

catena-Poly[[tetra- μ -acetato- κ^8 O:O'-dinickel(II)]- μ -2,3'-dipyridylamine- κ^2 N:N']

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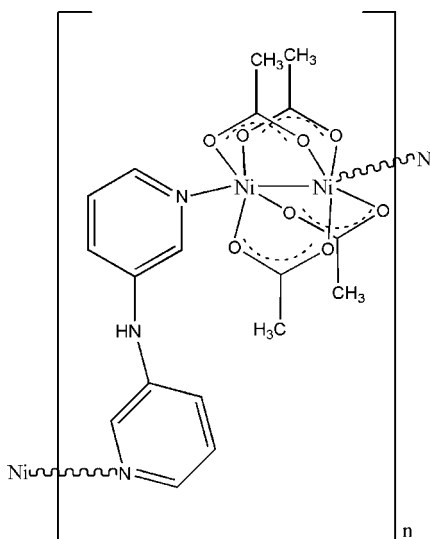
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.015$ Å; disorder in main residue; R factor = 0.054; wR factor = 0.132; data-to-parameter ratio = 13.4.

The title complex, $[\text{Ni}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_{10}\text{H}_7\text{N}_3)]_n$ or $[\text{Ni}_2(\text{dbp})(\text{C}_2\text{O}_2\text{H}_3)_4]_n$ ($\text{dbp} = 2,3'$ -dipyridylamine), forms a polymeric linear chain. The repeat unit of the chain is the dinuclear $[\text{Ni}_2(\text{C}_{10}\text{H}_7\text{N}_3)(\text{C}_2\text{O}_2\text{H}_3)_4]$ of the paddle-wheel type. The two Ni atoms are related by symmetry through an inversion center. Each Ni atom has a square-pyramidal coordination environment, with four O atoms from four acetate groups forming the basal plane, and a pyridyl N atom occupying the apex. The bridging dbp ligand is disordered over an inversion center, involving two alternative positions for the NH group.

Related literature

For related literature, see: Gudbjartson *et al.* (1999); Su *et al.* (2003).



Experimental

Crystal data

$[\text{Ni}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_{10}\text{H}_7\text{N}_3)]$
 $M_r = 522.78$
 Monoclinic, $C2/c$
 $a = 13.4008$ (13) Å
 $b = 8.4880$ (9) Å
 $c = 20.112$ (2) Å
 $\beta = 106.191$ (7)°

$V = 2196.9$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.76$ mm⁻¹
 $T = 298$ (2) K
 $0.27 \times 0.21 \times 0.16$ mm

Data collection

Bruker APEXII area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.648$, $T_{\max} = 0.766$

5441 measured reflections
 1973 independent reflections
 1098 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.095$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.132$
 $S = 1.00$
 1973 reflections

147 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.57$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.45$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O1}$	0.86	1.85	2.666 (10)	157

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXTL (Bruker, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2259).

References

- Bruker (2004). APEX2, SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.
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supplementary materials

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catena-Poly[[tetra- μ -acetato- $\kappa^8 O:O'$ -dinickel(II)]- μ -2,3'-dipyridylamine- $\kappa^2 N:N'$]

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Comment

The chemical structure of the organic ligands including the molecular angle, length, and relative orientation of the donor groups plays an extremely important role in dictating polymer topology. So far, much of the research has been concentrated on the exploitation of angular ligands with a molecular angle, such as ligands with a Tshape, V-shape *etc.*, in the construction of versatile coordination polymer architectures (Su *et al.*, 2003; Gudbjartson *et al.*, 1999). In this paper, we report the synthesis and crystal structure of the title complex, (I).

