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## **Synthesis and Structural Features of Hydrogen-Bonded Networks Based on The Combination of Tectons Triphenylboroxine Cation and Pt(SCN)<sub>4</sub> Anion Salts**

Fumet Duygu USTUNDAG<sup>1</sup>, Elif GUNGOR<sup>2</sup>, Hulya KARA SUBASAT<sup>3\*</sup>

**ABSTRACT:** In this work, a new compound [C<sub>15</sub>H<sub>16</sub>B<sub>3</sub>N<sub>3</sub>O<sub>5</sub>].[Pt(SCN)<sub>4</sub>].2(H<sub>2</sub>O) (**1**) has been synthesized and structurally characterized. The compound (**1**) crystallizes in monoclinic, space group P2<sub>1</sub>/c a=10.443 Å, b= 24.918 Å, c= 15.048 Å, α=90°, β=133.945°, γ=90°, V=2819.4 Å<sup>3</sup>, Z=4. The use of molecular tecton (building block) able to form thiocyno-based hydrogen-bonded synthons (OH···SCN) to synthesize a diverse range of crystal structures is described. In the crystal structure, molecules are linked by intermolecular SCN···HN and BO···HN hydrogen bonds, forming a three-dimensional network.

**Keywords:** Boroxine compound, single-crystal structure, hydrogen bonds synthons, supramolecular frameworks.

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## INTRODUCTION

In recent years, boronic acid compounds have been widely used in pharmaceuticals, agrochemicals, sensors for carbohydrates, optical and electrochemical sensors, biologically relevant materials, cell manipulation, including capture, culture, and protection of cells, inhibitors of proteases, therapeutic agents, enzyme inhibitors ( Pedireddi and SeethaLekshmi, 2004; Kara et al., 2006; Dai et al., 2010; Whyte et al., 2013; Brooks and Sumerlin, 2016). They are also used in the treatment of cancer with boron neutron capture therapy (BNCT), tumors, HIV, obesity, and diabetes in medicine ( Yang et al., 2003; Cambre and Sumerlin, 2011; Brooks and Sumerlin, 2016). Substituted pyridines are important components of drug candidates (Cai et al., 2002). The first boronic acid-containing anticancer agent Velcade proves increased interest in boronic acid in medicine (Hall, 2011). Boronic acids are rich in hydrogen bond derivatives and perform a wide variety of cross-coupling reactions with metals. Due to these properties, they have attracted great attention and gain importance in supramolecular chemistry (Aakeröy et al., 2005; Hall, 2011). At the same time, boronic acids are important compounds in the definition of molecules and anions in material science and crystal engineering (Braga et al., 2003; Deplano et al., 2004; Aakeröy and Salmon, 2005; Marinaro et al., 2012; Hezil, 2016). Boronic acid compounds are promising building-blocks in crystal engineering, mostly due to the formation of hydrogen bonds that seems to be the main force in producing supramolecular species in the solid phase ( Hall, 2011; Campos-Gaxiola et al., 2017). Ortho-substituted boronic acids seem to be especially prone to the creation of supramolecular structures. Boronic acids are useful building-blocks in the design and synthesis of supramolecular assemblies ( Aakeröy et al., 2005; Yahsi et al., 2015; Neochoritis et al., 2019). Water molecules play an important role as spacer molecules in the resulting supramolecular structure (Rodríguez-Cuamatzi et al., 2009).

Formation of boroxines or/and N–B dative bonds is also useful in crystal engineering (Aakeröy et al., 2005). Boroxines are cyclic trimers of organoboronic acids produced by dehydration of the acids. Boronic acids easily undergo spontaneous dehydration that can take place at purifying conditions or under storage, resulting in cyclic boroxines (Korich and Iovine, 2010; Hall, 2011). Due to this fact, most of the market-available boronic acids contain various amounts of anhydride. As the suppliers claim, the “impurity” does not disqualify the product as the dehydration process is reversible and therefore boronic acids and boroxines can be used interchangeably in most cases ( Rodríguez-Cuamatzi et al., 2009; Hall, 2011) .

