

catena-Poly[[diaquanickel(II)]- μ -2-hydroxyphosphonatoacetato]

Jing Li, Da-Peng Dong, Cui-Ying Huang, Zhen-Gang Sun* and Yan-Yu Zhu

Institute of Chemistry for Functionalized Materials, College of Chemistry and Chemical Engineering, Liaoning Normal University, Dalian 116029, People's Republic of China

Correspondence e-mail: szg188@163.com

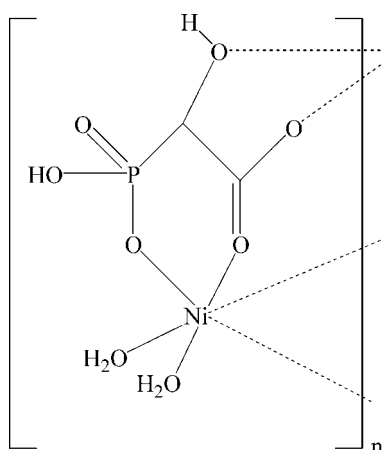
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.025; wR factor = 0.068; data-to-parameter ratio = 11.6.

The title compound, $[\text{Ni}\{\text{HO}_3\text{PCH}(\text{OH})\text{CO}_2\}(\text{H}_2\text{O})_2]_n$, was prepared by a hydrothermal reaction. The octahedral coordination geometry of Ni^{II} is made up of one phosphonate O atom, one hydroxyl O atom, two carboxylate O atoms and two water molecules. This architecture is further stabilized by a number of $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds involving the protonated hydroxyl groups, carboxylate O atoms, a phosphonate O atom and coordinated water molecules.

Related literature

For related literature, see: Alberti *et al.* (1992); Clearfield (1988, 1998); Hix & Harris (1998); Modi *et al.* (2005); Nonglaton *et al.* (2004); Riou *et al.* (2000); Sharma & Clearfield (2000); Song *et al.* (1999).



Experimental

Crystal data

$[\text{Ni}(\text{C}_2\text{H}_3\text{O}_6\text{P})(\text{H}_2\text{O})_2]$
 $M_r = 248.76$
 Orthorhombic, $Pbca$
 $a = 8.6776$ (9) Å
 $b = 9.7953$ (10) Å
 $c = 16.7592$ (16) Å

$V = 1424.5$ (2) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 2.96$ mm⁻¹
 $T = 295$ (2) K
 $0.20 \times 0.09 \times 0.07$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{\text{min}} = 0.589$, $T_{\text{max}} = 0.820$

7058 measured reflections
 1398 independent reflections
 1204 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.068$
 $S = 1.05$
 1398 reflections
 121 parameters
 4 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.48$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ni1—O5 ⁱ	2.0314 (17)	P1—O2	1.5123 (19)
Ni1—O2	2.0322 (18)	P1—O3	1.578 (2)
Ni1—O6	2.0419 (17)	P1—C1	1.837 (3)
Ni1—O4 ⁱ	2.0422 (17)	O4—C1	1.426 (3)
Ni1—O7	2.052 (2)	O5—C2	1.271 (3)
Ni1—O8	2.0881 (19)	O6—C2	1.247 (3)
P1—O1	1.4996 (19)	C1—C2	1.521 (3)
O5 ⁱ —Ni1—O2	173.98 (7)	O6—Ni1—O7	177.68 (8)
O5 ⁱ —Ni1—O6	87.48 (7)	O4 ⁱ —Ni1—O7	89.31 (8)
O2—Ni1—O6	92.57 (7)	O5 ⁱ —Ni1—O8	94.45 (7)
O5 ⁱ —Ni1—O4 ⁱ	78.82 (7)	O2—Ni1—O8	91.56 (7)
O2—Ni1—O4 ⁱ	95.17 (7)	O6—Ni1—O8	87.37 (7)
O6—Ni1—O4 ⁱ	89.40 (7)	O4 ⁱ —Ni1—O8	172.66 (7)
O5 ⁱ —Ni1—O7	90.37 (8)	O7—Ni1—O8	93.68 (8)
O2—Ni1—O7	89.47 (8)		

Symmetry code: (i) $x + \frac{1}{2}, y, -z + \frac{1}{2}$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O3—H3A \cdots O1 ⁱⁱ	0.82	1.80	2.621 (3)	173
O8—H8B \cdots O1 ⁱⁱ	0.843 (10)	1.902 (11)	2.741 (3)	173 (3)
O4—H4A \cdots O5 ⁱⁱⁱ	0.82	1.75	2.573 (2)	177
O7—H7A \cdots O8 ^{iv}	0.852 (10)	2.049 (11)	2.901 (3)	178 (3)
O7—H7B \cdots O3 ^v	0.846 (10)	2.135 (14)	2.958 (3)	164 (3)
O8—H8A \cdots O2 ^v	0.850 (10)	1.921 (12)	2.756 (3)	167 (3)

Symmetry codes: (ii) $-x + \frac{3}{2}, y - \frac{1}{2}, z$; (iii) $-x + \frac{3}{2}, y + \frac{1}{2}, z$; (iv) $-x + \frac{5}{2}, y + \frac{1}{2}, z$; (v) $-x + 2, -y, -z$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WK2072).

