

Ammonium ytterbium(III) diphosphate(V)

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Key indicators: single-crystal X-ray study; $T = 120$ K; mean $\sigma(\text{Yb}-\text{O}) = 0.003$ Å; R factor = 0.016; wR factor = 0.061; data-to-parameter ratio = 12.7.

The title compound, $\text{NH}_4\text{YbP}_2\text{O}_7$, crystallizes in the KAIP_2O_7 structure type and consists of distorted YbO_6 octahedra and bent $\text{P}_2\text{O}_7^{4-}$ diphosphate units forming together a three-dimensional network. There are channels in the structure running along the c axis, where the NH_4^+ cations are located. They are connected *via* $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds to the terminal O atoms of the diphosphate anions.

Related literature

Isotypic compounds were reported by Man-Rong *et al.* (2005), $[\text{NH}_4\text{LuP}_2\text{O}_7]$; Horchani-Naifer & Férid (2007), $[\text{YbP}_2\text{O}_7]$; Jansen *et al.* (1991), $[\text{CsYbP}_2\text{O}_7]$, that all crystallize with the KAIP_2O_7 structure type (Ng & Calvo, 1973). For the crystal structures of other isoformular rare earth diphosphates, see: Hamady & Jouini (1996), $[\text{NaYP}_2\text{O}_7]$; Férid *et al.* (2004), $[\text{NaEuP}_2\text{O}_7]$; Ferid *et al.* (2004), $[\text{NaYbP}_2\text{O}_7]$; Férid & Horchani-Naifer (2004), $[\text{NaLaP}_2\text{O}_7]$; Horchani-Naifer & Férid (2005), $[\text{NaCeP}_2\text{O}_7]$; Hamady *et al.* (1994) and Yuan *et al.* (2007), $[\text{KYP}_2\text{O}_7]$. Possible applications of rare earth phosphates were discussed by Yamada *et al.* (1974); Hong (1975); Bimberg *et al.* (1975). For background on crystallographic software, see: Becker & Coppens (1974).

Experimental

Crystal data

$\text{NH}_4\text{YbP}_2\text{O}_7$	$V = 692.04$ (3) Å ³
$M_r = 365$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 7.6468$ (2) Å	$\mu = 13.97$ mm ⁻¹
$b = 10.9119$ (2) Å	$T = 120$ K
$c = 8.6129$ (3) Å	$0.26 \times 0.08 \times 0.07$ mm
$\beta = 105.645$ (3)°	

Data collection

Oxford Diffraction XCalibur 2 diffractometer with Sapphire 2 area detector	(1995]
Absorption correction: analytical [implemented in <i>CrysAlis RED</i> (Oxford Diffraction, 2008), according to Clark & Reid	$T_{\min} = 0.169$, $T_{\max} = 0.545$
	8574 measured reflections
	1437 independent reflections
	1362 reflections with $I > 3\sigma(I)$
	$R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.016$	4 restraints
$wR(F^2) = 0.061$	Only H-atom coordinates refined
$S = 1.32$	$\Delta\rho_{\max} = 0.58$ e Å ⁻³
1437 reflections	$\Delta\rho_{\min} = -0.50$ e Å ⁻³
113 parameters	

Table 1

Selected geometric parameters (Å, °).

Yb1—O1	2.240 (3)	P1—O2	1.498 (5)
Yb1—O2 ⁱ	2.158 (4)	P1—O3	1.611 (4)
Yb1—O4 ⁱⁱ	2.230 (3)	P1—O4	1.525 (3)
Yb1—O5	2.224 (3)	P2—O3	1.622 (4)
Yb1—O6 ⁱⁱⁱ	2.191 (4)	P2—O5	1.532 (3)
Yb1—O7 ^{iv}	2.195 (3)	P2—O6	1.507 (4)
P1—O1	1.529 (3)	P2—O7	1.514 (3)

P1—O3—P2 127.40 (19)

Symmetry codes: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (iii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (iv) $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
N1—H1 ^v ⋯O7 ^v	0.87 (4)	2.52 (5)	3.381 (5)	168 (4)
N1—H2 ^{vi} ⋯O4 ^{vi}	0.88 (4)	2.03 (5)	2.888 (5)	166 (4)
N1—H3 ^{vii} ⋯O5 ^{vii}	0.87 (4)	2.00 (4)	2.873 (6)	177 (7)
N1—H4 ^{viii} ⋯O1	0.86 (5)	2.26 (4)	2.916 (5)	132 (4)

Symmetry codes: (v) $x - 1, y, z$; (vi) $-x + 1, -y + 1, -z + 1$; (vii) $x - 1, -y + \frac{1}{2}, z - \frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *JANA2006* (Petříček *et al.*, 2007); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *JANA2006*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2208).