The structure of (I) consists of one-dimensional polymeric $[\text{Ni}_2(\text{dbp})_2(\text{Ac})_4]_n$ chains built up from dinuclear Ni(II) subunit of the paddle-wheel type. The two nickel atoms are related by symmetry through inversion center (Fig. 1). Each nickel atom has a square pyramid coordination environment, with four oxygen atoms from four acetyl groups, forming the equatorial plane, and the pyridyl nitrogen atom, occupying the apex. This big paddle-wheel type arrangement prevents further torsion of the two pyridine rings of the bridging ligand, and then results in the formation of a neutral one-dimensional linear chain rather than a helical chain. The occurrence of intramolecular (N—H \cdots O) hydrogen bond stabilize the architecture (Table 1).

Experimental

Ni(AC)₂(0.036 g, 0.028 mmol), dbp (0.018 g, 0.013 mmol) a mixed solvent of acetonitrile, the mixture was heated for eight hours under reflux. During the process stirring and influx were required. The resultant was then filtered to give a pure solution which was infiltrated by diethyl ether freely in a closed vessel, weeks later some single crystals of the size suitable for X-Ray diffraction analysis.

Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.96 Å (methyl) and N—H = 0.86 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}_{\text{aromatic}} \text{ or } \text{N})$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

Some of the C atoms of the pyridyl group display very elongated ellipsoids, however no correct disordered models could be defined.

Figures

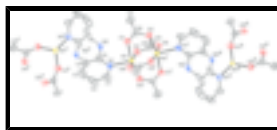


Fig. 1. ORTEP view of (I) showing the formation of the polymeric chain, with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $2 - x, 2 - y, 2 - z$; (ii) $-x + 2, y, -z + 3/2$; (iii) $x, -y + 2, 1/2 + z$]

Poly[(tetraacetyl- $\kappa^2N:N'$)dinickel(II)- μ -2,3'-dipyridylamine- $\kappa^2O:O'$]

Crystal data

[Ni₂(C₂H₃O₂)₄(C₁₀H₇N₃)]

$M_r = 522.78$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 13.4008\ (13)\ \text{\AA}$

$b = 8.4880\ (9)\ \text{\AA}$

$c = 20.112\ (2)\ \text{\AA}$

$\beta = 106.191\ (7)^\circ$

$V = 2196.9\ (4)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 1072$

$D_x = 1.581\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

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

Cell parameters from 1973 reflections

$\theta = 2.9\text{--}25.2^\circ$

$\mu = 1.76\ \text{mm}^{-1}$

$T = 298\ (2)\ \text{K}$

Block, green

$0.27 \times 0.21 \times 0.16\ \text{mm}$

Data collection

Bruker APEXII area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 0 pixels mm^{-1}

$T = 298\ (2)\ \text{K}$

φ and ω scan

Absorption correction: multi-scan
(SADABS; Sheldrick, 2004)