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supplementary materials

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catena-Poly[[diaquanickel(II)]- μ -2-hydroxyphosphonatoacetato]

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Comment

Metal phosphonates have been of increasing interest in the past decade due to their potential applications in the fields of catalysis (Sharma & Clearfield, 2000), ion exchange (Clearfield, 1988), proton conductivity (Alberti *et al.*, 1992), gas and liquid separations (Riou *et al.*, 2000), biology (Nonglaton *et al.*, 2004), and organic molecule sorption (Clearfield, 1998). Great efforts have been devoted to the syntheses of novel inorganic–organic hybrid materials based on metal phosphonates, which exhibit a variety of structures such as one-dimensional chains, two-dimensional layers, and three-dimensional network. To the best of our knowledge, however, reports on nickel phosphonates are rather scarce (Hix & Harris, 1998; Song *et al.*, 1999; Modi *et al.*, 2005).

The title compound (I) crystallizes in the orthorhombic space group *Pbca*. The molecular structure of (I) is shown in the Fig. 1. Each Ni atom adopts distorted octahedral coordination geometry with the six oxygen atoms from the two equivalent L^{3-} ($L = O_3PCH(OH)CO_2$) anions and two coordinated water molecule. The values of the Ni—O bond lengths and O—Ni—O angles are in the range of 2.0329 (19)–2.0888 (19) Å and 78.81 (7)–177.88 (8) °, respectively. This architecture is further stabilized by a number of O—H \cdots O hydrogen bonds involving the protonated hydroxyl oxygen atoms, carboxylate oxygen atoms, phosphonate oxygen atom, and coordinated water molecules (see Table 1, Table 2 and Fig. 2).

Experimental

A mixture of 0.27 g (1.0 mmol) $NiSO_4 \cdot 6H_2O$, 1.0 ml (4.0 mmol) 2-hydroxyphosphonoacetic acid (48.0 wt %) and 0.04 g (1.0 mmol) NaF (as a mineralizer) was dissolved in 8 ml of deionized water, and then 1,4-butylenediamine was added with stirring to adjust the pH of the mixture. The mixture (pH = 4.5) was sealed in a 23 ml Teflon-lined stainless steel autoclave, and then heated at 413 K for 72 h. Colorless block crystals were obtained, washed with distilled water, and dried in air at room temperature.

Refinement

During refinement, carbon bound H atoms were placed in calculated positions and allowed to ride on the parent atom, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. Oxygen bound hydrogen atoms were located from difference Fourier maps and refined with distance restraints.

Figures

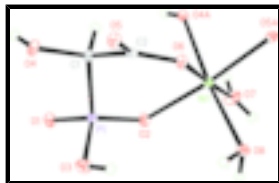


Fig. 1. Molecular structure of (I), showing displacement ellipsoids at the 30% probability level.

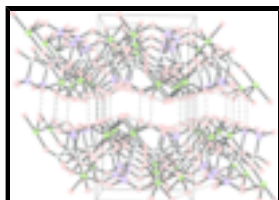


Fig. 2. View of the packing of (I) with the unit cell outlined along the *b* axis, showing the stacking of compound (I). H atoms have been omitted for clarity.

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Crystal data

[Ni(C₂H₃O₆P)(H₂O)₂]

M_r = 248.76

Orthorhombic, *Pbca*

a = 8.6776 (9) Å

b = 9.7953 (10) Å

c = 16.7592 (16) Å

V = 1424.5 (2) Å³

Z = 8

*F*₀₀₀ = 1008

D_x = 2.320 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 2474 reflections

θ = 2.4–28.2°

μ = 2.96 mm⁻¹

T = 295 (2) K

Block, green

0.20 × 0.09 × 0.07 mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 295(2) K

ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 2003)

