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Synthesis, thermal properties and structural characterisation, of the tetraphenylphosphonium pentaborate salt, $[PPh_4][B_5O_6(OH)_4]\cdot 1.5H_2O$

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ABSTRACT

$[Ph_4P][B_5O_6(OH)_4]\cdot 1.5H_2O$ (**1**) was obtained as colourless crystals in high yield from a MeOH/H_2O solution of $B(OH)_3$ and [PPh$_4$][OH] (5:1). A single-crystal X-ray study confirmed that the solid-state structure was comprised of supramolecular networks of interconnected anions (H-bonds) and cations (phenyl embraces). Crystals are triclinic, $P\bar{1}$ with $a = 13.3174(3)$ Å, $b = 13.3180(3)$ Å, $c = 16.2055(4)$ Å, $\alpha = 91.1950(10)°$, $\beta = 91.180(2)°$, $\gamma = 105.456(2)°$, $T = 120(2)$ K, $V = 2768.66(11)$ Å^3 and $Z = 4$. TGA/DSC analysis showed that **1** thermally decomposed in air at 800 °C to $2.5B_2O_3$, *via* a low temperature dehydration step to a condensed pentaborate, $[PPh_4][B_5O_8]$.

1. Introduction

Inorganic borate minerals are of great industrial importance, with applications as flame retardant [1], luminescence [2], piezoelectric [3], second harmonic generation [4], and non-linear optical materials [5,6]. Metal borates have a richly diverse structural chemistry [7–9], and non-metal cation based borates may serve as thermal precursors to new porous materials [10–12]. In the solid-state, non-metal cation pentaborate salts form H-bonded supramolecular anionic giant structures containing the cations within the cavities of the lattice [13–15]. The $[Ph_4P]^+$ cation is often used to aid the crystallisation of large inorganic anions [16], and the structures which result often contain a supramolecular network made up of multiple structure directing phenyl-phenyl embraces [17,18]. This manuscript details the synthesis and X-ray characterisation of the tetraphenylphosphonium pentaborate salt, $[PPh_4][B_5O_6(OH)_4]\cdot 1.5H_2O$ (**1**), and discusses the anion–anion and cation–cation structure directing effects present. The thermal properties of **1** are also described.

2. Experimental

2.1. Material and analyses

All chemicals were obtained commercially from Sigma Aldrich Chemical Company or Alfa Aesar Chemicals and were used as supplied. NMR spectra were obtained on a Bruker Avance-500 using XWIN-NMR 3.5. ^{11}B, ^{13}C and ^{31}P, ^{1}H NMR spectra were obtained at 500 MHz (^{1}H), 160 MHz (^{11}B), 125 MHz (^{13}C), 202 MHz (^{31}P), dissolved in D_2O. Fourier transform Infrared spectra (FTIR) were obtained as KBr pellets on a Perkin-Elmer 100 FTIR spectrometer over 450–4000 cm^{-1}. TGA and DSC analysis was performed between 10 and 800 °C (in air) on an SDT Q600 V4.1 Build 59 instrument using Al_2O_3 crucibles, with a ramp temperature rate of 10 °C min^{-1}. X-ray crystallography was carried out at the EPSRC National Crystallography service at the University of Southampton. CHN analysis was carried out at OEA laboratories Ltd., Callington, Cornwall.

2.2. Preparation of tetraphenylphosphonium pentaborate (**1**)

Tetraphenylphosphonium bromide (1.00 g, 2.38 mmol) was dissolved in distilled water (15 ml), to which excess Dowex 550A monosphere ion exchange resin (OH^- form) was added. The solution was stirred for 24 h, the ion exchange resin removed by filtration, and methanol (15 ml) was added to the filtrate. Boric acid (0.74 g, 11.92 mmol) was added to the H_2O/MeOH solution which was gently warmed for 1 h. The solvent was concentrated and crystals of **1** (0.56 g, 42%) precipitated from solution. The crystals were removed by filtration and allowed to dry in a desiccator. Re-crystallization of the crude product from distilled water yielded crystals suitable for single-crystal X-ray diffraction studies. **1**: $C_{24}H_{27}B_5O_{11.5}P$. *Anal.* Calc.: C = 49.31%, H = 4.67%. Found: C = 49.45%, H = 4.65%. m.p. 250 °C. NMR: (δ ^{1}H/ppm): 7.57

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(4H, s), 7.77 (1H, dd); (^{11}B/ppm) 1.2, 13.0, 17.7: (^{13}C/ppm) 117.45, 118.12, 130.08, 134.62, 135.23, 135.25; (^{31}P/ppm) 22.7. IR: ν_{max}/cm^{-1} 3476, 3416.1, 3069, 2346, 1638.2, 1617, 1442, 1361, 1107, 1026, 925, 782, 760, 721, 693, 662, 529.