For the past few years, our research group has been actively working on the structural and optical characterization of organic molecules containing various aromatic carboxylate and pyridine derivatives (Erkarslan et al., 2016; Coban et al., 2016; Kocak et al., 2017; Erkarslan et al., 2018). We have also reported geometrical and structural features of compounds formatting between pyridinium and bipyridinium cationic tectons and  $[\text{PtCl}_4]^{-2}$ ,  $[\text{PdCl}_4]^{-2}$ ,  $[\text{PtCl}_6]^{-2}$  anionic salts (Kara et al., 2006; 2011). These studies showed that caused the 1D, 2D and 3D networks formation according to the hydrogen bond donor ability of cations due to the isometric and isosteric use of 4-pyridinium cation. It was reported that it played an important role in determining their structure of stereochemistry of hydrogen bond donor groups in cations and caused different behaviours.

In this study, we have synthesized a new compound  $[\text{C}_{15}\text{H}_{16}\text{B}_3\text{N}_3\text{O}_5] \cdot [\text{Pt}(\text{SCN})_4] \cdot 2(\text{H}_2\text{O})$  (**1**) with the salt of the  $[\text{Pt}(\text{SCN})_4]^{-2}$  anion and dehydration of 4-Pyridine boronic acid to boroxine. We explored the use of molecular tecton of thiocyno-based hydrogen-bonded synthons ( $\text{Pt}-\text{SCN} \cdots \text{HO}$ ,  $\text{SCN} \cdots \text{HN}$  and  $\text{BO} \cdots \text{HN}$ ) to synthesis a diverse range of crystal structure.

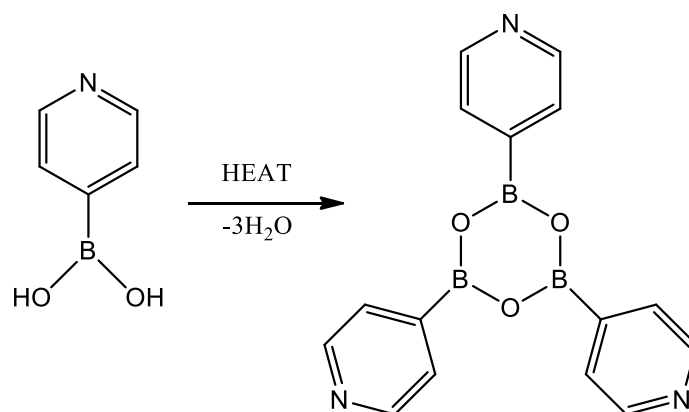
## MATERIALS AND METHODS

### Materials and Measurements

$[\text{C}_{15}\text{H}_{16}\text{B}_3\text{N}_3\text{O}_5] \cdot [\text{Pt}(\text{SCN})_4] \cdot 2(\text{H}_2\text{O})$  (**1**) was synthesized according to the published experimental method (Kara et. al., 2006). The organic compound was recrystallized from concentrated hydrochloric acid to give hydrochloride salts. Single crystal of **1** was obtained by slow diffusion of in stoichiometric quantities of boroxine and metal  $[\text{Pt}(\text{SCN})_4]$  salt reagent. Elemental analysis was performed in the Microanalytical Service of the Chemistry School.

