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Potassium nickel(II) gallium phosphate hydrate, $K[\text{NiGa}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$

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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{P}-\text{O}) = 0.001$ Å; R factor = 0.022; wR factor = 0.025; data-to-parameter ratio = 16.2.

The title compound, potassium nickel(II) digallium tris-(phosphate) dihydrate, $K[\text{NiGa}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$, was synthesized hydrothermally. The structure is constructed from distorted *trans*- $\text{NiO}_4(\text{H}_2\text{O})_2$ octahedra linked through vertices and edges to GaO_5 trigonal bipyramids and PO_4 tetrahedra, forming a three-dimensional framework of formula $[\text{NiGa}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]^-$. The K, Ni and one P atom lie on special positions (Wyckoff position 4e, site symmetry 2). There are two sets of channels within the framework, one running parallel to the $[10\bar{1}]$ direction and the other parallel to $[001]$. These intersect, forming a three-dimensional pore network in which the water molecules coordinated to the Ni atoms and the K^+ ions required to charge balance the framework reside. The K^+ ions lie in a highly distorted environment surrounded by ten O atoms, six of which are closer than 3.1 Å. The coordinated water molecules are within hydrogen-bonding distance to O atoms of bridging Ga—O—P groups.

Related literature

For reviews of open-framework phosphate materials, see: Cheetham *et al.* (1999); Harrison (2002); Maspoch *et al.* (2007). For background to heterometal-substituted gallophosphates, MGAPOs , see: Baerlocher *et al.* (2001); Lin & Wang (2005). For related octahedral-trigonal bipyramidal silicate structures, see: Rocha & Lin (2005). For ammonium gallophosphates isostructural to the title compound, see: Chippindale *et al.* (1996, 1998); Bieniok *et al.* (2008). For the aluminium analogue, $\text{K}[\text{NiAl}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$, see: Meyer & Haushalter (1994). The same structure type occurs in $\text{Cs}[\text{Fe}_3(\text{PO}_4)_3(\text{H}_2\text{O})_2]$ (Lii & Huang, 1995) and $\text{NH}_4[\text{CoAl}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$ (Panz *et al.*, 1998) and is related to that of $(\text{NH}_4)_3\text{Ga}_2(\text{PO}_4)_3$ (Lesage *et al.*, 2004). For bond-valence sums, see: Brese & O'Keeffe (1991); For the weighting scheme, see: Prince (1982); Watkin (1994).

Experimental

Crystal data

$\text{K}[\text{NiGa}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$	$V = 1128.4$ (2) Å ³
$M_r = 558.17$	$Z = 4$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 13.2095$ (13) Å	$\mu = 7.27$ mm ⁻¹
$b = 10.1733$ (9) Å	$T = 150$ K
$c = 8.8130$ (9) Å	$0.09 \times 0.08 \times 0.06$ mm
$\beta = 107.680$ (10)°	

Data collection

Oxford Diffraction Xcalibur diffractometer	3597 measured reflections
Absorption correction: multi-scan (<i>CrysAlis RED</i> ; Oxford Diffraction, 2006)	1913 independent reflections
$T_{\min} = 0.54$, $T_{\max} = 0.65$	1734 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.022$	3 restraints
$wR(F^2) = 0.025$	Only H-atom coordinates refined
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.84$ e Å ⁻³
1667 reflections	$\Delta\rho_{\text{min}} = -0.91$ e Å ⁻³
103 parameters	

Table 1

Selected geometric parameters (Å, °).

Ga1—O1	1.8556 (11)	Ni1—O4 ⁱ	2.1030 (12)
Ga1—O3	1.9994 (11)	P1—O1 ⁱⁱ	1.5279 (11)
Ga1—O5	1.8541 (10)	P1—O1 ⁱⁱⁱ	1.5279 (11)
Ga1—O6	1.9455 (11)	P1—O3	1.5452 (11)
Ga1—O7	1.8357 (11)	P1—O3 ⁱ	1.5452 (11)
Ni1—O2	1.9951 (11)	P2—O2	1.5135 (11)
Ni1—O2 ⁱ	1.9951 (11)	P2—O5 ^{iv}	1.5510 (11)
Ni1—O3	2.1374 (11)	P2—O6 ^v	1.5348 (12)
Ni1—O3 ⁱ	2.1374 (11)	P2—O7	1.5434 (12)
Ni1—O4	2.1030 (12)		

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H1 \cdots O5 ⁱⁱ	0.85 (1)	1.896 (11)	2.741 (2)	172
O4—H2 \cdots O6 ^v	0.85 (1)	2.163 (17)	2.976 (2)	161

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

Data collection: *CrysAlis Pro* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FI2077).

