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Crystal structure of methyl 3-(1*H*-naphtho[1,8-*de*][1,3,2]diazaborinin-2(3*H*)-yl)benzoate, $C_{18}H_{15}BN_2O_2$

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.12 × 0.10 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.5°, 97%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9847, 3446, 0.054
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1995
$N(\text{param})_{\text{refined}}$:	209
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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Abstract

$C_{18}H_{15}BN_2O_2$, monoclinic, $P2_1/n$ (no. 14), $a = 13.61(2)$ Å, $b = 7.389(11)$ Å, $c = 16.34(2)$ Å, $\beta = 113.91(2)^\circ$, $V = 1502(4)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0544$, $wR_{\text{ref}}(F^2) = 0.1651$, $T = 273$ 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, {bis(pinacolato)diboron}-{naphthalene-1,8-diamino boronamide} (0.1 mmol, 1.0 eq.), methyl 3-aminobenzoate (0.2 mmol, 2.0 eq.), TBAI (Tetrabutylammonium iodide, 0.01 eq.), NaOAc (0.15 eq.), and dibenzoyl peroxide (BPO)

(0.01 eq.) were sequentially weighed and added to a Schlenk tube containing a magnetic stir bar. The vessel was evacuated and refilled with nitrogen for three times. *t*BuONO (0.2 eq.) and MeCN (0.6 mL) were added in turn under a N₂ atmosphere using syringes through a septum. The reaction mixture was then vigorously stirred at 80 °C. 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 to dryness and the residue was purified by column chromatography to yield the desired product as a colorless 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 have aroused the research interest of chemists, because of their widely use in synthetic chemistry, medicinal chemistry, and material chemistry [5]. Some of these compounds can be used to construct new C–C or C–X bonds [6, 7]. The very common boron-containing reagent Bpin {bis(pinacolato)diboron} has a relatively high activity, and its activity cannot be easily controlled during the reaction. Therefore, chemists developed the Bdan group (naphthalene-1,8-diamino boronamide) which is robust enough to avoid the

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

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
B1	0.3328 (2)	0.7297 (3)	0.41103 (16)	0.0368 (6)
C1	0.20274 (18)	0.7487 (3)	0.48247 (14)	0.0394 (5)
C2	0.1014 (2)	0.7246 (3)	0.47989 (17)	0.0539 (6)
H2A	0.045896	0.686866	0.427071	0.065*
C3	0.0808 (2)	0.7566 (4)	0.55683 (19)	0.0650 (8)
H3	0.012106	0.737255	0.554374	0.078*
C4	0.1602 (2)	0.8154 (4)	0.63441 (17)	0.0599 (7)
H4	0.144871	0.836093	0.684171	0.072*
C5	0.2658 (2)	0.8456 (3)	0.64055 (15)	0.0450 (6)
C6	0.3504 (2)	0.9139 (3)	0.71885 (15)	0.0527 (7)
H6	0.337944	0.936839	0.769798	0.063*
C7	0.4500 (2)	0.9461 (3)	0.71995 (15)	0.0501 (6)
H7	0.503357	0.995696	0.770914	0.060*
C8	0.47331 (19)	0.9054 (3)	0.64514 (14)	0.0435 (6)
H8	0.541887	0.926049	0.647612	0.052*
C9	0.39440 (17)	0.8352 (3)	0.56859 (13)	0.0365 (5)
C10	0.28805 (18)	0.8092 (3)	0.56359 (13)	0.0374 (5)
C11	0.36102 (17)	0.6797 (3)	0.32931 (13)	0.0365 (5)
C12	0.45549 (18)	0.5862 (3)	0.34277 (14)	0.0417 (5)
H12	0.501184	0.553513	0.400757	0.050*
C13	0.48375 (18)	0.5402 (3)	0.27264 (14)	0.0419 (5)
H13	0.546657	0.475813	0.283929	0.050*
C14	0.41833 (17)	0.5901 (3)	0.18644 (14)	0.0390 (5)
H14	0.437666	0.561703	0.139502	0.047*
C15	0.32252 (17)	0.6840 (3)	0.16959 (13)	0.0357 (5)
C16	0.29385 (18)	0.7259 (3)	0.24082 (14)	0.0378 (5)
H16	0.229284	0.785212	0.229200	0.045*
C17	0.25050 (19)	0.7392 (3)	0.07734 (14)	0.0386 (5)
C18	0.2414 (2)	0.7974 (4)	−0.06885 (15)	0.0571 (7)
H18A	0.282696	0.773588	−0.103397	0.086*
H18B	0.224427	0.924008	−0.071956	0.086*
H18C	0.176122	0.728251	−0.092427	0.086*
N1	0.41435 (15)	0.7893 (2)	0.49340 (11)	0.0410 (5)
H1	0.479147	0.797530	0.497292	0.049*
N2	0.22806 (15)	0.7175 (2)	0.40874 (11)	0.0417 (5)
H2	0.176466	0.689001	0.358737	0.050*
O1	0.15652 (14)	0.7757 (2)	0.05299 (11)	0.0575 (5)
O2	0.30327 (13)	0.7467 (2)	0.02341 (10)	0.0506 (5)

formation of undesirable by-products in the Suzuki–Miyaura coupling reaction. By-products can be hydrolyzed by placing in an acidic aqueous solution to form the corresponding boric acid. The above characteristics make the Bdan groups very important as the building blocks in

several synthetic protocols [8–11]. So here we report the crystal structure of the title compound (*cf.* figure).

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

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