### Synthesis of $[(4\text{-HNC}_5\text{H}_4)_3\text{B}_3\text{O}_3]$ , Boroxine

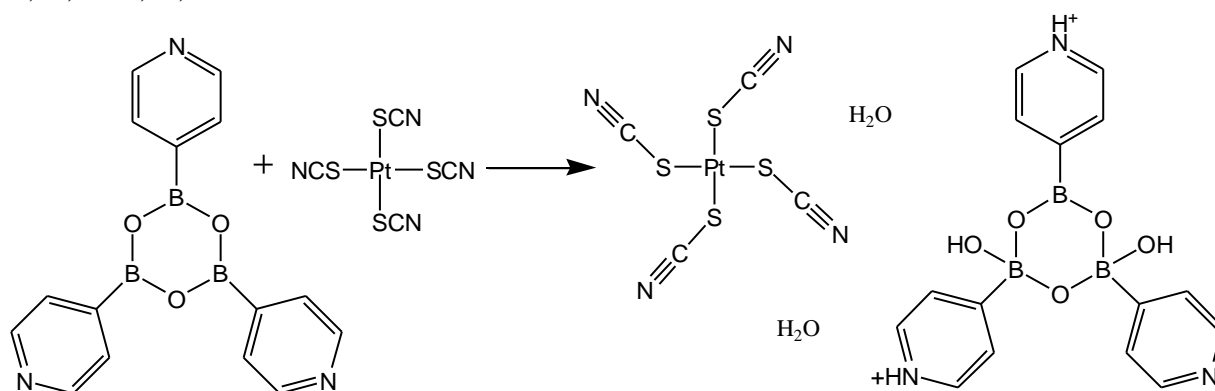
4-Pyridinylboronic acid, (1 mmol) was dissolved in %37 hydrochloric acid solution and water. The solution was maintained at the boiling point with stirring for 1 h. The solvent was evaporated, and the white crystals were obtained upon filtration. The dehydration of 4-Pyridine boronic acid to boroxine is outlined in Scheme 1.



**Scheme 1** The dehydration of 4-Pyridine boronic acid to boroxine

### Synthesis of $[\text{C}_{15}\text{H}_{16}\text{B}_3\text{N}_3\text{O}_5] \cdot [\text{Pt}(\text{SCN})_4] \cdot 2(\text{H}_2\text{O})$ (**1**)

To a solution of  $\text{Pt}(\text{SCN})_4$  (0.4274 mg, 1 mmol) in 10 ml of water, a solution of  $[(4\text{-HNC}_5\text{H}_4)_3\text{B}_3\text{O}_3]$  (0.1229 g, 1 mmol) in 10 ml water was added dropwise with stirring. The orange precipitate was obtained, collected by filtration and dried. The synthetic route of **1** is outlined in Scheme 2. Yield: 55%. Elemental analysis (%). Found: C, 27.89; H, 2.61; N, 12.01. Calculated: C, 27.99; H, 2.59; N, 12.03.



**Scheme 2.** The synthetic route of **1**

## X-Ray Crystallography

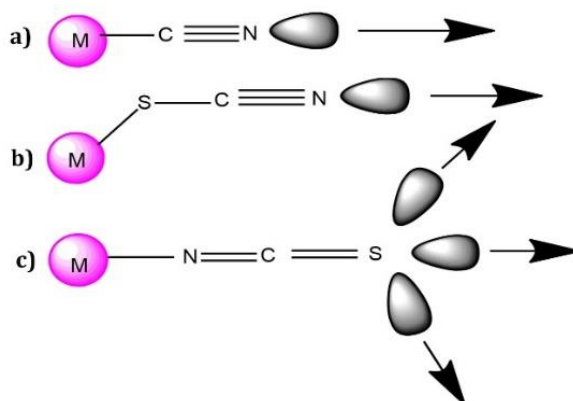
Diffraction measurements were made on three-circle Bruker Apex II Kappa CCD diffractometers using graphite monochromated Mo-K $\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ) at 100 K for **1**. The intensity data were integrated using the SAINT ( Bruker-AXS, 2008) program. The data was collected for Lorentz-polarization and absorption effects (Sheldrick, 2008). Absorption corrections were applied based on equivalent reflections using SADABS (Sheldrick, 2008). The structures were solved by direct methods using SHELXS (Sheldrick, 2008) and were refined with SHELXL (Sheldrick, 2015) using full-matrix least-squares in Olex2 (Dolomanov et al., 2009) program. All non-hydrogen atoms were assigned anisotropic displacement parameters and refined without positional constraints. Hydrogen atoms were included in idealized positions. Crystal data for **1** and its structural refinement parameters are given in Table 1.

