différents selon qu'ils sont dans les cages simples [Cs(2) et Cs(4)] ou doubles [Cs(1) et Cs(3)]. Cs(2) et Cs(4) sont à proximité de quatre atomes d'oxygène tous situés du même côté à des distances variant de 3,15 à 3,34 Å. La distance qui vient ensuite, supérieure à 3,5 Å, ne peut être considérée comme constituant une liaison. Si l'on s'en tient à cette limite de 3,5 Å, Cs(1) et Cs(3) sont à proximité de sept atomes d'oxygène, également situés du même côté par rapport à eux. L'augmentation de ce nombre de voisins se traduit par une agitation thermique moins élevée (Tableau 1). Les cations sont proches les uns des autres: les plus courtes distances Nb–Nb, 3,492 et 3,509 Å, se trouvent entre l'atome de niobium du tétraèdre et celui des octaèdres accolés. La distance minimale Cs–Cs est de 4,15 Å. Toutes les distances O–O ont été calculées. La plus courte est de 2,64 Å. Dans le tétraèdre NbO_4, la distance entre atomes d'oxygène est proche de 3 Å. Bien qu'évoquant le type pyrochlore, cette structure qui réunit tout au long de l'axe *c* un assemblage d'octaèdres et de tétraèdres entourant l'ion Nb^{5+} est tout à fait originale.

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The α Phase of Ammonium Pentaborate Tetrahydrate

By V. Domenech* and J. Solans

Departamento de Cristalografía y Mineralogía, Universidad de Oviedo, Arias de Velasco s/n, Oviedo, Spain

and X. Solans

Departamento de Cristalografía y Mineralogía, Universidad de Barcelona, Gran Via 585, Barcelona 7, Spain

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Abstract. $NH_4[B_5O_6(OH)_4].2H_2O$, monoclinic, *Pn*, $a = 7{\cdot}115\,(1)$, $b = 11{\cdot}301\,(2)$, $c = 7{\cdot}183\,(3)$ Å, $\beta = 99{\cdot}92\,(1)°$, $Z = 2$, $D_c = 1{\cdot}57$ Mg m^{-3}. The structure was solved by isomorphous replacement from potassium pentaborate tetrahydrate coordinates, and refined by block-diagonal least squares to $R = 0{\cdot}058$ for all observed reflections. The compound is pseudo-isostructural with potassium pentaborate tetrahydrate. The ammonium ion is eight-coordinated.

Introduction. The structure of the title compound has been investigated to examine any differences between the α phases of ammonium and potassium pentaborate.

Colourless prismatic crystals of the title compound were obtained by direct crystallization from the reaction mixture. A crystal 0·2 × 0·2 × 0·3 mm was used for crystal-data and intensity measurements on a Philips PW 1100 four-circle diffractometer. The unit cell was measured by automatically centring 23 independent reflections and refining the orientation matrix and unit-cell parameters by least squares. Intensities were collected with Mo $K\alpha$ radiation using the ω-scan technique [scan width = $1{\cdot}0°(\theta)$, scan speed = $0{\cdot}05°(\theta)$ s^{-1}]. 828 independent reflections were measured within the range $3 \leq \theta \leq 22°$, 769 of which were treated as observed according the condition $I \geq 2\sigma(I)$.

The structure was solved by isomorphous replacement, using the pentaborate ion coordinates of potassium pentaborate tetrahydrate (Zachariasen & Plettinger, 1963). A subsequent Fourier synthesis revealed peaks for all non-hydrogen atoms. The structure was isotropically and anisotropically refined by block-diagonal least squares with a modified version of *SFLS* (Ahmed, Hall, Pippy & Huber, 1966). The function minimized as $\sum w||F_o| - |F_c||^2$, with the weights of Cruickshank (1965). A difference synthesis revealed the peaks for the H atoms, which were refined isotropically, whereas the non-hydrogen atoms were refined anisotropically. The refinement was terminated at $R = 0{\cdot}058$ for all observed reflections, where R is defined as $\sum||F_o| - |F_c||/\sum|F_o|$.†

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* This paper reports part of the research work undertaken to satisfy the requirements for a DSc degree.