2.3. X-ray crystallography

Crystallisation of **1** from distilled water yielded crystals in the triclinic space group *P*1. Numerical details of the solution and refinement of its X-ray structure are described in Table 1. A suitable crystal was selected and data collected on a Bruker Nonius KappaCCD Area Detector at the window of a Bruker Nonius FR591 rotating anode (Mo Kα = 0.71073 Å) driven by COLLECT [19] and DENZO [20] software at 120 K. The structures were determined in SHELXS-97 [21] and refined using SHELXL-97 [21]. All non-hydrogen atoms were refined anisotropically, with all non-water hydrogen atoms placed geometrically using standard riding models. The water hydrogen atoms were restrained to a fixed geometry but with free rotation of the water molecule. The crystal used was a merohedral, inversion twin (matrix 0 −1 0 −1 0 0 0 0 1) in the following ratio (18.86(7): 18.35(11):30.87(7):31.92(11)).

3. Results and discussion

3.1. Synthesis

The title compound, $[Ph_4P][B_5O_6(OH)_4]\cdot 1.5H_2O$ (**1**), was obtained as analytically pure colourless crystals in high yield from a MeOH/H_2O solution of $[PPh_4][OH]$ and $B(OH)_3$ in the molar ratio 1:5. The salt was characterized by NMR (^{11}B, ^{13}C, and ^{1}H) and IR spectroscopy, and by a single-crystal XRD study. Spectroscopic data are in agreement with the single-crystal XRD structure (see below) and with previously reported NMC pentaborates [12,13].

3.2. Crystal structure of $[PPh_4][B_5O_6(OH)_4]\cdot 1.5H_2O$

Compound **1** is ionic, comprised of discrete $[PPh_4]^+$ cations with $[B_5O_6(OH)_4]^-$ anions, has 1.5 molecules of interstitial water per formula unit, and displays supramolecular networks of interconnected anions (H-bonds) and cations (phenyl embraces). The structure is best refined in the space group *P*1, although there exists high degrees of pseudo-symmetry (~90%) for inversion centres, 2-fold rotations and translations, meaning the structure can be solved to a reasonable degree in either $P\bar{1}$ or C2, but with extra disorder observed instead. The reasons for these apparent symmetry anomalies lie in the supramolecular networks described below. There are four independent cations and four independent anions per unit cell, and a total of six interstitial H_2O molecules. One of the anions, together with the H_2O molecules associated with it, is disordered. The anion containing B131 [occupancy factor 0.675(5)], is disordered over two sites with the alternative anion containing B31 [occupancy factor 0.325(5)]. Two of the interstitial water molecules (O201 and O202) have an occupancy factor of 1, whereas the other four water molecules are disordered over eight sites with an occupancy of 0.325(5) [O203, O204, O205, and O206] and 0.675(5) [O303, O304, O305, and O306] and are associated with either the pentaborate anion containing B31 or B131, respectively. A drawing of the formula unit of **1** (containing B1 and P31) is shown in Fig. 1. The anions containing B11, B21 and B131 (and B31) are very similar in structure to the one containing B1, and the cations containing P1, P61 and P91 are very similar in structure to the cation containing P31.

Table 1
Crystallographic and structure refinement parameters for **1**.