*T*_{min} = 0.589, *T*_{max} = 0.820

7058 measured reflections

1398 independent reflections

1204 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.030

θ_{max} = 26.0°

θ_{min} = 3.4°

h = -10→10

k = -11→12

l = -12→20

Refinement

Refinement on *F*²

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$$R[F^2 > 2\sigma(F^2)] = 0.025$$

$$wR(F^2) = 0.068$$

$$S = 1.05$$

1398 reflections

121 parameters

4 restraints

Primary atom site location: structure-invariant direct methods

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 1.2128P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.41 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.48 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	1.12817 (4)	-0.00201 (3)	0.134842 (19)	0.01264 (13)
P1	0.79917 (7)	0.13589 (7)	0.13742 (4)	0.01399 (17)
O1	0.7275 (2)	0.27285 (19)	0.12260 (11)	0.0205 (4)
O2	0.9474 (2)	0.10843 (19)	0.09274 (10)	0.0190 (4)
O3	0.6732 (2)	0.0253 (2)	0.11674 (12)	0.0227 (4)
H3A	0.7113	-0.0511	0.1198	0.034*
O4	0.7059 (2)	0.14671 (17)	0.28985 (10)	0.0165 (4)
H4A	0.7019	0.2294	0.2973	0.025*
O5	0.81452 (19)	-0.09508 (17)	0.31593 (10)	0.0149 (4)
O6	1.00059 (19)	-0.08174 (17)	0.22600 (11)	0.0152 (4)
O7	1.2642 (3)	0.0773 (2)	0.04608 (12)	0.0287 (5)
H7A	1.316 (3)	0.150 (2)	0.053 (2)	0.034*
H7B	1.294 (4)	0.037 (3)	0.0042 (13)	0.034*
O8	1.0632 (2)	-0.17173 (19)	0.06738 (11)	0.0180 (4)
H8A	1.044 (3)	-0.154 (3)	0.0186 (8)	0.022*
H8B	0.9736 (17)	-0.194 (3)	0.0817 (17)	0.022*
C1	0.8399 (3)	0.1148 (3)	0.24425 (15)	0.0131 (5)
H1A	0.9228	0.1776	0.2594	0.016*
C2	0.8903 (3)	-0.0304 (3)	0.26311 (15)	0.0134 (5)

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0133 (2)	0.0128 (2)	0.0118 (2)	0.00033 (12)	-0.00064 (12)	0.00038 (12)
P1	0.0154 (3)	0.0132 (3)	0.0134 (3)	0.0021 (3)	0.0003 (3)	0.0013 (2)
O1	0.0215 (10)	0.0165 (10)	0.0235 (10)	0.0046 (8)	-0.0004 (8)	0.0036 (8)
O2	0.0180 (9)	0.0251 (10)	0.0137 (10)	0.0071 (8)	0.0018 (8)	0.0035 (8)
O3	0.0240 (10)	0.0189 (10)	0.0251 (11)	-0.0024 (8)	-0.0054 (9)	0.0021 (8)
O4	0.0216 (9)	0.0090 (9)	0.0190 (10)	0.0020 (7)	0.0069 (8)	0.0007 (7)
O5	0.0166 (9)	0.0117 (9)	0.0166 (9)	-0.0003 (7)	0.0032 (7)	0.0021 (7)
O6	0.0146 (9)	0.0136 (9)	0.0173 (10)	0.0015 (7)	0.0032 (7)	0.0011 (7)
O7	0.0370 (12)	0.0299 (12)	0.0192 (11)	-0.0115 (10)	0.0092 (10)	-0.0022 (9)
O8	0.0183 (9)	0.0212 (10)	0.0144 (9)	-0.0011 (8)	-0.0011 (8)	-0.0011 (8)
C1	0.0140 (12)	0.0133 (13)	0.0122 (13)	0.0007 (10)	0.0032 (10)	0.0005 (9)
C2	0.0129 (12)	0.0152 (13)	0.0120 (13)	-0.0019 (10)	-0.0037 (10)	-0.0017 (10)

