

# $[\text{NH}_3\text{CH}_2\text{CHCH}_3\text{NH}_3][\text{B}_8\text{O}_{11}(\text{OH})_4] \cdot \text{H}_2\text{O}$ : Synthesis and characterization of the first 1D borate templated by 1,2-diaminopropane

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## Abstract

A new hydrated borate compound,  $[\text{NH}_3\text{CH}_2\text{CHCH}_3\text{NH}_3][\text{B}_8\text{O}_{11}(\text{OH})_4] \cdot \text{H}_2\text{O}$  **1**, has been synthesized in the presence of 1,2-diaminopropane acting as a structure-directing agent under mild conditions. Its structure was determined by single crystal X-ray diffraction and further characterized FTIR, elemental analysis, powder X-ray diffraction and thermogravimetric analysis. Compound **1** crystallizes in the monoclinic system, space group  $P2_1/c$  (No. 14),  $a = 10.0787(7) \text{ \AA}$ ,  $b = 8.8482(6) \text{ \AA}$ ,  $c = 19.3097(4) \text{ \AA}$ ,  $\beta = 91.352(6)^\circ$ ,  $V = 1721.53(2) \text{ \AA}^3$ , and  $Z = 4$ . The structure consists of infinite open-branched borate chains constructed from  $[\text{B}_3\text{O}_6(\text{OH})]$  units, onto which the  $[\text{B}_5\text{O}_7(\text{OH})_3]$  groups are grafted. It represents the first example of one-dimensional borate templated by an organic amine. The adjacent borate chains are further linked together by extensive hydrogen bonds to form a 3D supramolecular network. The diprotonated organic amines and guest water molecules are filled in the free space of the hydrogen-bonded network and interact with the inorganic framework by extensive hydrogen bonds.

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**Keywords:** Borates; Hydrothermal synthesis; Crystal structure

## 1. Introduction

Borate materials have attracted a great deal of attention in the past decades because of their rich structural chemistry and potential applications in mineralogy and industry [1–5]. From the structural point of view, boron atoms may coordinate with oxygen atoms not only in three-fold coordination (triangular,  $\text{BO}_3$ ) but also in four-fold coordination (tetrahedral,  $\text{BO}_4$ ). And the  $\text{BO}_3$  and  $\text{BO}_4$  groups may be further linked via common oxygen atoms to form polynuclear anions, including isolated rings (or cages), infinite chains, sheets and frameworks [4,6–12]. Burns et al. [2,4] have developed a comprehensive description based on fundamental building blocks (FBBs) to have a clearer nomenclature for the borates with more complicated borate anions.

To date, borate materials with various alkali metal, alkaline earth metal, rare earth and transition metal, traditionally prepared under high temperature/pressure solid-state conditions, have been extensively studied. In contrast, the template synthesis of borate materials is still a particularly unexplored area, though a very few cases have been reported recently [12–24]. The interest of our work is to explore the possibility of devising novel borates with useful properties under various structure directing agents (SDAs), such as inorganic cations, organic amines or transition metal complexes, etc. During our investigations of such borate materials, we apply self-assembly process of organic and inorganic moieties under mild solvothermal conditions, which has recently proved to be very effective. Following the successful introduction of various organic amines, such as 1,4-diaminobutane, diethylenetriamine, triethylenetetramine, *trans*-1,4-diaminocyclohexane and piperazine into borate system, we continue to explore the construction of other interesting borate network in the presence of different organic moieties. Herein, we luckily

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obtained another new organically templated borate compound,  $[\text{NH}_3\text{CH}_2\text{CHCH}_3\text{NH}_3][\text{B}_8\text{O}_{11}(\text{OH})_4] \cdot \text{H}_2\text{O}$  **1**, which represents the first instance of one-dimensional borate templated by an organic amine with  $[\text{B}_8\text{O}_{11}(\text{OH})_4]^{2-}$  as FBBs.

## 2. Experimental section

### 2.1. Synthesis

The title compound was synthesized hydrothermally under autogenous pressure. Typically, the mixture of 0.960 g  $\text{H}_3\text{BO}_3$ , 0.2 mL water and 0.4 mL 1,2-dimino propane, was sealed in a Teflon-lined autoclave and heated at  $180^\circ\text{C}$  for 7 days and then cooled to room temperature. The resulting colorless crystals were recovered by filtration, washed with distilled water, and dried in air (28% yield based on boron). C, H, N analysis: found: C 8.49, H 4.25, N 6.32 wt%; Calculated: C 8.48, H 4.27, N 6.60 wt%. The X-ray powder diffraction pattern for the bulk product is in good agreement with the pattern based on single-crystal X-ray solution in position, indicating the phase purity of the as-synthesized samples of the title compound (Fig. 1). The difference in reflection intensities between the simulated and experimental patterns was due to the variation in preferred orientation of the powder sample during collection of the experimental XRD data.

