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Ammonium ytterbium(III) diphosphate(V)

Karla Fejfarová,[a] Rachid Essehli,[b] Brahim El Bali[b] and Michal Dušek[a]*

[a]Institute of Physics, Na Slovance 2, 182 21 Praha 8, Czech Republic, and [b]Department of Chemistry, Faculty of Sciences, University Mohammed 1st, PO Box 717, 60 000 Oujda, Morocco
Correspondence e-mail: fejfarov@fzu.cz

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

The title compound, $NH_4YbP_2O_7$, crystallizes in the $KAlP_2O_7$ structure type and consists of distorted YbO_6 octahedra and bent $P_2O_7^{4-}$ 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* N—H···O hydrogen bonds to the terminal O atoms of the diphosphate anions.

Related literature

Isotypic compounds were reported by Man-Rong *et al.* (2005), [$NH_4LuP_2O_7$]; Horchani-Naifer & Férid (2007), [YbP_2O_7]; Jansen *et al.* (1991), [$CsYbP_2O_7$], that all crystallize with the $KAlP_2O_7$ structure type (Ng & Calvo, 1973). For the crystal structures of other isoformular rare earth diphosphates, see: Hamady & Jouini (1996), [$NaYP_2O_7$]; Férid *et al.* (2004), [$NaEuP_2O_7$]; Ferid *et al.* (2004), [$NaYbP_2O_7$]; Férid & Horchani-Naifer (2004), [$NaLaP_2O_7$]; Horchani-Naifer & Férid (2005), [$NaCeP_2O_7$]; Hamady *et al.* (1994) and Yuan *et al.* (2007), [KYP_2O_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

$NH_4YbP_2O_7$
M_r = 365
Monoclinic, $P2_1/c$
a = 7.6468 (2) Å
b = 10.9119 (2) Å
c = 8.6129 (3) Å
β = 105.645 (3)°
V = 692.04 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 13.97 mm^{-1}
T = 120 K
0.26 × 0.08 × 0.07 mm

Data collection

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

Refinement

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

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—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O7^{v}	0.87 (4)	2.52 (5)	3.381 (5)	168 (4)
N1—H2···O4^{vi}	0.88 (4)	2.03 (5)	2.888 (5)	166 (4)
N1—H3···O5^{vii}	0.87 (4)	2.00 (4)	2.873 (6)	177 (7)
N1—H4···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).

References

Becker, P. J. & Coppens, P. (1974). *Acta Cryst.* A**30**, 129–147.
Bimberg, D., Robbins, D. J., Wight, D. R. & Jeser, J. P. (1975). *Appl. Phys. Lett.* **27**, 67–68.
Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Burla, M. C., Camalli, M., Carrozzini, B., Cascarano, G., Giacovazzo, C., Polidori, G. & Spagna, R. (2003). *J. Appl. Cryst*. **36**, 1103.

Clark, R. C. & Reid, J. S. (1995). *Acta Cryst.* A**51**, 887–897.

Férid, M., Horchani, K. & Amami, J. (2004). *Mater. Res. Bull.* **39**, 1949–1955.

Férid, M. & Horchani-Naifer, K. (2004). *Mater. Res. Bull.* **39**, 2209–2217.

Ferid, M., Horchani-Naifer, K. & Trabelsi-Ayedi, M. (2004). *Z. Kristallogr. New Cryst. Struct.* **219**, 353–354.

Hamady, A., Faouzi Zid, M. & Jouini, T. (1994). *J. Solid State Chem.* **113**, 120–124.

Hamady, A. & Jouini, T. (1996). *Acta Cryst.* C**52**, 2949–2951.

Hong, H. Y. P. (1975). *Mater. Res. Bull.* **10**, 1105–1110.

Horchani-Naifer, K. & Férid, M. (2005). *Solid State Ionics*, **176**, 1949–1953.

Horchani-Naifer, K. & Férid, M. (2007). *Acta Cryst.* E**63**, i33–i34.

Jansen, M., Wu, G. Q. & Koenigstein, K. (1991). *Z. Kristallogr.* **197**, 245–246.

Man-Rong, L., Wei, L., Hao-Hong, C., Xin-Xin, Y., Zan-Bin, W., Dun-Hua, C., Mu, G. & Jing-Tai, Z. (2005). *Eur. J. Inorg. Chem.* pp. 4693–4696.

Ng, H. N. & Calvo, C. (1973). *Can. J. Chem.* **51**, 2613-2620.

Oxford Diffraction (2005). *CrysAlis CCD*. Oxford Diffraction Ltd, Abingdon, England.

