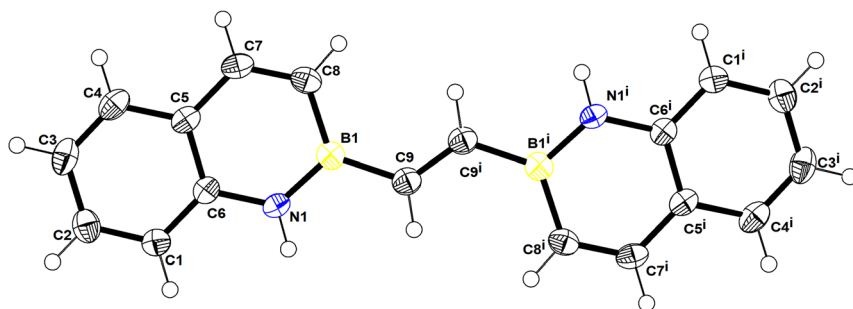


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The crystal structure of (*E*)-1,2-bis(benzo[*e*][1,2]azaborinin-2(1*H*)-yl)ethene, C₁₈H₁₆B₂N₂



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Abstract

C₁₈H₁₆B₂N₂, monoclinic, *C*2/*c* (no. 15), *a* = 34.048(12) Å, *b* = 5.720(2) Å, *c* = 7.666(3) Å, β = 96.803°, *V* = 1482.5(9) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0412, *wR*_{ref}(*F*²) = 0.1152, *T* = 150 K.

CCDC no.: 2299849

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.

1 Source of materials

The title compound was synthesized from a mixture of catalytic Grubbs 2nd generation catalyst and 2-vinyl-1,2-dihydrobenzo[*e*][1,2]azaborinine [1] suspended in dichloroethane under N₂ atmosphere at room temperature for 1 week. Until the completion of this reaction, the resulting residue was adsorbed onto silica gel by rotary evaporation of a DCM solution, loaded directly onto a silica gel column by eluting with EtOAc:PE 1:10 to get the target product as white solid. The crystal structure of this target product was

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.20 × 0.20 mm
Wavelength:	Mo <i>K</i> α radiation (0.71073 Å)
μ:	0.07 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	26.7°, >99 %
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	7378, 1574, 0.029
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1093
<i>N</i> (<i>param</i>) _{refined} :	100
Programs:	Bruker [2], SHELX [3–5], Olex2 [1]

formed and obtained in a mixed system of n-hexane and dichloromethane.

2 Experimental details

Semi-empirical absorption corrections were applied using SADABS program [2]. The structures were solved with SHELXT-2018, and the non-hydrogen atoms were refined anisotropically using the SHELXL-2019 crystallographic software [1, 4]. All the hydrogen atoms were introduced at the calculated positions.

3 Comment

Stilbene derivatives are a series of molecules with a double bond connecting two benzene rings as a skeleton structure. Stilbenes are widely found in natural products, and have

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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}
N1	0.40650 (3)	0.4701 (2)	0.50002 (15)	0.0259 (3)
H1	0.406508	0.603313	0.557510	0.031*
C2	0.37019 (4)	0.3649 (2)	0.45040 (17)	0.0246 (3)
C3	0.36942 (4)	0.1472 (2)	0.36151 (17)	0.0261 (3)
C4	0.40597 (4)	0.0396 (2)	0.32636 (18)	0.0293 (3)
H4	0.405008	−0.107744	0.268749	0.035*
C5	0.33468 (4)	0.4694 (3)	0.48504 (18)	0.0286 (3)
H5	0.335260	0.614212	0.545966	0.034*
C6	0.44159 (4)	0.1411 (3)	0.37260 (19)	0.0299 (4)
H6	0.465107	0.065007	0.347821	0.036*
C7	0.48169 (4)	0.5275 (3)	0.51745 (18)	0.0296 (3)
H7	0.478661	0.668996	0.579352	0.035*
C8	0.33238 (4)	0.0435 (3)	0.30865 (18)	0.0328 (4)
H8	0.331292	−0.102090	0.248570	0.039*
C9	0.29892 (4)	0.3633 (3)	0.4312 (2)	0.0344 (4)
H9	0.274984	0.435886	0.454514	0.041*
C1	0.29775 (5)	0.1490 (3)	0.3424 (2)	0.0370 (4)
H1	0.273062	0.076521	0.305533	0.044*
B1	0.44387 (5)	0.3794 (3)	0.4652 (2)	0.0269 (4)

many biological activities such as anti-oxidation, antibacterial, anti-inflammatory, anti-platelet agglutination, and preventing cell cancerization [6]. When the two benzene rings on stilbene are changed to two naphthalene rings, the skeleton is 1,2-di(naphthalen-2-yl)ethene (DNE). Naphthalene is an extensively explored aromatic conjugated system with applications in various pathophysiological conditions [7]. Vinyl naphthalene can be widely used as pharmaceutical intermediates, dye intermediates, and optical component coating materials. BN 2-vinylnaphthalene, as a polar olefin monomer, has attracted the research interest of polymer scholars due to its unique structural characteristics [8–10]. However, the chemistry and biological activity of DNE are rarely studied. Especially, BN substituted 1,2-di(naphthalen-2-yl)ethene (BN-DNE) has never been constructed. Here we reported the synthesis and unique structure of BN substituted 1,2-di(naphthalen-2-yl)ethene. Furthermore, we compared the crystal structure of DNE (CCDC number, 1555496) and BN-DNE.

Single-crystal X-ray diffraction (SC-XRD) measurement result showed that each asymmetric unit contains one half (E)-1,2-bis(benzo[e][1,2]azaborinin-2(1H)-yl)ethene molecule. As shown in the Figure, the central boron atom B1 adopts a typical three-coordinated planar triangular geometry surrounded by two carbon atoms (C6, C7) and one nitrogen atom (N1). The C/N–B bond lengths and C/N–B–C/N bond angles are in the range of 1.4284(19)–1.554(2) and 114.32(13)–126.76(13)°, respectively, which are comparable to some previously

reported boron-containing compounds with the similar structure [5–7]. Due to the planar geometry of boron atoms and the linkage of C=C bond, all the atoms of this molecule are closely coplanar. In addition, in the crystal structure of 1, adjacent molecules are assembled by C–H... π interactions into two-dimensional (2D) supramolecular layers, which are further stacked to the final 3D structure.

The crystal of (E)-1,2-di(naphthalen-2-yl)ethene (DNE) crystallized in the space group *P*2₁/*c* and each asymmetric unit consists of one half DNE molecule. Meanwhile, all the atoms in compound 1 and DNE are both closely coplanar due to the planar geometry of C=C bond and the aromatic rings. In addition, significant C–H... π interactions could be observed between the adjacent DNE molecules as well with the similar dihedral angle to that of compound 1, but the interacted H atoms are located on the same six-membered ring, which is quite different from compound 1. On the other hand, the adjacent two molecules in the crystal of compound 1 are intertwined with each other and their projections on the *ab* plane are X shape, while the neighboring two molecules in DNE are side-by-side with the projections nearly parallel to each other. Further inspection revealed that the adjacent DNE molecules are also assembled by the C–H... π interactions into layers, which is similar to the crystal structure of 1.

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