### 2.2. Characterization

Infrared spectra were obtained from sample powder pelletized with KBr on an ABB Bomen MB 102 series FTIR spectrophotometer over a range  $400\text{--}4000\text{ cm}^{-1}$ . Powder X-ray diffraction (XRD) data were obtained

using a Philips X'Pert-MPD diffractometer with  $\text{CuK}\alpha_1$  radiation ( $\lambda = 1.54076 \text{ \AA}$ ). The elemental analysis was carried out on an Elemental Vario EL III CHNOS elemental analyzer. The thermogravimetric analysis (TGA) was performed on a Mettler Toledo TGA/SDTA 851e analyzer in  $\text{O}_2$  atmosphere with a heating rate of  $5^\circ\text{C}/\text{min}$ .

### 2.3. Determination of crystal structure

A suitable single crystal of as-synthesized compound with the dimensions of  $0.58 \times 0.08 \times 0.05 \text{ mm}^3$  was carefully selected under an optical microscope and glued to thin glass fiber with epoxy resin. Crystal structure determination by X-ray diffraction was performed on a Siemens SMART CCD diffractometer with graphite-monochromated  $\text{MoK}\alpha$  ( $\lambda = 0.71073 \text{ \AA}$ ) in the  $\omega$  scanning mode at room temperature. An empirical absorption correction was applied using the SADABS program [25]. The structure was solved by direct methods using SHELXS-97 [26]. The boron, oxygen, carbon and nitrogen atoms were found in the successive difference Fourier map. The C(3) atom is disordered over two positions with the site-occupation factor (SOF) of 0.7 and 0.3, respectively. All the hydrogen atoms were located in a difference Fourier map and their positions and isotropic displacement parameters were refined. The structures were refined on  $F^2$  by full-matrix least-squares methods using the SHELXL-97 program package [26]. All non-hydrogen atoms were refined anisotropically. Crystallographic data for **1** is presented in Table 1. The final atomic coordinates and the selected bond distances are listed in Tables 2 and 3, respectively. CCDC 610280 for **1** contains the supplementary crystallographic data for this paper.

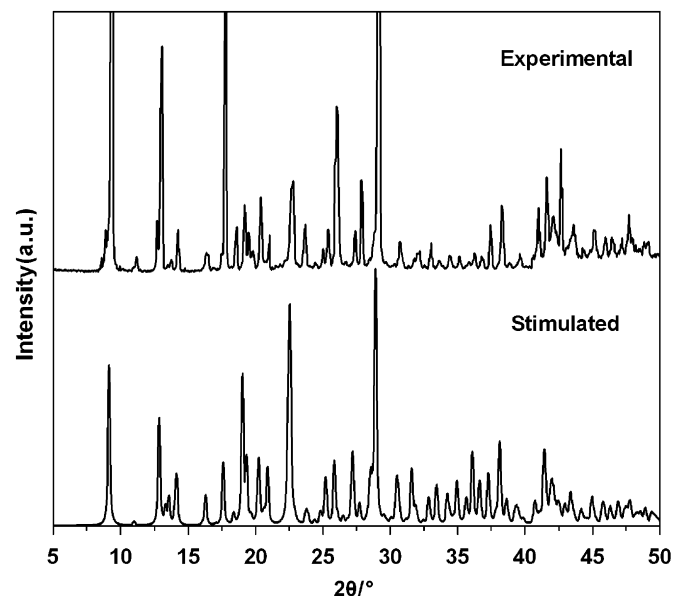


Fig. 1. Experimental and simulated X-ray powder diffraction pattern of **1**.