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supplementary materials

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Ammonium ytterbium(III) diphosphate(V)

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Comment

Rare earth phosphates have many potential applications in the field of optical materials including laser phosphors (Yamada *et al.*, 1974; Hong, 1975; Bimberg *et al.*, 1975). Their crystal structures depend on the ionic radii of the alkali metal and the rare earth ions. The two $AYbP_2O_7$ ($A = Cs$ (Jansen *et al.*, 1991), K (Horchani-Naifer & Férid, 2007)) structures known so far belong to the $KAlP_2O_7$ structure type (Ng & Calvo, 1973) and crystallize in space group $P2_1/c$. For the correspondent isoformular sodium rare earth diphosphates, several other structures have been described, for instance $NaYP_2O_7$ in space group $P2_1$ (Hamady & Jouini, 1996), $NaLnP_2O_7$ ($Ln = Eu$ (Férid, Horchani & Amami, 2004), Yb (Ferid *et al.*, 2004)) in space group $P2_1/n$, and $NaLnP_2O_7$ ($Ln = La$ (Férid & Horchani-Naifer, 2004), Ce (Horchani-Naifer & Férid, 2005) in space group $Pnma$. KYP_2O_7 is dimorphic and can adopt the $KAlP_2O_7$ structure type (Yuan *et al.*, 2007), or a structure in space group $Cmcm$ (Hamady *et al.*, 1994).

In the present paper we report the crystal structure of $NH_4YbP_2O_7$. This compound is isotypic with $NH_4LuP_2O_7$ (Man-Rong *et al.* 2005), $KYbP_2O_7$ (Horchani-Naifer & Férid, 2007) and $CsYbP_2O_7$ (Jansen *et al.*, 1991). The Yb atom is coordinated by six oxygen atoms forming a distorted octahedron that belong to five symmetry-related $P_2O_7^{4-}$ anions (Fig. 1). The average Yb—O bond length is 2.206 Å (Table 1). The diphosphate anion is bent with a bridging angle of 127.40 (19)°. The three-dimensional network of YbO_6 and $P_2O_7^{4-}$ units forms channels running along the c direction in which the NH_4^+ cations are located (Fig. 2). Each NH_4^+ cation is connected *via* N—H...O hydrogen bonds to four different $P_2O_7^{4-}$ anions (Table 2).

Experimental

Three solutions have been mixed in a beaker to prepare the title compound: NH_4OH (20 ml, 0.1 mmol), $YbCl_3 \cdot 6H_2O$ (20 ml, 0.1 mmol) and $Na_4P_2O_7$ (20 ml, 0.1 mmol). The pH of the mixture was controlled with diluted hydrochloric acid to be slightly acidic, and the solution was stirred for two hours at room temperature. Crystals suitable for X-ray analysis were formed after a few days.

Refinement

All hydrogen atoms were discernible in difference Fourier maps and could be refined to reasonable geometry. The N—H distances were restrained to 0.87 Å with σ of 0.02. The isotropic atomic displacement parameters of all hydrogen atoms were refined with $1.2 \times U_{eq}$ of the N atom.

Figures

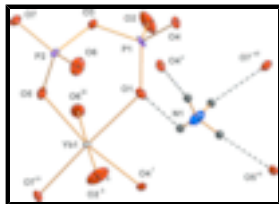


Fig. 1. Part of the structure of $\text{NH}_4\text{YbP}_2\text{O}_7$. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $1 - x, -1/2 + y, 1.5 - z$; (ii) $x, 0.5 - y, -1/2 + z$; (iii) $x, 0.5 - y, 1/2 + z$; (iv) $2 - x, -1/2 + y, 1.5 - z$; (v) $1 - x, 1 - y, 1 - z$; (vi) $-1 + x, 0.5 - y, -1/2 + z$; (vii) $-1 + x, y, z$.]

