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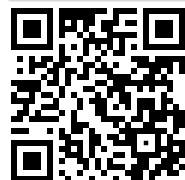
# Synthesis and crystal structure determination of a new 1D polymer adduct of 1,2-di(pyridin-4-yl)ethane, based on B-N dative bonded eight-membered cyclo-1,3,3,5,7,7-hexaphenyl-1,5-dibora-3,7-disiloxane

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## RESEARCH ARTICLE



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## ABSTRACT

A novel 1D polymer of 1,2-di(pyridin-4-yl)ethane ( $L_1$ ), connected via B-N dative-bonded adduct with an eight-membered cyclo-1,3,3,5,7,7-hexaphenyl-1,5-dibora-3,7-disiloxane [ $Ph_6B_2Si_2O_4$ ]- $L_1$  was synthesized and characterized. The new compound [ $Ph_6B_2Si_2O_4$ ]- $L_1$  was prepared by the reaction of cyclo-1,3,3,5,7,7-hexaphenyl-1,5-dibora-3,7-disiloxane and bis(pyridinyl)ethane in a mixture of diethyl ether and petroleum ether solvents at reflux. The 1D polymer [ $Ph_6B_2Si_2O_4$ ]- $L_1$  was characterized by single-crystal X-ray diffraction, nuclear magnetic resonance, and FT-IR spectroscopy. The single crystal X-ray diffraction studies reveal that the aforementioned compound crystallized in the monoclinic crystal system with a centrosymmetric space group of  $P2_1/n$  (no. 14),  $a = 16.5378(4) \text{ \AA}$ ,  $b = 12.6201(3) \text{ \AA}$ ,  $c = 20.4904(5) \text{ \AA}$ ,  $\beta = 96.689(2)^\circ$ ,  $V = 4247.43(18) \text{ \AA}^3$ ,  $Z = 4$ ,  $T = 173.0 \text{ K}$ ,  $\mu(\text{MoK}\alpha) = 0.130 \text{ mm}^{-1}$ ,  $D_{\text{calc}} = 1.233 \text{ g/cm}^3$ , 14630 reflections measured ( $4.96^\circ \leq 2\theta \leq 56.424^\circ$ ), 8424 unique ( $R_{\text{int}} = 0.0235$ ,  $R_{\text{sigma}} = 0.0460$ ) which were used in all calculations, the final  $R_1$  was 0.0455 ( $I > 2\sigma(I)$ ) and  $wR_2$  was 0.1201 (all data). Furthermore, the compound exhibits various noncovalent interactions in the crystal packing, such as intermolecular and intramolecular, as well as hydrogen bonding. This study demonstrates the potential for making novel materials via the combination of cyclodiboradisiloxanes (Lewis acid) and nitrogen-containing ligands (Lewis bases).

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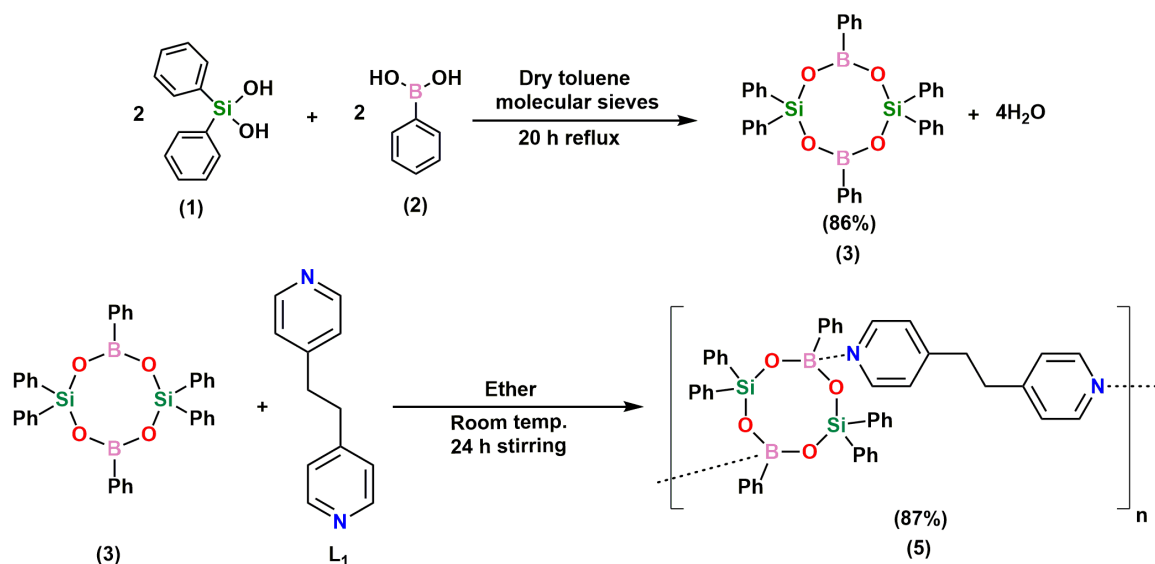
## 1. Introduction