Table 1  
Crystal data and structure refinement for **1**

Empirical formula	$\text{C}_3\text{H}_{18}\text{N}_2\text{B}_8\text{O}_{16}$
Formula weight	424.67
Crystal system	Monoclinic
Space group	$P2_1/c$
$a$ (Å)	10.0787(7)
$b$ (Å)	8.8482(6)
$c$ (Å)	19.3097(4)
$\alpha$ (°)	90.00
$\beta$ (°)	91.352(6)
$\gamma$ (°)	90.00
$V$ (Å <sup>3</sup> )	1721.53(2)
$Z$	4
$D_c$ (g cm <sup>-3</sup> )	1.639
$\mu$ (Mo- $K\alpha$ ) (mm <sup>-1</sup> )	0.153
$\theta$ range for data collection (°)	3.26–26.50
Reflection collected	12172
Independent reflections	3549 [R(int) = 0.0328]
Parameters refined	327
Limiting indices	$-11 \leq h \leq 12$ , $-10 \leq k \leq 11$ , $-24 \leq l \leq 21$
Goodness-of-fit on $F^2$	1.102
Final $R_1$ , $wR_2$ [ $I > 2\sigma(I)$ ]	0.0476, 0.1254

Table 2

Final atomic coordinates ( $\times 10^4$ ) and equivalent thermal isotropic displacement  $U(\text{eq})$  ( $\text{\AA}^2 \times 10^3$ ) with e.s.d.'s in parentheses of **1**

Atoms	<i>x</i>	<i>y</i>	<i>z</i>	$U(\text{eq})^a$
B(1)	5194(2)	4119(2)	2489(1)	16(1)
B(2)	7429(2)	4638(3)	2702(1)	21(1)
B(3)	3887(2)	1703(2)	2702(1)	16(1)
B(4)	2778(2)	3003(3)	3708(1)	19(1)
B(5)	1488(2)	3693(3)	4678(1)	23(1)
B(6)	1677(2)	5459(3)	3703(1)	19(1)
B(7)	366(2)	7617(3)	3246(1)	20(1)
B(8)	2459(2)	8117(3)	3796(1)	22(1)
O(1)	4207(1)	3114(2)	2353(1)	17(1)
O(1W)	4874(3)	4281(4)	5691(2)	97(1)
O(2)	6381(1)	3639(2)	2793(1)	29(1)
O(3)	8571(2)	4022(2)	3131(1)	33(1)
O(4)	4976(1)	592(2)	2693(1)	17(1)
O(5)	7307(1)	6101(2)	2680(1)	21(1)
O(6)	3559(1)	1921(2)	3431(1)	21(1)
O(7)	2426(2)	2764(2)	4395(1)	30(1)
O(8)	1049(2)	3303(2)	5319(1)	36(1)
O(9)	1020(1)	4921(2)	4337(1)	24(1)
O(10)	2338(1)	4235(2)	3359(1)	23(1)
O(11)	2651(1)	6627(2)	3902(1)	26(1)
O(12)	625(1)	6112(2)	3246(1)	21(1)
O(13)	-808(2)	8181(2)	2999(1)	28(1)
O(14)	1286(1)	8649(2)	3502(1)	27(1)
O(15)	3440(2)	9092(2)	3993(1)	31(1)
C(1)	3447(3)	4827(3)	756(1)	40(1)
C(2)	2404(3)	5906(3)	1014(1)	42(1)
C(3)	1081(4)	5826(5)	687(2)	49(1)
C(3')	2500(13)	7217(13)	663(6)	64(3)
N(1)	3256(2)	3229(3)	965(1)	37(1)
N(2)	2317(2)	5861(2)	1788(1)	27(1)

<sup>a</sup> $U(\text{eq})$  is defined as one-third of the trace of the orthogonalized  $U_{ij}$  tensor.

### 3. Results and discussion

#### 3.1. Infrared (IR) spectrum

The FTIR spectrum of the title compound is shown in Fig. 2. The broad bands at  $3250\text{--}2922\text{ cm}^{-1}$  correspond to the stretching bands of the  $\text{NH}_2$  and  $\text{CH}_2$ . The bending bands of  $\text{NH}_2$  and  $\text{CH}_2$  are present at the about  $1625\text{--}1440\text{ cm}^{-1}$ . The strong bands at  $\sim 1312, 1268\text{ cm}^{-1}$  in the spectra are consistent with the existence of trigonally coordinated boron, while the bands at 1038, 1060, 940 and  $950\text{ cm}^{-1}$  are characteristic of tetrahedral boron [27]. In addition, the stretching vibration of the O–H is observed at  $\sim 3507$  and  $3287\text{ cm}^{-1}$ , respectively.