Empirical formula	$C_{24}H_{27}B_5O_{11.5}P$
Formula weight	584.48
T (K)	120(2)
Crystal system	triclinic
Space group	*P*1 Merohedral twin ratio: 18.86(7):18.35(11):30.87(7):31.92(11)
Twin law	(0 −1 0 −1 0 0 0 0 −1)
a (Å)	13.3174(3)
b (Å)	13.3180(3)
c (Å)	16.2055(4)
α (°)	91.1950(10)
β (°)	91.180(2)
γ (°)	105.456(2)
V (Å^3)	2768.66(11)
Z	4
D_c (g cm^{-3})	1.402
μ (mm^{-1})	0.160
F(000)	1212
Crystal size (mm)	0.14 × 0.10 × 0.05
2θ max (°)	54.96
Reflections collected	47412
Independent reflections [R_{int}]	22986 [0.0444]
Completeness (%)	99.3
Data/restraints/ parameters	22986/965/1577
Goodness-of-fit on F^2	1.098
Final *R* indices [$F^2 > 2\sigma(F^2)$]	R_1 = 0.0667, wR_2 = 0.1194
R indices (all data)	R_1 = 0.0979, wR_2 = 0.1368
Max, min difference (eÅ^{-3})	0.370, −0.310

Molecular dimensions within the cations and anions (exemplified with data for ions illustrated in Fig. 1) are discussed before the crystal supramolecular architecture. The distance between the *ipso* C of the phenyl rings and P31 range from 1.790(11) to 1.821(14) Å (av. C-P31 1.805 Å) and, when all P–C bonds considered the range increases to 1.761(11)–1.821(11) Å. Bond angles about P31 range from 107.1(5) to 111.4(5)° (av. 109.5°) with angles about all P centres in the range 106.4(4)–112.3(5)°, confirming tetrahedral P atoms. These data, and C–C bond lengths and C–C–C bond angles within the cation's phenyl rings are in accordance with previously reported bond distances and angles for the $[PPh_4]^+$ cation [22].

The pentaborate anion, as illustrated in Fig. 1, contains one 4-coordinate B centre and four 3-coordinate B centres, with all B atoms bound to either four or three O atoms, respectively. The B–O distances to the 4-coordinate B1 range from 1.457(9) to 1.499(9) Å (av. 1.471 .Å) with corresponding data for all 4-coordinate B centres of 1.427(10)–1.508(15) Å (av. 1.468 Å). The OBO angles at B1 range from 108.2(7) to 110.5(6)° (av. 109.45°) with corresponding data for all 4-coordinate B centres of 105.0(12)–112.1(7)° (av. 109.45°) consistent with (distorted) sp^3 hybridized tetrahedral B centres. The B–O bond-lengths at trigonal B centres within the anion containing B1 range from 1.331(9) to 1.401(9) Å (av. 1.366 Å) with corresponding data for all 3-coordinate B centres of 1.322(13)–1.434(9) Å (av. 1.367 Å). These bond distances are generally significantly shorter than those involving 4-coordinate B centres. OBO at trigonal B centres and O ring angles (BOB) within the anion containing B1 range within 116.5(8)–123.9(7)° (av. 120.0°) and 119.0(6)–125.3(7)° (av. 122.5°) respectively. The variation in these bond-lengths and angles are in accord with bond lengths and angles that have been previously reported in pentaborate [12–14,23,24] and related [25–31] structures.

As noted above, the supramolecular structure of **1** consists of two interpenetrating networks of interconnecting anions (H-bonds) and cations (phenyl embraces). A common feature of pentaborate structures is an H-bonded anion-anion lattice with cations situated within the cavities. Each pentaborate anion is able to form 4 donor

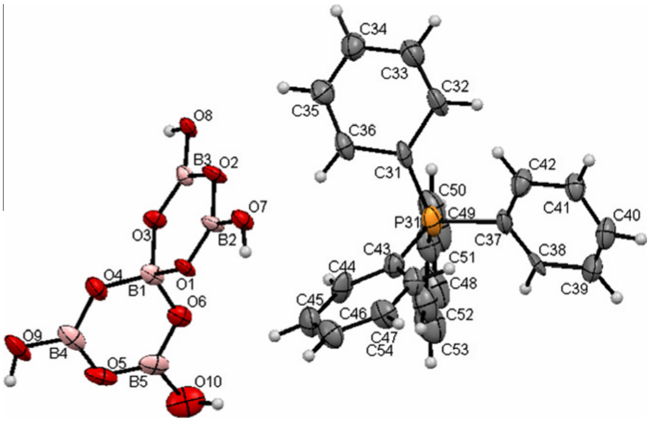

Fig. 1. ORTEP drawing of the cation containing P31 and the anion containing B1 of $[PPh_4][B_5O_6(OH)_4]$ (**1**) showing numbering scheme. The unit cell of **1** contains four independent cations and four independent anions, one of which is disordered. The numbering schemes for the other cations (containing P1, P61, and P91) and anions (containing B11, B21, and B31/B131) follow a similar pattern.