In recent decades, cyclodiboradisiloxanes and their derivatives as a class of organoborasilicon compounds have garnered great attention due to their exceptional structural architecture, magnetic properties, electronic properties, optical properties, quasiaromatic character, as well as their aptitude for the formation of extended intramolecular and intermolecular interactions [1]. These groups of compounds consist of mainly alkyl Si-O-B and O-B-O ring systems in which the boron centres (if tricoordinated) are further ligated with Lewis bases such as pyridines, phosphines, carboxylates, ethers, aryl-sulphides, thioethers, imines, oximes etc. leading to the formation of B-N, B-P, B-O, B-S and so forth dative bonds, which imparts remarkable stability and reactivity. The presence of silicon and oxygen atoms within the cyclodiboradisiloxane affords the framework with unusual high elasticity and plasticity when subjected to sudden and slow stress. Boron-nitrogen adducts, which as those formed between cyclodiboradisiloxanes and nitrogen-containing ligands, have shown great potential applications in various fields, such as: conductive polymers for advanced energy devices, catalysis, carbon capture, gas separation, and adsorption, lightweight,

high-strength materials for application in spacecraft, pharmaceuticals, and drug delivery. The ability to form stable covalent bonds between boron and nitrogen atoms enables the creation of novel materials with tailored properties. Furthermore, polymers, in particular, have revolutionized modern technology, finding applications in everything from packaging materials to advanced electronics. The development of novel polymers with unique properties, such as conductivity, luminescence, or stimulus responsiveness, is an active area of research [2]. The incorporation of cyclodiboradisiloxanes and boron-nitrogen adducts into polymer backbones or side chains offers a promising route to the preparation of materials with enhanced performance and functionality [3-10]. Regardless of the functions and uses of these borasiloxane materials, as well as their promising future prospects, only a few of these materials are available in the literature [4,11-15]. This dearth of literature and materials could be due to the availability of a handful of commercially available silanols, difficulties in structural modification of available materials, and problems of stability. The methods for the preparation of these materials are discussed in detail in the literature [4,7,14,16]. However, the method used in this study is similar to that described in the literature [4], in which molecular sieves were used to remove

**Table 1.** Crystal data and structure refinement for  $[\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4]\cdot\text{L}_1$  (5).

Empirical formula	$\text{C}_{48}\text{H}_{42}\text{B}_2\text{N}_2\text{O}_4\text{Si}_2$
Formula weight (g/mol)	788.63
Temperature (K)	173.0
Crystal system	Monoclinic
Space group	$P2_1/n$
$a$ , (Å)	16.5378(4)
$b$ , (Å)	12.6201(3)
$c$ , (Å)	20.4904(5)
$\alpha$ (°)	90
$\beta$ (°)	96.689(2)
$\gamma$ (°)	90
Volume (Å <sup>3</sup> )	4247.43(18)
$Z$	4
$\rho_{\text{calc}}$ (g/cm <sup>3</sup> )	1.233
$\mu$ (mm <sup>-1</sup> )	0.130
$F(000)$	1656.0
Crystal size (mm <sup>3</sup> )	$0.6 \times 0.32 \times 0.18$
Radiation	$\text{MoK}\alpha$ ( $\lambda = 0.71073$ )
$2\theta$ range for data collection (°)	4.96 to 56.424
Index ranges	$-21 \leq h \leq 14, -16 \leq k \leq 15, -26 \leq l \leq 21$
Reflections collected	14630
Independent reflections	8424 [ $R_{\text{int}} = 0.0235, R_{\text{sigma}} = 0.0460$ ]
Data/restraints/parameters	8424/0/523
Goodness-of-fit on $F^2$	1.025
Final R indexes [ $I \geq 2\sigma(I)$ ]	$R_1 = 0.0455, wR_2 = 0.1070$
Final R indexes [all data]	$R_1 = 0.0682, wR_2 = 0.1201$
Largest diff. peak/hole (e.Å <sup>-3</sup> )	0.77/-0.29

**Scheme 1.** Synthesis of  $(\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4)$  (3) and  $[\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4]\cdot\text{L}_1$  (5) adduct polymer.

water, as shown in [Scheme 1](#), to obtain the crude compound 3. Compound 3 (Lewis acid) was ligated with 1,2-di(pyridin-4-yl)ethane ( $\text{L}_1$ ) leading to the formation of the aforementioned 1D polymer (5) following a literature procedure [4]. Therefore, the main purpose of the study was to synthesize and determine the crystal structure of a new 1D polymer adduct of 1,2-di(pyridin-4-yl)ethane, based on B-N dative bonded eight-membered cyclo-1, 3, 3, 5, 7, 7-hexaphenyl-1, 5-dibora-3, 7-disiloxane ( $\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4$ ) (3) as shown in [Scheme 1](#).

## 2. Experimental

### 2.1. Chemicals and reagents

All chemicals, reagents, and solvents were purchased from Sigma-Aldrich or Alfa-Aesar and used as received unless otherwise stated. Molecular sieves (1.6 mm rods and 0.4 nm pores) were preheated to dryness in the oven at 120 °C for two weeks prior to use.

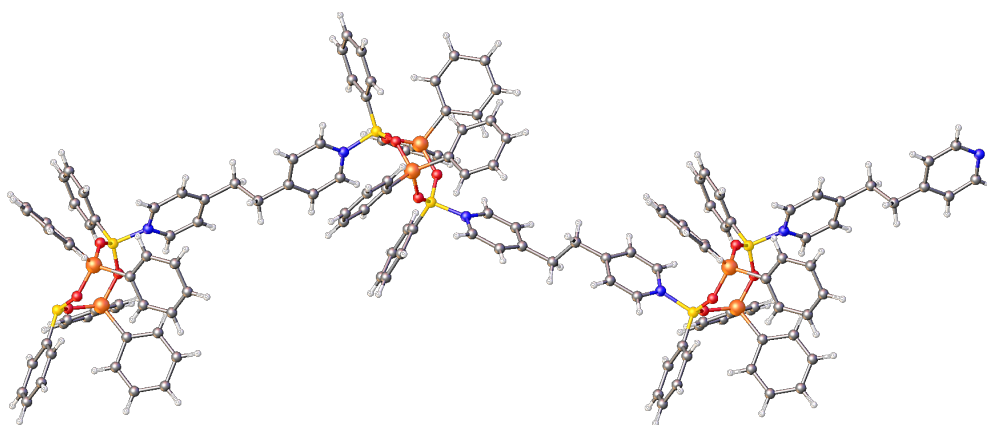
### 2.2. Instrumentation

$^1\text{H}$ ,  $^{13}\text{C}\{^1\text{H}\}$ ,  $^{11}\text{B}\{^1\text{H}\}$ , and  $^{29}\text{Si}\{^1\text{H}\}$  NMR spectra were recorded on Bruker AVANCE III HD 400 MHz or 500 MHz spectrometers in  $\text{CDCl}_3$  solvent unless otherwise stated. The chemical shifts ( $\delta$ ) for  $^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$ ,  $^{29}\text{Si}\{^1\text{H}\}$ , and  $^{11}\text{B}\{^1\text{H}\}$ , are quoted in ppm with reference to  $\text{Me}_4\text{Si}$  and  $\text{BF}_3\text{OEt}_2$ , respectively. Coupling constants are reported in Hz. Infrared spectra were obtained on a Perkin Elmer Spectrum 100 FTIR Spectrometer operating in ATR mode.