#### 3.2. Crystal structure

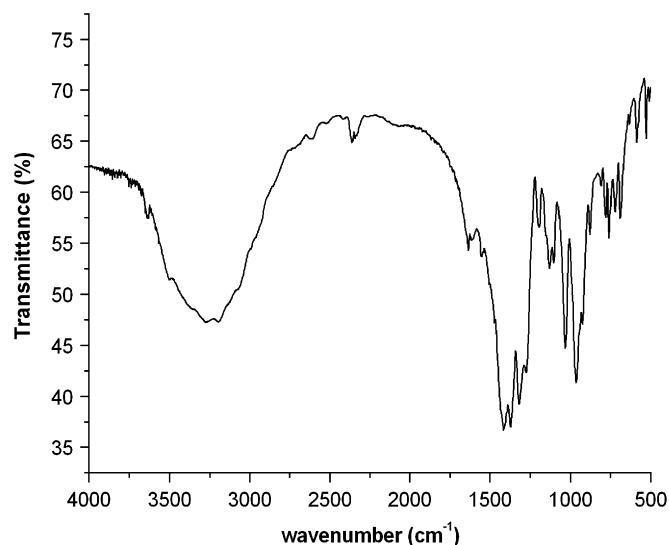
The asymmetric unit of the title compound contains 29 independent non-hydrogen atoms, including 8 boron atoms, 16 oxygen atoms, 3 carbon atoms and 2 nitrogen atoms, as shown in Fig. 3. Of the eight unique B atoms, B(3) and B(6) both adopt tetrahedral oxygen coordination,

Table 3

Selected bond length ( $\text{\AA}$ ) and angles ( $^\circ$ ) for **1**

B(1)–O(1)	1.355(3)	B(5)–O(9)	1.350(3)
B(1)–O(4)	1.360(3)	B(5)–O(8)	1.367(3)
B(1)–O(2)	1.387(3)	B(5)–O(7)	1.375(3)
B(2)–O(5)	1.346(3)	B(6)–O(10)	1.442(3)
B(2)–O(3)	1.365(3)	B(6)–O(11)	1.470(3)
B(2)–O(2)	1.382(3)	B(6)–O(12)	1.481(3)
B(3)–O(1)#1	1.459(2)	B(6)–O(9)	1.484(3)
B(3)–O(6)	1.465(3)	B(7)–O(12)	1.358(3)
B(3)–O(4)	1.474(2)	B(7)–O(13)	1.360(3)
B(3)–O(5)	1.494(2)	B(7)–O(14)	1.384(3)
B(4)–O(10)	1.350(3)	B(8)–O(11)	1.347(3)
B(4)–O(6)	1.358(3)	B(8)–O(15)	1.360(3)
B(4)–O(7)	1.398(3)	B(8)–O(14)	1.382(3)
O(1)–B(1)–O(4)	119.44(18)	O(9)–B(5)–O(8)	121.9(2)
O(1)–B(1)–O(2)	120.21(18)	O(9)–B(5)–O(7)	121.5(2)
O(4)–B(1)–O(2)	120.32(18)	O(8)–B(5)–O(7)	116.7(2)
O(5)–B(2)–O(3)	123.66(19)	O(10)–B(6)–O(11)	109.53(16)
O(5)–B(2)–O(2)	121.70(18)	O(10)–B(6)–O(12)	110.52(17)
O(3)–B(2)–O(2)	114.64(19)	O(11)–B(6)–O(12)	110.23(17)
O(1)#1–B(3)–O(6)	112.87(16)	O(10)–B(6)–O(9)	111.06(17)
O(1)#1–B(3)–O(4)	113.09(15)	O(11)–B(6)–O(9)	108.58(17)
O(6)–B(3)–O(4)	106.49(15)	O(12)–B(6)–O(9)	106.87(16)
O(1)#1–B(3)–O(5)	105.12(15)	O(12)–B(7)–O(13)	121.70(19)
O(6)–B(3)–O(5)	108.90(15)	O(12)–B(7)–O(14)	121.33(18)
O(4)–B(3)–O(5)	110.35(15)	O(13)–B(7)–O(14)	116.97(19)
O(10)–B(4)–O(6)	124.0(2)	O(11)–B(8)–O(15)	118.55(19)
O(10)–B(4)–O(7)	120.50(18)	O(11)–B(8)–O(14)	120.92(19)
O(6)–B(4)–O(7)	115.49(18)	O(15)–B(8)–O(14)	120.5(2)

<sup>a</sup>Symmetry transformations used to generate equivalent atoms: #1  $-x+1, y+1/2, -z-1/2$ .

