (16)	0.0307 (3)
H2	0.327517	0.536335	0.221577	0.037*
C1	0.02859 (14)	0.32723 (13)	−0.0560 (2)	0.0348 (4)
H1A	0.008478	0.267592	−0.011895	0.042*
C2	−0.02229 (15)	0.35676 (14)	−0.2017 (2)	0.0384 (4)
H2A	−0.077093	0.316757	−0.254909	0.046*
C3	0.00500 (15)	0.44165 (14)	−0.2690 (2)	0.0360 (4)
H3	−0.031563	0.460370	−0.367243	0.043*
C4	0.08753 (13)	0.50203 (13)	−0.19321 (19)	0.0302 (4)
C5	0.12021 (14)	0.58956 (14)	−0.2595 (2)	0.0346 (4)
H5	0.085338	0.610306	−0.357705	0.042*
C6	0.20165 (15)	0.64457 (13)	−0.1833 (2)	0.0361 (4)
H6	0.223083	0.702846	−0.229988	0.043*
C7	0.25406 (14)	0.61651 (13)	−0.0376 (2)	0.0329 (4)
H7	0.310378	0.655713	0.013643	0.040*
C8	0.22390 (13)	0.53203 (12)	0.03126 (19)	0.0273 (4)
C9	0.13975 (13)	0.47275 (12)	−0.04552 (19)	0.0266 (4)
C10	0.10786 (13)	0.38467 (12)	0.02361 (19)	0.0283 (4)
C11	0.30601 (13)	0.38850 (12)	0.4133 (2)	0.0295 (4)
C12	0.25604 (14)	0.37617 (13)	0.5445 (2)	0.0328 (4)
H12	0.179881	0.375303	0.534287	0.039*
C13	0.31500 (14)	0.36505 (13)	0.6908 (2)	0.0317 (4)
H13	0.278831	0.358825	0.779153	0.038*
C14	0.42587 (14)	0.36303 (12)	0.70801 (19)	0.0270 (4)
C15	0.47829 (13)	0.37064 (11)	0.57555 (19)	0.0255 (3)
C16	0.41904 (13)	0.38522 (12)	0.43239 (19)	0.0278 (4)
H16	0.455252	0.393264	0.344366	0.033*
C17	0.64335 (14)	0.36790 (13)	0.4699 (2)	0.0329 (4)
H17A	0.617374	0.315787	0.397050	0.049*
H17B	0.720540	0.359699	0.502286	0.049*
H17C	0.630038	0.431586	0.419250	0.049*
C18	0.44416 (16)	0.37066 (14)	0.9829 (2)	0.0376 (4)
H18A	0.410357	0.435368	0.977589	0.056*
H18B	0.499561	0.367553	1.073451	0.056*
H18C	0.389763	0.320423	0.991816	0.056*
B1	0.24398 (15)	0.41580 (15)	0.2511 (2)	0.0296 (4)

boronamide), which is robust enough to avoid the formation of undesirable by-products in the Suzuki–Miyaura coupling reaction. They can be hydrolyzed by placing in an acidic aqueous solution to form its corresponding boric acid. The above characteristics make the Bdan group very important as a building block [8–11]. So here we report the crystal structure of the title compound (*cf.* Figure).

There is one molecule in the symmetric unit (see Figure). All bonds and angles in the crystal structure are within the normal range [9]. In conclusion, we have developed a facile synthetic protocol for the synthesis of 2-(3,4-dimethoxyphenyl)-2,3-dihydro-1*H*-naphtho [1,8-*de*][1,3,2]diazaborinine, C₁₈H₁₇BN₂O₂.

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

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