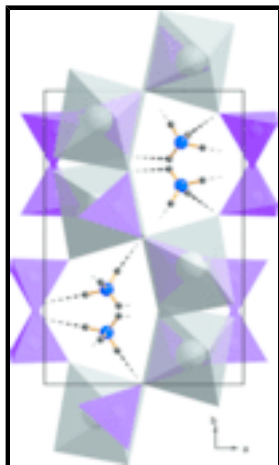


Fig. 2. The packing of $\text{NH}_4\text{YbP}_2\text{O}_7$ viewed along c . Colors: Pink (P_2O_7), grey (YbO_6), blue balls (N), black balls (H).

Ammonium ytterbium(III) diphosphate(V)

Crystal data

$\text{NH}_4\text{YbP}_2\text{O}_7$

$M_r = 365$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 7.6468\ (2)\ \text{\AA}$

$b = 10.9119\ (2)\ \text{\AA}$

$c = 8.6129\ (3)\ \text{\AA}$

$\beta = 105.645\ (3)^\circ$

$V = 692.04\ (3)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 668$

$D_x = 3.502\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 8929 reflections

$\theta = 2.8\text{--}26.5^\circ$

$\mu = 13.97\ \text{mm}^{-1}$

$T = 120\ \text{K}$

Prism, colorless

$0.26 \times 0.08 \times 0.07\ \text{mm}$

Data collection

Oxford Diffraction XCalibur 2 with Sapphire 2 area detector diffractometer

1437 independent reflections

Radiation source: X-ray tube

1362 reflections with $I > 3\sigma(I)$

Monochromator: graphite

$R_{\text{int}} = 0.023$

Detector resolution: $8.3438\ \text{pixels mm}^{-1}$

$\theta_{\text{max}} = 26.5^\circ$

$T = 120\ \text{K}$

$\theta_{\text{min}} = 2.8^\circ$

Rotation method data acquisition using ω scans $h = -9 \rightarrow 9$
 Absorption correction: analytical
 [implemented in CrysAlis RED (Oxford Diffraction, $k = -13 \rightarrow 13$
 2008), according to Clark & Reid (1995)]
 $T_{\min} = 0.169$, $T_{\max} = 0.545$ $l = -10 \rightarrow 10$
 8574 measured reflections

Refinement

Refinement on F^2	Only H-atom coordinates refined
$R[F^2 > 2\sigma(F^2)] = 0.016$	Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(I) + 0.0016I^2]$
$wR(F^2) = 0.061$	$(\Delta/\sigma)_{\max} = 0.039$
$S = 1.32$	$\Delta\rho_{\max} = 0.58 \text{ e } \text{\AA}^{-3}$
1437 reflections	$\Delta\rho_{\min} = -0.50 \text{ e } \text{\AA}^{-3}$
113 parameters	Extinction correction: B-C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
4 restraints	Extinction coefficient: 170 (60)
4 constraints	

Special details

Refinement. The refinement was carried out against all reflections. The conventional R -factor is always based on F . The goodness of fit as well as the weighted R -factor are based on F and F^2 for refinement carried out on F and F^2 , respectively. The threshold expression is used only for calculating R -factors *etc.* and it is not relevant to the choice of reflections for refinement.