Fig. 2. IR spectrum of **1**.

while the remaining six B atoms adopt trigonal oxygen coordination. The connectivity of the triangular  $\text{BO}_3$  and tetrahedral  $\text{BO}_4$  units creates the octoborate anion  $[\text{B}_8\text{O}_{11}(\text{OH})_4]^{2-}$ , as also found in  $\text{M}_2[\text{B}_8\text{O}_{13}]$  ( $M = \text{Na}, \text{Ag}$ ) [10,28]. It is formed by a triborate  $[\text{B}_3\text{O}_6(\text{OH})]$  unit constituted by two  $\text{BO}_3$  triangles (B(1) and B(2)) and one  $\text{BO}_4$  tetrahedron (B(3)) linked to a pentaborate

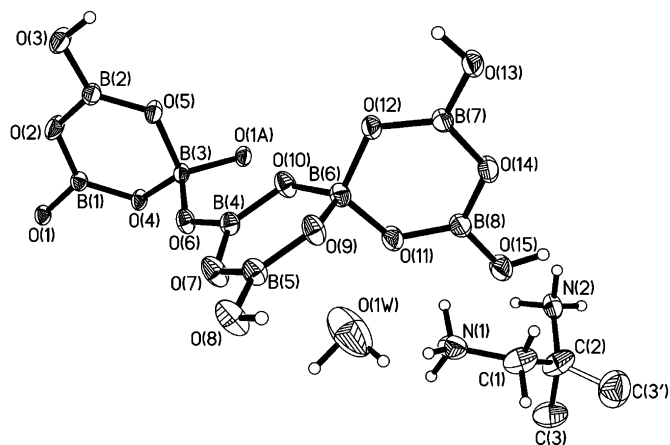


Fig. 3. ORTEP view of **1** showing the atom-labeling scheme and 50% thermal ellipsoids. Atom labels having “A” refer to symmetry-generated atoms.

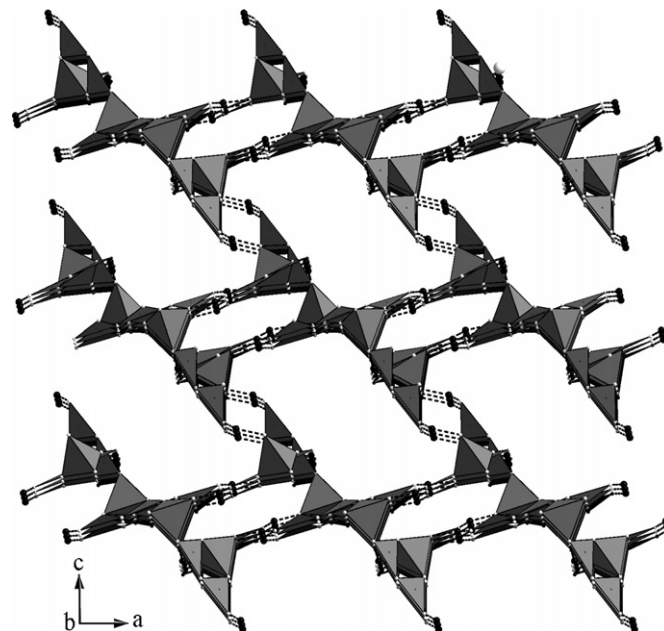


Fig. 5. View of the packing structure of **1** along the *b*-axis.

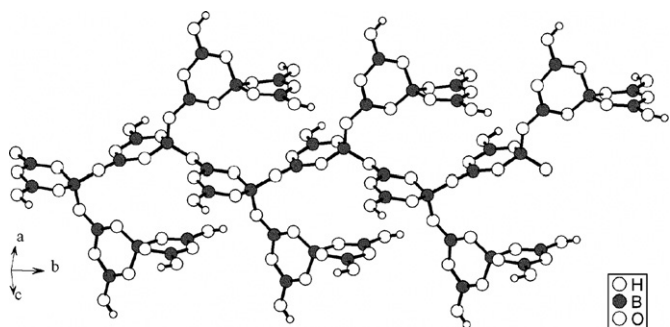


Fig. 4. View of the open-branched borate chains constructed from  $[B_3O_6(OH)]$  units, onto which  $[B_5O_7(OH)_3]$  groups are grafted.