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Potassium nickel(II) gallium phosphate hydrate, $K[\text{NiGa}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$

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S1. Comment

Although the majority of heterometal substituted gallophosphates, MeGaPOs , contain MO_4 ($M = \text{Me}$ or Ga) and PO_4 units (Baerlocher *et al.*, 2001), some are known in which the metal atoms have non-tetrahedral geometries. For example, NGP-1 (Lin & Wang, 2005), the only organically templated NiGaPO reported to date, contains Ni and Ga disordered over the same octahedral metal sites, together with GaO_5 and PO_4 units. $K[\text{NiGa}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$ is assembled from the same polyhedra as NGP-1 and is isostructural with a number of ammonium transition-metal gallophosphates, $\text{NH}_4[\text{MeGa}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$ ($\text{Me} = \text{Mn}$ (Chippindale *et al.*, 1998), Fe , Ni (Bieniok *et al.*, 2008), Co (Chippindale *et al.*, 1996)) and has an aluminium analogue, $K[\text{NiAl}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$ (Meyer & Haushalter, 1994). The same structure type occurs in $\text{Cs}[\text{Fe}_3(\text{PO}_4)_3(\text{H}_2\text{O})_2]$ (Lii & Huang, 1995) and $\text{NH}_4[\text{CoAl}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$ (Panz *et al.*, 1998) and is related to that of $(\text{NH}_4)_3\text{Ga}_2(\text{PO}_4)_3$ (Lesage *et al.*, 2004).

The two phosphorus atoms have tetrahedral geometry (P1, point symmetry C_{2v} ; P2,1). Unlike in NGP-1, the metal atoms are located in two distinct sites; Ga1 in a GaO_5 trigonal bipyramid (1) and Ni1 in an $\text{NiO}_4(\text{H}_2\text{O})_2$ distorted octahedron (C_{2v}), in which the terminal water molecules, $\text{H}_2\text{O}4$, lie *trans* to each other (Fig. 1). Two of the other oxygen atoms in the NiO_6 unit, O2 and O2ⁱ, are two coordinate whilst O3 and O3ⁱ are three coordinate. As expected, the M—O3 ($M = \text{Ni}$, Ga) and P1—O3 bond lengths are the longest M—O and P—O bond lengths observed in the structure. Bond-valence sums (Brese & O'Keeffe, 1991) for P1, P2, Ga1 and Ni1 are 4.80, 4.81, 3.22 and 1.93, respectively, which together with the yellow colour of the crystals, confirm the presence of Ni^{2+} in the structure.

The GaO_5 , NiO_6 and PO_4 units link together through edge- and vertex-sharing arrangements to give a three-dimensional framework of composition $[\text{NiGa}_2(\text{PO}_4)_3(\text{H}_2\text{O})_2]$. A set of approximately circular channels, running parallel to the $[10\bar{1}]$ direction, contain the water molecules and potassium ions (Fig. 2). A second set of channels, elliptical in shape, run parallel to the c axis (Fig. 3) and intersect with the first set to generate a three-dimensional pore network.

The potassium atom lies in a very distorted coordination environment with six near oxygen atoms ($\text{K1}\cdots\text{O}$ in range 2.865 (1) to 3.072 (1) Å) with two additional O atoms at 3.397 (1) and two at 3.456 (1) Å). $\text{H}_2\text{O}4$ is also involved in hydrogen bonding to Ga—O—P bridging oxygen atoms.

S2. Experimental

The title compound was prepared hydrothermally from a gel of composition Ga_2O_3 : $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$: $10\text{H}_3\text{PO}_4$: $156\text{H}_2\text{O}$: $0.1\text{Si}(\text{OEt})_4$: $5\text{KH}_2\text{PO}_4$ which was heated at 433 K for 7 d in a Teflon-lined stainless steel autoclave. The solid product was collected by filtration, washed with deionized water and dried in air. The product consisted of large yellow faceted blocks of the title compound, which could be easily separated from unidentified white powder.

S3. Refinement

Prior to refinement, reflections with $I < 3\sigma(I)$ were omitted. The H atoms of the water molecule, H_2O_4 , were located in a difference Fourier map. Their fractional coordinates were refined subject to bond length and angle restraints [$\text{O}-\text{H} = 0.85(1) \text{ \AA}$, $\text{H}-\text{O}-\text{H} = 109(5)^\circ$] with isotropic displacement parameters fixed at 0.05 \AA^2 .