$T_{\min} = 0.648$, $T_{\max} = 0.766$

5441 measured reflections

1973 independent reflections

1098 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.095$

$\theta_{\max} = 25.2^\circ$

$\theta_{\min} = 2.9^\circ$

$h = -14 \rightarrow 16$

$k = -10 \rightarrow 8$

$l = -24 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.132$

$S = 1.00$

1973 reflections

147 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.57\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.45\ \text{e \AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Ni1	0.95385 (5)	0.92235 (8)	0.94293 (4)	0.0423 (3)	
N1	0.8818 (4)	0.7990 (6)	0.8453 (3)	0.0577 (14)	
N2	1.0307 (9)	0.7655 (13)	0.8155 (5)	0.061 (3)	0.50
H2	1.0613	0.7845	0.8584	0.074*	0.50
O1	1.0991 (3)	0.8984 (5)	0.9401 (2)	0.0644 (12)	
O2	1.1772 (3)	1.0270 (5)	1.0372 (3)	0.0688 (13)	
O3	0.9695 (3)	0.7416 (5)	1.0053 (2)	0.0603 (12)	
O4	1.0480 (3)	0.8712 (5)	1.1020 (2)	0.0654 (13)	
C1	0.7828 (6)	0.7539 (8)	0.8293 (4)	0.077 (2)	
H1	0.7445	0.7658	0.8611	0.092*	
C2	0.7364 (8)	0.6873 (11)	0.7633 (6)	0.121 (4)	
H21	0.6677	0.6535	0.7516	0.145*	
C3	0.7940 (15)	0.6729 (14)	0.7163 (7)	0.165 (8)	
H3	0.7632	0.6323	0.6724	0.198*	
C4	0.8911 (11)	0.7158 (18)	0.7332 (5)	0.170 (8)	
C5	0.9334 (7)	0.7787 (12)	0.7980 (4)	0.098 (3)	
C6	1.1793 (5)	0.9576 (7)	0.9823 (4)	0.0533 (17)	
C7	1.2802 (5)	0.9406 (8)	0.9663 (4)	0.079 (2)	
H7A	1.3101	1.0428	0.9647	0.118*	
H7B	1.2695	0.8897	0.9222	0.118*	
H7C	1.3265	0.8782	1.0015	0.118*	
C8	1.0076 (5)	0.7494 (8)	1.0691 (4)	0.0573 (17)	
C9	1.0085 (6)	0.6030 (8)	1.1110 (4)	0.074 (2)	
H9A	0.9487	0.6024	1.1284	0.111*	
H9B	1.0704	0.6009	1.1491	0.111*	
H9C	1.0069	0.5120	1.0824	0.111*	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0396 (5)	0.0408 (5)	0.0436 (5)	-0.0011 (4)	0.0069 (3)	-0.0036 (4)
N1	0.054 (4)	0.054 (4)	0.062 (4)	0.002 (3)	0.011 (3)	-0.003 (3)

supplementary materials

N2	0.074 (9)	0.085 (8)	0.022 (5)	-0.016 (7)	0.008 (5)	0.004 (5)
O1	0.051 (3)	0.068 (3)	0.072 (3)	0.003 (2)	0.014 (2)	-0.009 (2)
O2	0.050 (3)	0.077 (3)	0.078 (3)	-0.003 (2)	0.015 (2)	-0.019 (3)
O3	0.074 (3)	0.049 (3)	0.053 (3)	-0.008 (2)	0.011 (2)	-0.003 (2)
O4	0.082 (3)	0.043 (3)	0.062 (3)	-0.003 (2)	0.006 (2)	0.005 (2)
C1	0.057 (5)	0.064 (5)	0.098 (6)	-0.007 (4)	0.001 (4)	0.016 (4)
C2	0.090 (8)	0.082 (7)	0.138 (10)	-0.040 (6)	-0.053 (7)	0.006 (7)
C3	0.25 (2)	0.111 (9)	0.082 (9)	0.083 (11)	-0.048 (10)	-0.036 (7)
C4	0.167 (12)	0.259 (16)	0.044 (6)	0.143 (12)	-0.038 (7)	-0.032 (8)
C5	0.067 (6)	0.163 (9)	0.053 (5)	0.029 (6)	-0.002 (4)	0.005 (5)
C6	0.042 (4)	0.050 (4)	0.070 (5)	0.003 (3)	0.019 (4)	0.006 (4)
C7	0.063 (5)	0.072 (5)	0.112 (6)	-0.006 (4)	0.041 (4)	-0.003 (4)
C8	0.050 (4)	0.053 (5)	0.072 (5)	0.009 (3)	0.022 (4)	0.001 (4)
C9	0.095 (6)	0.056 (5)	0.072 (5)	-0.005 (4)	0.027 (4)	0.013 (4)

Geometric parameters (Å, °)