### 2.3. Synthesis

#### 2.3.1. Synthesis of $\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4$

In a round bottom flask equipped with a magnetic stirrer bar, diphenylsilanediol (1) (1.082 g, 5.0 mmol) was mixed with 0.609 g (5.0 mmol) of phenylboronic acid (2) in dry toluene (50 mL) and 0.5 g of molecular sieves.



**Figure 1.** 1-D polymeric structure of compound  $[\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4]\cdot\text{L}_2$  (5). Colour identity, Yellow = B, orange = Si, grey = C, blue = N, red= O and white = H.

The mixture was refluxed under  $\text{N}_2$  for 20 h. Thereafter, the round bottom flask and its contents were allowed to cool to room temperature. The mixture was filtered, and the filtrate was collected. The molecular sieves were washed with dry toluene ( $3 \times 25$  mL) followed by filtration and collection of the filtrate. The filtrates were combined and concentrated under reduced pressure and the solid residue was recrystallized from diethyl ether and petroleum ether (3:1 ratio) to give compound 3. Cyclo-1,3,3,5,7,7-hexaphenyl-1,5-dibora-3,7-disiloxane (3): Colour: White. Yield: 1.30 g, 86%. M.p.: 162.0-163.0 °C [Lit. 161-162 °C [17]].  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ,  $\delta$ , ppm): 7.31-7.54 (m, 18H, *m*, *p*- $\text{C}_6\text{H}_5$ ), 7.73-7.75 (m, 8H *o*- $\text{C}_6\text{H}_5\text{Si}$ ), 8.08-8.10 (m, 4H, *o*- $\text{C}_6\text{H}_5\text{B}$ ).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ,  $\delta$ , ppm): 127.93, 128.06, 130.60, 131.74, 133.40, 134.12, 135.65 ( $\text{C}_6\text{H}_5\text{Si}$ ,  $\text{C}_6\text{H}_5\text{B}$ ).  $^{11}\text{B}$  NMR (160 MHz,  $\text{CDCl}_3$ ,  $\delta$ , ppm): 25.69.  $^{29}\text{Si}\{^1\text{H}\}$  NMR (99 MHz,  $\text{CDCl}_3$ ,  $\delta$ , ppm): -45.00. MS (70 eV, EI,  $m/z$ ): 604 ( $\text{M}^+$ ), 527 ( $\text{M-Ph}$ ) $^+$ , 423 ( $\text{M-Ph-PhBO}$ ) $^+$ , 406 ( $\text{M-Ph}_2\text{SiO}$ ) $^+$ . FT-IR (ATR,  $\nu$ ,  $\text{cm}^{-1}$ ): 3023 (C-H<sub>Ar</sub>), 1349 (B-O), 1307 (Si-C str.), 1071 (Si-O str.).

### 2.3.2. Synthesis of $[\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4]\cdot\text{L}_1$

In a round bottom flask,  $[\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4]$  (3) (0.125 g, 0.208 mmol) was dissolved in ether (20 mL) and stirred with a magnetic stirrer bar. In another round bottom flask, 1,2-bis(pyridyl)ethane ( $\text{L}_1$ ) (0.038 g, 0.208 mmol) was dissolved in ether (20 mL) and stirred. The  $[\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4]$  solution was added to 1,2-bis(pyridyl)ethane while stirring. The reaction mixture was stirred for 24 h during which time a milky solution formed. The removal of ether via filtration yielded a colourless powder. Recrystallisation from  $\text{CH}_2\text{Cl}_2$ :ether (2:1, v:v) through slow evaporation gave colourless crystals after 24 h of  $[\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4]\cdot\text{L}_1$  (5). Colour: White. Yield: 0.14 g, 87%. M.p.: 196-197 °C.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ,  $\delta$ , ppm): 2.94 (s, 4H,  $\text{CH}_2\text{CH}_2$ ), 7.09-7.10 (m, 4H, *m*- $\text{C}_5\text{H}_4\text{N}$ ), 7.31-7.49 (m, 18H, *m*, *p*- $\text{C}_6\text{H}_5$ ), 7.68-7.70 (m, 8H, *o*- $\text{C}_6\text{H}_5\text{Si}$ ), 8.02-8.04 (m, 4H, *o*- $\text{C}_6\text{H}_5\text{B}$ ), 8.55-8.56 (m, 4H, *o*- $\text{C}_5\text{H}_4\text{N}$ ).  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ,  $\delta$ , ppm): 150.64, 148.36, 135.27, 134.32, 134.12, 133.88, 133.59, 131.17, 130.33, 127.90, 127.79, 127.51, 124.06, 33.52.  $^{11}\text{B}\{^1\text{H}\}$ , NMR:  $\delta$  ( $\text{CDCl}_3$ , 160 MHz): 19.86.  $^{29}\text{Si}\{^1\text{H}\}$ , NMR:  $\delta$  ( $\text{CDCl}_3$ , 99 MHz): -45.60. FT-IR (KBr,  $\nu$ ,  $\text{cm}^{-1}$ ): 3017 (C-H Ar), 1477 ( $\text{CH}_2$  scissor), 1412 (B-O), 1379 (C-N), 1251 ( $\text{CH}_2$  rock), 1114 ( $\text{CH}_2$  wag), 1073 (Si-O).