Figure 1

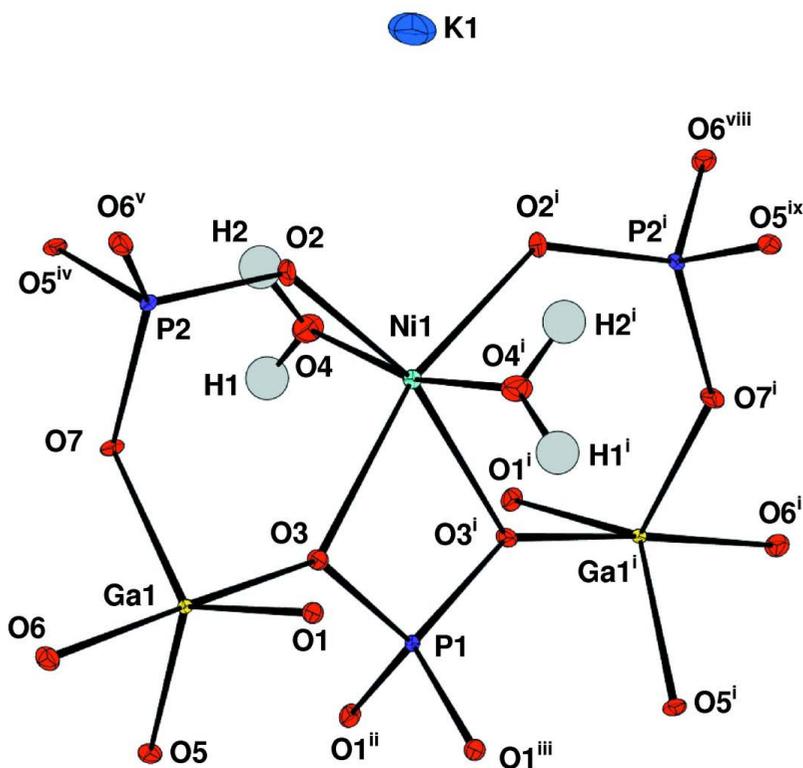
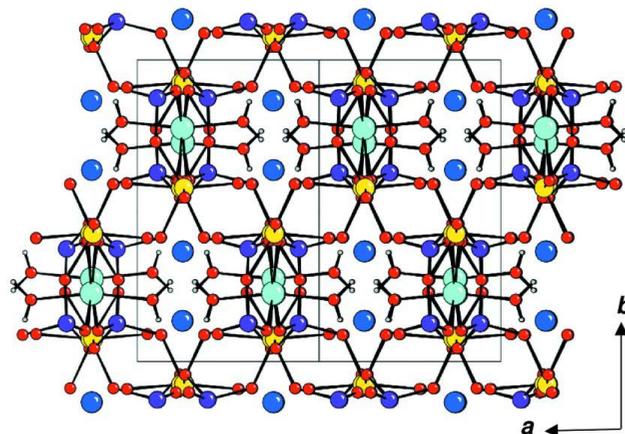


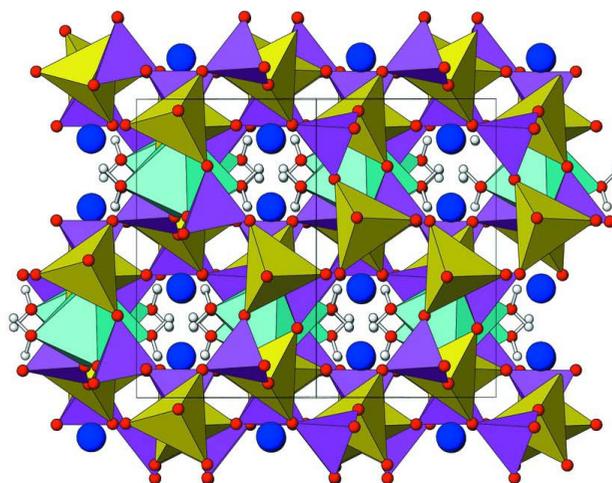
Figure 1

Local coordination of the title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius. [Symmetry codes: (i) $-x, y, -z + 1/2$; (ii) $x, -y, z + 1/2$; (iii) $-x, -y, -z$; (iv) $-x + 1/2, y + 1/2, -z + 1/2$; (v) $-x + 1/2, -y + 1/2, -z$; (viii) $x - 1/2, 1/2 - y, 1/2 + z$; (ix) $-x + 1/2, y + 1/2, z$].