$[B_5O_7(OH)_3]$  group, which consists of four  $BO_3$  triangles (B(4), B(5), B(7) and B(8)) and one  $BO_4$  tetrahedron (B(6)). According to the crystal chemical classification scheme proposed by Christ and Clark [29] and Heller [30], the shorthand notation for  $[B_8O_{11}(OH)_4]^{2-}$  can be written as “8:  ${}^1_{\infty}[(3:2 \Delta + T) + (5:4 \Delta + T)]$ ”. The B–O bond lengths and O–B–O bond angles are in the range of 1.346(3)–1.494(3) Å and 105.1(2)–124.0(2)°, which are in good agreement with those reported previously for other borate compounds.

The structure of **1** consists of infinite one-dimensional borate chains, which are further linked together to form a three-dimensional supramolecular network by extensive H-bonding. As shown in Fig. 4, the  $[B_8O_{11}(OH)_4]^{2-}$  anions are jointed together through the exocyclic oxygen atoms O(1), to form a novel open-branched anion chain  $\infty \{ [B_8O_{11}(OH)_4]^{2-} \}$ . Interestingly, only the  $[B_3O_6(OH)]$  units in the FBBs are responsible for the expansion of the borate chains. The  $[B_5O_7(OH)_3]$  groups, however, act as the decoration of the chains and protrude alternately above and below the borate chains through the common O(6) atoms. To the best of our knowledge, such a chain-like borate architecture has not been found in any other borates. Besides, it is also worthy to note that the title compound represents the first example of one-dimensional

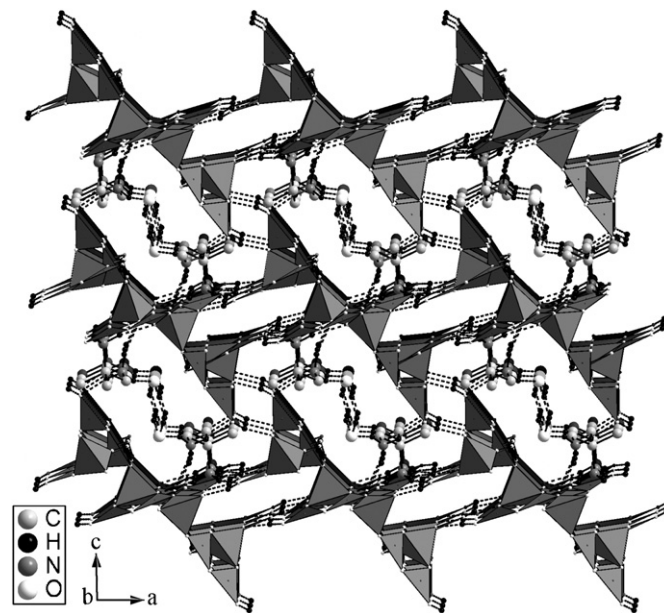


Fig. 6. View of the diprotonated organic amines and guest water molecules in the inorganic borate network along the  $[010]$  directions.

borate with an organic amine as template. Such adjacent borate chains are further connected with each other through the strong O–H⋯O hydrogen bonds, forming a three-dimensional framework with rhombus-like channels (Fig. 5). The guest water molecules and diprotonated organic molecules, however, reside in the channels and further interact with the inorganic framework through hydrogen bonding interactions as O(1W)–H(14)⋯O(1W) [ $d = 2.08$ ], N(1)–H(7)⋯O(1) [ $d = 1.88$ ], N(1)–H(5)⋯O(1W) [ $d = 1.97$ ], N(1)–H(6)⋯O(8) [ $d = 1.92$ ], N(2)–H(10)⋯O(3) [ $d = 2.14$ ], N(2)–H(11)⋯O(4) [ $d = 1.97$ ],