## 3. Results and discussion

Cyclo-1, 3, 3, 5, 7, 7 -hexaphenyl-1, 5-dibora-3, 7-disiloxane ( $\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4$ ) (3) was obtained as colourless crystalline solids and readily soluble in nonpolar organic solvents such as benzene and hexane. Compound 3 was characterized using

standard analytical and spectroscopic methods. The  $^1\text{H}$  NMR spectrum of compound 3 showed well-resolved resonances for which aromatic proton signals were found within the range of  $\delta$  7.31-8.10 ppm, and this range of values is comparable to the values reported in the literature [4,11,17,18]. Furthermore, the  $^{13}\text{C}\{^1\text{H}\}$  NMR spectrum of compound 3 showed signals for the aromatic carbon atoms in the range of  $\delta$  127.93-135.65 ppm, these are also in accordance with the values reported in the literature [4,11,17,18]. The  $^{11}\text{B}\{^1\text{H}\}$  NMR of compound 3 showed a broad singlet at  $\delta$  25.69 ppm. This value is symbolic of a single boron chemical environment. Furthermore, the  $^{29}\text{Si}$  NMR of compound 3 showed a singlet at  $\delta$  -45.00 ppm. Furthermore, the IR spectrum obtained from compound 3 showed strong absorption peaks in the regions:  $\nu(\text{cm}^{-1})$  3023, C-H aromatic; 1349, B-O; 1307, Si-C; and 1071 for Si-O stretching mode [4,19]. Subsequently, compound 3 was treated with a 1,2-bis(pyridyl) ethane ligand to give compound 5. The  $^{11}\text{B}\{^1\text{H}\}$  NMR and  $^{29}\text{Si}\{^1\text{H}\}$  NMR spectra of compound 5 showed singlets at  $\delta$  19.86 and -45.60 ppm, respectively. Data obtained through single crystal X-ray diffraction studies reveal that compound 5 comprises the eight-membered ring structure, (i.e. compound 3), and linker  $\text{L}_1$  (i.e. 1,2-di(pyridin-4-yl)ethane) as shown in Figure 1. Compound 5 crystallized in the monoclinic crystal system with a centrosymmetric space group of  $P2_1/n$ , as shown in Table 1, while the bond lengths and angles are shown in Table 2.

As shown in Figure 1, the two B atoms are tetracoordinate. Each boron atom is bonded to two O atoms, one C atom from a phenyl ring, and one N via a coordination (dative bond) to the 1,2-di(pyridin-4-yl)ethane ( $\text{L}_1$ ) to give a 1-D polymeric structure compound 5. The B-O-Si angles of compound 5 range from 133.24(12) to 143.93(13)° with an average value of 139.69(13)°, which are comparable to those of compound [4], and show distortion at the O atoms in the ring as revealed in the ellipsoid plot Figure 2, compared to compound 3.

The four B-O distances in compound 5 range from 1.321(2) to 1.442(3) Å with a mean value of 1.436 Å. These B-O bond distances are longer than the B-O length for a tricoordinated B in  $\text{Si}_2\text{B}_2\text{O}_4$  rings, which range from (1.36-1.39 Å) [11,19,20] but similar to those of the compound reported in the literature [4] discussed earlier. The longer bond length observed at the tetrahedrally bonded B site compared to the tricoordinated B may be linked to a reduction in the B-O  $\pi$ -bonding component on changing from trigonal to tetrahedral coordination geometry at the B atom. Furthermore, the four lengths of Si-O bonds in compound 5 range from 1.604(14) to 1.618(13) Å, which are similar to those found in compound in the literature [4,21-23].

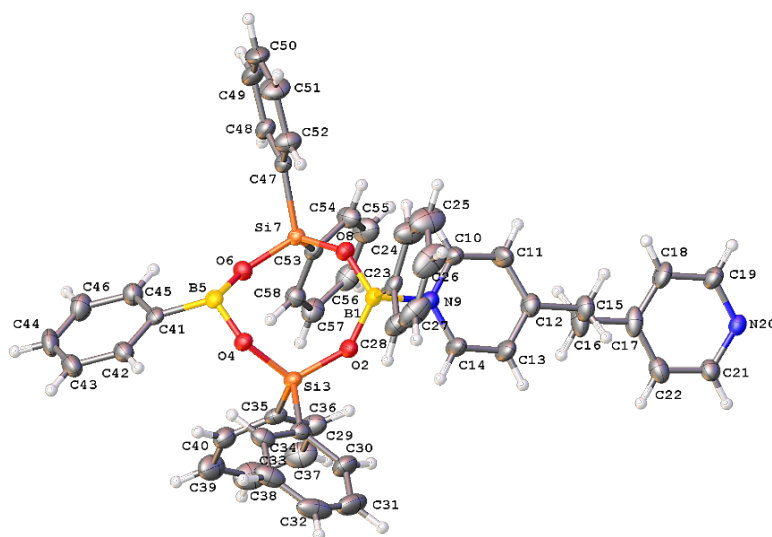
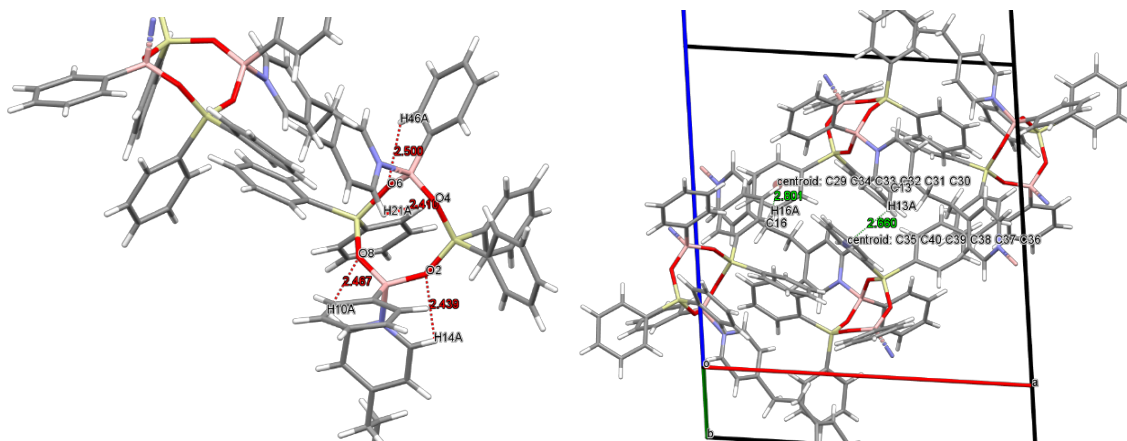
**Table 2.** Bond lengths and bond angles of [Ph<sub>6</sub>B<sub>2</sub>Si<sub>2</sub>O<sub>4</sub>]-L<sub>1</sub> (5).