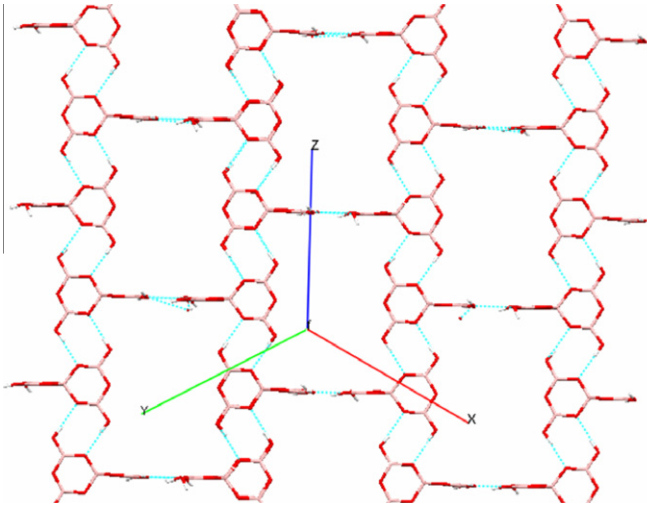

Fig. 2. A view of the supramolecular $[B_5O_6(OH)_4]^-$ anion network in **1**.

Table 2
Details of hydrogen bonds for $[PPh_4][B_5O_6(OH)_4]\cdot 1.5H_2O$.

D–H…*A*	*d*(*D*–H)	*d*(H…*A*)	*d*(*D*…*A*)	∠(*D*H*A*)
O7–H7…O21^{i}	0.84	1.88	2.714(9)	173
O8–H8…O33^{ii}	0.84	2.06	2.776(15)	142.8
O8–H8…O133^{ii}	0.84	2.14	2.709(10)	124.6
O9–H9…O139	0.84	2.19	2.835(8)	133.8
O9–H9…O206	0.84	2.27	3.059(11)	157.3
O10–H10…O201	0.84	2.44	2.919(8)	117.4
O17–H17…O31^{iii}	0.84	1.89	2.723(16)	170.5
O17–H17…O131^{iii}	0.84	1.94	2.756(11)	164.5
O18–H18…O23^{ii}	0.84	1.91	2.736(8)	166.8
O19–H19…O30	0.84	2.11	2.778(11)	136.7
O20–H20…O303	0.84	2.12	2.84(2)	143.7
O20–H20…O203	0.84	2.14	2.87(3)	145.2
O27–H27…O1^{iv}	0.84	1.9	2.697(9)	158
O28–H28…O13^{v}	0.84	1.89	2.716(9)	169.7
O29–H29…O10^{vi}	0.84	2.08	2.754(9)	137.3
O30–H30…O304	0.84	2.18	2.717(10)	121.4
O30-H30…O204	0.84	2.55	3.098(10)	123.9
O37–H37…O11^{vii}	0.84	2.11	2.75(3)	133.6
O38–H38…O3^{v}	0.84	2.1	2.75(3)	134.6
O39–H39…O206	0.84	2.1	2.733(18)	131.5
O40–H40…O20	0.84	2.09	2.707(16)	129.5
O137–H137…O11^{vii}	0.84	1.88	2.695(16)	162.6
O138–H138…O3^{v}	0.84	1.93	2.749(17)	165.8
O139–H139…O305	0.84	2.6	3.021(8)	112.3
O140–H140…O306	0.84	2.07	2.779(11)	141.6
O201–H21A…O19^{viii}	0.85	2.12	2.700(9)	124.8
O201–H21B…O25^{viii}	0.85	2.09	2.875(5)	153.9
O202–H22A…O9	0.85	2.08	2.652(9)	124.5
O202–H22B…O39	0.85	2.07	2.811(13)	144.8
O202–H22B…O135	0.85	2.16	2.964(8)	157.9
O203–H23A…O35	0.85	2.43	3.279(14)	172.4
O203–H23B…O202	0.85	2.1	2.489(19)	107
O204–H24A…O15	0.85	2.11	2.945(7)	167.6
O204–H24B…O40	0.85	2.11	2.810(15)	139.3
O205–H25A…O5	0.85	2.11	2.839(9)	143
O205–H25B…O29^{viii}	0.85	2.09	2.505(15)	109.2
O206–H26A…O205	0.85	2.1	2.647(11)	122.2
O303–H33A…O140	0.85	2.12	2.865(8)	146.3
O303–H33B…O202	0.85	2.1	2.728(13)	130.3
O304–H34A…O15	0.85	2.07	2.858(6)	154.9
O304–H34B…O306	0.85	2.11	2.616(9)	117.8
O305–H35A…O5	0.85	2.06	2.892(7)	167.7
O305–H35B…O29^{viii}	0.85	2.1	2.853(8)	146.7
O306–H36A…O20	0.85	2.17	2.974(8)	156.6