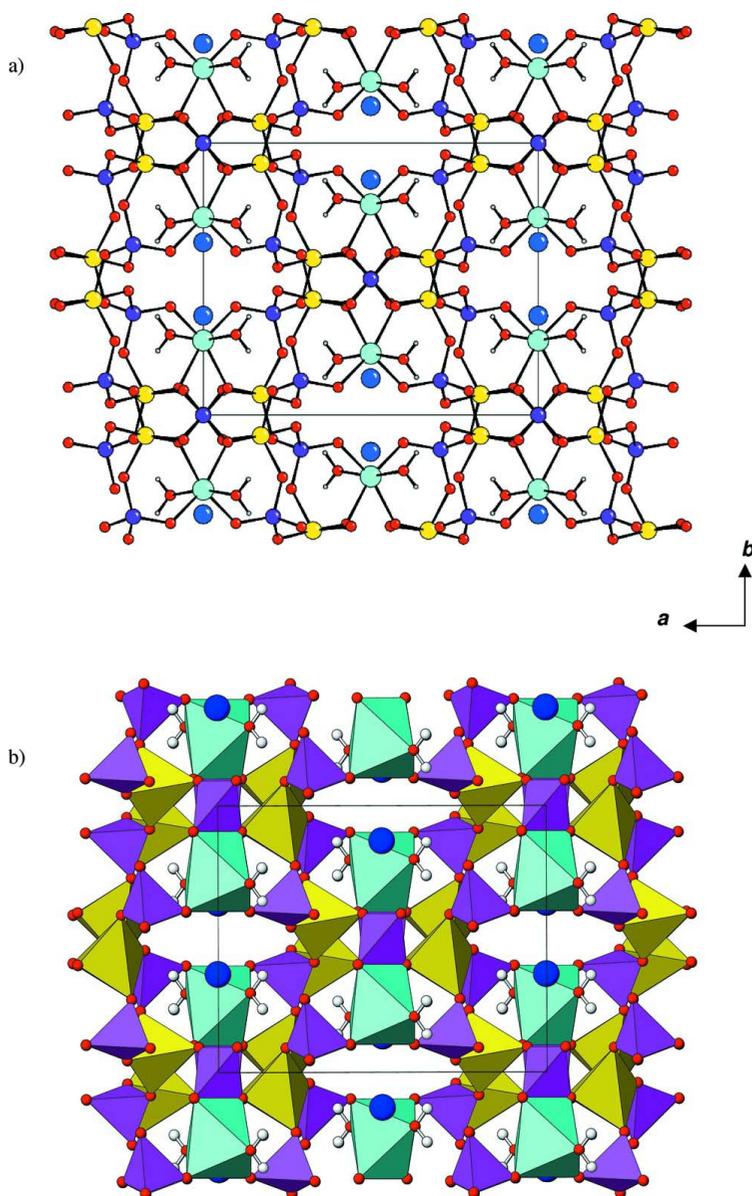
a)



b)

**Figure 2**

View of the main channels running parallel to the $[1\ 0\ \bar{1}]$ direction shown as (a) ball and stick and (b) polyhedral representation. The cross pore distance $O1\cdots O5^x$ is $7.251(1)\ \text{\AA}$. The colour of each polyhedron corresponds to the colour of the central atom as defined in Fig. 1. [Symmetry codes: (x) $1/2 - x, 1/2 - y, -z$]

**Figure 3**

View along the c axis as (a) ball and stick and (b) polyhedral representation showing the elliptical channels bounded by eight membered rings. The shortest cross channel distances are 4.092 (2) and 4.360 (1) Å for $O2 \cdots O2^{xi}$ and $O2 \cdots O6^{ix}$ respectively. These channels intersect with the main channels shown in Fig.2 to form a 3-D pore network. [Symmetry codes: (ix) $-x + 1/2, y + 1/2, z$; (xi) $-x, 1 - y, z$].

potassium nickel(II) digallium tris(phosphate) dihydrate

Crystal data

$K[NiGa_2(PO_4)_3(H_2O)_2]$

$M_r = 558.17$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 13.2095$ (13) Å