Atom	Atom	Length (Å)	Atom	Atom	Length (Å)		
B1	O2	1.442(3)	C24	C25	1.380(3)		
B1	O8	1.431(2)	C25	C26	1.382(4)		
B1	N9	1.671(3)	C26	C27	1.366(4)		
B1	C23	1.608(3)	C27	C28	1.384(3)		
O2	Si3	1.6057(14)	C29	C30	1.397(3)		
Si3	O4	1.6085(13)	C29	C34	1.394(3)		
Si3	C29	1.871(2)	C30	C31	1.390(3)		
Si3	C35	1.866(2)	C31	C32	1.364(4)		
O4	B5	1.437(3)	C32	C33	1.376(4)		
B5	O6	1.432(2)	C33	C34	1.382(3)		
B5	N20 <sup>1</sup>	1.702(3)	C35	C36	1.397(3)		
B5	C41	1.603(3)	C35	C40	1.392(3)		
O6	Si7	1.6035(14)	C36	C37	1.381(3)		
Si7	O8	1.6047(14)	C37	C38	1.367(4)		
Si7	C47	1.868(2)	C38	C39	1.388(4)		
Si7	C53	1.8689(19)	C39	C40	1.384(3)		
N9	C10	1.337(3)	C41	C42	1.388(3)		
N9	C14	1.335(2)	C41	C46	1.389(3)		
C10	C11	1.374(3)	C42	C43	1.388(3)		
C11	C12	1.384(3)	C43	C44	1.380(3)		
C12	C13	1.381(3)	C44	C45	1.375(3)		
C12	C15	1.508(3)	C45	C46	1.388(3)		
C13	C14	1.368(3)	C47	C48	1.389(3)		
C15	C16	1.508(3)	C47	C52	1.391(3)		
C16	C17	1.505(3)	C48	C49	1.387(3)		
C17	C18	1.378(3)	C49	C50	1.368(4)		
C17	C22	1.385(3)	C50	C51	1.376(4)		
C18	C19	1.374(3)	C51	C52	1.387(3)		
C19	N20	1.339(3)	C53	C54	1.392(3)		
N20	B5 <sup>2</sup>	1.702(3)	C53	C58	1.393(3)		
N20	C21	1.332(3)	C54	C55	1.386(3)		
C21	C22	1.379(3)	C55	C56	1.376(3)		
C23	C24	1.388(3)	C56	C57	1.372(3)		
C23	C28	1.392(3)	C57	C58	1.388(3)		
Atom	Atom	Atom	Angle (°)	Atom	Atom	Atom	Angle (°)
O2	B1	N9	103.26(15)	C19	N20	B5 <sup>2</sup>	121.11(16)
O2	B1	C23	116.02(17)	C21	N20	B5 <sup>2</sup>	120.79(16)
O8	B1	O2	115.78(17)	C21	N20	C19	118.08(17)
O8	B1	N9	105.17(15)	N20	C21	C22	122.3(2)
O8	B1	C23	110.21(16)	C21	C22	C17	120.2(2)
C23	B1	N9	104.89(15)	C24	C23	B1	119.84(19)
B1	O2	Si3	141.00(12)	C24	C23	C28	116.3(2)
O2	Si3	O4	114.88(7)	C28	C23	B1	123.85(19)
O2	Si3	C29	109.82(8)	C25	C24	C23	122.4(2)
O2	Si3	C35	108.10(9)	C24	C25	C26	119.6(3)
O4	Si3	C29	105.63(8)	C27	C26	C25	119.5(2)
O4	Si3	C35	110.74(8)	C26	C27	C28	120.3(2)
C35	Si3	C29	107.43(9)	C27	C28	C23	121.8(2)
B5	O4	Si3	133.24(12)	C30	C29	Si3	120.77(17)
O4	B5	N20 <sup>1</sup>	101.82(15)	C34	C29	Si3	122.33(16)
O4	B5	C41	114.24(17)	C34	C29	C30	116.9(2)
O6	B5	O4	115.49(16)	C31	C30	C29	121.5(2)
O6	B5	N20 <sup>1</sup>	107.20(15)	C32	C31	C30	119.9(2)
O6	B5	C41	111.22(17)	C31	C32	C33	120.2(2)
C41	B5	N20 <sup>1</sup>	105.67(14)	C32	C33	C34	120.1(2)
B5	O6	Si7	143.93(13)	C33	C34	C29	121.4(2)
O6	Si7	O8	113.07(7)	C36	C35	Si3	122.31(17)
O6	Si7	C47	109.47(8)	C40	C35	Si3	120.53(16)
O6	Si7	C53	106.46(8)	C40	C35	C36	117.1(2)
O8	Si7	C47	104.60(8)	C37	C36	C35	121.2(2)
O8	Si7	C53	111.75(8)	C38	C37	C36	120.4(2)
C47	Si7	C53	111.58(9)	C37	C38	C39	120.1(2)
B1	O8	Si7	140.58(13)	C40	C39	C38	119.1(2)
C10	N9	B1	121.11(16)	C39	C40	C35	122.0(2)
C14	N9	B1	120.53(16)	C42	C41	B5	121.58(19)
C14	N9	C10	118.07(17)	C42	C41	C46	116.91(19)
N9	C10	C11	122.2(2)	C46	C41	B5	121.50(18)
C10	C11	C12	120.1(2)	C43	C42	C41	122.0(2)
C11	C12	C15	122.2(2)	C44	C43	C42	119.7(2)
C13	C12	C11	116.86(19)	C45	C44	C43	119.7(2)
C13	C12	C15	120.9(2)	C44	C45	C46	120.0(2)
C14	C13	C12	120.3(2)	C45	C46	C41	121.8(2)
N9	C14	C13	122.4(2)	C48	C47	Si7	122.74(15)
C16	C15	C12	111.61(19)	C48	C47	C52	116.96(19)
C17	C16	C15	112.06(19)	C52	C47	Si7	120.20(16)
C18	C17	C16	121.3(2)	C49	C48	C47	121.9(2)
C18	C17	C22	116.75(19)	C50	C49	C48	119.8(2)
C22	C17	C16	121.9(2)	C49	C50	C51	119.9(2)
C19	C18	C17	120.5(2)	C50	C51	C52	120.0(2)
N20	C19	C18	122.2(2)	C51	C52	C47	121.4(2)