Symmetry transformations used to generate equivalent atoms:
(i) $x, y+1, z+1$; (ii) $x, y+1, z$; (iii) $x-1, y, z$; (iv) $x, y-1, z-1$.
(v) $x, y-1, z$; (vi) $x-1, y-1, z-1$; (vii) $x+1, y, z$; (viii) $x+1, y+1, z+1$.

H-bonds to neighbouring pentaborates with acceptor sites designated as α, β and γ depending on distance from the central tetrahedral boron atom [13,14]. A commonly occurring H-bonding motif, encountered in 'herringbone' and 'brickwall' structures, is designated α,α,α,β [12]. With compound **1** the 'interstitial' H_2O molecules form additional H-bonds between anions and the situation is complicated further by anion/H_2O disorder. The four H-bond donor interactions from the O7, O8, O9, and O10 sites on the borate anion containing B1 can be designated α,α(α),β(H_2O),H_2O, with the lower s.o.f. acceptor site in parentheses for disordered interactions. The other pentaborates follow a similar numbering scheme and their interactions are α(α),α,β,H_2O(H_2O) (for anion containing B11), α,α,β,H_2O(H_2O) for β21, and α,α,H_2O,H_2O (for B131). The alternative anion to that containing B131 (i.e. containing B31) may be designated α,α,H_2O,β. A view of the anion/H_2O H-bonding network is illustrated in Fig. 2. Details of H-bond interactions can be found in Table 2. An alternative system for designating H-bond interactions has been devised by Etter [32]. This system describes all the α interactions in **1** as reciprocal pairs, $R_2{}^2(8)$; the β interactions are not the simple C(8) chains as observed in the herringbone and brickwall structures, but include the interstitial H_2O molecules. The inclusion of H_2O molecules within the anionic lattice produces a unique lattice structure which is expanded and adapted to accept the interpenetrating cation lattice.

The crystal structure of **1** also displays a supramolecular network of non-covalent phenyl embraces between $[Ph_4P]^+$ cations. A view of this cation network, in the same orientation as the anion network, is shown in Fig. 3. The three main types of phenyl embraces which have been reported for $[PPh_4]^+$ cations are offset face-to-face, edge-to-face, and vertex-to-face [17,18,33]. The network in **1** has every Ph group of every cation forming an offset face-to face embrace with a neighbouring cation. Thus, the four phenyl groups on P1 embrace phenyl groups on P31, P31', P91 and P91' with the other embraces as follows: P31 phenyls with phenyls on P1, P1', P61, P61'; P61 phenyls with phenyls on P31, P31', P91 and P91'; P91 phenyls with phenyls on P61, P61', P1 and P1'. The prime notation indicates a cation in an adjacent unit cell. All phenyl interactions are between 3.90(1) and 4.49(1) Å, and the interaction between a phenyl ring of P1 with a phenyl ring on P31 is shown in Fig. 4. P…P separations range from 7.33(1) to 8.33(1) Å, and are consistent with strong phenyl embrace interactions and are significantly shorter than 13.6 Å which is twice the van der Waals radius when the cation is regarded as a sphere [34].

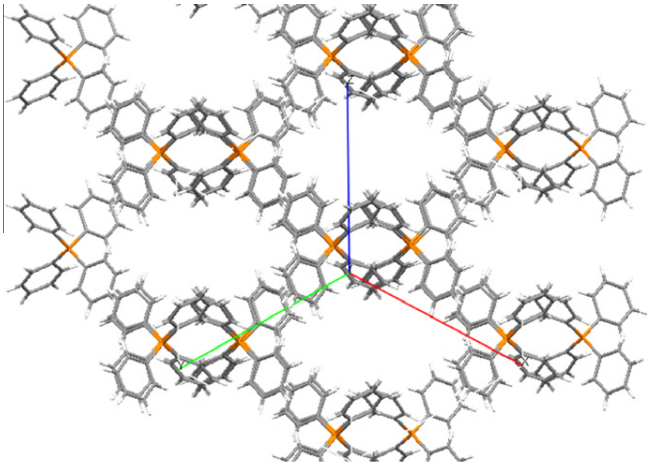

Fig. 3. A view of the supramolecular $[PPh_4]^+$ cation network in **1**.