$b = 10.1733$ (9) Å

$c = 8.8130$ (9) Å

$\beta = 107.68$ (1)°

$V = 1128.4$ (2) Å³

$Z = 4$

$F(000) = 1080$

$D_x = 3.286$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 3597 reflections
 $\theta = 3\text{--}32.4^\circ$
 $\mu = 7.27 \text{ mm}^{-1}$

$T = 150 \text{ K}$
 Block, yellow
 $0.09 \times 0.08 \times 0.06 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur
 diffractometer
 Graphite monochromator
 $\omega/2\theta$ scans
 Absorption correction: multi-scan
 (CrysAlis RED; Oxford Diffraction, 2006)
 $T_{\min} = 0.54, T_{\max} = 0.65$
 3597 measured reflections

1913 independent reflections
 1734 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$
 $\theta_{\max} = 32.6^\circ, \theta_{\min} = 2.6^\circ$
 $h = -15 \rightarrow 19$
 $k = -15 \rightarrow 15$
 $l = -13 \rightarrow 11$

Refinement

Refinement on F
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.022$
 $wR(F^2) = 0.025$
 $S = 1.04$
 1667 reflections
 103 parameters
 3 restraints
 Primary atom site location: structure-invariant
 direct methods
 Hydrogen site location: inferred from
 neighbouring sites

Only H-atom coordinates refined
 Method, part 1, Chebychev polynomial,
 (Watkin, 1994, Prince, 1982) [weight] =
 $1.0/[A_0 * T_0(x) + A_1 * T_1(x) \dots + A_{n-1} * T_{n-1}(x)]$
 where A_i are the Chebychev coefficients listed
 below and $x = F / F_{\max}$ Method = Robust
 Weighting (Prince, 1982) $W = [\text{weight}] * [1 - (\Delta/F/6 * \sigma F)^2]^2$ A_i are: 18.9 -12.3 16.6
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.84 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.91 \text{ e \AA}^{-3}$

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ga1	0.172478 (11)	0.075592 (15)	0.069169 (16)	0.0036
Ni1	0.0000	0.27385 (3)	0.2500	0.0053
K1	0.0000	0.63513 (6)	0.2500	0.0264
P1	0.0000	0.00118 (5)	0.2500	0.0036
P2	0.20870 (3)	0.37358 (4)	0.17941 (4)	0.0043
O1	0.05847 (8)	0.09109 (11)	-0.11504 (12)	0.0063
O2	0.09754 (9)	0.39793 (11)	0.18796 (14)	0.0082
O3	0.07314 (9)	0.09888 (11)	0.19895 (13)	0.0064
O4	0.09834 (9)	0.29514 (12)	0.48612 (14)	0.0111
O5	0.20652 (8)	-0.08860 (10)	0.16197 (12)	0.0061
O6	0.27707 (8)	0.04382 (11)	-0.04158 (13)	0.0070
O7	0.23679 (8)	0.22862 (11)	0.15963 (13)	0.0070
H1	0.135 (3)	0.236 (3)	0.547 (4)	0.0500*
H2	0.136 (3)	0.361 (3)	0.481 (5)	0.0500*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ga1	0.00279 (8)	0.00233 (9)	0.00525 (8)	-0.00019 (4)	0.00068 (5)	0.00009 (4)
Ni1	0.00413 (11)	0.00400 (12)	0.00818 (12)	0.0000	0.00228 (8)	0.0000
K1	0.0287 (3)	0.0109 (2)	0.0465 (4)	0.0000	0.0215 (3)	0.0000

P1	0.00246 (18)	0.0030 (2)	0.00514 (19)	0.0000	0.00089 (14)	0.0000
P2	0.00345 (15)	0.00312 (15)	0.00631 (14)	-0.00077 (10)	0.00159 (11)	-0.00031 (10)
O1	0.0048 (4)	0.0058 (4)	0.0066 (4)	0.0003 (3)	-0.0007 (3)	-0.0014 (3)
O2	0.0042 (4)	0.0074 (4)	0.0140 (4)	0.0001 (3)	0.0042 (3)	0.0016 (4)
O3	0.0058 (4)	0.0043 (4)	0.0108 (4)	-0.0002 (3)	0.0051 (3)	0.0010 (3)
O4	0.0106 (5)	0.0083 (4)	0.0115 (5)	-0.0003 (4)	-0.0009 (4)	0.0023 (4)
O5	0.0063 (4)	0.0039 (4)	0.0072 (4)	0.0008 (3)	0.0006 (3)	0.0018 (3)
O6	0.0071 (4)	0.0062 (4)	0.0090 (4)	0.0011 (3)	0.0042 (3)	0.0013 (3)
O7	0.0068 (4)	0.0038 (4)	0.0104 (4)	-0.0017 (3)	0.0029 (3)	-0.0030 (3)