<sup>1</sup>-1/2+x, 1/2-y, 1/2+z; <sup>2</sup>1/2+x, 1/2-y, -1/2+z.

**Table 3.** Hydrogen bonds and C-H... $\pi$  contacts for compound 5.

Donor-H...Acceptor	D-H, Å	H...A, Å	D...A, Å	D-H...A, °	Symmetry	
C10-H10A...O8	0.95	2.47	2.825(3)	102	-	
C14-H14A...O2	0.95	2.44	2.793(3)	102	-	
C21-H21A...O4	0.95	2.41	2.755(3)	101	1/2+x, 1/2-y, -1/2+z	
C46-H46A...O6	0.95	2.50	2.865(3)	103	-	
C-H...Cg <sup>a</sup>	H...Cg, Å	H-Perp <sup>b</sup>	$\gamma^c$	$\angle$ C-H...Cg, °	C...Cg, Å	Symmetry
C13-H13A...Cg(5)	2.66	-2.62	10.17	137	3.414(2)	1-x, 1-y, 1-z
C16-H16A...Cg(4)	2.80	2.68	17.11	169	3.777(3)	1-x, 1-y, 1-z

<sup>a</sup> Cg: Center of gravity of ring J, Cg(5): C35/C40, Cg(4): C29/C34.<sup>b</sup> Perpendicular distance of H to the ring plane J.<sup>c</sup> Angle between Cg-H vector and ring J normal.**Figure 2.** An ORTEP diagram of  $[\text{Ph}_6\text{B}_2\text{Si}_2\text{O}_4]\cdot\text{L}_2$  (5) with atom labelling. Colour identity, Yellow = B, orange = Si, grey = C, blue = N, red= O and white = H.**Figure 3.** Two polymer chains with C-H... $\pi$  and C-H...O interactions in the crystal packing of compound 5. Colour identity, pink = B, orange = Si, grey = C, blue = N, red= O and white = H.

The B-N bond lengths in compound 5 are 1.671(3) Å and 1.702(3) Å with an average value of 1.686(3) Å. Just like compound [4], the B-N bond lengths for compound 5 are slightly longer than those of simple borosiloxane such as  $\text{Ph}(\text{OSiR}_2\text{R}')\text{B}\{\text{OCH}_2\}_3\text{N}$  [24] (R or R' = Ph or CH<sub>3</sub>) and  $[\text{Bu}^t\text{Si}(\text{O}^i\text{PhBO})_3\text{Si}^i\text{Bu}^t]\cdot\text{NC}_5\text{H}_5$  [19]. The Si-C bond lengths in compound 5 range 1.866(2) to 1.871(2) Å with an average bond distance of 1.868(2) Å.

The ring angles of O2-B1-O8 and O4-B5-O6 are 115.78(17)° and 115.49(16)°, respectively, with a mean value of 115.64(16)°. The mean internal ring angle of compound 5 is higher than 109.5° expected for a tetrahedral geometry but similar to compound [4] as well as the observation reported in the literature [19]. Similarly, the ring angles of silicon; O2-Si3-

O4 and O6-Si7-O8 are 114.88(7)° and 113.07(7)°, respectively, with a mean value of 113.98(7)° which is also slightly higher than 109.5° for a tetrahedral silicon. The other bond angles of Si as well as the Si-C bond lengths are comparable to compounds [4] as well as other borosiloxane compounds and simple adducts in the literature [11,19,25].

Molecular interactions determine the supramolecular structure of the compound 5 (Table 3). In addition to the covalent and dative bonds found in compound 5, the crystal packing of the compound also shows noncovalent interactions C-H... $\pi$  (2.80 Å) and C-H...O (2.44 Å) [26,27] as shown in Figure 3. These two interactions differ from those observed in similar compounds reported in the literature [4].

