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Diammonium tris[hexaaquanickel(II)] tetrakis[hydrogenphosphate(III)], $(NH_4)_2[Ni(H_2O)_6]_3(HPO_3)_4$

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(P–O) = 0.002 Å
R factor = 0.020
wR factor = 0.060
Data-to-parameter ratio = 11.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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$(NH_4)_2[Ni(H_2O)_6]_3(HPO_3)_4$ is a member of the isotypic $(NH_4)_2[T(H_2O)_6]_3(HPO_3)_4$ family, where T = Co, Ni or Mg. The structure of $(NH_4)_2[Ni(H_2O)_6]_3(HPO_3)_4$ is composed of the units $[Ni(H_2O)_6]^{2+}$, $(HPO_3)^{2-}$ and NH_4^+, which interact *via* an intricate network of hydrogen bonds. One of the Ni atoms is located at a site of 2/*m* symmetry, whereas the other central atoms, *viz*. the second Ni, the two P and ammonium N atom, are located at sites of *m* symmetry.

Comment

$(NH_4)_2[Ni(H_2O)_6]_3(HPO_3)_4$ is a member of the $(NH_4)_2[T(H_2O)_6]_3(HPO_3)_4$ family that has currently three isotypic representatives with T = Co (Ouarsal *et al.*, 2005), Ni and Mg (Messouri *et al.*, 2005). A detailed structure description of the $(NH_4)_2[T(H_2O)_6]_3(HPO_3)_4$ family has already been given for the Mg compound (Messouri *et al.*, 2005).

Fig. 1 shows the $[Ni(H_2O)_6]^{2+}$, $(HPO_3)^{2-}$ and NH_4^+ coordination polyhedra. The packing of the main building units is shown in Figs. 2 and 3. No classical chemical bonds exist between the building units, which are connected solely by hydrogen bonds (Table 2).

The average P—O and P—H distances [1.527 (2) and 1.27 (3) Å, respectively] are virtually the same as the average distances in the isotypic Mg and Co compounds of 1.527 (2)/1.25 (2) and 1.527 (2)/1.26 (3) Å.

The average Ni—O distance in $(NH_4)_2[Ni(H_2O)_6]_3(HPO_3)_4$ is 2.057 (2) Å, comparable to the average Ni—O distance of 2.094 Å reported for $Ni(HPO_3)\cdot H_2O$ (Marcos, Amoros, Sapina *et al.*, 1993) or 2.070 Å for $Ni_{11}(HPO_3)_8(OH)_6$ (Marcos, Amoros, Beltran-Porter *et al.*, 1993). The shortest Ni···Ni distance in the title compound, of 6.1550 (3) Å, is considerably

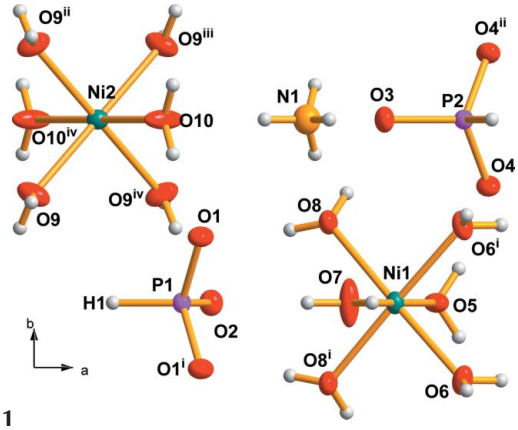

Figure 1
View of the $Ni(H_2O)_6$, HPO_3 and NH_4 coordination polyhedra. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) x, $-y$, z; (ii) x, $1-y$, z; (iii) $-x$, $1-y$, $-z$; (iv) $-x$, y, $-z$.]