Ni1—O2 ⁱ	1.955 (5)	C1—C2	1.418 (12)
Ni1—O3	1.956 (4)	C1—H1	0.9300
Ni1—O4 ⁱ	1.968 (4)	C2—C3	1.381 (16)
Ni1—O1	1.974 (4)	C2—H21	0.9300
Ni1—N1	2.197 (5)	C3—C4	1.30 (2)
Ni1—Ni1 ⁱ	2.6371 (14)	C3—H3	0.9300
N1—C1	1.332 (8)	C4—C5	1.376 (13)
N1—C5	1.333 (10)	C4—N2 ⁱⁱ	1.675 (18)
N2—C5	1.258 (12)	C6—C7	1.482 (9)
N2—C4 ⁱⁱ	1.675 (18)	C7—H7A	0.9600
N2—H2	0.8600	C7—H7B	0.9600
O1—C6	1.273 (7)	C7—H7C	0.9600
O2—C6	1.258 (7)	C8—C9	1.501 (9)
O2—Ni1 ⁱ	1.955 (5)	C9—H9A	0.9600
O3—C8	1.242 (7)	C9—H9B	0.9600
O4—C8	1.265 (8)	C9—H9C	0.9600
O4—Ni1 ⁱ	1.968 (4)		
O2 ⁱ —Ni1—O3	89.00 (19)	C3—C2—H21	120.2
O2 ⁱ —Ni1—O4 ⁱ	90.07 (19)	C1—C2—H21	120.2
O3—Ni1—O4 ⁱ	167.99 (17)	C4—C3—C2	120.3 (12)
O2 ⁱ —Ni1—O1	168.00 (18)	C4—C3—H3	119.9
O3—Ni1—O1	90.45 (18)	C2—C3—H3	119.8
O4 ⁱ —Ni1—O1	87.97 (19)	C3—C4—C5	118.2 (14)
O2 ⁱ —Ni1—N1	95.3 (2)	C3—C4—N2 ⁱⁱ	131.3 (11)
O3—Ni1—N1	98.13 (18)	C5—C4—N2 ⁱⁱ	107.3 (13)
O4 ⁱ —Ni1—N1	93.87 (18)	N2—C5—N1	121.0 (8)
O1—Ni1—N1	96.7 (2)	N2—C5—C4	110.5 (10)
O2 ⁱ —Ni1—Ni1 ⁱ	86.66 (14)	N1—C5—C4	124.6 (11)

O3—Ni1—Ni1 ⁱ	83.87 (13)	O2—C6—O1	123.3 (6)
O4 ⁱ —Ni1—Ni1 ⁱ	84.13 (13)	O2—C6—C7	118.8 (6)
O1—Ni1—Ni1 ⁱ	81.36 (13)	O1—C6—C7	118.0 (7)
N1—Ni1—Ni1 ⁱ	177.22 (17)	C6—C7—H7A	109.5
C1—N1—C5	118.1 (7)	C6—C7—H7B	109.5
C1—N1—Ni1	120.8 (5)	H7A—C7—H7B	109.5
C5—N1—Ni1	120.9 (5)	C6—C7—H7C	109.5
C5—N2—C4 ⁱⁱ	128.9 (9)	H7A—C7—H7C	109.5
C5—N2—H2	115.6	H7B—C7—H7C	109.5
C4 ⁱⁱ —N2—H2	115.6	O3—C8—O4	125.2 (6)
C6—O1—Ni1	126.6 (4)	O3—C8—C9	118.2 (6)
C6—O2—Ni1 ⁱ	121.6 (4)	O4—C8—C9	116.6 (6)
C8—O3—Ni1	124.0 (4)	C8—C9—H9A	109.5
C8—O4—Ni1 ⁱ	122.5 (4)	C8—C9—H9B	109.5
N1—C1—C2	119.1 (8)	H9A—C9—H9B	109.5
N1—C1—H1	120.4	C8—C9—H9C	109.5
C2—C1—H1	120.4	H9A—C9—H9C	109.5
C3—C2—C1	119.6 (10)	H9B—C9—H9C	109.5

Symmetry codes: (i) $-x+2, -y+2, -z+2$; (ii) $-x+2, y, -z+3/2$.

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2 \cdots O1	0.86	1.85	2.666 (10)	157

Fig. 1

