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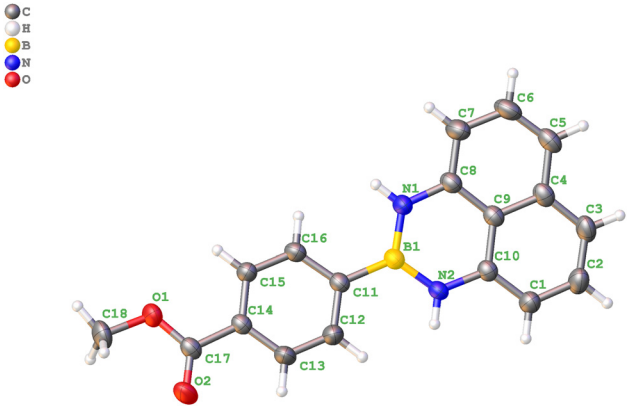
The crystal structure of methyl 4-(1*H*-naphtho [1,8-*de*][1,3,2]diazaborinin-2(3*H*)-yl)benzoate, $C_{18}H_{15}BN_2O_2$



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Abstract

$C_{18}H_{15}BN_2O_2$, monoclinic, $P2_1/c$ (no. 14), a = 6.8522(9) Å, b = 8.4333(11) Å, c = 13.1908(18) Å, β = 95.880(5)°, V = 758.24(17) Å^3, Z = 2, $R_{gt}(F)$ = 0.0381, $wR_{ref}(F^2)$ = 0.1068, 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.

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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:	0.44 mm^{-1}
Diffractometer, scan mode:	Bruker D8 VENTURE, φ and ω
θ_{max}, completeness:	60.6°, 97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5876, 3468, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2341
$N(param)_{refined}$:	209
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

In air, {bis(pinacolato)diboron}-{naphthalene-1,8-diamino boronamide} (0.1 mmol, 1.0 eq.), methyl 4-aminobenzoate (0.2 mmol, 2.0 eq.), TBAI (0.01 eq.), NaOAc (0.15 eq.), and BPO (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 N_2 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 colorless solid.

Experimental details

All the H atoms were placed geometrically and refined using a riding model.

Comment

Recently, organoboron materials have catched the researchers' eyes because of their widely use in material

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.0130 (2)	0.8066 (2)	0.81392 (10)	0.0469 (4)
O2	0.0943 (2)	0.7559 (3)	0.97598 (9)	0.0530 (4)
N1	0.8402 (2)	0.4511 (2)	0.63501 (11)	0.0397 (4)
H1	0.760563	0.507403	0.592562	0.048*
N2	0.9227 (2)	0.3374 (2)	0.80155 (10)	0.0341 (3)
H2	0.896788	0.322850	0.864858	0.041*
C1	1.2137 (3)	0.1746 (3)	0.83539 (14)	0.0422 (5)
H1A	1.186378	0.159272	0.903863	0.051*
C2	1.3795 (3)	0.1048 (3)	0.80048 (16)	0.0492 (5)
H2A	1.461809	0.039591	0.845363	0.059*
C3	1.4258 (3)	0.1283 (3)	0.70349 (16)	0.0458 (5)
H3	1.540386	0.080662	0.682100	0.055*
C4	1.3041 (3)	0.2233 (2)	0.63418 (14)	0.0379 (4)
C5	1.3483 (3)	0.2548 (3)	0.53359 (16)	0.0452 (5)
H5	1.464463	0.212679	0.510529	0.054*
C6	1.2256 (3)	0.3451 (3)	0.46977 (15)	0.0515 (5)
H6	1.257807	0.364589	0.402533	0.062*
C7	1.0522 (3)	0.4103 (3)	0.50076 (16)	0.0493 (5)
H7	0.967404	0.471382	0.454534	0.059*
C8	1.0066 (3)	0.3847 (2)	0.59880 (13)	0.0370 (4)
C9	1.1314 (3)	0.2906 (2)	0.66759 (13)	0.0327 (4)
C10	1.0898 (2)	0.2660 (2)	0.76986 (12)	0.0325 (4)
C11	0.6083 (2)	0.5142 (2)	0.77518 (13)	0.0326 (4)
C12	0.5804 (3)	0.5177 (3)	0.87892 (14)	0.0474 (5)
H12	0.673720	0.467455	0.926647	0.057*
C13	0.4209 (3)	0.5922 (3)	0.91339 (13)	0.0466 (5)
H13	0.405640	0.592847	0.984161	0.056*
C14	0.2821 (2)	0.6665 (2)	0.84506 (12)	0.0319 (4)
C15	0.3050 (3)	0.6642 (2)	0.74190 (13)	0.0360 (4)
H15	0.210898	0.714089	0.694397	0.043*
C16	0.4664 (3)	0.5886 (3)	0.70854 (13)	0.0372 (4)
H16	0.480678	0.587586	0.637655	0.045*
C17	0.1137 (2)	0.7465 (2)	0.88640 (13)	0.0348 (4)
C18	−0.1750 (3)	0.8951 (3)	0.84780 (18)	0.0559 (6)
H18A	−0.244907	0.950501	0.789654	0.084*
H18B	−0.124903	0.972668	0.899350	0.084*
H18C	−0.265092	0.822157	0.877334	0.084*
B1	0.7927 (3)	0.4324 (3)	0.73664 (15)	0.0334 (4)

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, which were often found in compounds with more complex structure [6, 7]. The two nitrogen atoms in the naphthalene-1,8-diaminato (dan) molecule can interact with boron to form the naphthalene-1,8-diamino boronamide (Bdan). And Bdan compounds are relatively stable, and in most organic reactions they can avoid unnecessary side reactions. More importantly, Bdan 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].

Now we report the crystal structure of the title compound (cf. figure). There is one molecule in the symmetric unit. All bonds and angles in the crystal structure are within the normal range [9, 11, 12]. In conclusion, we have developed a facile process for the synthesis of methyl 4-(1*H*-naphtho[1,8-*de*][1,3,2]diazaborinin-2(3*H*)-yl)benzoate.

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

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