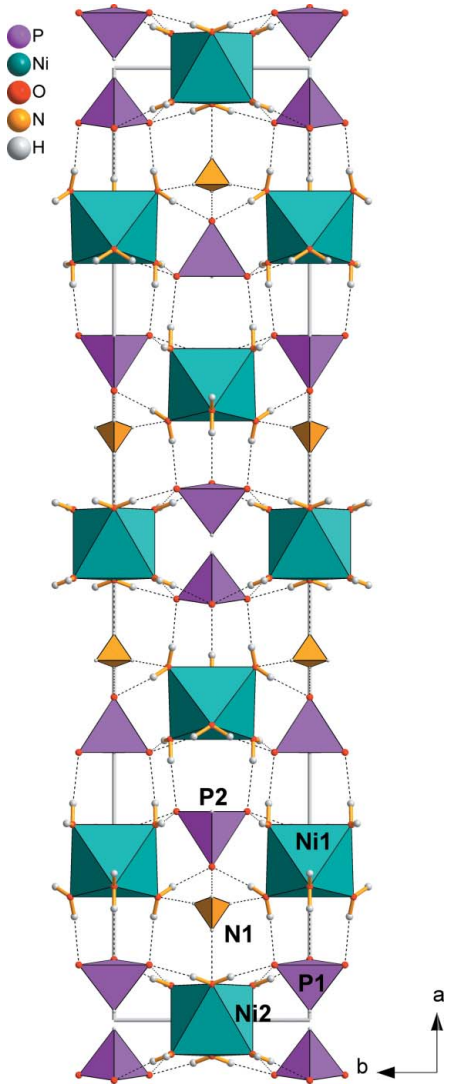

Figure 2
The packing of the title structure, viewed along the *c* axis.

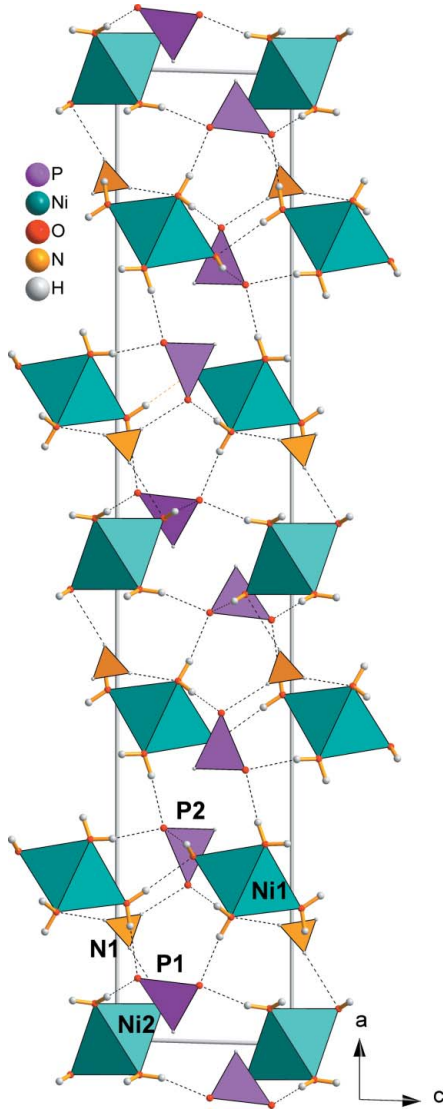

Figure 3
The packing of the title structure, viewed along the *b* axis.

longer than the values observed in the above-mentioned phosphites, *viz*. $Ni(HPO_3)\cdot H_2O$ (3.037 Å) and $Ni_{11}(HPO_3)_8(OH)_6$ (2.680 Å).

Experimental

In a solution of H_3PO_3 (10 ml), mixed with ammonia (0.4 *M*, 5 ml) to adjust the pH, $NiCl_2\cdot 6H_2O$ (10 mg) was dissolved. The mixture was heated to 300 K for 2 h and then left at room temperature for one week. Colourless crystals deposited and were filtered off and washed with an 80:20 ethanol–water solution.

Crystal data

$(NH_4)_2[Ni(H_2O)_6]_3(HPO_3)_4$
M_r = 856.3
Monoclinic, *C*2/*m*
a = 34.1405 (15) Å
b = 7.0197 (3) Å
c = 6.1550 (3) Å
β = 91.371 (4)°
V = 1474.66 (12) Å^3
Z = 2
D_x = 1.928 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 9240 reflections
θ = 3.3–26.4°
μ = 2.22 mm^{-1}
T = 296 K
Block, colorless
0.17 × 0.13 × 0.06 mm