† Lists of structure factors, anisotropic thermal parameters and H atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35750 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Non-hydrogen atomic parameters* ($\times 10^4$), *with e.s.d.'s in parentheses, and equivalent isotropic thermal parameters* ($Å^2$)

	x	y	z	U_{eq}*
N	8103 (11)	2497 (6)	9879 (10)	0·046
B(1)	8973 (13)	7511 (6)	10762 (12)	0·034
O(2)	8718 (8)	6684 (4)	9137 (6)	0·034
B(3)	8624 (14)	5519 (7)	9301 (10)	0·027
O(4)	8723 (8)	4968 (4)	11056 (6)	0·032
B(5)	8949 (12)	5669 (7)	12674 (11)	0·021
O(6)	9043 (7)	6850 (4)	12543 (6)	0·033
O(7)	10743 (7)	8148 (4)	10831 (7)	0·036
B(8)	10821 (11)	8342 (7)	10697 (10)	0·021
O(9)	9258 (7)	10023 (4)	10507 (8)	0·041
B(10)	7488 (11)	9514 (8)	10430 (13)	0·044
O(11)	7336 (7)	8313 (4)	10508 (7)	0·028
O(12)	8413 (10)	4725 (4)	7853 (7)	0·054
O(13)	8998 (9)	5123 (4)	14331 (6)	0·051
O(14)	12532 (7)	9877 (4)	10801 (9)	0·049
O(15)	6052 (8)	10253 (4)	10236 (10)	0·067
O(16)	12286 (8)	12236 (5)	10564 (7)	0·035
O(17)	8775 (8)	2767 (4)	14090 (7)	0·055

* $U_{eq} = 1/(6\pi^2) \sum_i \sum_j B_{ij} \mathbf{a}_i \mathbf{a}_j$.

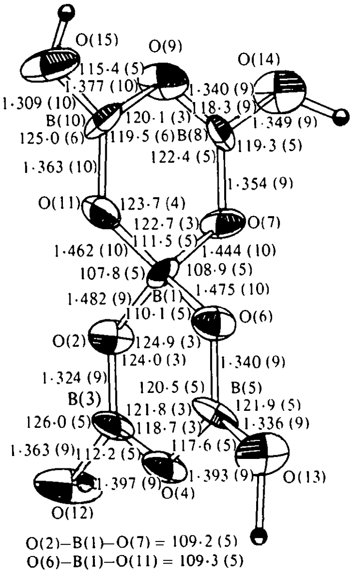

Fig. 1. View of the pentaborate ion with bond distances (Å) and angles (°).

The final atomic parameters are listed in Table 1. Fig. 1 (*ORTEP*, Johnson, 1965) shows a view of the pentaborate ion, bond distances and angles and the numbering of the atoms.

Discussion. The unit-cell parameters and the space group of ammonium pentaborate tetrahydrate were determined in early crystallographic literature [from Groth (1908) to Cook & Jaffe (1957)] by morphological studies. The differences between the values of Cook & Jaffe and an equivalent cell obtained with our values are shown in Table 2 ($\mathbf{a}' = \mathbf{b}$, $\mathbf{b}' = \mathbf{a} - \mathbf{c}$, $\mathbf{c}' = \mathbf{a} + \mathbf{c}$).

Table 2. *Unit-cell parameters and space group obtained by Cook & Jaffe* (1957) *and in this work for the* α *phase, and the values for the* β *phase*

	Cook & Jaffe	This work	β phase
a'	11·324 Å	11·301 Å	11·40 Å
b'	11·029	10·947	11·65
c'	9·325	9·198	8·66
α'	90·00°	90·55°	90·00°
β'	90·00	90·00	90·00
γ'	90·00	90·00	93·15
Space group	*Aba*2	*Ab*11	*A*112/*a*

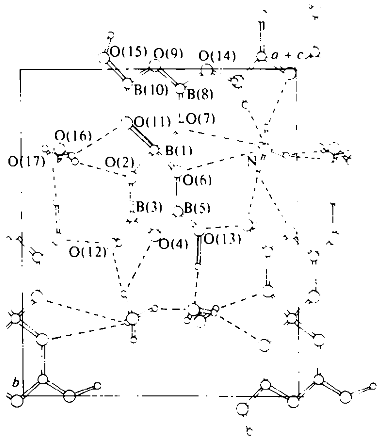

Fig. 2. Projection of the unit-cell contents down the [10$\bar{1}$] axis, showing the short intermolecular distances.

The bond distances in the pentaborate ion are similar to those obtained for the α phase of potassium pentaborate tetrahydrate (Zachariasen & Plettinger, 1963) and for the β phase of ammonium pentaborate tetrahydrate (Merlino, 1969). Fig. 2 shows a view of the unit-cell contents, and the shorter intermolecular distances are listed in Table 3. The ammonium ion is linked to eight O atoms, with six distances of about 2·95 and two of about 3·17 Å. This coordination of the ammonium ion is analogous to those observed in the previously mentioned compounds, while it is different from that in ammonium dihydrogen phosphate (Khan & Baur, 1973) where the ammonium ion is linked to eight O atoms with four distances of about 2·91 and four of about 3·17 Å.