**Table 1** Crystal data of **1**

	<b>1</b>
CCDC deposit no.	1993503
Chemical Formula	C <sub>15</sub> H <sub>16</sub> B <sub>3</sub> N <sub>3</sub> O <sub>5</sub> C <sub>4</sub> N <sub>4</sub> PtS <sub>4</sub> .2(H <sub>2</sub> O)
Crystal System	Monoclinic
Space Group	P2 <sub>1</sub> /c
	<i>a</i> = 10.443(2) Å
	<i>b</i> = 24.918(5) Å
	<i>c</i> = 15.048(4) Å
Unit cell parameters	$\alpha = 90^\circ$
	$\beta = 133.945(17)^\circ$
	$\gamma = 90^\circ$
V	2 819.4 (14) Å <sup>3</sup>
T	100 K
Z	4
$\mu$	5.327 mm <sup>-1</sup>
Reflections collected	19 798
Independent reflections (R <sub>int</sub> )	6 459 [R <sub>int</sub> = 0.0695]
Final R <sub>1</sub> [I > 2 $\sigma$ (I)]	0.0640

## RESULTS AND DISCUSSION

Molecular networks are infinite periodic molecular formed under self-assembly conditions between molecular tectons. To create hydrogen bonded 1D, 2D, 3D networks, it is necessary to combine anionic and cationic components. When hydrogen bonding acceptor thiocyanometallate anions and metal centers are combined, they exhibit interesting coordination modes leading to various H-bond patterns (Sheldrick, 2015). Although thiocyanometallate anions and cyanometallate analogs are different sizes and geometries, they are similar hydrogen bonding properties (Figure 1). Although the isothiocyanometallate complexes and cyanometallate anions as H-bond acceptors have similar coordination geometry, nevertheless they offer different possibilities ( Bowmaker et al., 2009; Tan et al., 2013; Otgonbaatar et al., 2015).



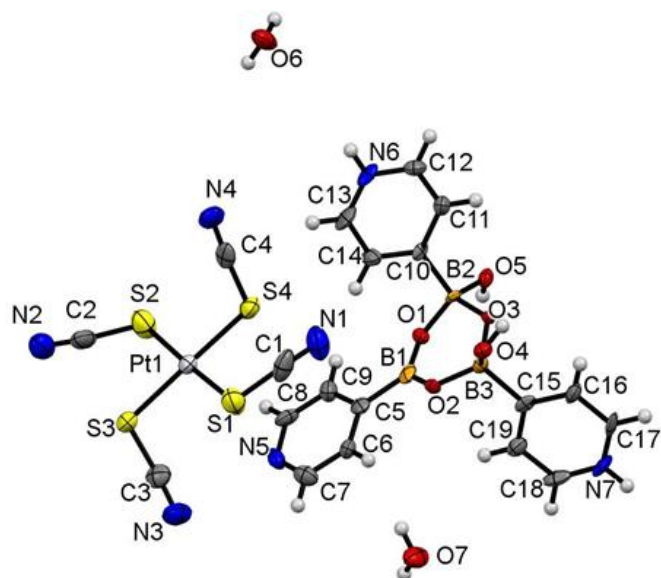
**Figure 1.** Coordination and possible H-bond acceptor modes of cyanide (a) thiocyanate (b) and isothiocyanate (c) metal complexes. The arrow represents the direction of the H-bond.