Data collection

Oxford Diffraction Sapphire 2 CCD diffractometer
ω scans
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2004)
T_{min} = 0.578, T_{max} = 0.776
9240 measured reflections
1642 independent reflections
1377 reflections with $I > 3\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.4°
h = −42 → 42
k = −8 → 8
l = −7 → 7

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.020
$wR(F^2)$ = 0.060
S = 1.08
1642 reflections
143 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(I) + 0.0016I^2]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—O1	1.5240 (13)	Ni1—O5	2.0801 (19)
P1—O2	1.5245 (19)	Ni1—O6	2.0433 (14)
P1—H1	1.32 (3)	Ni1—O7	2.067 (2)
P2—O3	1.5342 (19)	Ni1—O8	2.0770 (13)
P2—O4	1.5278 (14)	Ni2—O9	2.0557 (15)
P2—H2	1.22 (3)	Ni2—O10	2.037 (2)
O1—P1—O1^{i}	112.30 (8)	O6—Ni1—O8^{i}	85.66 (5)
O1—P1—O2	112.32 (6)	O7—Ni1—O8	89.00 (5)
O1—P1—H1	106.1 (6)	O8—Ni1—O8^{i}	95.82 (5)
O2—P1—H1	107.1 (11)	O9—Ni2—O9^{iii}	180
O3—P2—O4	111.66 (6)	O9—Ni2—O9^{iv}	91.57 (6)
O3—P2—H2	107.9 (13)	O9—Ni2—O9^{ii}	88.43 (6)
O4—P2—O4^{ii}	112.95 (8)	O9—Ni2—O10	93.34 (6)
O4—P2—H2	106.1 (6)	O9—Ni2—O10^{iii}	86.66 (6)
O5—Ni1—O6	90.63 (5)	O10—Ni2—O10^{iii}	180
O5—Ni1—O7	177.38 (8)	H110—N1—H112	117.7 (16)
O5—Ni1—O8	89.25 (5)	H110—N1—H112^{ii}	117.7 (16)
O5—Ni1—O8^{i}	89.25 (5)	H110—N1—H113	98 (3)
O6—Ni1—O6^{i}	92.85 (6)	H112—N1—H112^{ii}	97.6 (16)
O6—Ni1—O7	91.17 (5)	H112—N1—H113	113.6 (18)
O6—Ni1—O8	178.51 (5)		

Symmetry codes: (i) $x, -y, z$; (ii) $x, -y+1, z$; (iii) $-x, -y+1, -z$; (iv) $-x, y, -z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O4^{i}	0.81 (2)	1.89 (2)	2.6790 (17)	163 (2)
O6—H61···O4^{v}	0.82 (2)	1.93 (1)	2.7311 (18)	166 (2)
O6—H62···O4^{vi}	0.80 (1)	1.94 (1)	2.7372 (18)	172 (2)
O7—H71···O5^{vii}	0.81 (2)	2.08 (2)	2.890 (3)	176 (3)
O7—H72···O2^{vii}	0.81 (2)	1.87 (1)	2.677 (3)	176 (4)
O8—H81···O3	0.83 (2)	1.90 (2)	2.7199 (19)	174 (2)
O8—H82···O1	0.82 (1)	1.87 (1)	2.6906 (18)	173 (2)
O9—H91···O2^{iv}	0.82 (2)	1.90 (2)	2.7111 (18)	176 (2)
O9—H92···O1^{viii}	0.81 (1)	1.96 (2)	2.7378 (18)	159 (2)
O10—H100···O1	0.81 (1)	1.85 (2)	2.6577 (17)	172 (2)
N1—H110···O3	0.87 (2)	1.95 (2)	2.820 (3)	177 (3)
N1—H112···O8^{ix}	0.88 (2)	2.13 (2)	2.995 (2)	168 (2)
N1—H113···O10	0.87 (2)	2.31 (2)	3.171 (4)	168 (3)

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

The H atoms were located in a difference Fourier map. Their coordinates were refined independently. The O—H distances were restrained to 0.82 (1) Å and the N—H distances were restrained to 0.87 (1) Å. The H-1tom isotropic displacement parameters were set at $1.2U_{eq}$ of the parent atom.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2004); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *JANA2000* (Petricek *et al.*, 2000); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *JANA2000*.

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