Geometric parameters (Å, °)

Ga1—O1	1.8556 (11)	P1—O1 ⁱⁱ	1.5279 (11)
Ga1—O3	1.9994 (11)	P1—O1 ⁱⁱⁱ	1.5279 (11)
Ga1—O5	1.8541 (10)	P1—O3	1.5452 (11)
Ga1—O6	1.9455 (11)	P1—O3 ⁱ	1.5452 (11)
Ga1—O7	1.8357 (11)	P2—O2	1.5135 (11)
Ni1—O2	1.9951 (11)	P2—O5 ^{iv}	1.5510 (11)
Ni1—O2 ⁱ	1.9951 (11)	P2—O6 ^v	1.5348 (12)
Ni1—O3	2.1374 (11)	P2—O7	1.5434 (12)
Ni1—O3 ⁱ	2.1374 (11)	O4—H1	0.850 (10)
Ni1—O4	2.1030 (12)	O4—H2	0.848 (10)
Ni1—O4 ⁱ	2.1030 (12)		
O1—Ga1—O3	89.49 (5)	O4 ⁱ —Ni1—O4	168.18 (7)
O1—Ga1—O5	119.09 (5)	O1 ⁱⁱ —P1—O1 ⁱⁱⁱ	104.18 (9)
O1—Ga1—O6	94.97 (5)	O1 ⁱⁱ —P1—O3	114.14 (6)
O1—Ga1—O7	116.97 (5)	O1 ⁱⁱⁱ —P1—O3	112.42 (6)
O3—Ga1—O5	88.16 (5)	O3 ⁱ —P1—O1 ⁱⁱ	112.42 (6)
O3—Ga1—O6	174.93 (4)	O3 ⁱ —P1—O1 ⁱⁱⁱ	114.14 (6)
O3—Ga1—O7	87.06 (5)	O3 ⁱ —P1—O3	99.94 (8)
O5—Ga1—O6	87.57 (5)	O2—P2—O7	115.61 (6)
O5—Ga1—O7	123.66 (5)	O5 ^{iv} —P2—O2	111.12 (6)
O6—Ga1—O7	93.05 (5)	O5 ^{iv} —P2—O6 ^v	110.43 (6)
O2 ⁱ —Ni1—O2	101.49 (7)	O5 ^{iv} —P2—O7	101.91 (6)
O2—Ni1—O3	95.65 (4)	O6 ^v —P2—O2	107.68 (6)
O2 ⁱ —Ni1—O3	162.84 (5)	O6 ^v —P2—O7	110.02 (6)
O2—Ni1—O4	87.12 (5)	Ga1—O1—P1 ⁱⁱⁱ	135.47 (7)
O2 ⁱ —Ni1—O4	85.41 (5)	Ga1—O3—Ni1	129.51 (5)
O3 ⁱ —Ni1—O2	162.84 (5)	Ga1—O3—P1	131.86 (7)
O3 ⁱ —Ni1—O2 ⁱ	95.65 (4)	Ni1—O2—P2	128.84 (7)
O3 ⁱ —Ni1—O3	67.23 (6)	Ni1—O3—P1	96.42 (5)
O3 ⁱ —Ni1—O4	93.46 (4)	Ni1—O4—H1	128 (3)
O3 ⁱ —Ni1—O4 ⁱ	96.38 (5)	Ni1—O4—H2	103 (3)
O4 ⁱ —Ni1—O2	85.41 (5)	H1—O4—H2	111 (3)
O4 ⁱ —Ni1—O2 ⁱ	87.12 (5)	Ga1—O5—P2 ^{vi}	129.28 (7)

O3—Ni1—O4	96.38 (5)	Ga1—O6—P2 ^v	125.62 (7)
O4 ⁱ —Ni1—O3	93.46 (4)	Ga1—O7—P2	139.82 (7)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H1 \cdots O5 ⁱⁱ	0.85 (1)	1.90 (1)	2.741 (2)	172
O4—H2 \cdots O6 ^{iv}	0.85 (1)	2.16 (2)	2.976 (2)	161

Symmetry codes: (ii) $x, -y, z+1/2$; (iv) $-x+1/2, y+1/2, -z+1/2$.