Compound **1** crystallizes in the monoclinic crystal system and  $P2_1/c$  space group. The asymmetric unit of compound contains a  $[\text{Pt}(\text{SCN})_4]^{2-}$  anion, a boroxine, and two water molecules (Figure 2). The Pt ion in the **1** is four-coordinated by four SCN bounds and located on an inversion centre. The cis S-Pt-S bond angles are between  $83.28(11)^\circ$  and  $95.25(12)^\circ$  for **1** which deviates from the ideal square geometry angle ( $90^\circ$ ). Therefore,  $[\text{Pt}(\text{SCN})_4]^{2-}$  anion of **1** have slightly distorted square planar geometry around the Pt centre with the average Pt-S bond lengths of  $2.32(7)$  Å. The average Pt-C and C≡N bond lengths of compounds are  $1.68(8)$  Å and  $1.15(10)$  Å, respectively. The S-Pt-S angle varies from **1** is between  $83.28(11)^\circ$  and  $95.25(12)^\circ$ . As shown in Table 2, the Pt-S bond is bent with the PtSC angle varying between  $101.30(3)^\circ$  and  $109.60(5)^\circ$ . The NCS angle in the thiocyanide anion of **1** is almost linear with an average  $177.30(8)^\circ$ .

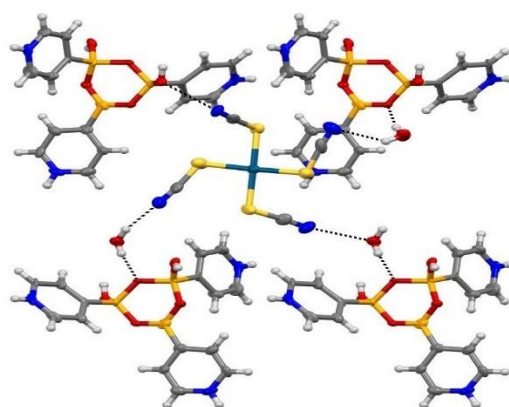
The triphenylboroxin in **1** contains one three-coordinate, two four-coordinate boron. The phenyl rings attached to the three-coordinate B atom in the triphenylboroxin structure of **1** is approximately coplanar with the  $\text{B}_3\text{O}_3$  ring. The average B-O-B bond angles in the  $\text{B}_3\text{O}_3$  ring of **1** is  $124.25(10)^\circ$  which are display deviates from  $120^\circ$  values expected if the bonds are formed from  $sp^2$  hybrid orbitals. The four-coordinate B atoms in the  $\text{B}_3\text{O}_3$  rings of **1** is distorted from planarity, resulting in the formation of two twisted six-membered rings after coordination with OH of B atoms. Therefore, the three-coordinate boron atom has slightly distorted trigonal planar geometry while the four-coordinate boron atoms have slightly distorted tetrahedral geometry.

Compound **1** is formed 1-D chain as infinite ribbons by the mutual interconnection of the cationic and anionic units with hydrogen bonds as shown in Figure 3a. The four ends of the  $[\text{Pt}(\text{SCN})_4]^{2-}$  anion of both compounds are linked to four cations by one Pt-SCN $\cdots$ HO (boroxine) and three Pt-SCN $\cdots$ HO (water) hydrogen bonds. The Pt-SCN $\cdots$ HO (boroxine) and Pt-SCN $\cdots$ HO (water) distances are  $2.838$  Å and  $2.887$  Å for **1** and formed these hydrogen bonds are strong.