#### 4. Conclusions

In this article, the successful synthesis and determination of the crystal structure of a new 1D polymer (5) adduct of 1,2-di(pyridin-4-yl)ethane ( $L_1$ ), based on an eight-membered cyclo-1,3,3,5,7,7-hexaphenyl-1,5-dibora-3,7-disiloxane ( $Ph_6B_2Si_2O_4$ ) (3) was reported here. The crystal structure of compound 5 was determined using single-crystal X-ray diffraction. The structure has intermolecular and intramolecular interactions. This novel 1D polymer has promising potential to enhance the performance and functionality of borasiloxane backbone materials. Therefore, further work is recommended to explore some functions and uses of this unique cyclo-1,3,3,5,7,7-hexaphenyl-1,5-dibora-3,7-disiloxane ( $Ph_6B_2Si_2O_4$ ) (3) 1D B-N adduct polymer in areas such as heterogeneous catalyst in the conversion of cooking oil to biodiesel, adsorption of heavy metals from waste water, optoelectronic applications such as Organic Light-Emitting Diodes (OLEDs), Organic Photovoltaics (OPVs) and sensor, energy storage and conversion, biomedical and biotechnology etc.

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#### Supporting information

CCDC-2403230 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif), or by e-mailing [data\\_request@ccdc.cam.ac.uk](mailto:data_request@ccdc.cam.ac.uk), or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

#### Disclosure statement

Conflict of interest: The authors declare that they have no conflict of interest. Ethical approval: All ethical guidelines have been adhered.


Sample availability: Samples of the compounds are available from the author.


#### CRedit authorship contribution statement

Conceptualization: Okpara Sergeant Bull; Conceptualization: Okpara Sergeant Bull; Methodology: Okpara Sergeant Bull; Software: Okpara Sergeant Bull; Validation: Chioma Don-Lawson, Okpara Sergeant Bull; Formal Analysis: Okpara Sergeant Bull; Investigation: Okpara Sergeant Bull; Resources: Chioma Don-Lawson; Data Curation: Okpara Sergeant Bull; Writing - Original Draft: Okpara Sergeant Bull; Writing - Review and Editing: Okpara Sergeant Bull, Chioma Don-Lawson; Visualization: Chioma Don-Lawson; Funding acquisition: Chioma Don-Lawson; Supervision: Chioma Don-Lawson; Project Administration: Okpara Sergeant Bull.