The program used for refinement, Jana2006, uses the weighting scheme based on the experimental expectations, see `_refine_ls_weighting_details`, that does not force S to be one. Therefore the values of S are usually larger than the ones from the *SHELX* program.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Yb1	0.73470 (3)	0.100256 (15)	0.753623 (18)	0.00555 (9)
P1	0.63175 (18)	0.40147 (9)	0.81812 (14)	0.0080 (4)
P2	0.93914 (15)	0.36312 (10)	0.68708 (13)	0.0070 (3)
O1	0.5777 (4)	0.2746 (3)	0.7457 (4)	0.0124 (10)
O2	0.6416 (6)	0.4080 (3)	0.9940 (5)	0.0254 (14)
O3	0.8335 (4)	0.4300 (3)	0.8037 (4)	0.0126 (9)
O4	0.5107 (4)	0.5010 (3)	0.7202 (3)	0.0097 (9)
O5	0.9555 (4)	0.2277 (3)	0.7359 (4)	0.0121 (10)
O6	0.8260 (6)	0.3855 (3)	0.5169 (5)	0.0183 (12)
O7	1.1241 (4)	0.4235 (3)	0.7273 (4)	0.0142 (10)
N1	0.3131 (6)	0.3233 (4)	0.4381 (5)	0.0183 (13)
H1	0.281 (7)	0.348 (5)	0.523 (4)	0.0219*
H2	0.365 (7)	0.385 (3)	0.403 (6)	0.0219*
H3	0.206 (4)	0.306 (5)	0.375 (5)	0.0219*
H4	0.373 (7)	0.267 (4)	0.501 (5)	0.0219*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Yb1	0.00611 (15)	0.00443 (15)	0.00616 (16)	-0.00018 (5)	0.00173 (9)	0.00025 (5)
P1	0.0101 (6)	0.0088 (6)	0.0056 (5)	0.0056 (4)	0.0027 (5)	-0.0006 (4)
P2	0.0066 (5)	0.0058 (5)	0.0084 (5)	-0.0016 (4)	0.0017 (4)	0.0016 (4)
O1	0.0142 (16)	0.0076 (15)	0.0178 (16)	0.0019 (12)	0.0085 (13)	0.0023 (12)
O2	0.034 (3)	0.039 (2)	0.0043 (17)	0.0210 (17)	0.0062 (17)	0.0013 (14)
O3	0.0073 (15)	0.0117 (14)	0.0158 (16)	0.0000 (12)	-0.0019 (13)	-0.0036 (13)
O4	0.0093 (14)	0.0097 (13)	0.0102 (14)	0.0026 (12)	0.0028 (11)	0.0041 (12)
O5	0.0088 (15)	0.0113 (15)	0.0166 (16)	-0.0007 (12)	0.0041 (12)	0.0045 (12)
O6	0.017 (2)	0.0277 (19)	0.0086 (18)	-0.0005 (14)	0.0015 (15)	0.0090 (14)
O7	0.0093 (16)	0.0101 (13)	0.0228 (17)	-0.0036 (13)	0.0035 (14)	0.0014 (12)
N1	0.017 (2)	0.021 (2)	0.013 (2)	-0.0069 (17)	-0.0014 (17)	0.0046 (16)

Geometric parameters (\AA , $^\circ$)

Yb1—O1	2.240 (3)	P1—O4	1.525 (3)
Yb1—O2 ⁱ	2.158 (4)	P2—O3	1.622 (4)
Yb1—O4 ⁱⁱ	2.230 (3)	P2—O5	1.532 (3)
Yb1—O5	2.224 (3)	P2—O6	1.507 (4)
Yb1—O6 ⁱⁱⁱ	2.191 (4)	P2—O7	1.514 (3)
Yb1—O7 ^{iv}	2.195 (3)	N1—H1	0.87 (5)
P1—O1	1.529 (3)	N1—H2	0.87 (5)
P1—O2	1.498 (5)	N1—H3	0.87 (3)
P1—O3	1.611 (4)	N1—H4	0.86 (4)
O1—Yb1—O2 ⁱ	88.83 (13)	O2—P1—O3	106.3 (2)
O1—Yb1—O4 ⁱⁱ	87.53 (11)	O2—P1—O4	112.5 (2)
O1—Yb1—O5	82.94 (12)	O3—P1—O4	105.70 (17)
O1—Yb1—O6 ⁱⁱⁱ	89.43 (12)	O3—P2—O5	106.33 (19)
O1—Yb1—O7 ^{iv}	175.63 (13)	O3—P2—O6	106.2 (2)
O2 ⁱ —Yb1—O4 ⁱⁱ	91.87 (14)	O3—P2—O7	104.64 (18)
O2 ⁱ —Yb1—O5	89.99 (15)	O5—P2—O6	113.98 (18)
O2 ⁱ —Yb1—O6 ⁱⁱⁱ	178.23 (14)	O5—P2—O7	110.79 (17)
O2 ⁱ —Yb1—O7 ^{iv}	93.37 (13)	O6—P2—O7	114.1 (2)
O4 ⁱⁱ —Yb1—O5	170.25 (11)	P1—O3—P2	127.40 (19)
O4 ⁱⁱ —Yb1—O6 ⁱⁱⁱ	88.39 (13)	P1—O4—H2 ^v	115.2 (13)
O4 ⁱⁱ —Yb1—O7 ^{iv}	88.63 (12)	P2—O5—H3 ^{vi}	109.5 (14)
O5—Yb1—O6 ⁱⁱⁱ	89.47 (13)	H1—N1—H2	108 (5)
O5—Yb1—O7 ^{iv}	100.82 (12)	H1—N1—H3	99 (4)
O6 ⁱⁱⁱ —Yb1—O7 ^{iv}	88.39 (12)	H1—N1—H4	85 (5)
O1—P1—O2	113.0 (2)	H2—N1—H3	113 (4)
O1—P1—O3	107.61 (19)	H2—N1—H4	123 (5)
O1—P1—O4	111.26 (16)	H3—N1—H4	119 (4)