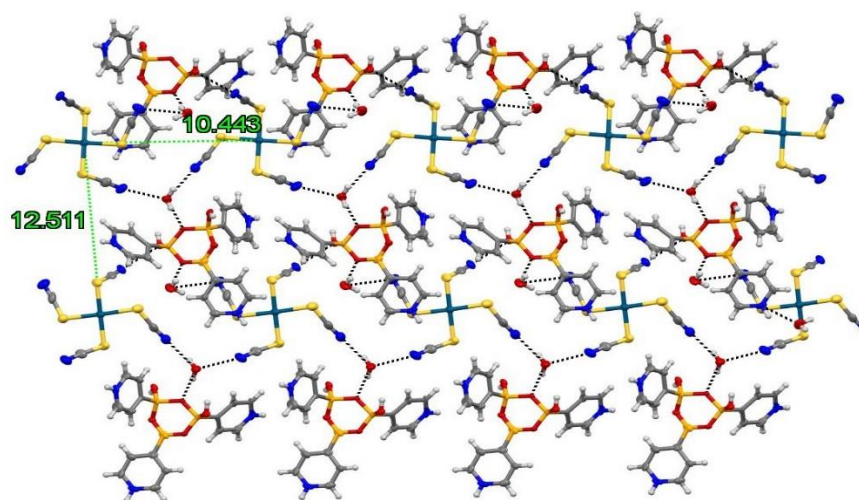
The 1D chain of **1** are arranged as double ribbons in the  $ab$  plane from the interconnection of dications and dianions with O-H $\cdots$ O hydrogen bonds, forming 2D networks (Table 3, Figure 3b). In the structure, 2D sheets are packed in parallel in the  $ab$  plane. The distance between two metal centres within the 1-D networks is  $10.443$  Å for **1** while the distance between two metal centres belonging to two serial chains is observed  $12.511$  Å for **1**. Additionally, ribbon motifs with SCN $\cdots$ HN and BO $\cdots$ HN hydrogen bond interactions can increase the stability of solid-state structure and form 3D networks (Figure 4).



**Figure 2.** The molecular structure of **1**.

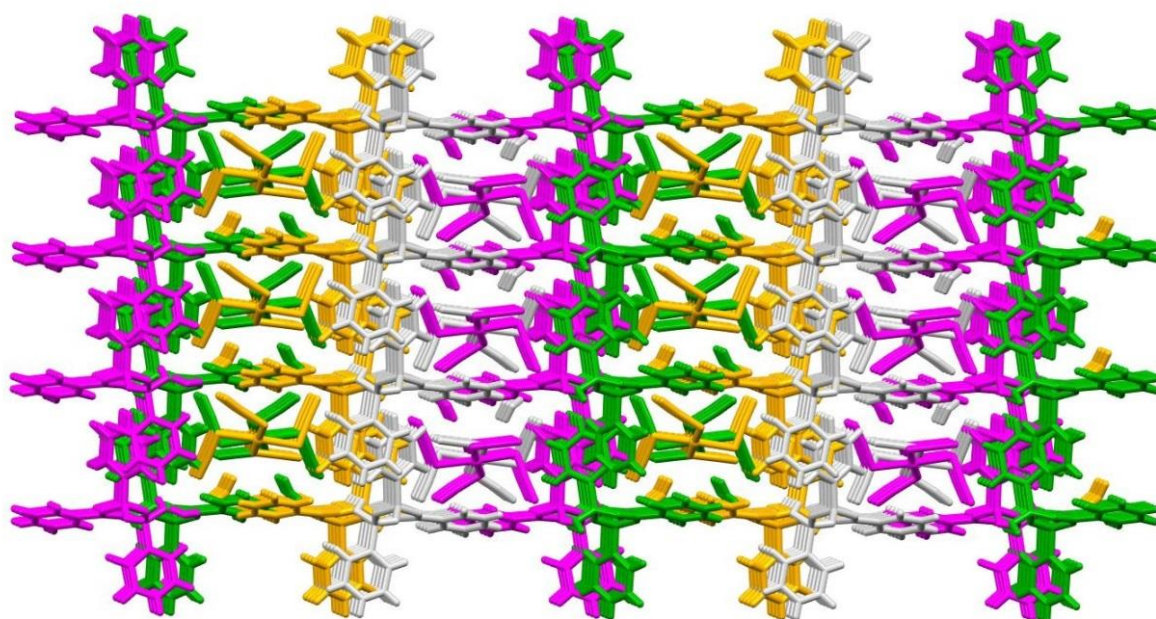


(a)



(b)

**Figure 3.** (a) Part of the crystal structure of formed between  $[\text{Pt}(\text{SCN})_4]^{2-}$  anion and boroxine cation (b) 2D sheets of  $\text{Pt-SCN}\cdots\text{HO}$  (boroxine) and  $\text{Pt-SCN}\cdots\text{HO}$  (water) hydrogen bonded double ribbons in the structure.