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

Ni1—O5 ⁱ	2.0314 (17)	O4—Ni1 ⁱⁱ	2.0422 (17)
Ni1—O2	2.0322 (18)	O4—H4A	0.8200
Ni1—O6	2.0419 (17)	O5—C2	1.271 (3)
Ni1—O4 ⁱ	2.0422 (17)	O5—Ni1 ⁱⁱ	2.0314 (17)
Ni1—O7	2.052 (2)	O6—C2	1.247 (3)
Ni1—O8	2.0881 (19)	O7—H7A	0.852 (10)
P1—O1	1.4996 (19)	O7—H7B	0.846 (10)
P1—O2	1.5123 (19)	O8—H8A	0.850 (10)
P1—O3	1.578 (2)	O8—H8B	0.843 (10)
P1—C1	1.837 (3)	C1—C2	1.521 (3)
O3—H3A	0.8200	C1—H1A	0.9800
O4—C1	1.426 (3)		
O5 ⁱ —Ni1—O2	173.98 (7)	P1—O3—H3A	109.5
O5 ⁱ —Ni1—O6	87.48 (7)	C1—O4—Ni1 ⁱⁱ	116.37 (14)
O2—Ni1—O6	92.57 (7)	C1—O4—H4A	109.5
O5 ⁱ —Ni1—O4 ⁱ	78.82 (7)	Ni1 ⁱⁱ —O4—H4A	126.6
O2—Ni1—O4 ⁱ	95.17 (7)	C2—O5—Ni1 ⁱⁱ	118.09 (16)
O6—Ni1—O4 ⁱ	89.40 (7)	C2—O6—Ni1	129.43 (17)
O5 ⁱ —Ni1—O7	90.37 (8)	Ni1—O7—H7A	121 (2)
O2—Ni1—O7	89.47 (8)	Ni1—O7—H7B	127 (2)
O6—Ni1—O7	177.68 (8)	H7A—O7—H7B	111 (3)
O4 ⁱ —Ni1—O7	89.31 (8)	Ni1—O8—H8A	114 (2)
O5 ⁱ —Ni1—O8	94.45 (7)	Ni1—O8—H8B	108 (2)
O2—Ni1—O8	91.56 (7)	H8A—O8—H8B	99 (3)
O6—Ni1—O8	87.37 (7)	O4—C1—C2	109.16 (19)
O4 ⁱ —Ni1—O8	172.66 (7)	O4—C1—P1	109.94 (16)
O7—Ni1—O8	93.68 (8)	C2—C1—P1	111.29 (17)

O1—P1—O2	115.47 (11)	O4—C1—H1A	108.8
O1—P1—O3	106.88 (11)	C2—C1—H1A	108.8
O2—P1—O3	111.01 (11)	P1—C1—H1A	108.8
O1—P1—C1	110.00 (11)	O6—C2—O5	122.9 (2)
O2—P1—C1	107.41 (11)	O6—C2—C1	119.6 (2)
O3—P1—C1	105.67 (11)	O5—C2—C1	117.5 (2)
P1—O2—Ni1	125.40 (10)		

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O3—H3A···O1 ⁱⁱⁱ	0.82	1.80	2.621 (3)	173
O8—H8B···O1 ⁱⁱⁱ	0.843 (10)	1.902 (11)	2.741 (3)	173 (3)
O4—H4A···O5 ^{iv}	0.82	1.75	2.573 (2)	177
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O7—H7B···O3 ^{vi}	0.846 (10)	2.135 (14)	2.958 (3)	164 (3)
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Symmetry codes: (iii) $-x+3/2, y-1/2, z$; (iv) $-x+3/2, y+1/2, z$; (v) $-x+5/2, y+1/2, z$; (vi) $-x+2, -y, -z$.

Fig. 1

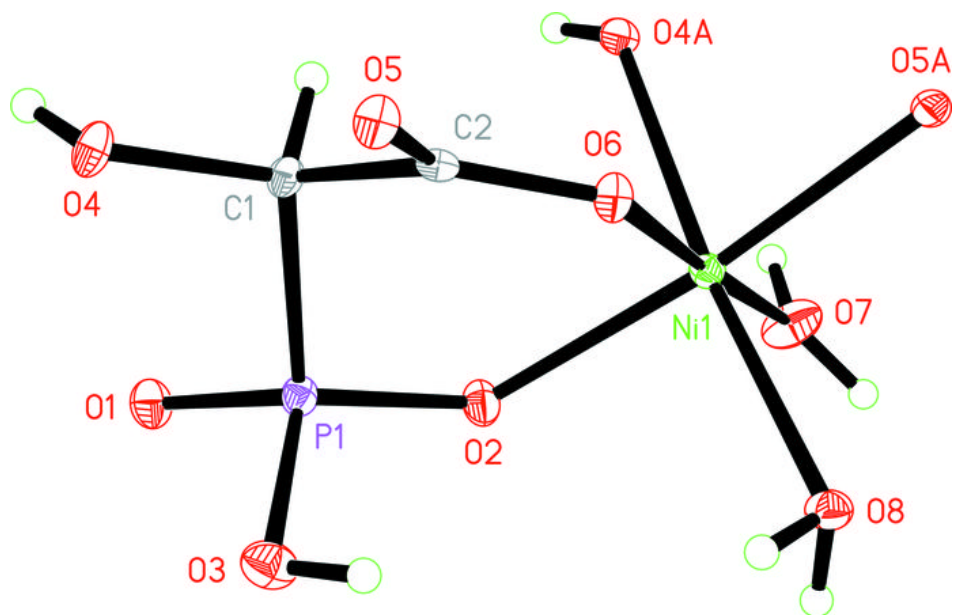


Fig. 2