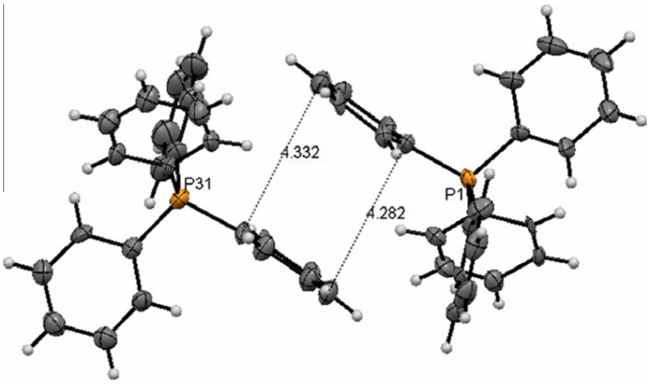

Fig. 4. A drawing showing an offset phenyl embrace between a phenyl on P1 and P31. The P1…P31 separation is 7.438 Å.

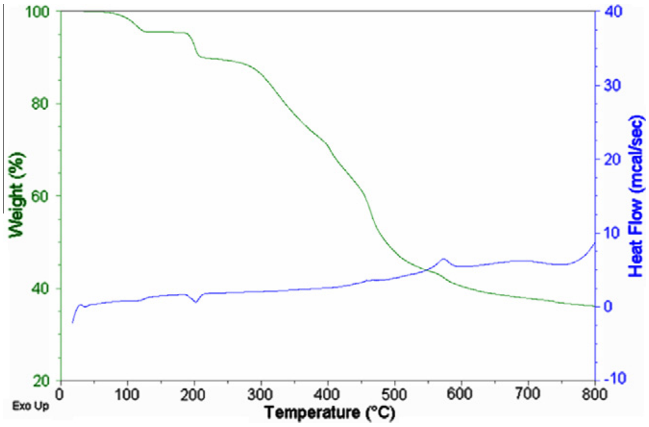

Fig. 5. DSC/TGA plot of a sample of **1** heated in 20-800 °C in air (ramp trrate 10 °C min^{-1}).

3.3. Thermal properties of ***1***

Pentaborate salts undergo a well-known two stage thermal decomposition [11–13,35], with the first stage of decomposition cross linking of the pentaborate anions with the release of two water molecules per pentaborate anion. The second stage is the oxidative decomposition of the organic component. Compound **1** was analysed using TGA/DSC, and results are shown in Fig. 5. The first event is when there is a small weight loss ca. 3–4% around 60–70 °C, corresponding to the loss of interstitial water in the structure (calc. 3.3%). The second weight loss event (ca. 5–6%) commences in the temperature range of 200–250 °C, and corresponds to the dehydration and crosslinking of the pentaborate anion network to form an anhydrous pentaborate (calc. 6.1%), $[Ph_4P][B_5O_8]$. There is then a steady weight loss until 800 °C which is the decomposition/oxidation of the cation leaving 2.5 molar equivalents of boron oxide (B_2O_3) which has a percentage weight remaining of ca. 32–33% (calc. 29.8%).

4. Conclusions

Tetraphenylphosphonium pentaborate, $[PPh_4][B_5O_6(OH)_4]\cdot 1.5\ H_2O$ crystallized in high yield form an aqueous methanol solution of $[PPh_4][OH]$ and $B(OH)_3$ (1:5). The solid-state structure is comprised of interpenetrating supramolecular networks of interconnected pentaborate anions (H-bonds) and tetraphenylphosphonium cations (phenyl embraces). TGA/DSC analysis revealed that $[PPh_4][B_5O_6(OH)_4]\cdot 1.5H_2O$ is thermally decomposed in air at 800 °C to $2.5B_2O_3$.

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Appendix A. Supplementary material

CCDC 832938 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2011.11.001.

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