Oxford Diffraction (2008). *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.

Petříček, V., Dušek, M. & Palatinus, L. (2007). *JANA2006*. Institute of Physics, Praha, Czech Republic.

Yamada, T., Otsuka, K. & Nakano, J. (1974). *J. Appl. Phys.* **45**, 5096–5097.

Yuan, J., Zhang, H., Chen, H., Yang, X., Zhao, J. & Gu, M. (2007). *J. Solid State Chem.* **180**, 3381–3387.

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supporting information

Acta Cryst. (2008). E**64**, i85–i86 [doi:10.1107/S1600536808039664]

Ammonium ytterbium(III) diphosphate(V)

Karla Fejfarová, Rachid Essehli, Brahim El Bali and Michal Dušek

S1. 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$_2$O$_7$ (A = Cs (Jansen *et al.*, 1991), K (Horchani-Naifer & Férid, 2007)) structures known so far belong to the KAlP$_2$O$_7$ 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$_2$O$_7$ in space group $P2_1$ (Hamady & Jouini, 1996), NaLnP$_2$O$_7$ (Ln = Eu (Férid, Horchani & Amami, 2004), Yb (Ferid *et al.*, 2004)) in space group $P2_1/n$, and NaLnP$_2$O$_7$ (Ln = La (Férid & Horchani-Naifer, 2004), Ce (Horchani-Naifer & Férid, 2005) in space group *Pnma*. KYP$_2$O$_7$ is dimorphic and can adopt the KAlP$_2$O$_7$ 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$_4$YbP$_2$O$_7$. This compound is isotypic with NH$_4$LuP$_2$O$_7$ (Man-Rong *et al.* 2005), KYbP$_2$O$_7$ (Horchani-Naifer & Férid, 2007) and CsYbP$_2$O$_7$ (Jansen *et al.*, 1991). The Yb atom is coordinated by six oxygen atoms forming a distorted octahedron that belong to five symmetry-related P$_2$O$_7^{4-}$ anions (Fig. 1). The average Yb—O bond lenght 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$_2$O$_7^{4-}$ units forms channels running along the *c* direcion 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$_2$O$_7^{4-}$ anions (Table 2).

S2. Experimental

Three solutions have been mixed in a beaker to prepare the title compound: NH$_4$OH (20 ml, 0.1 mmol), YbCl$_3$·6H$_2$O (20 ml, 0.1 mmol) and Na$_4$P$_2$O$_7$ (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.

S3. 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×$U_{\rm eq}$ of the N atom.

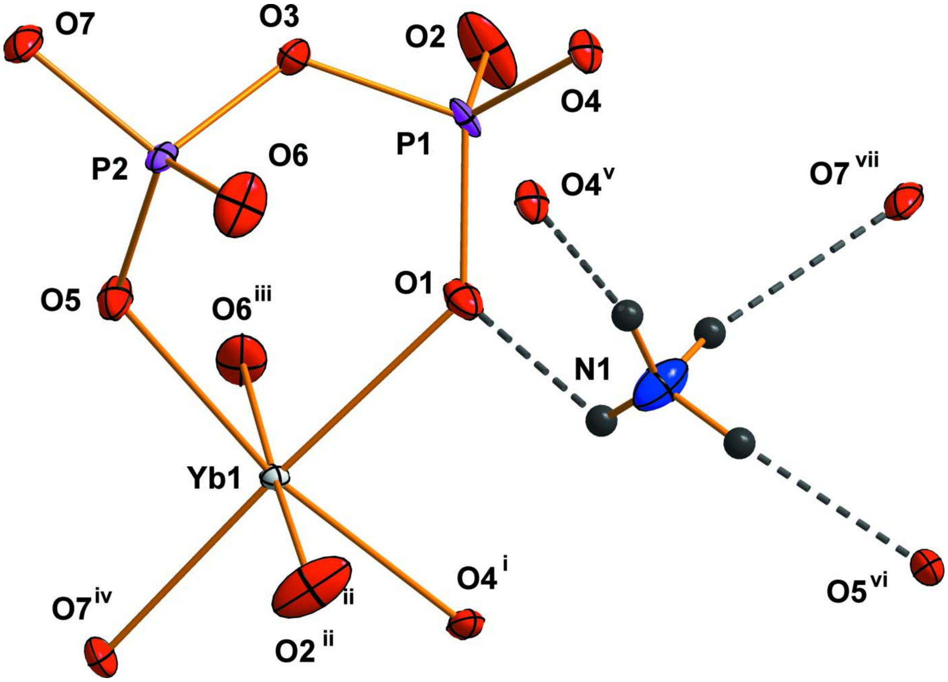

Figure 1

Part of the structure of $NH_4YbP_2O_7$. 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*.]

