Wenqi Song and Zongcheng Miao*

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

Abstract
C_{18}H_{15}BN_{2}O_{2}, monoclinic, C2/c (no. 15), a = 23.976(3) Å, b = 7.7690(9) Å, c = 32.940(4) Å, β = 95.17(1)°, V = 2482.26(15) Å³, Z = 4, R_{ref}(F) = 0.0525, wR_{ref}(F^2) = 0.1415, T = 193 K.

Table 1: Data collection and handling.

<table>
<thead>
<tr>
<th>Crystal:</th>
<th>Colorless block</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size:</td>
<td>0.12 × 0.10 × 0.10 mm</td>
</tr>
<tr>
<td>Wavelength:</td>
<td>Ga Kα radiation (1.34139 Å)</td>
</tr>
<tr>
<td>μ:</td>
<td>0.44 mm⁻¹</td>
</tr>
<tr>
<td>Diffractometer, scan mode:</td>
<td>Bruker APEX-II, φ and ω</td>
</tr>
<tr>
<td>R_{max}, completeness:</td>
<td>54.1°, &gt;99%</td>
</tr>
<tr>
<td>N(hkl)<em>{measured}, N(hkl)</em>{unique}, R_{int}:</td>
<td>32,343, 5654, 0.111</td>
</tr>
<tr>
<td>Criterion for I_{obs}, N(hkl)_{gt}:</td>
<td>I_{obs} &gt; 2σ(I_{obs}), 3971</td>
</tr>
<tr>
<td>N(param)_{refined}:</td>
<td>417</td>
</tr>
<tr>
<td>Programs:</td>
<td>Bruker [1], Olex2 [2], SHELX [3, 4]</td>
</tr>
</tbody>
</table>

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–B(dan) (Bpin = Bis(pinacolato)diboron; B(dan) = naphthalene-1,8-diamino boronamide; 0.1 mmol; 1.0 eq.), methyl 2-aminobenzoate (0.2 mmol 2.0 eq.), TBAI (0.01 eq.), NaOAc (0.15 eq.) and benzoylperoxide (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. 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 without any constraints or restraints.
All the authors have accepted the chemical reactions. The naphthalene-1,8-diamino boronamide therefore it is not easy to control the reactivity in the chemical reactions. The naphthalene-1,8-diamino boronamide has a weak reactivity and has received much attention because it is robust enough to avoid the side reactions. The reagent will be converted to the corresponding boronic acid under acidic hydrolysis conditions in order to participate in the following reactions [8–11].

There are two molecules in the symmetric unit (see Figure). All bonds and angles in the crystal structure are within the normal ranges [8, 9, 12, 13]. The molecules are connected by hydrogen bonds to form a chain along the [010] direction. In conclusion we have shown that a further diazaborinin compound: methyl 2-(1H-naphtho[1,8-de] [1,3]2 diazaborinin-2(3H-yl)benzoate can be easily obtained.

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

References

1. Bruker. APEX-2 SAINT+ Version 6.02 (Includes XPREP and SADABS); Bruker AXS Inc. Madison WI USA, 2016.
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13. Ren H., Miao Z.-C. The crystal structure of methyl 4-(1H-naphtho[1,8-de][1,3,2]diazaborinin-2(3H)-yl)benzoate, C_{18}H_{15}BF_{2}N_{2}. Z. Kristallogr. N. Cryst. Struct. 2021, 236, 807–808.