**Figure 4.** 3-D packed structure of SCN···HN and BO···HN hydrogen-bonded ribbons of **1**.

**Table 2.** Some bond lengths (Å) and angles (°) for **1**

<b>Bond lengths (Å)</b>			
Pt1–S1	2.297(5)	O1–B2	1.459(9)
Pt1–S2	2.315(5)	O2–B1	1.345(16)
Pt1–S3	2.312(2)	O2–B3	1.462(14)
Pt1–S4	2.329(2)	O3–B2	1.511(16)
S1–C1	1.688(11)	O3–B3	1.541(14)
S2–C2	1.689(13)	N1–C1	1.133(16)
S3–C3	1.690(15)	N2–C2	1.134(18)
S4–C4	1.664(13)	N3–C3	1.144(19)
O1–B1	1.365(13)	N4–C4	1.168(16)
<b>Bond angles (°)</b>			
S2–Pt1–S4	83.28(11)	B1–O1–B2	121.20(8)
S3–Pt1–S4	176.99(10)	B1–O2–B3	124(8)
S1–Pt1–S3	86.85(12)	B2–O3–B3	127.20(7)
S1–Pt1–S4	94.68(11)	O1–B1–O2	124(9)
S1–Pt1–S2	177.62(9)	O1–B2–O3	108(8)
S2–Pt1–S3	95.25(12)	O2–B3–O3	107.60(1)
Pt1–S1–C1	107.10(5)	S1–C1–N1	176.30(1)
Pt1–S2–C2	109.60(5)	S2–C2–N2	174.30(1)
Pt1–S3–C3	103.40(4)	S3–C3–N3	176 (10)
Pt1–S4–C4	101.30(3)	S4–C4–N4	176.20(9)

**Table 3.** Hydrogen bond geometry (Å, °) for **1**.

D–H...A*	D–H	H...A	D...A	D–H...A	Symmetry
O4–H4...N6	0.84	2.38	2.661	100	1-x,1-y,1-z
N5–H5...O7	0.88	2.01	2.729	138	x,y,1+z
N5–H5...N4	0.88	2.61	3.209	126	1+x,3/2-y,1/2+z
O5–H5A...O1	0.84	2.52	2.878	107	2-x,1-y,2-z
N6–H6A...O4	0.88	1.80	2.661	164	1-x,1-y,1-z
O6–H6C...N2	0.87	2.06	2.829	147	-x,-1/2+y,3/2-z
N7–H7A...O5	0.88	1.81	2.661	163	3-x,1-y,2-z
O7–H7B...N1	0.87	2.29	2.910	129	x,3/2-y,-1/2+z
O7–H7C...O2	0.87	2.10	2.961	168	x,3/2-y,-1/2+z
C11–H11...N2	0.95	2.56	3.486	165	1-x,-1/2+y,3/2-z
C12–H12...N1	0.95	2.54	3.311	139	1-x,1-y,1-z
C14–H14...O1	0.95	2.58	2.924	102	
C19–H19...O2	0.95	2.54	2.901	103	

## CONCLUSION

We have demonstrated the generation of hydrogen-bonded networks based on the combination of tectons triphenylboroxine cationic and Pt(SCN)<sub>4</sub> anionic in crystal synthesis. The combination of the boroxine with thiocyanometallate Pt(SCN)<sub>4</sub> leads to the formation of neutral, 1D and 2D, 3D, periodic, H-bonded, infinite architectures. Based on structural features of the cationic and anionic partners, the infinite 1D networks by Pt-SCN...HO (boroxine) and Pt-SCN...HO (water) hydrogen bonds were formed. The formation of the 2D network resulted from the interconnection of dications and dianions through O-H...O hydrogen. 3D networks were generated by the ribbon motifs with SCN...HN and BO...HN hydrogen bond interactions.

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## Conflict of Interest

The article authors declare that there is no conflict of interest between them.

## Author's Contributions

The authors declare that they have contributed equally to the article.

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