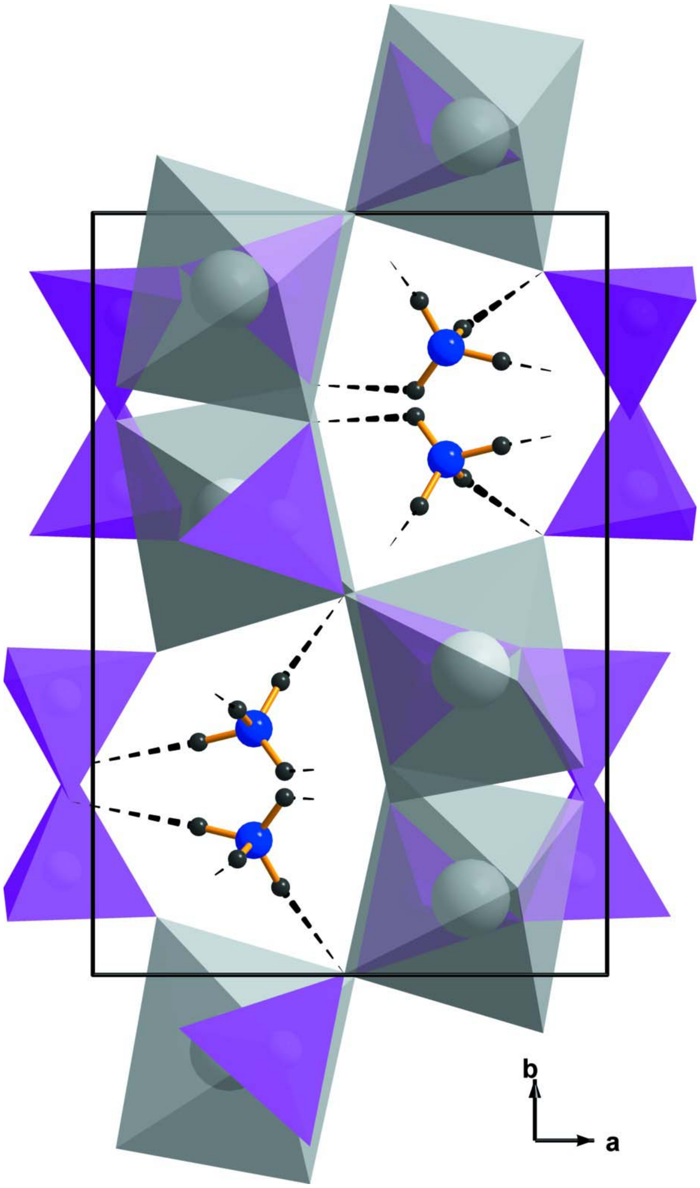

Figure 2

The packing of $NH_4YbP_2O_7$ viewed along *c*. Colors: Pink (P_2O_7), grey (YbO_6), blue balls (N), black balls (H).

Ammonium ytterbium(III) diphosphate(V)

Crystal data

$NH_4YbP_2O_7$	$F(000) = 668$
$M_r = 365$	$D_x = 3.502$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 8929 reflections
$a = 7.6468$ (2) Å	$\theta = 2.8–26.5°$
$b = 10.9119$ (2) Å	$\mu = 13.97$ mm^{-1}
$c = 8.6129$ (3) Å	$T = 120$ K
$\beta = 105.645$ (3)°	Prism, colorless
$V = 692.04$ (3) Å^3	0.26 × 0.08 × 0.07 mm
$Z = 4$	

Data collection

Oxford Diffraction XCalibur 2 with Sapphire 2 area detector diffractometer
Radiation source: X-ray tube
Graphite monochromator
Detector resolution: 8.3438 pixels mm^{-1}
Rotation method data acquisition using ω scans
Absorption correction: analytical [implemented in *CrysAlis RED* (Oxford Diffraction, 2008), according to Clark & Reid (1995)]
T_{min} = 0.169, T_{max} = 0.545
8574 measured reflections
1437 independent reflections
1362 reflections with $I > 3\sigma(I)$
R_{int} = 0.023
θ_{max} = 26.5°, θ_{min} = 2.8°
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -10 \rightarrow 10$

Refinement

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

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{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*

Atomic displacement parameters (Å^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 (Å, º)

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 (Å, º)

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

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