Table 4  
Details of Hydrogen Bonds for **1**

D–H...A	$d(\text{D–H})$ (Å)	$d(\text{H...A})$ (Å)	$d(\text{D...A})$ (Å)	$\angle(\text{DHA})$ (°)
N(2)–H(11)...O(4) [#1]	0.95(4)	1.97(4)	2.895(2)	163(3)
N(2)–H(12)...O(13) [#1]	0.97(3)	1.92(3)	2.852(3)	161(2)
N(1)–H(5)...O(1W) [#2]	0.93(4)	1.97(4)	2.814(4)	149(3)
O(1W)–H(14)...O(1W) [#3]	1.049(2)	2.08(2)	2.971(7)	141(2)
O(3)–H(1)...O(12) [#4]	0.98(4)	1.81(5)	2.781(2)	176(4)
O(13)–H(3)...O(5) [#5]	0.90(4)	1.82(4)	2.705(2)	167(3)
O(15)–H(4)...O(6) [#6]	0.88(3)	1.87(4)	2.733(2)	165(3)
N(1)–H(6)...O(8) [#7]	0.97(4)	1.92(4)	2.866(3)	167(3)
N(1)–H(7)...O(1)	1.00(4)	1.88(4)	2.826(3)	158(3)
O(8)–H(2)...O(9) [#8]	0.90(4)	1.82(4)	2.707(2)	167(4)
N(2)–H(12)...O(13) [#9]	0.97(3)	1.92(3)	2.852(3)	161(2)

[#1]  $-x+1, y+1/2, -z+1/2$ ; [#2]  $-x+1, y-1/2, -z+1/2$ ; [#3]  $-x+1, -y+1, -z+1$ ; [#4]  $x+1, y, z$ ; [#5]  $x-1, y, z$ ; [#6]  $x, y+1, z$ ; [#7]  $x, -y+1/2, z-1/2$  [#8]  $-x, -y+1, -z+1$ ; [#9]  $-x, y-1/2, -z+1/2$ .

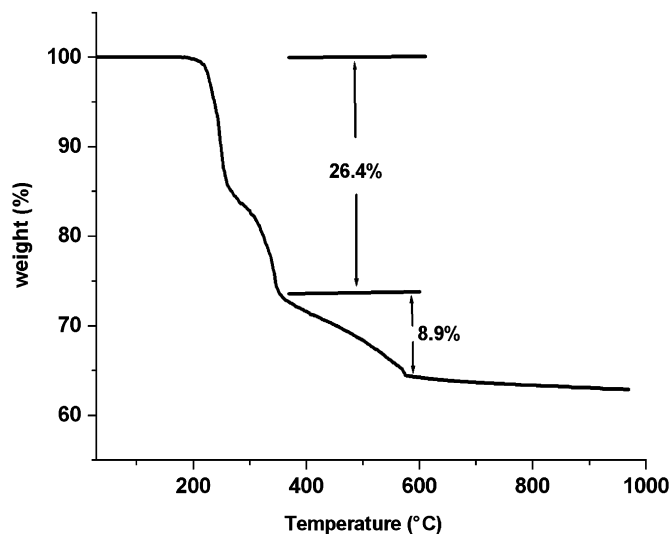


Fig. 7. TG curve of **1**.

N(2)–H(12)...O(13) [ $d = 1.92$ ], as shown in Fig. 6. The details of H-bonds are given in Table 4.

### 3.3. Thermal property

Thermogravimetric analysis (TGA) was performed in dry O<sub>2</sub> atmosphere from 40 to 1000 °C with a heating rate of 5 °C/min. As shown in Fig. 7, the TG curve of **1** showed that the compound was stable up to about 210 °C. On further heating, a two-step weight loss was observed. The initial weight loss between 210 and 352 °C corresponds to the removal of organic amine and two water molecules (observed: 26.4%; expected: 25.9%). The second step, occurring between 352 and 580 °C, is attributed to the dehydration process of hydroxyls (observed: 8.9%; expected: 8.5%).

## 4. Conclusions

In summary, we have described the synthesis, crystal structure and thermal property of new organically templated borates [NH<sub>3</sub>CH<sub>2</sub>CHCH<sub>3</sub>NH<sub>3</sub>][B<sub>8</sub>O<sub>11</sub>(OH)<sub>4</sub>]·H<sub>2</sub>O. The structure features an unusual open-branched borate chains constructed from [B<sub>3</sub>O<sub>6</sub>(OH)] groups, onto which the [B<sub>5</sub>O<sub>7</sub>(OH)<sub>3</sub>] groups are grafted. Adjacent borate chains are linked together by multipoint hydrogen bonds to form interesting three-dimensional network with rhombus-like channels, in which the guest water and diprotated organic amine molecules reside. It represents the first example of one-dimensional borate with an organic amine as template and considered many kinds of organic amines that can be chosen as template agents, it is expected that more borate materials with novel structural features will be generated in the near future. And further work on this subject is in progress.

## Acknowledgments

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## Appendix A. Supplementary Materials

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jssc.2007.01.038.

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