The differences between the title compound and the α phase of potassium pentaborate tetrahydrate and the β phase of ammonium pentaborate tetrahydrate are minimal. A projection of these two compounds down the b' axis is similar to Fig. 2. The main differences are in the locations of the cation and the water molecules (Table 3). In the potassium derivative the interionic K···O distances are shorter than the values obtained for the α phase of the ammonium derivative, which

Table 3. *Short intermolecular distances* (Å) *in the* α *and* β *phases of ammonium pentaborate tetrahydrate and in the* α *phase of potassium pentaborate tetrahydrate*

	α-NH_4	β-NH_4	α-K
$N\cdots O(4)^{i}$	2·930 (8)	2·998 (3)	2·819 (3)
$N\cdots O(9)^{iii}$	2·927 (8)		
$N\cdots O(12)^{i}$	2·935 (8)	2·935 (3)	2·856 (3)
$N\cdots O(15)^{iii}$	2·959 (9)		
$N\cdots O(16)^{iii}$	2·946 (10)	3·018 (4)	2·902 (4)
$N\cdots O(17)^{i}$	2·995 (9)		
$N\cdots O(6)^{ii}$	3·168 (7)	3·231 (5)	3·034 (4)
$N\cdots O(7)^{ii}$	3·182 (9)		
$O(17)\cdots O(13)^{i}$	2·671 (7)	2·696 (3)	2·676 (5)
$O(16)\cdots O(14)^{i}$	2·675 (7)		
$O(17)\cdots O(7)^{vii}$	2·867 (7)	2·926 (3)	2·920 (5)
$O(16)\cdots O(6)^{vi}$	2·877 (7)		
$O(17)\cdots O(11)^{viii}$	2·839 (7)	2·788 (4)	2·827 (6)
$O(16)\cdots O(2)^{v}$	2·866 (7)		
$O(14)\cdots O(15)^{iv}$	2·640 (8)	2·665 (4)	2·661 (6)
$O(12)\cdots O(13)^{ix}$	2·672 (7)		

Symmetry code

(i)	x,y,z	(vi)	$x+\frac{1}{2}, 1-y, z-\frac{1}{2}$
(ii)	$x-\frac{1}{2}, \bar{y}, z-\frac{1}{2}$	(vii)	$x-\frac{1}{2}, \bar{y}, z+\frac{1}{2}$
(iii)	$x, y-\frac{1}{2}, z$	(viii)	$\frac{1}{2}+x, \bar{y}, z+\frac{1}{2}$
(iv)	$x+1, y, z$	(ix)	$x, y, z-1$
(v)	$\frac{1}{2}+x, 1-y, z+\frac{1}{2}$		

indicates that the ammonium ion is more bulky than the K^+ ion. The structure of the β phase shows a pseudo *b* plane normal to the *c* axis (*a'* axis in Table 2), but the main difference from the α phase is in the locations of the water molecules.

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Structure du Trimétaphosphate–Tellurate de Sodium Hexahydraté $Te(OH)_6.2Na_3P_3O_9.6H_2O$

PAR N. BOUDJADA, M. T. AVERBUCH-POUCHOT ET A. DURIF

Laboratoire de Cristallographie, CNRS, 166 *X,* 38042 *Grenoble CEDEX, France*

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Abstract. $Te(OH)_6.2Na_3P_3O_9.6H_2O$ is hexagonal, $P6_3/m$, with $a = 11{\cdot}67$ (1), $c = 12{\cdot}12$ (1) Å and $Z = 2$. The structure has been solved by the Patterson method with 741 independent reflections to a final *R* value of 0·04. The main feature of this atomic arrangement is the coexistence of two different and independent types of anions: P_3O_9 rings and TeO_6 octahedra linked by NaO_6 octahedra. This compound is the first example of a phosphate–tellurate containing a condensed phosphate ion.

Introduction. Des études structurales récentes entreprises dans le domaine des phosphates–tellurates montrent que dans ce type de composé, il n'y a pas formation d'anions mixtes condensés phosphotelluriques mais coexistence de groupements phosphates et d'octaèdres TeO_6 indépendants. Ces études étaient jusqu'à présent limitées aux composés enfermant des groupements monophosphate (tétraèdre PO_4): $Te(OH)_6.2(NH_4)_2HPO_4$ et $Te(OH)_6.Na_2HPO_4.H_2O$ par Durif, Averbuch-Pouchot & Guitel (1979), $Te(OH)_6.Rb_2HPO_4.RbH_2PO_4$ par Averbuch-Pouchot, Durif & Guitel (1979), $Te(OH)_6.Cs_2HPO_4$ et $Te(OH)_6.Cs_2HPO_4.2CsH_2PO_4$ par Averbuch-Pouchot, Durif & Guitel (1980).

Il était intéressant de vérifier si les groupements phosphates condensés existant dans les polyphosphates ou les métaphosphates, pouvaient de la même manière coexister avec des octaèdres TeO_6. L'objet du présent travail est l'étude structurale du

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