Symmetry codes: (i) $x, -y+1/2, z-1/2$; (ii) $-x+1, y-1/2, -z+3/2$; (iii) $x, -y+1/2, z+1/2$; (iv) $-x+2, y-1/2, -z+3/2$; (v) $-x+1, -y+1, -z+1$; (vi) $x+1, -y+1/2, z+1/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O7^{vii}$	0.87 (4)	2.52 (5)	3.381 (5)	168 (4)
$N1-H2\cdots O4^v$	0.88 (4)	2.03 (5)	2.888 (5)	166 (4)
$N1-H3\cdots O5^{viii}$	0.87 (4)	2.00 (4)	2.873 (6)	177 (7)
$N1-H4\cdots O1$	0.86 (5)	2.26 (4)	2.916 (5)	132 (4)

Symmetry codes: (vii) $x-1, y, z$; (v) $-x+1, -y+1, -z+1$; (viii) $x-1, -y+1/2, z-1/2$.

Fig. 1

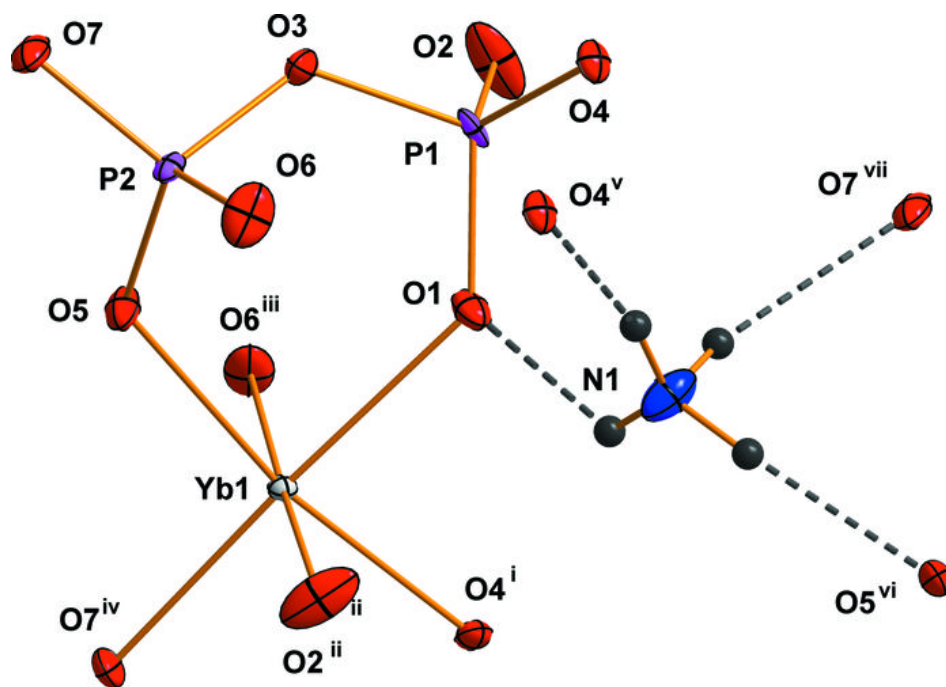


Fig. 2