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#### References

- Le, V.; Kim Lien, V. T.; Pham, V. T.; Tran, Q. T.; Thuy, P. T.; Ha, C. V.; Doanh, V. V.; Ha, L. T.; Hanh, C. H.; Thao, P. N.; Luc, N. Effect of extended  $\pi$ -conjugation on photophysical characteristics of chalcone and cinnamylideneacetophenone. *Mater. Sci. Semicond. Process.* **2023**, *162*, 107507.
- Viswanathan, T.; Gopalakrishnan, M.; Thirumoorthy, K.; Prakash, M.; Palanisami, N. Enhancement of Second-Order Nonlinear Optical Properties of Centrosymmetric Ferrocenyl Borasiloxane by a Broken-Symmetry Approach. *J. Phys. Chem. C.* **2021**, *125* (16), 8732–8740.
- Purushothaman, P.; Mohanapriya, D.; Thenmozhi, K.; Karpagam, S. Designing a ferrocene biphenyl pyridine modified electrode for the non-enzymatic electrochemical detection of catechol. *New J. Chem.* **2024**, *48* (15), 6893–6901.
- Bull, O. S.; Don-Lawson, C.; Nweke-Maraizu, U. Synthesis and Characterization of a Novel Eight-Membered Cyclo-1,3,3,5,7,7-Hexaphenyl-1,5-Dibora-3,7-Disiloxane and 4,4'-Bipyridine, 1D Adduct. *Eur. J. Chem.* **2024**, *15* (3), 232–238.
- Bull, O. S.; Don-Lawson, C. Facile Heck coupling synthesis and characterization of a novel tris(4-(pyridine-4-vinyl)phenyl)methyl silane tridentate core. *Eur. J. Chem.* **2024**, *15* (1), 71–73.
- Sergeant Bull, O.; Monsuru Adewale, S.; Okpa, E. Production of Biodiesel from Waste Cooking Oil using A Zinc-Based Metal-Organic Framework (Zn-MOF) As Catalyst. *SSRG-IJAC* **2024**, *11* (1), 1–6.
- Bull, O.; Bull, I.; Amadi, G. Global Warming and Technologies for Carbon Capture and Storage. *Jasem* **2020**, *24* (9), 1671–1686.
- Bull, O.; Bull, I.; Amadi, G.; Odu, C. Covalent Organic Frameworks (COFs): A Review. *J. Appl. Environ. Manag.* **2022**, *26* (1), 145–179.
- Bull, O. S.; George, D. M. C. Assessment of Fuel Properties of Biodiesel Obtained From African Pear (Dacryodeseludis) Seeds Oil. *Int. J. Adv. Res. Sci. Eng. Technol.* **2015**, *2* (10), 894–898.
- Hiscock, L.; Maly, Kenneth E.; Dawe, Louise N., Synthesis, Properties, and Solid-State Structures of a Series of 6,13-Dicyanoheteropentacene Analogues: Towards New Liquid Crystalline Materials Theses and Dissertations (Comprehensive). (2018) 2073. <https://scholars.wlu.ca/etd/2073> (accessed Jan 7, 2024).
- Gopalakrishnan, M.; Thirumoorthy, K.; Bhuvanesh, N. S.; Palanisami, N. Eight membered cyclic-borasiloxanes: synthesis, structural, photophysical, steric strain and DFT calculations. *RSC. Adv.* **2016**, *6* (61), 55698–55709.
- Sheepwash, E.; Zhou, K.; Scopelliti, R.; Severin, K. Self-Assembly of Arylboronate Esters with Pyridyl Side Chains. *Eur. J. Inorg Chem* **2013**, *2013* (14), 2558–2563.
- Beckett, M. A.; Hibbs, D. E.; Hursthouse, M. B.; Malik, K.; Owen, P.; Varma, K. cyclo-Boratrilsiloxane and cyclo-diboratetrasiloxane derivatives and their reactions with amines: crystal and molecular structure of [p-BrC6H4BO]2(Ph2SiO)2. *J. Organomet. Chem.* **2000**, *595* (2), 241–247.
- Bull, O. S.; Okpa, E. Application of Green Chemistry for the One-pot Preparation of Tris (4-bromophenyl) Chlorosilane. *SSRG-IJAC* **2023**, *10* (2), 1–5.
- Bull, O. S. Silicon-Containing COFs and MOFs for CO2 Capture. Imperial College London 2018. <https://doi.org/10.25560/83674> (accessed Jan 7, 2024).
- Gontarczyk, K.; Durka, K.; Klimkowski, P.; Luliński, S.; Serwatowski, J.; Woźniak, K. Synthesis and characterization of di-, tri- and tetraboronic acids based on phenyl- and thienylsilane cores. *J. Organomet. Chem.* **2015**, *783*, 1–9.
- Brisdon, B. J.; Mahon, M. F.; Molloy, K. C.; Schofield, P. J. Synthesis and structural characterization of cycloborasiloxanes: The X-ray crystal structures of cyclo-1,3,3,5,5-pentaphenyl-1-bora-3,5-disiloxane and cyclo-1,3,3,5,7,7-hexaphenyl-1,5-dibora-3,7-disiloxane. *J. Organomet. Chem.* **1992**, *436* (1), 11–22.
- Foucher, D. A.; Lough, A. J.; Manners, I. Synthesis, properties, and the ring-ring transformation reactions of cyclic siloxanes incorporating skeletal boron atoms: x-ray crystal structures of the strained boracycloborasiloxane (PhBO)(Ph2SiO)2 and the boracycloborasiloxane (PhBO)(Ph2SiO)3. *Inorg. Chem.* **1992**, *31* (14), 3034–3043.
- Ferguson, G.; Lawrence, S. E.; Neville, L. A.; O'Leary, B. J.; Spalding, T. R. Synthetic and X-ray diffraction studies of borosiloxane cages [R'Si(ORBO)3SiR'] and the adducts of [BtSi{O(Ph)O}3SiBut] with pyridine or N,N,N',N'-tetramethylethylenediamine. *Polyhedron* **2007**, *26* (12), 2482–2492.
- O'Dowd, A. T.; Spalding, T. R.; Ferguson, G.; Gallagher, J. F.; Reed, D. Synthesis and crystal structure of the novel borosilicate cage compound [B(OSiPh2OSiPh2O)3B]. *J. Chem. Soc., Chem. Commun.* **1993**, 1816–1817.
- Baykov, S. V.; Mikhredov, A. S.; Novikov, A. S.; Geyl, K. K.; Tarasenko, M. V.; Gureev, M. A.; Boyarskiy, V. P.  $\pi$ - $\pi$  Noncovalent Interaction Involving 1,2,4- and 1,3,4-Oxadiazole Systems: The Combined Experimental, Theoretical, and Database Study. *Molecules* **2021**, *26* (18), 5672–5686.
- Tskhovrebov, A. G.; Novikov, A. S.; Odintsova, O. V.; Mikhaylov, V. N.; Sorokoumov, V. N.; Serebryanskaya, T. V.; Starova, G. L. Supramolecular polymers derived from the PtII and PdII Schiff base complexes via C(sp<sup>2</sup>)-H...Hal hydrogen bonding: Combined experimental and theoretical study. *J. Organomet. Chem.* **2019**, *886*, 71–75.
- Nenajdenko, V. G.; Shikhaliyev, N. G.; Maharramov, A. M.; Bagirova, K. N.; Suleymanova, G. T.; Novikov, A. S.; Khrustalev, V. N.; Tskhovrebov, A. G. Halogenated Diazabutadiene Dyes: Synthesis, Structures, Supramolecular Features, and Theoretical Studies. *Molecules* **2020**, *25* (21), 5013–5027.

- [24]. Ferguson, G.; Lough, A. J.; Sheehan, J. P.; Spalding, T. R. Structure of 2-(diphenylmethylsiloxy)-2-phenyl-1,3,2-oxazaborinane. *Acta Crystallogr C. Cryst Struct Commun* **1991**, *47* (2), 379–381.
- [25]. Foucher, D. A.; Lough, A. J.; Manners, I. Synthesis, Properties, and the Ring-Ring Transformation Reactions of Cyclic Siloxanes Incorporating Skeletal Boron Atoms: X-Ray Crystal Structures of the Strained Boracyclobutrisiloxane (PhBO)(Ph<sub>2</sub>SiO)<sub>2</sub> and the Boracyclopentasiloxane (PhBO)(Ph<sub>2</sub>SiO)<sub>3</sub>. *Inorg. Chem.* **1992**, *31* (14), 3034–3043.
- [26]. Arunan, E.; Desiraju, G. R.; Klein, R. A.; Sadlej, J.; Scheiner, S.; Alkorta, I.; Clary, D. C.; Crabtree, R. H.; Dannenberg, J. J.; Hobza, P.; Kjaergaard, H. G.; Legon, A. C.; Mennucci, B.; Nesbitt, D. J. Definition of the hydrogen bond (IUPAC Recommendations 2011). *Pure Appl. Chem.* **2011**, *83* (8), 1637–1641.
- [27]. Sarveswari, S.; Srikanth, A.; Arul Murugan, N.; Vijayakumar, V.; Jasinski, J. P.; Beauchesne, H. C.; Jarvis, E. E. Synthesis, characterization of (3E)-1-(6-chloro-2-methyl-4-phenyl quinolin-3-yl)-3-aryl prop-2-en-1-ones through IR, NMR, single crystal X-ray diffraction and insights into their electronic structure using DFT calculations. *Spectrochim. Acta A: Mol. Biomol. Spectrosc.* **2015**, *136*, 1010–1017.



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